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NRIM

Research Activities

1997

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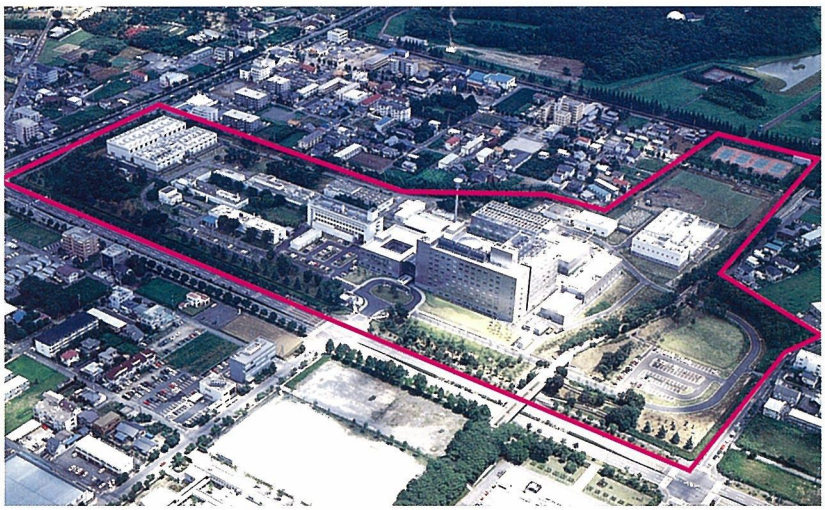

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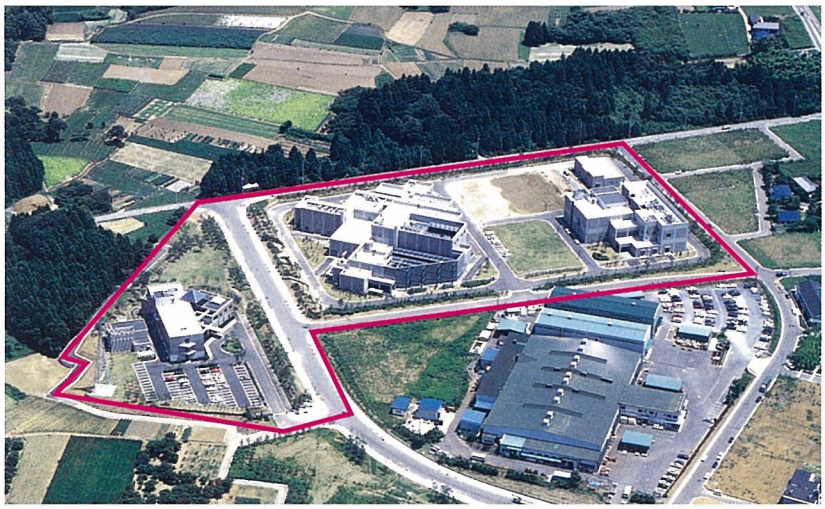

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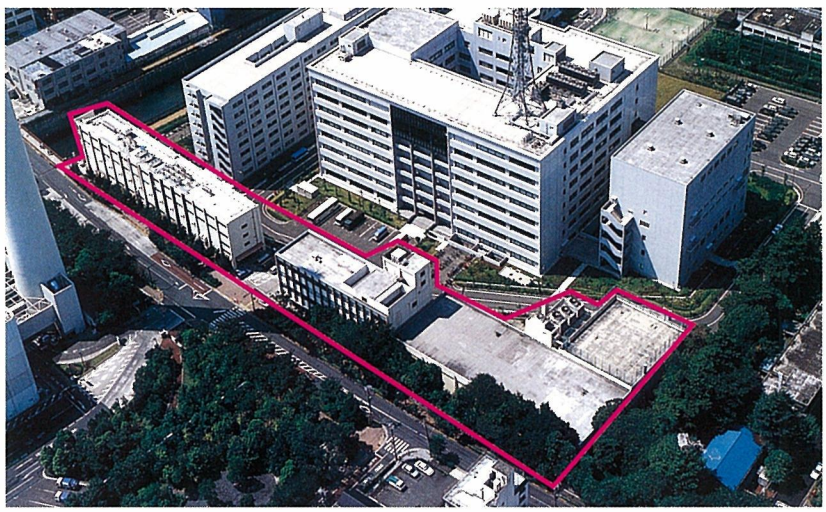

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Preface

I am pleased to present "NRIM Research Activities 1997." This report summarizes the annual activities of the National Research Institute for Metals (NRIM), focusing on our research programs and the progress we have made over the past year.

NRIM is a research institution attached to the Science and Technology Agency of the government of Japan and was established in 1956. Since then, NRIM has played an essential role in the field of research and development of metals, alloys and other materials. An ongoing effort has been to improve the reliability of existing materials, which is documented in the "NRIM Creep and Fatigue Data Sheet" series of publications.

On July 1, 1995, NRIM moved its headquarters from Tokyo to Tsukuba Science City, where there are many world-class experimental facilities and various supercomputers. With the support and efforts of sponsors, staff, and the community, we have built a Center of Excellence for fundamental materials research in Tsukuba. Responding to the present social and economic needs of Japan, we are establishing a "Frontier Structural Materials Research Center" as part of the "New Century Structural Materials Project." We have already started an R&D program to create future "ultra steels" at the center, making full use of the excellent resources available at NRIM. In this way, NRIM continues to expand its activities for advanced materials research.

To all of you, who have supported NRIM, we greatly appreciate your continued understanding and encouragement

Masatoshi Okada

Dr. Masatoshi OKADA
Director-General

NRIM Research Activities
1997

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❒ Effect of Atomic Pairs on the Longteam Creep Strength of Carbon Steels

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Introduction

Recent environmental problems in a global scale seem to originate from strains existing in a large materials and energy consumption type of society. Now, it is a time for us to think not only of better performance but also how materials and related technologies can become less hazardous to environment. In the case of metallic materials, reduction of alloying elements is desirable from view points of saving energy and mineral resource. The possibility of minimum alloying is suggested from the NRIM Creep Data Sheets for ferritic steels where the long term creep strength depends mainly on the minute contents of Mo and C[1]. In these alloys, solid solution strengthening plays important role[2]. Thus, effects of solute elements in the ferrite matrix on the long term creep strength of carbon steels were studied from view points of atomic configurations such as atomic pairs, in order to make clear the factors controlling the long term creep strength required for improving Ecobalance of heat resistant steels.

Calculation of Atomic Configuration in the Ferrite

The equilibrium concentrations of solute elements in the ferrite matrix were esti-mated by thermodynamic calculations with the sublattice model (Thermo-calc.(3)). The concentrations of atomic pairs were estimated by the central atoms model(4) which can calculate the atom configurations in the nearest neighbor atom shell. In the case of b.c.c. ferrite, the nearest neighbour atom shell is shown in Fig.1. The Wager interaction coefficients reported for Fe-Mo-C(5) and Fe-Cr-C(6) systems were used in CAM calculation (Table 1). The parameter for Mn was estimated from the binding energy for Mn-C reported by Abe et al(7).

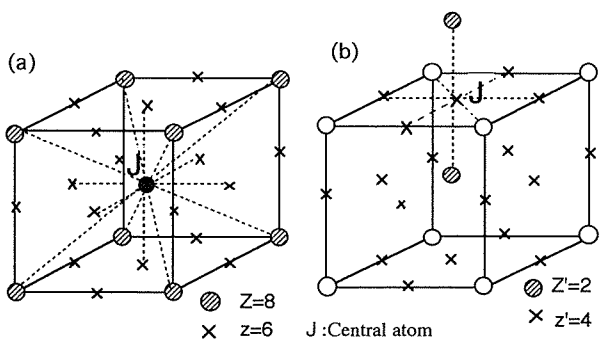

Fig. 1 Geometry of the b.c.c. lattice. O and X represent substitutional and interstitial sites, respectively. Z and z designate respectively the numbers of substitutional and interstitial nearest neighbors to a substitutional site (a), while Z' and z' degignate respectively these numbers to an interstitial site (b).

Table 1 Wagner interaction coefficients for carbon used in the present study

Alloying Element	ε_C^X	Reference
Mo	-100	H. Wada(5)
Cr	-72	H. Wada(6)
Mn	-26*	
Ni	0	

* Estimated from the binding energy for Mn-C, 0.27eV/pair, reported by H.Abe et al.(7)

Effect of Atomic Pairs on the Long Term Creep Strength of Carbon Steels

Effects of solute elements in the ferrite matrix on the long term creep strength were examined for sixteen heats of 0.2 and 0.3mass%C steels having minute and different contents of Mo, Mn, and Cr as shown in Table 2. In the analysis, the creep rupture lives at 773K

Table 2 Chemical compositions (mass %)

Alloy	Haet	C	Si	Mn	Cr	Mo	N
	CAA	0.20	0.31	0.59	0.05	0.01	0.006
	CAB	0.20	0.28	0.60	0.05	0.01	0.007
	CAC	0.20	0.29	0.55	0.05	0.01	0.006
STB	CAG	0.21	0.21	0.62	0.05	0.02	0.003
410	CAH	0.24	0.24	0.64	0.07	0.02	0.006
	CAJ	0.20	0.20	0.47	0.06	0.01	0.011
	CAL	0.21	0.32	0.48	0.02	0.01	0.004
	CAM	0.20	0.31	0.50	0.02	0.01	0.004
	CaC	0.28	0.30	0.70	0.07	0.02	0.010
	CaD	0.20	0.22	0.68	0.11	0.03	0.008
SB	CaE	0.34	0.22	0.70	0.10	0.03	0.011
480	CaF	0.28	0.19	0.80	0.06	0.22	0.007
	CaG	0.29	0.20	0.93	0.03	0.36	0.005
	CaH	0.29	0.22	1.00	0.07	0.34	0.004
	CaM	0.22	0.22	0.82	0.06	0.18	0.008
	CaN	0.26	0.23	0.74	0.04	0.15	0.011

and 88MPa were taken from the NRIM Creep Data Sheets(8). The equilibrium composition of ferrite matrix at 773K, which is the temperature of creep test, are calculated by Thermo-Calc. for sixteen carbon steels. Si shows the maximum solubility in the ferrite, ranging from 0.4 to 0.7 at%, and the solubility decreases in the order of Mn, Cr, Mo, C, and N. Figure 2 shows correlations between the long term creep rupture life at 773K and 88MPa, which seems to be Inherent creep strength, and the concentration of solute element in the ferrite matrix. The creep strength is proportional to concentrations of Mn and Mo and inversely proportional to concentrations of C and Si in solution.

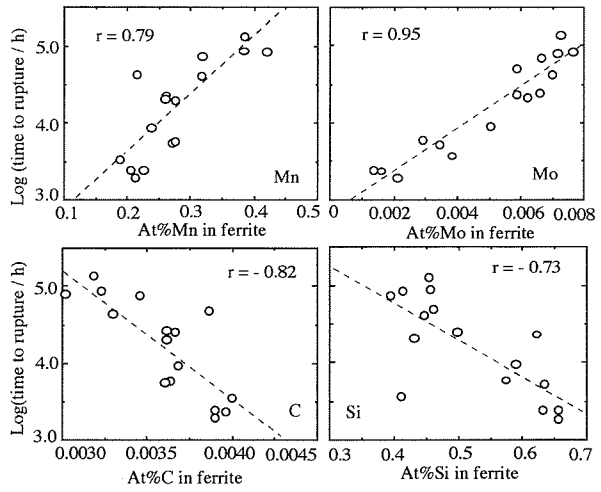

Fig. 2 Correlations between the creep rupture life at 773K and 88MPa and the concentration of solute element in the ferrite matrix.

The concentration of each atomic pair in the ferrite matrix was calculated by CAM. The relationship between the creep strength and the concentration of these atomic pairs were examined by the multiple regression analysis. In this analysis, the concentration of Cr-C pair was not 1% significant, and the best regression equation of Eq.(1) was obtained by the concentrations of Mn-C and Mo-C atomic pairs .

$$\mathrm{Log}(t_R) = 1.11X_{Mn\text{-}C} + 13.60X_{Mo\text{-}C} + 1.45 \quad (1)$$

t_R: the time to rupture (h) at 773K and 88MPa.
$X_{Mn\text{-}C}$, $X_{Mo\text{-}C}$: concentration of Mn-C and Mo-C pairs (atppm).

A good correlation was observed between the creep rupture life predicted by this regression equation and the experimental ones as shown in Fig.3. The rupture life of sixteen steels estimated by the regression equation increases with increasing Mo content, but it sutturates at very minute content of about 0.03mass%Mo as shown in Fig.4. This can explain experimental results(1) very well. Thus, it is concluded that the Inherent creep strength of carbon steels is con-

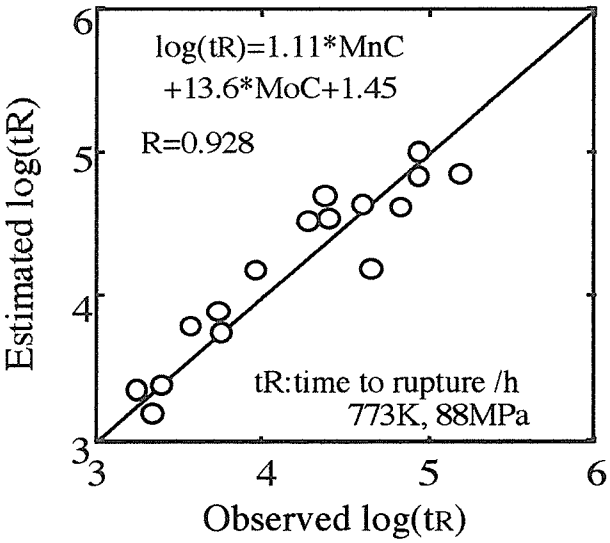

Fig. 3 Correlation between estimated and observed values of the creep rupture life

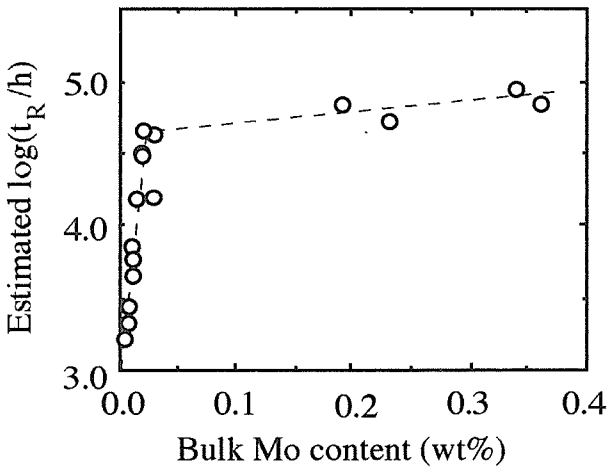

Fig. 4 Variation of the estimated creep rupture life with the bulk Mo content.

trolled by Mn-C and Mo-C atomic pairs. These M-C atomic pairs seem to have strong interaction with dislocation. If a M-C pair is trapped at a jog of dislocation, the absorption of vacancy at jog will be prevented due to the strong binding energy of M-C pair with dislocation. Usioda et al.(9) analyzed the effect of solute atom, Mn, and Mn-C pair on the climb rate of dislocation assuming that the climb rate of dislocation is controlled by the rate at which vacancies diffuse to or from the dislocation. They reported that the climb rate of dislocation in the ferrite containing Mn-C pairs is about 10 times slower than that in pure Fe. Thus, the M-C pair seems to be very effectve to reduce the climb velocity of dislocation.

Conclusion

This paper describes an analysis of effects of short range ordering in the b.c.c. ferrite phase on the long term creep strength of carbon steels based on CAM. Obtained results are follows.

Effects of solute elements in the ferrite matrix on the long term creep strength of 0.2 and 0.3 mass% car-

bon steels were studied from a view point of atomic configurations such as atomic pairs. The equilibrium concentrations of solute elements and atomic pairs in the ferrite matrix were estimated by thermodynamic calculations. A good correlation is observed between the long term creep strength and the concentrations of Mn-C and Mo-C atomic pairs suggesting that the Inherent creep strength of carbon steels is controlled by Mn-C and Mo-C atomic pairs.

Keywords: Inherent creep strength, Solute elements, M-C atomic pairs, climb velocity of dislocations, carbon steels

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❒ Desorption dynamics of mono-oxide species in reactive scattering on semiconductor surfaces

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Reaction dynamics on semiconductor surfaces has been attracting much attention not only due to fundamental interest but also due to practical application in semiconductor technology. Molecular beam reactive scattering (MBRS) technique makes it possible to simulate gas-surface reaction on highly controlled conditions. Combination of MBRS and state-selective measurements by using laser spectroscopy provide a wealth information on surface reaction dynamics. However, there are few studies of state selective measurements on reaction dynamics on semiconductor surfaces. In this work, we report the state selective measurements of mono-oxide species (SiO and GeO) desorbed from the high-temperature surface using the resonance enhanced multiphoton ionization (REMPI) mass spectroscopy to understand the desorption dynamics.

The apparatus of REMPI measuements consists of a UHV reaction chamber and a differentially pumped time-of-flight (TOF) detection chamber. O_2 molecular beam was introduced from pulse valve and operated at 10 Hz with synchronization to a laser. The translational energy of O_2 beam was below 0.07 eV. Pulse width of the O_2 beam was about 250 ms (FWHM). The probe laser system consisted of a Nd:YAG pumped a dye laser and an autotracker . The laser repetition rate was 10 Hz and the pulse duration was 8 ns. The output of the dye laser was doubled in a KD*P set in the autotracker to produce about 2 mJ/pulse. The laser was focused by a quartz lens (f=350 mm) into the ionization region of the TOF mass spectrometer which is about 5 cm apart from the surface .

A pulse O_2 molecular beam was irradiated onto a Si(100) surface at the substrate temperature of 1273±50 K in the UHV chamber and the volatile reaction products desorbed from the surface were detected by REMPI. Only the SiO was observed as reaction products in a TOF spectrum. In the TOF spectrum signals of ^{28}SiO, ^{29}SiO and ^{30}SiO were well separated. In the REMPI study we measured the ^{28}SiO species and used a wavelength between 282 and 294 nm (~34483 cm-1), where the ionization potential of SiO is 11.4 eV (91941cm-1) [5]. The $F^1\Sigma^+$-$X^1\Sigma^+$ and $H^1\Sigma^+$-$X^1\Sigma^+$ transitions are possible with two photon absorption in the used wavelength region. Figure 1 shows a typical 2+1 REMPI spectrum of the SiO molecule at wavelength which matches HX(0,0), HX(1,1), and HX(2,2) transitions.

Figure 2 shows a Boltzmann plot of the SiO intensity of each vibrational bands versus the vibrational energy spacing of the electronic ground state $X^1\Sigma^+$. The plot is linear and the vibrational temperature (T_v) was estimated to be 1250±150 K ($T_v \approx T_s$). The vibrational distribution of the desorbed SiO would be in thermaly equilibrium with the surface.

Rotational distributions were estimated by fitting a calculated spectrum to the observed one. The observed spectrum was well characterized by the Q branch tran-

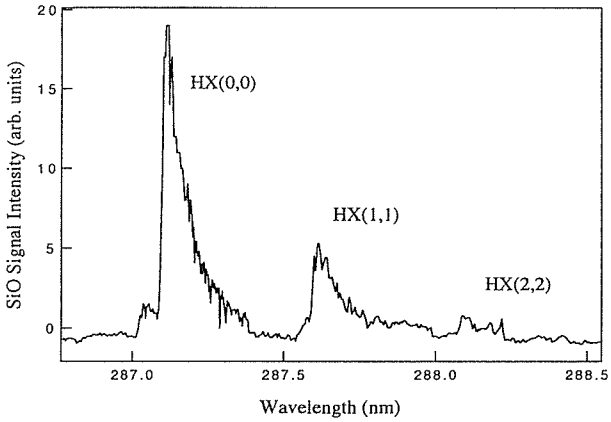

Fig. 1 A typical example of REMPI spectrum of SiO desorbed from Si(100) in reaction with O_2 at a surface temperature of 1273±50 K.

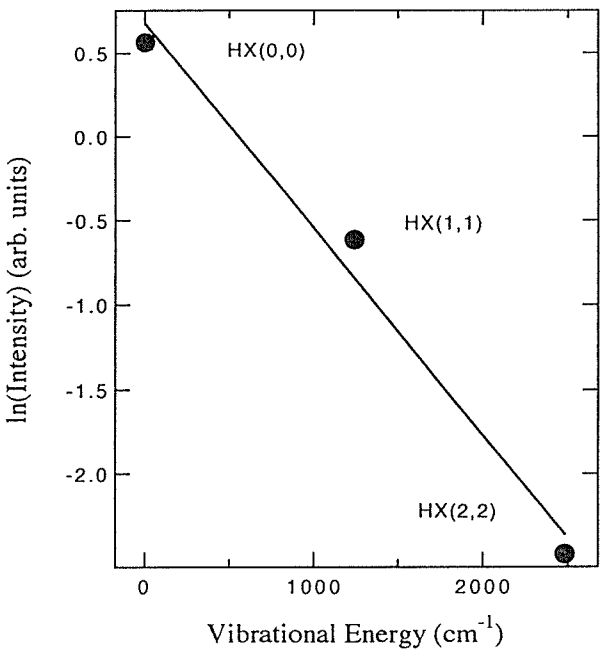

Fig. 2 Boltzmann plot of the peak intensities of HX(0,0), HX(1,1), and HX(2,2) transitions versus the vibtarional energy spacing. The vibrational temperature is estimated to be 1300±250 K.

sitions with the Boltzmann distribution. The rotational temperature was estimated to be 1200±250 K (T_R T_s). In order to study surface orientation effect we measured a REMPI spectrum of SiO desorbed from Si(111) at Ts=1273±50 K. No significant surface orientation effect has been observed in the internal state distributions of the desorbed SiO. An angular distribution was also measured on the flux of SiO desorbing from Si(100) at Ts=1273 K. The obtained angular distribution was well approximated by a cosine function. The vibrational and rotational distribution of the desorbed SiO estimated from the REMPI spectra was independent of the desorption angle. Therefore we believe that the SiO desorption is isotropic.

Figure 3 shows a typical example of REMPI spectrum of the GeO desorbed from Ge(100) surface at the substrate temperature of 970±30 K. The strong enhancement in ion signal intensities is observed 296.85 nm, which matches to the two photon absorption of FX(0,0) transition. A small peak was observed at 297.51 nm and was assigned to be FX(1,1) transition peak. The intensity ratio of the FX(1,1) band versus the FX(0,0) band was estimated to be 0.24±0.07. This value is comparable to the vibrational state distribution ratio of v=1/v=0 (0.23) which is expected for the GeO molecule thermalized at surface temperature (970 K). The angular distribution of the GeO desorption was also measured and found to be cosine distribution for the desorption from the virgin surface.

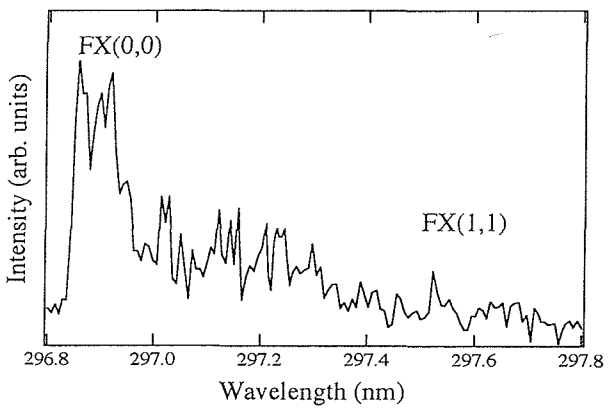

Fig. 3 A typical example of REMPI spectrum of GeO desorbed from Ge(100) in reaction with O_2 at a surface temperature of 970±30 K.

The present results show that the vibrational and rotational state distributions of SiO desorbed from the Si surface in the reactive scattering of O_2 at surface temperature of 1273 K are thermally equilibrium with the surface temperature and the vibrational distribution of GeO was also supposed to be thermalized to the surface temperature at 970 K. In the case of the SiO and GeO desorption, the thermalized internal state distributions and isotropic spatial distribution of the desorption suggests that a memory of configuration at the adsorbed state may be lost in desorption trajectories. Then we suppose that the mono-oxides pass through a nearly free rotational state, such as physisorbed state, during the desorption trajectories. A quantum mechanical calculation suggested an existence of a shallow minimum corresponding to a weakly bounded state in which the SiO can rotate in the potential energy surface for the SiO desorption from the Si(111) surface . In the state, the rotational and vibrational motions would be thermalized to the surface temperature by a weak interaction with the surface.

Keywords : desorption dynamics, oxidation, semiconductor surfaces, resonance enhanced multiphoton ionization, pulse molecular beam

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❒ Phase Transformation and Microstructure of Intermetallic Compound TiAl Studied by High-Resolution Electron Microscopy

E. Abe, T. Kumagai and M. Nakamura

The α -Ti (disordered h.c.p.) γ -TiAl ($L1_0$: ordered fcc) phase transformations in two-phase γ - α_2-Ti_3Al ($D0_{19}$: ordered hcp) intermetallic alloys involve two processes which are hcp - fcc structural change and rearrangement of atoms for ordering. From this viewpoint, these transformations are interesting and worth studying as occuring in intermetallic systems, and we have studied the α - γ phase transformation mechanisms based on the systematic microstructural analysis from a micron scale to an atomic scale using high-resolution electron microscopy [1-6] . The results are briefly summarized below.

We used a 400kV transmission electron microscope (Jeol JEM-4000EX) having the point-ti-point resolution of 0.17nm. The investigations were performed with a Ti-48at.%Al alloy prepared by arc-melting.

Fine lamellae formation : $\alpha \rightarrow \alpha_2 + \gamma$

The lamellar structure formed by ice-water quenching consists of thin plates (a few nm - a few tens nm) of the ordered α_2 and twin-related γ crystals. The characteristic features are as follows.

1) Partial dislocation ledges to accomplish the fundamental lattice change from hcp to fcc are observed at the α_2 - γ interfaces in the growth edge of the γ -plate (Fig. 1) [1].
2) The α_2 - γ interfacial lattice misfit is very small, and the α_2 - γ and γ - γ twin interfaces are atomically flat in a wide range [6].
3) Antiphase domain boundaries (APDBs) are formed both in the α_2 and γ plates, and they are continuous across the α_2 - γ and γ - γ twin interfaces at the atomic level [6].
4) Considerable compositional change has occurred between the α_2 and γ phases during quenching[2, 6].

On these bases, the formation process of the present fine lamellar structure is described as $\alpha \rightarrow \alpha_2 \rightarrow \alpha_2 + \gamma$. Although a long-range diffusion process is involved in this transformation, it is concluded that the mechanism is strongly dominated by the atomic movement involving shear displacements.

Massive transformation : $\alpha \rightarrow \gamma_m$

The $\alpha \rightarrow \gamma$ massive transformation which occurs by rapid cooling is a composition invariant one and produces the microstructure of fine γ grains with random orientations. The features characteristic of the microstructure are below.

1) High-density of microtwins which are aligned parallel to one another is formed in some places. Extremely thin h.c.p. plates with the thickness of about 0.8-2.0nm are found to exist in the massively transformed γ phase, which are also aligned to the microtwins mentioned above [3].
2) Twin boundaries are irregular and are not on the

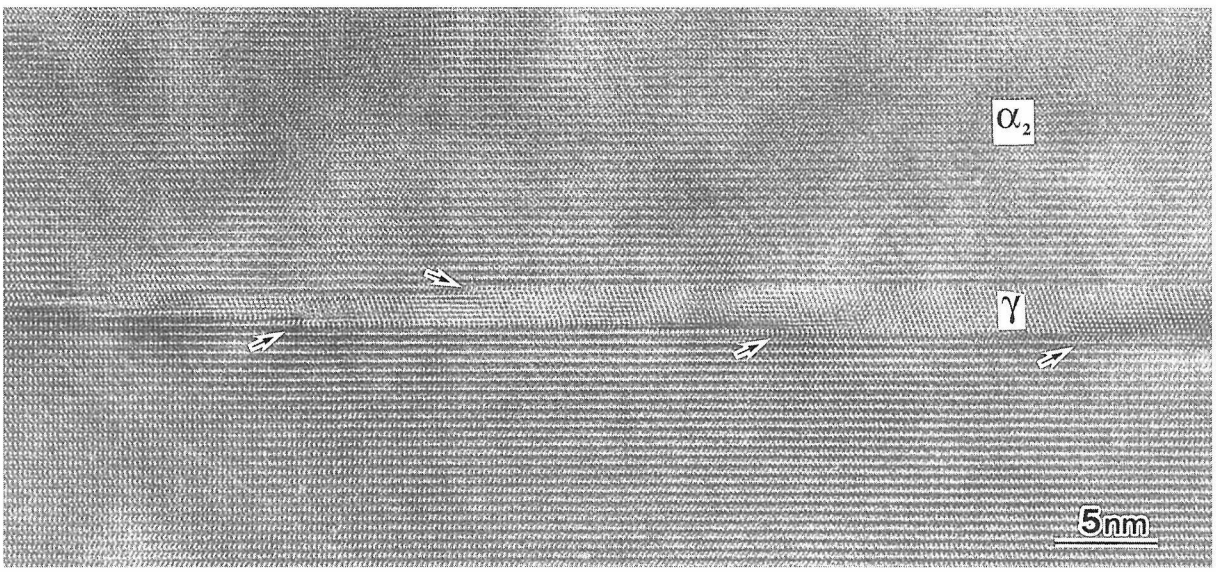

Fig. 1 High-resolution image of the growth edge of the γ -plate in the $\alpha_2 + \gamma$ lamellar structure. The steps of two close-packed planes high are clearly visible as indicated by arrows.

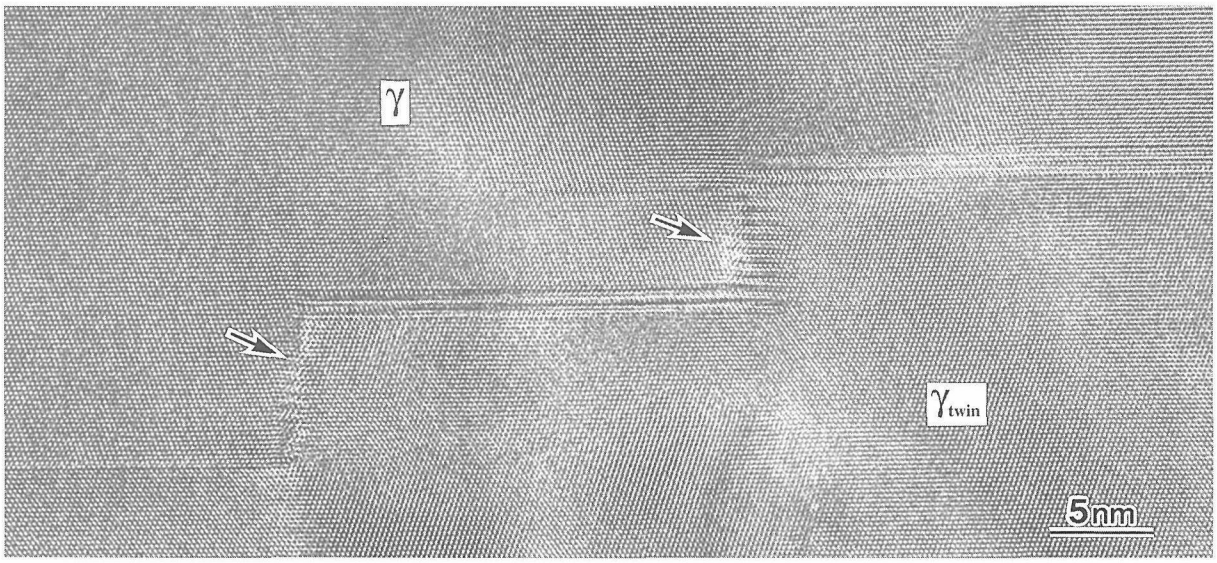

Fig. 2 High-resolution image of a twin boundaries in the massively transformed γ grain. Large ledges due to piling upof a number of twinning dislocations are formed at the boundary as indicated by arrows.

(111) mirror plane of the twinning operation. Large ledges with the height of about a few tens nm are formed at the twin boundaries (Fig. 2). These features are attributed to pilimg up of twinning partial dislocations [3].

3) Extremely thin rotated domain walls are mostly formed at the APDBs. These rotated domains are considered to be grown from the simple APDB in order to reduce the high APB energy of the γ phase [4].

From these facts, it is concluded that the mechanism of the present massive transformation is based on the hcp to fcc shear displacement, although the mobility of individual atoms is high due to a nature of high-temperature transformation. Short-range diffusion process should be involved in the present massive transformation, which accomplishes the rearrangement of atoms including ordering for the $L1_0$ and growth of rotated domains.

Formation of the α_2 plate from the γ_m phase : $\gamma_m \rightarrow \alpha\ (\alpha_2)$

Since the γ_m phase is in metastable state with Ti supersaturation, the α_2 phase is formed by subsequent aging. By this treatment, both the particles and fine plates of the α_2 phase are formed, and the present paper describes the formation mechanism of the plate growth.

1) By isothermal aging at 1473K, the fine α_2 plates are formed in the grain interiors lying on the {111} planes of the γ phase. The formation process is described as $\gamma_m \rightarrow \gamma + \alpha \rightarrow \gamma + \alpha_2$. The interfaces were found to be not flat but to have a complex structure [5].
2) The fine α_2 plates formed in the early stage of isothermal aging exhibit a very large aspect ratio, indicating lengthening growth has been fast during the early stage, just like the case of the γ plate precipitation mentioned earier [6].

These facts indicate that this transformation mechanism is also based on the fcc - hcp change accomplished by migration of ledges, which dominates the early stage of platelet precipitation.

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Keywords : intermetallic compound, phase transformation, high-resolution electron microscopy

❐ Addition of new functions to conventional PTCR material.

T.Dan, M.Egashira, J.Kyono, H.Fudouzi and N.Shinya
5th Research Group

Our research group has already proposed the concept of powder particles assembly for the creation of multi-functional materials in 1994.

This concept enumerates some principal processes, for example, arrangement of particles onto the electrified pattern on a substrate by electron beam, manipulation of particles with a tungsten microprobe, coating of a conducting polymer onto metal particles[1)]

Particle hybridization by forced electrification is one of the above principal processes[2)]. By use of this process, it is expected that the new multi-functional materials, different from conventional ones, be obtained.

Forced electrification process can be applied to whatever a particle combination is. In this process, high electrical potentials are imposed to two kinds of particles, reciprocally; the electrical charge on the particles can overwhelm the inherent contact potential differences of the combination.

Forced electrification processing

An electrification apparatus consists of a cylindrical brass electrode which was 4 mm inside diameter and 21 mm long, a grounded counter-electrode, piezoelectric devices which vibrated the above-mentioned electrode, a high voltage electric source for particle charging and another electric source for the vibration. A pair of cylindrical electrodes were prepared for reciprocal charging, as shown in Fig. 1.

Electrification processing was carried out as follows. At first two kinds of particles were inserted into the cylindrical electrodes, respectively. The high voltage of direct current was loaded to each electrode positively or negatively, respectively. The electric voltage for vibration of these electrodes was also imposed in order to promote the particles movement. After holding these conditions, the electrode ends were opened at the same time in order to spray the two kinds of particles into the same region. They were mixed and combined each other by the electrostatic force.

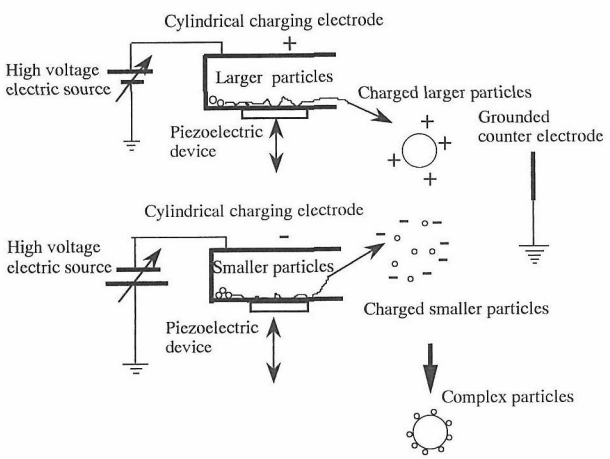

Fig. 1 Schematic diagram of particle electrification apparatus

Experimentals

Semiconductlve $BaTiO_3$ particles used in the present experiment were pressed at the load of 320 kgf/cm^2, heat-treated at 1330°C for an hour and cooled down at the rate of ll0°C/hr in the atmosphere. The raw materials were supplied by Mitsui Mining Material Co.,Ltd. Metallic In particles under 20 μm diameter fabricated using the centrifugal spray method were supplied by Mitsuwa Chemical Co.,Ltd. Figure 2 shows the morphology of the typical semiconductive $BaTiO_3$ particle.

The determination of charge quantity of particles is described below. A Faraday cage, which was electrostatically shielded by a stainless steel mesh with 1.5 mm opening, was located just under the open end of the cylindrical electrode. This mesh prevented gaseous ions from flowing into the cage together with the charged particles. The charge quantity in the Farady cage was measured by a digital electrometer.

The PTCR characteristics of complex particles were measured as follows. The cell for the measurements consisted of alumina tube (1.8 mm inner diameter, 7 mm long), In foils and stainless steel rods as electrodes. The room in the cell was filled with particles to be determined. This cell was set in an oven and heated. The resistivity of this cell was measured under a constant electric current by a coulombmeter from the room tem-

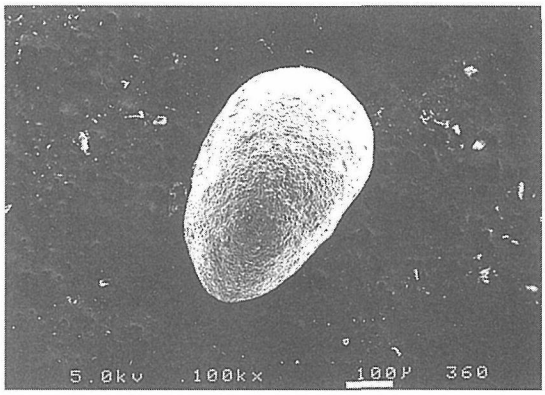

Fig. 2 Scanning electron micrograph of semiconductive $BaTiO_3$ particle.

perature to near the melting point of metallic indium.

Results and discussion

It is very important in the electrification process of particles that the charge quantity of particles is measured precisely. Gold particles, which are stable physically and chemically, were used for the determination of the charge quantity. A distinguished jump in the charge quantity was observed. The obtained charge quantity is considerably larger than one estimated theoretically described below. The theoretical relationship between the maximum charge quantity q_i by the induction charging and the electrostatic field strength E is

$$q_i = 1.65 \times 4 \pi \varepsilon_0 r^2 E \quad (1)$$

On the other hand, that by the corona charging is

$$q_c = 4 \pi p \varepsilon_0 r^2 E \quad (2)$$

where q_c is the maximum charge quantity by corona charging, ε_0 is the dielectricity in vacuum(8.854 1 x 10^{-10} F/m), r is the radius of particle(m), E is the electrostatic field strength(V/m), p is a constant depending on materials and defined by the relationship

$$p = 2(\varepsilon_s - 1)/(\varepsilon_s + 2) + 1 \quad (3)$$

ε_s is the specific dielectric constant of the particle. As the particle is conductive in the present case, ε_s becomes ∞. Therefore, p is equal to 3. The present results may be ascribed to the concentration of the electrostatic field at the end of the charging electrode.

A scanning electron microscopic image of the complex particles which were created by use of this forced electrification apparatus($\pm$ 4 kV, 30 min) is shown in Fig.3.It shows that metallic In particles are attached to the surface of a semiconductive $BaTiO_3$ particle dispersively or discontinuously by the coulomb's force. The morphology of the complex particles does not depend on the polarity of constituent particles during the processing.

Figure 4 shows the PTCR characteristics of the complex particles cell(a) and the only semiconductive $BaTiO_3$ particles cell(b). It is definite that the electric resistivity of the complex particles cell is smaller than

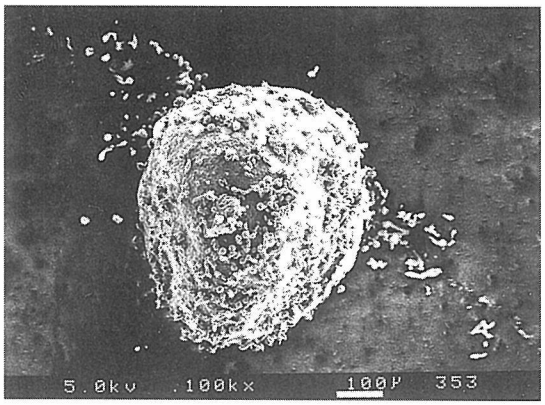

Fig. 3 Scanning electron micrograph of complex particle.

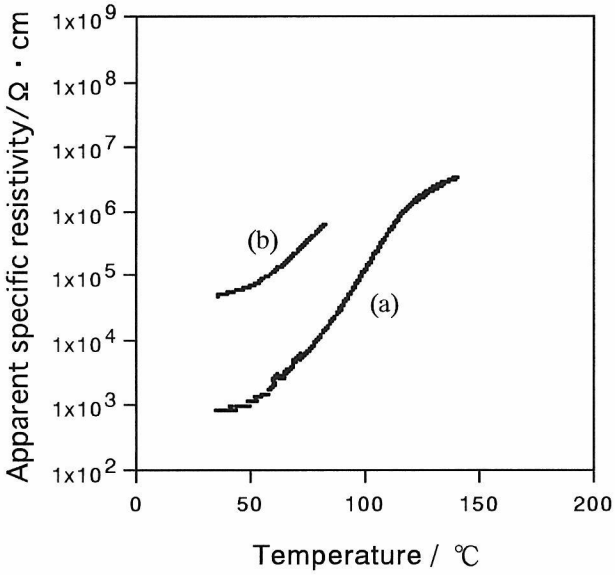

Fig. 4 PTC characteristics of In-semiconductive $BaTiO_3$ complex particles cell(a) and only semiconductive $BaTiO_3$ particles one(b).

that of the only semiconductive $BaTiO_3$ particles cell at the room temperature. These results are ascribed to the low resistivitv of metallic In particles between semiconductive $BaTiO_3$ particles and a good ohmic contact between In and semiconductive $BaTiO_3$. These complex particles can be used in arbitrary shapes and forms, different from conventional PTC materials which were sintered rigidly.

Conclusion

In order to develop the fundamental techniques of powder particle assembly, a forced electrification apparatus with vibrating cylindrical electrodes was introduced. By use of this apparatus, the charging behavior of the several kinds of particles and the preparation of complex particles were investigated.

The results obtained are as follows.

(1) The forced electrification apparatus electrified several kinds of particles, metals, semiconductors and insulators, positively or negatively.

(2) In consideration of the effective electrostatic field strength, the charge quantity per particle is estimated theoretically.

(3) By means of this forced electrification processing complex particles were prepared. This complex particle consists of semiconductive $BaTiO_3$ particle as the core and metallic In particles which were adhered to the surface of the core particle dispersively and discontinuously.

(4) Agglomeration consisting of such complex particles can be used as PTC materials.

(5) This PTC material can be used in arbitrary forms and shapes, different from conventional PTC materials.

Keywords : positive temperature coefficient of resistivity (PTCR), semiconductive $BaTiO_3$ particle, metallic indium particle, ohmic contact, forced electrification, powder particles assembly, multi-functional materials

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❒ Electrode Potential Measurement of Galvanic corrosion using Noncontact Electrode.

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In outdoor or indoor atmosphere, metallic corrosion proceeds electrochemically through thin water film on metal surfaces. The reaction rate, in this case, is expected to be larger than that in bulk solution because of accelerated mass transfer or diffusion. However little information has been available on the mass-transfer: current flow and electrode potential in water film corrosion because of experimental difficulties. For the measurement of electrode potential of metals covered with thin water film, a Kelvin technique [1][2] was applied.

Fig. 1 shows the apparatus of Kelvin probe system. The apparatus consists of three parts; 1)Kelvin probe and specimen within a chamber, 2) a driving system of the probe and specimen, and 3) a personal computer system as a controller and for data collection. A gold wire of 0.5 mm diameter was used as he Kelvin probe for potential detection. The gold probe placed in air was oscillated sinusoidally with respect to metal surface using a piezo-actuator so that it generates AC current proportional to electrostatic potential difference between the probe and metal surfaces. It is thus possible to measure relative electrode potential without immersing the probe in the electrolyte.

A galvanic couple model comprising of iron and zinc was used for demonstrating potential transition at the boundary. Experiments were carried out under dry and wet condition with variety of water film thickness ranging 100 to 3000 um, and water composition of pure water to 5%NaCl solution. Fig. 2 shows Kelvin potentials plotted as function of distance from zinc/iron

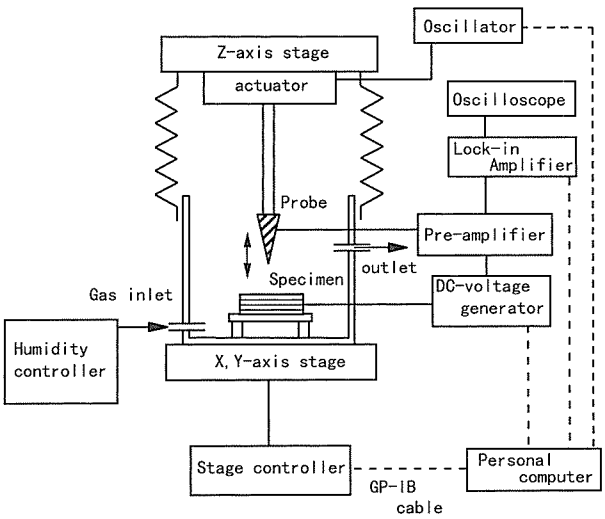

Fig. 1 Schemiatic of a potential measurement system using a non-contact electrode (Kelvin probe).

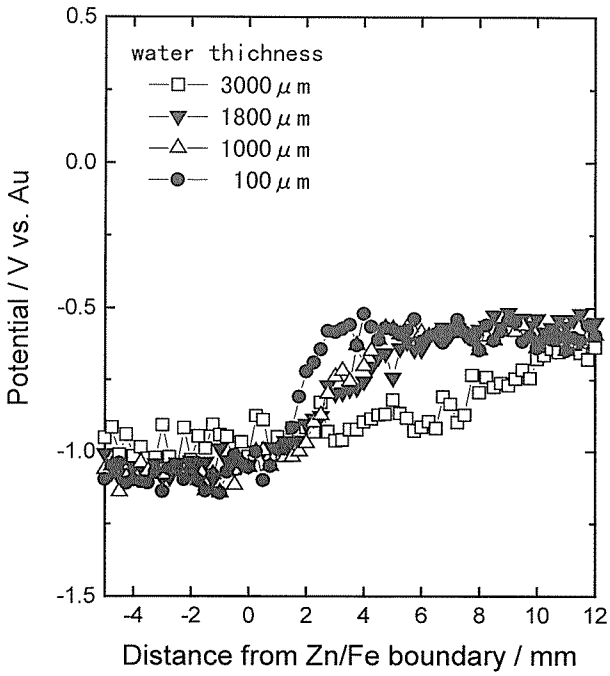

Fig. 2 Changes in Kelvin potential of a galvanic couple as functions of water thickness and distance from the boundary

boundary and as a parameter of thickness of the pure water films. Under the pure water of 100 um thick, potential on zinc was about -1.1 V while that on iron was about -0.6 V. The zone of potential transition was 3.0 mm wide penetrating into Fe surface from the boundary. This zone is formed by the action of zinc as sacrificial anode[3]. The width of potential transition was influenced by water film thickness and increased with the thickness of water films. Film thickness affects the electrical resistance of water layer, which in turn affects the potential transition mode at the boundary. The width of transition zone is also influenced by solution concentration. The width of potential transition zone under the film of the NaCl solution was larger than that under pure water. This indicates that galvanic effect reaches further into Fe surface in salt solutions.

Potential distribution at Zn/Fe boundary was explained in terms of "transmission line model" [4] as shown in Fig.3(a), where Rs denotes a solution resistance per unit length of solution on a specimen and Rp denotes electrochemical polarization resistance per unit length. The following differential equation is attained for the small length dx perpendicular to the boundary.

$$-dV = IRsdx \quad (1)$$

$$-dl = V/Rpdx \quad (2)$$

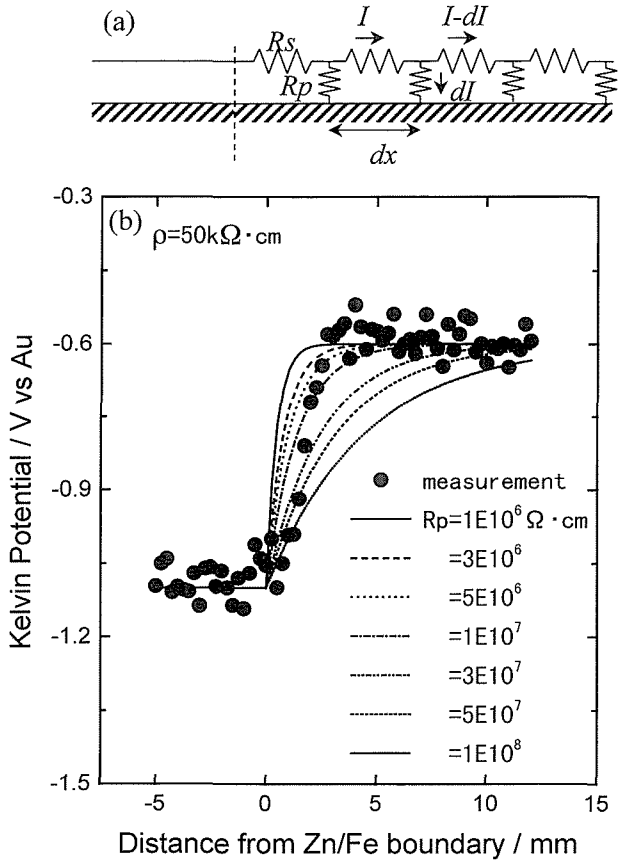

Fig. 3 Analysis by transmission line model ;
(a) schematic diagram of transmission line model,
(b) comparison of measured potentials with those calculated by transmission line model.

Potential distribution or current distribution near the boundary was obtained from the upper equation. When a thickness of water film equals to d(cm) and a parallel unit length to a Zn/Fe boundary of water film equals to u(cm), the following relationship between Rs and ordinary (three-dimensional) specific resistively p exists;

$$Rs = p/(ud) \quad (3)$$

Fig.3(b) shows an application of this model to water film of 100 um thick, where the following assumptions were made; an electrolyte on a specimen is saturated with carbon dioxide gas in air, and the dissolved zinc caution in the electrolyte is in equilibrium with the precipitates of zinc hydroxide (white rust). From this assumption, the specific resistively p of this electrolyte estimated to be 50 k Ω・cm. In the case of water film of 100 um/thick, the value of solution resistance Rs is about 5000 k Ω・cm^{-1}. Curves in Fig.3(b) show the calculated potential distribution at the transition zone where it is assumed that the polarization resistance Rp ranged from 1 x 10^4 to 1 x 10^6 Ω・cm. When the polarization resistance Rp equals to about 100k Ω・cm, the calculated curve fits the experimental results. The reported polarization resistance of iron in a neutral solution, however, is of the order of 10 k Ω・cm, which is tenth of the estimation of this investigation. This difference was due to the following factors: 1)On iron surface near the boundary, catholic reaction is rate determining, where polarization resistance is much higher than that of spontaneous corrosion because the limiting current of oxygen diffusion is controlling polarization resistance of the system: 2) The real area of exposed iron surface is smaller than the apparent area by the precipitation of white rust of zinc hydroxide.

Keywords : Kelvin probe, thin water film, galvanic couple, transmission line model

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□ Monitoring of Argon-Hydrogen Mixed Gas Arc Plasma

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Advanced Materials Processing Division

Thermal plasma under argon-hydrogen mixed gas atmosphere has been utilized for the formation of the ceramic coatings in plasma spraying process, and for the production of ultra-fine particles. The characteristics of the argon-hydrogen mixed gas arc plasma is still poorly understood. In this research, the argon-hydrogen mixed gas arc plasma structure is discussed by the emission spectroscopic measurement.

In temperature measurements of arc plasma by the emission spectroscopy, it is important whether the plasma is in the local thermodynamic equilibrium (LTE) or not. It has been discussed in Intenational Institute for Welding (IIW) Study Groap-212 meetings that temperatures measured by some methods, such as Off-axis, ArII (singly ionized argon line spectrum)/ArI (argon atom line spectrum) intensity ratio and Boltzmann-plot methods, are different each other.

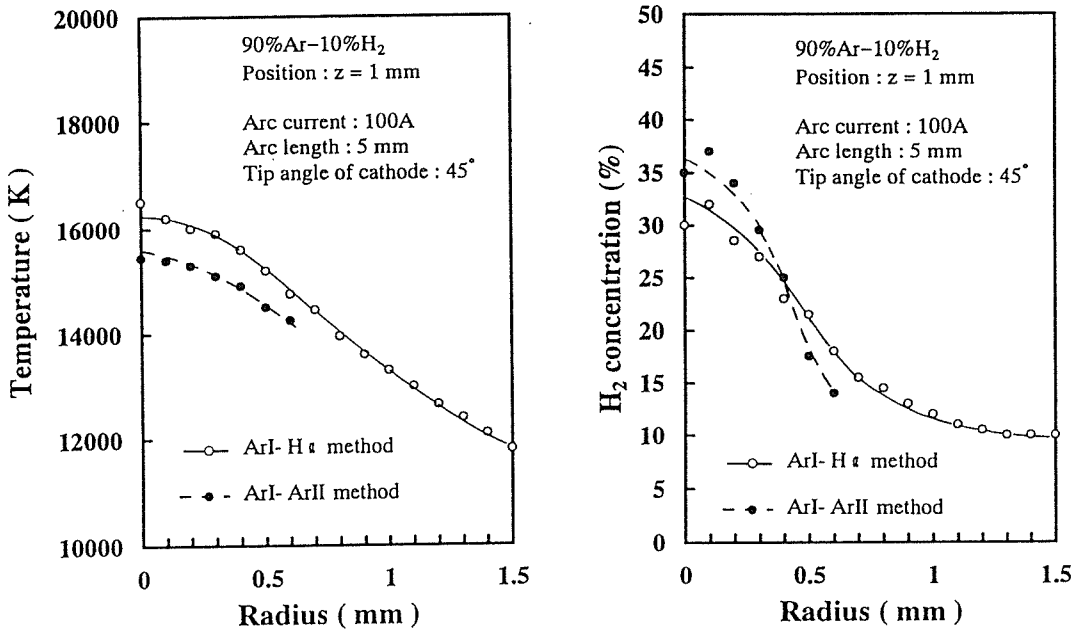
From precision measurements, it was found that the temperature distributions in pure argon arcs of 1 atm measured by Off-axis, ArII/ArI intensity ratio and Boltzmann-plot methods were almost the same. It can be expected from the results that pure argon arc plasmas are almost in the existence of LTE[1].

On the other hand, the temperature distributions in 90%Ar-10%H_2 arcs determined by Off-axis method using ArI and HI (hydrogen atom line spectrum) intensities and ArII/ArI intensity ratio method were different each other. It is suggested that the results have inconsistency in the correlation among intensities of ArI, ArII and HI line spectra in 90%Ar-10%H_2 arcs.

New Emission Spectroscopic Method

In 93's topics of NRIM Research Activities, it was proposed that the gas composition in the Ar-He mixed gas arc plasma was not homogeneous. In the present papar, the temperature (T) and hydrogen gas concentration (α) profiles of argon-hydrogen mixed gas arc plasma are discussed by developing the new spectroscopic measurements.

In Ar-H_2 mixed gas plasma at 10,000K～20,000K, the plasma components are neutral argon Ar, single ionized argon Ar^+, double ionized argon Ar^{++}, neutral hydrogen H, single ionized hydrogen H^+ and electron e^-, because the dissociation of hydrogen molecule to neutral atom becomes sufficiently significant. The densities of these species are related as a function of plasma temperature and hydrogen gas content of atmosphere in Saha's equations. And the intensities of line spectra ArI, ArII and HI are formulated with the plasma tem-



(a) Temperature distribution (b) H_2 concentration distribution

Fig. 1 Distributions of temperature and H_2 concentration at z=1 mm in 90%ar-10%H_2 arcs

perature and densities of argon atom, argon ion and hydrogen atom. Unknown parameters, T and α can be solved from Saha's equations and spectral intensity equations of ArI and ArII (ArI-ArII correlative intensities method[2]) or from Saha's equations and spectral intensity equations of ArI and HI (ArI-HI correlative intensities method[3]).

Figure 1 shows the temperature and hydrogen concentration profiles measured by ArI-Hα and ArI-ArII correlative intensities methods. Similar results are obtained by two spectroscopic methods. It is suggest that there is no inconsistency in the correlation among intensities of ArI, ArII and HI line spectra, under the assumption of LTE and the consideration in which local gas concentration of a 90%Ar-10%H_2 arc plasma is not homogeneous.

Electron Density Measurements

The results mentioned above are verified by electron densities using the Stark broadening and the infrared plasma diagnostic methods in which the electron density can be measured without the assumption of LTE.

Electron density can be determined from the full half width of H_β line profile after Abel inversion in Stark broadening method. In Infrared plasma diagnostic method (IR method) proposed by Ohji and Eagar[4], bremsstrahlung can be measured by a pyrometer with 2 μ m PbS sensor. Using Planck's equation, the intensity of bremsstrahlung can be calculated from the temperature measured by the pyrometer.

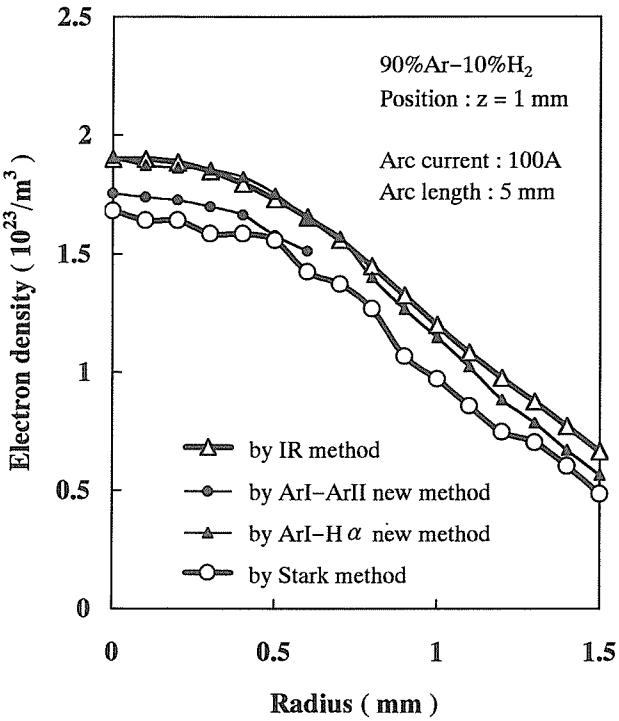
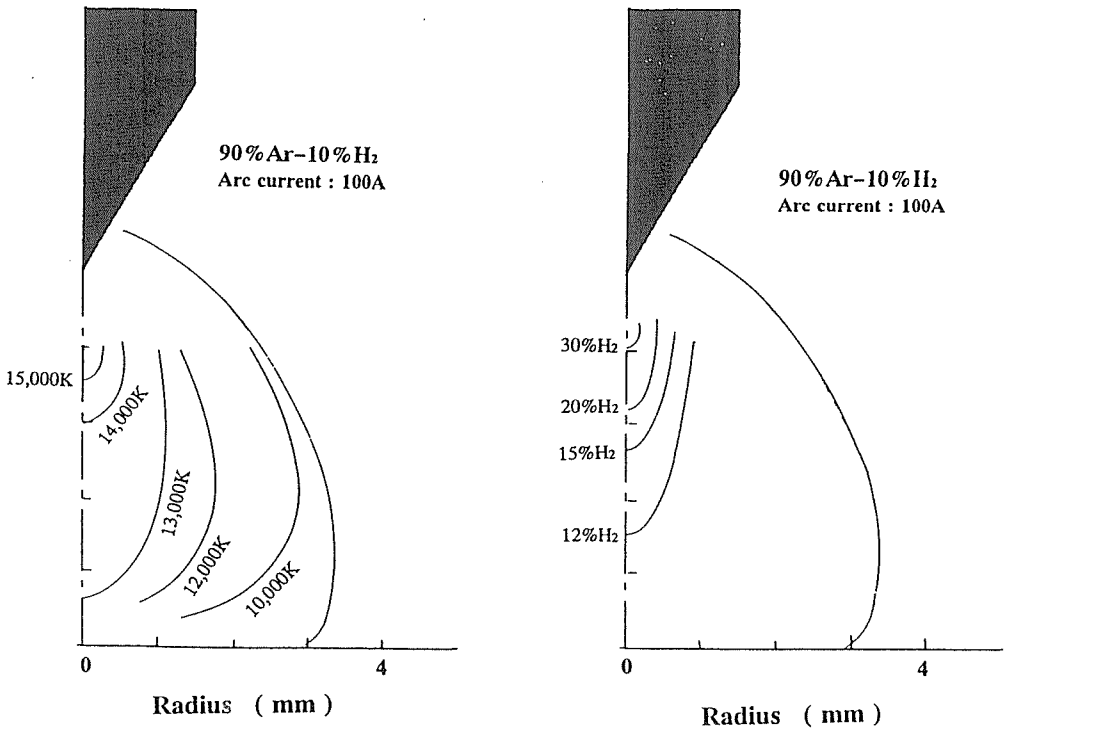


Fig. 2 Comparison among electron clensity profiles measured by stark broadening, infrared radiation and newly developed method

Figure 2 shows the electron density profiles measured by Stark broadening (○ marks) and IR (△ marks) methods in 90%Ar-10%H_2 arcs. And the plotted marks (▲ and ●) shown in Fig. 1 are the electron densities calculated from temperature and hydrogen



(a) Isotherms of plasma

(b) Contours of H_2 concentration distribution

Fig. 3 Contours of Temperature and H_2 concentration in 90%Ar-10%H_2 arcs

content by Saha's relations. In this figure, it can be found that the electron density profiles measured by each method are nearly the same.

Conclusions

All of the results led to the conclusion that the performed spectroscopic analysis for argon-hydrogen gas arc plasma, under the assumption of LTE, was valid and effective, and that the hydrogen concentration was not homogeneous in Ar-H_2 arc plasma.

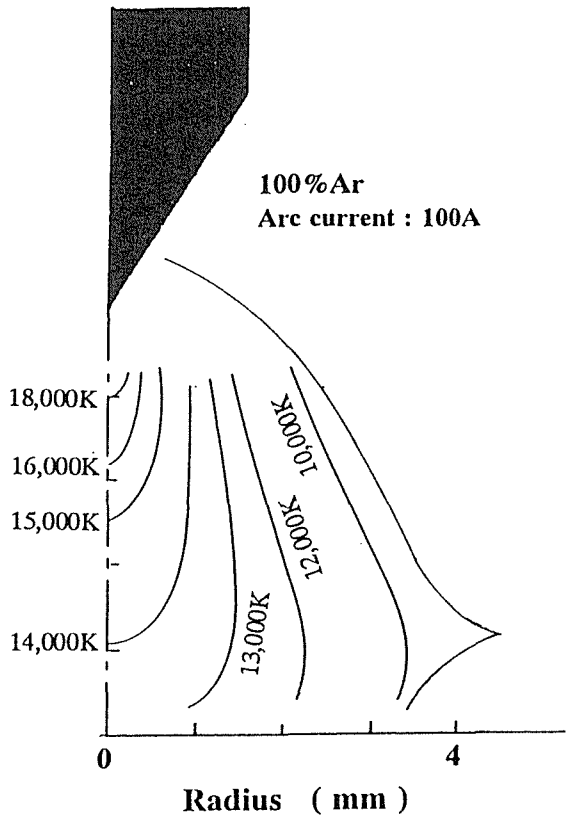


Fig. 4 Isotherms of argon arc plasma

Finally, the contours of temperature and hydrogen concentration in 90%Ar-10%H_2 arc plasma were measured by ArI-Hα correlative intensities method as shown in Fig. 3. It was found that the hydrogen gas concentration remarkably increased near the cathode on the arc center axis. When the isotherms were compared with that in pure argon arc plasma as shown in Fig. 4 measured by Off-axis method, it was found that the peak temperature near the cathode tip decreased and the plasma region near the anode constricted by the introduction of hydrogen gas into argon gas arc plasma.

Keywords: gas tungsten arc, argon-hydrogen mixed gas, emission spectroscopy, temperature, demixing, electron density

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❐ Alloy Design of Advanced Ferritic Heat Resisting Steels for 650°C USC Boilers (R&D of Structural Materials for 21st Century)

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Introduction

The strategic research on advanced ferritic heat resisting steels started in April 1997 for application to thick section boiler components such as main steam pipe and header in ultra-supercritical (USC) electric power plant which will be operated at 650 °C and 350 atmospheric pressure. The thermal efficiency is evaluated to increase to 43.8% for the USC steam conditions of 650 °C and 350 atm pressure from 38.9% for the present supercritical plants at 538 °C and 246 atm pressure. The enhanced thermal efficiency results in reducing carbon dioxide emissions and reducing fuel cost. For construction of large USC plant suitable for cyclic operation, it is necessary to develop advanced ferritic heat resisting steels, because austenitic steels are highly susceptible to thermal fatigue resulting from their low thermal conductivity and high thermal expansion. The major limiting factors to an increase in steam temperature up to 650 °C are the availability of ferritic steels with sufficient creep strength and sufficient resistance to steam oxidation. This paper describes alloy design philosophy for the development of advanced ferritic steels for 650 °C USC boilers.

Creep strength and toughness of W-stabilized ferritic/martensitic steels

The author and co-workers have investigated the microstructure-mechanical property relationship for Cr-W base ferritic/martensitic steels [1][2]. Figure 1 shows the effect of W concentration on the creep rupture strength for simple 9Cr-W steels together with solute-modified 9Cr-WVTa steels at 600 °C. The concentration of carbon was about 0.1% in each steel. The logarithm of creep rupture strength increases linearly with W concentration up to about 3% where the steels composed of 100% tempered martensite. Fine MC carbides, (V, Ta)C in the 9Cr-W-0.2V-0.1Ta steels, significantly increases the creep rupture strength. The increase in creep rupture strength by W and MC carbides correlated with the stabilization of tempered martensitic microstructure during creep. At high W concentrations above 3% where the δ -ferrite formed, the creep rupture strength scarcely increases. The 9Cr-4W-0.1C steel contained 10 vol% δ -ferrite. The ductile-to-brittle transition temperature increased with increasing δ -ferrite volume; Δ DBTT / Δ δ -ferrite

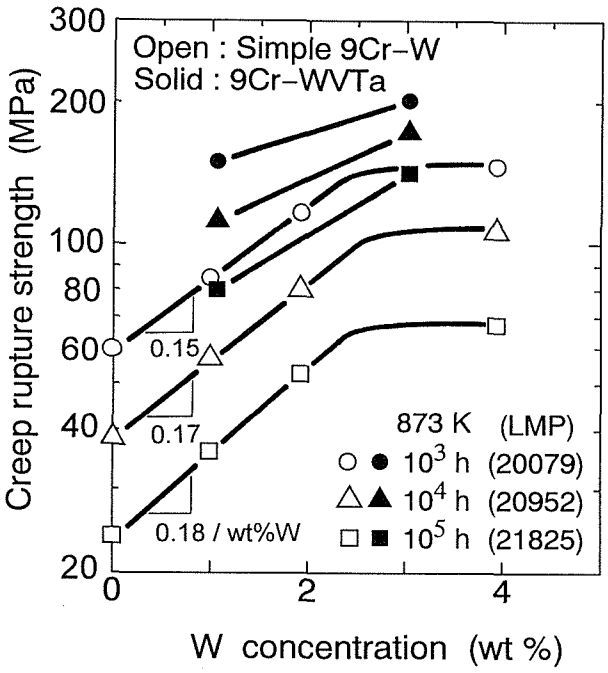

Fig. 1 Creep rupture strength of 9Cr steels at 600°C as a function of W concentration.

= 2 K / vol% δ -ferrite.

Alloy designing of high-Cr ferritic steels for 650 °C USC boilers

The steels to be developed should consist of 100% tempered martensite and the δ -ferrite should be eliminated from the view point of sufficient creep strength and toughness. The alloy compositions producing 100% tempered martensite are evaluated by Thermo-Calc. The addition of Cr and or Si suppresses the growth of oxide scale [3]. However, excess addition of these elements enhances the agglomeration of carbides as well as the formation of δ -ferrite (Figure 2), causing a decrease in creep strength and toughness. Therefore, key issues for the development of ferritic steels for 650 °C USC boilers are the stabilization of tempered martensitic microstructure for long time at 650 °C for ferritic steels containing high-Cr and or high-Si. Figure 3 shows the philosophy for the alloy designing. At high Cr above 9%, the addition of a large amount of austenite-stabilizing elements is required to suppress the formation of δ -ferrite. The austenite-stabilizing elements Ni, Cu and Co can accelerate dislocation recovery and softening at 650 °C, causing a decrease in creep strength. Co also promotes the spalling of oxide

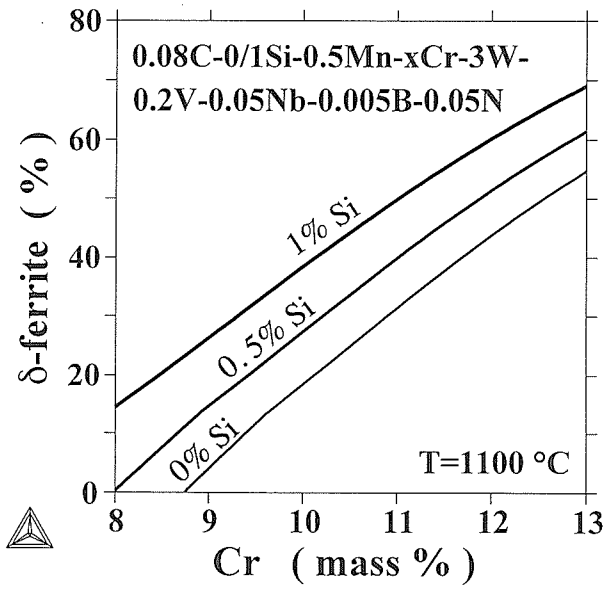

Fig. 2 Evaluation of δ -ferrite volume by Thermo-calc for (8-13) Cr-steels with 0-1% Si.

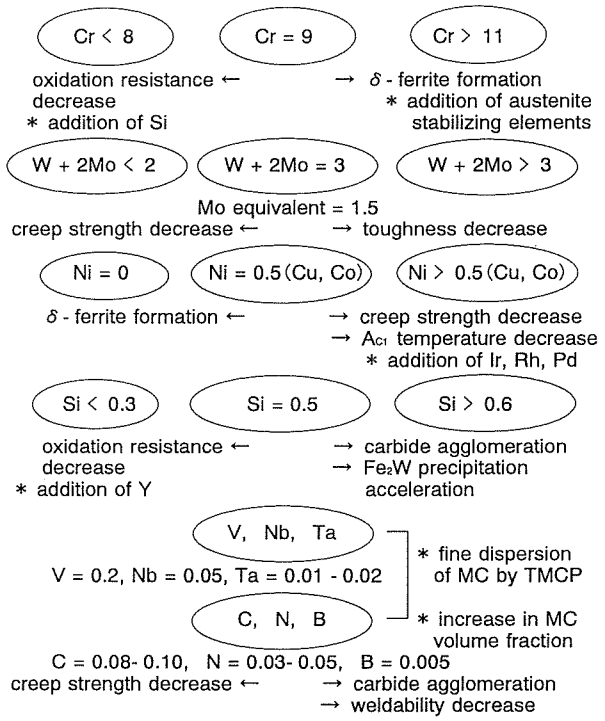

Fig. 3 Alloy design of advanced ferritic steels for 650°C USC boilers.

scale. Ir, Rh and Pd can be used to substitute for less desirable elements Ni, Cu and Co. The higher melting points and larger atomic sizes of Ir, Rh and Pd than Ni, Cu and Co lead to lower diffusion rates and hence higher resistance to softening for Ir, Rh and Pd than Ni, Cu and Co in ferritic steels. For the improvement of oxidation resistance, Y can be used to substitute for Si without any enhancement of carbide agglomeration. Several heats are being now examined in terms of tempered martensitic microstructure, resistance to softening during tempering and creep strength. In parallel with the alloy composition selection, optimization of the distributions of lath subgrains and carbides in the tempered martensite toward the stabilization of microstructure during creep at 650 °C is also examined by combination of appropriate TMCP. The preliminary results show that Ir is effective for the improvement of creep strength of ferritic steels than Ni, Cu and Co.

Keywords : ferritic heat resisting steel, ultra-supercritical power plant, alloy design, tempered martensite, creep, steam oxidation

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❒ Anisotropic Standing Wave Formation on an Au(111) Reconstructed Surface

D. Fujita, K. Amemiya, T. Yakabe, H. Nejoh
Extremely High Vacuum Station

Noble metal (111) surfaces have a Shockley surface-state, whose energy bands show parabolic dispersions around $\bar{\Gamma}$ point of surface Brillouin zone. It means that the surface-state electrons can be considered as a two-dimensional (2D) nearly-free-electron gas. Recently, scanning tunneling microscopy (STM) has revealed that standing-wave formation caused by quantum interference of the 2D electrons at potential barriers results in the spatial modulation of the surface-state local-density-of-states (LDOS). Compared with the other (111) surfaces, it is well known that the surface reconstruction of Au(111) is extremely complicated, consisting of both a short-range ($23 \times \sqrt{3}$) superlattice and a long-range "herringbone" structure. Since this reconstruction offers periodic adsorption sites, initial nucleation study of C_{60} molecules has been performed in order to fabricate ordered 2D arrays of nanoclusters. In spite of the peculiarity of the herringbone reconstruction, its potential effects on standing-wave formation of the 2D electron-waves have not been considered yet. Here, we have clarified for the first time the significant effects of the long-range reconstruction using a low temperature STM at 30 K.

Figure 1(A) shows a STM image of the Au(111) surface (V_t = 30 mV, I_t = 0.3 nA), which exhibits the reconstruction patterns consisting of several pairs of bright stripes running in a zigzag way. The observed superlattice can be interpreted as a manifestation of quasi one-dimensional soliton-like misfit structures, indicating that 23 gold surface atoms are packed on 22 lattice-spacings in a rectangular ($23 \times \sqrt{3}$) unit cell. Consequently, the surface atoms are forced to occupy the three atomic-stacking sites such as normal fcc (C+ABC...;"f"), hcp(B+ABC...;"h"), and bridge sites ("s"). Since the transition between fcc and hcp can be described by soliton expression, the transition regions are named as soliton walls. The complex long-range reconstruction stems from spontaneous formation of elastic stress-domains in order to relieve the anisotropic stress in a more uniform manner. Figure 1(B) is a constant-current STM image obtained by approaching the bias voltage very close to zero (V_t = -2 mV, I_t = 0.3 nA). In marked contrast with the topographic character in Fig. 1(A), the image shows emergence of long-range spatial oscillations. With such a low tunneling voltage, constant-current imaging can probe directly the Fermi-level LDOS contour of the surface-state electrons. On the upper terrace, especially near the step

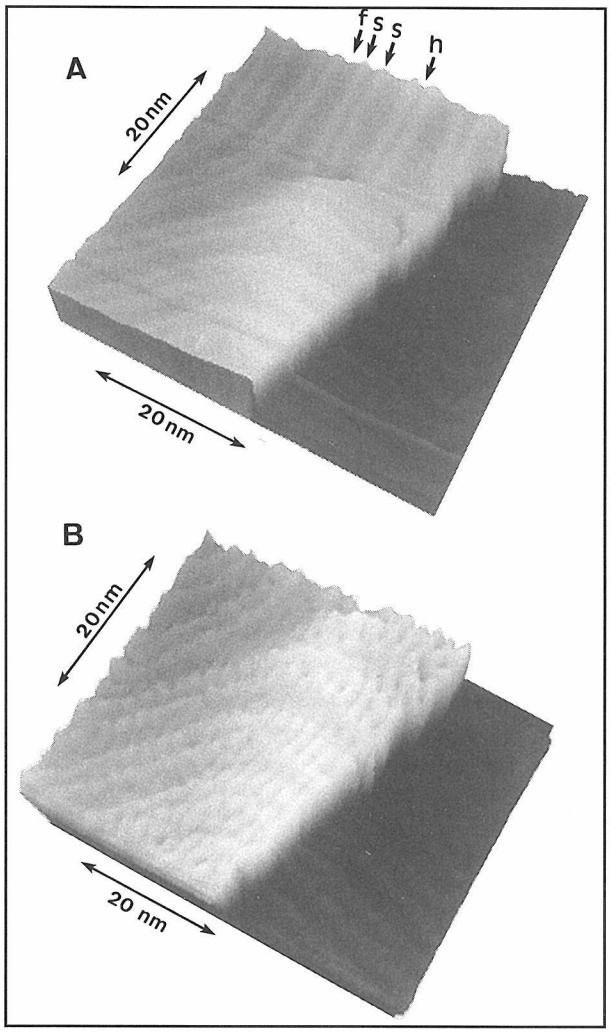

Fig. 1 STM images of a reconstructed Au(111) surface.
(A):V_t = 30mV, (B):V_t = -2mV

edge, the ripples with almost constant periodicity are clearly observed. The oscillatory structure can be explained by formation of standing waves in the LDOS ρ ($k_{//}$, x), caused by interference of electron waves at a step edge. Modulation in the surface-state LDOS can be expressed as follows:

$$\rho\ (k_{//}, x) = \rho_0\ (1-J_0(2k_{//}x)) \qquad (1)$$

where x is the distance from the potential barrier, $k_{//}$ is the 2D wavenumber of surface-state electron, ρ_0 is the averaged surface LDOS, and J_0 is the zeroth-order Bessel function. The observed oscillation has a characteristic periodicity L of 1.8 $\pm$ 0.1 nm, from which the lateral wavenumber can be deduced to be $\sim$ 1.7 nm^{-1}, corresponding well to the 2D Fermi-level wavenumber (k_F = 1.73 nm^{-1}). The standing waves are

also observable at the natural nanometer-scale structures. In the case of a monolayer island, more enhanced oscillation is observed because of stronger confinement effect. On the contrary, in the case of a monolayer depression, more faint oscillations are found as shown in Fig.2, which suggests the weaker potential barrier for this configuration.

Although the general feature of standing-wave oscillations can be explained by equation (1), it should be noted that they are strongly affected by the herringbone reconstruction. Careful observation suggests that, in the vicinity of the step, the amplitudes of the standing waves in the hcp region and the soliton walls are larger than that in the fcc region. This observation suggests that the surface-state LDOS is distributed inhomogeneously on the surface. Moreover, one-dimension-like wave propagation along the pairwise soliton walls can be deduced. On the terrace relatively farther from step edges, we can observe the enhanced oscillation only along the soliton walls. That is, the surface-state electron waves look like propagating quasi-one-dimensionally along the soliton walls. In order to show the feasibility of the quasi one-dimensional state, we have developed a simple one-dimensional wave-guide model as shown in Fig. 3. Here, it is assumed that the potential of surface-state electrons is distributed inhomogeneously, depending on the three different regions. It is also assumed that the surface-state electrons are confined in an infinitely long wave-guide of width d, which is composed of a pair of soliton walls and a hcp region. Although this model is a rough approximation, the calculated probability density can qualitatively reproduce a similar behavior to the observed spatial modulation of the LDOS. This result suggests the possible anisotropic nature in the local Fermi surface. In fact, by Fourier-transforming the STM images of the standing waves, an ellipsoidal Fermi surface can be extracted for the surface-state electrons, instead of an isotropic Fermi circle.

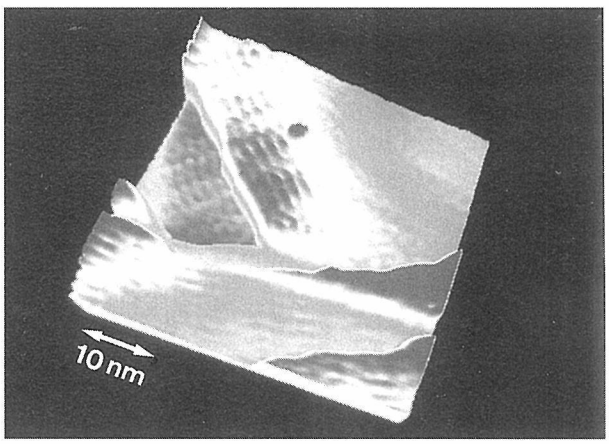

Fig. 2 Standing wave in a monolayer depression of an Au(111) surface.

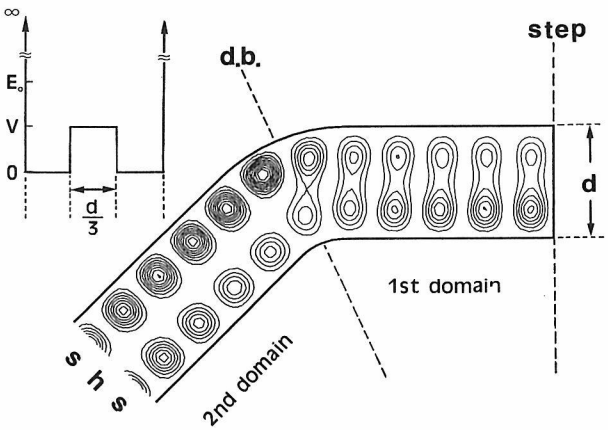

Fig. 3 Model for an enhanced standing-wave formation along soliton walls.

In summary, overall results clearly show that the quantum interference of electron waves can be visualized with our Fermi-level STM imaging technique with an extremely low tunneling voltage. Combined with a 2D Fourier transform, it will offer a novel evaluation method for quantum interference of other 2D electron systems.

Keywords: Au(111); Quantum Interference; Scanning Tunneling Microscopy; Surface Reconstruction; Surface State; 2D Nearly Free Electron Gas

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❒ Extreme High Vacuum Substrate Delivery System among Six Instruments for Nanoscale Material Research

M.Tosa
Extreme High Vacuum Station

Introduction

Smooth creation of advanced materials with an atomically controlled structure which is expected to exhibit excellent quantum properties requires the development of the advance physical environment of the extreme high vacuum (XHV) integrated process. The obstacle of the fabrication of atomic scale structure material is atomic scale contamination by gas adsorption from the environment. The XHV integrated process can offer an atomically clean environment during such multi-operation as material processing and analyses necessary for atomic scale materials fabrication due to the super clean atmosphere with little gas molecules of extreme high vacuum. The research is, therefore, carried out on the construction of XHV integrated process offering the super clean environment on an atomic scale necessary for the fabrication of nano structure .

XHV Integrated Process

The XHV integrated process with six instruments

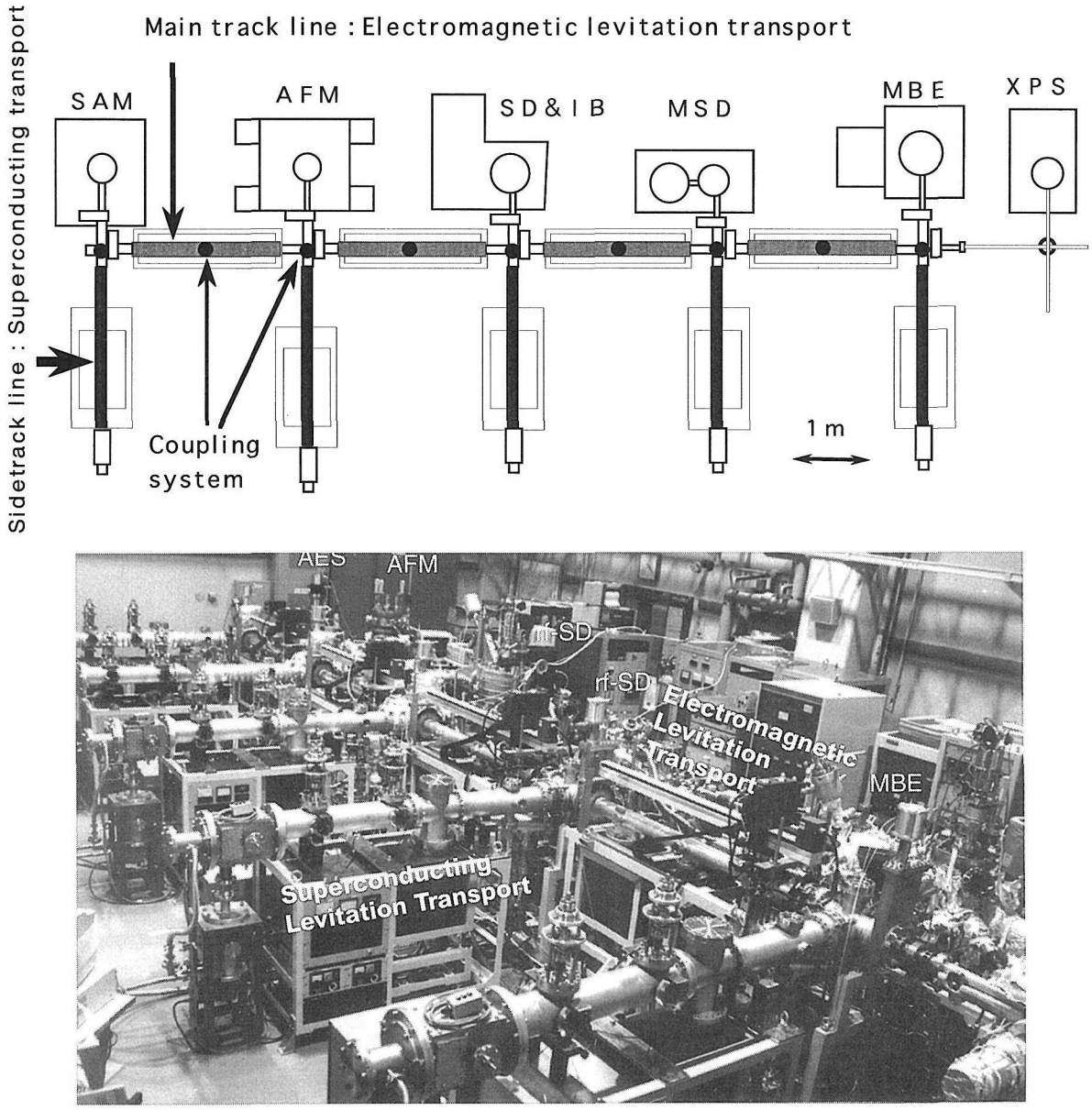

Fig. 1 The shematic diagram and the photograph of the XHV integrated process

was developed for nano scale continuous processing. Figure 1 show the schematic diagram and the whole photograph of the integrated process. The process consists of a main track line chambers for magnetic levitation long-distance transport, a sidetrack line chambers for superconducting magnetic levitation short-distance transport, interconnecting chambers, pumps, valves, and gauges. This process is a unit to combine three film preparation chambers (MBE and sputter deposition systems) and three surface analysis chambers (SAM, XPS and AFM/STM), and can deliver samples among these connecting instruments chambers.

Production of XHV environment requires a very low outgassing chamber, high sensitivity gauge system, high performance vacuum pumps and so on. Type 304L stainless steel employed as a chamber material has little intergranular corrosion and outgassing sources at welds because of low carbon content. The surface of the inside wall of the chambers was electrolytically polished in phosphoric-sulfuric acid solution and the chambers were annealed at 823 K in a high vacuum for enough outgassing operation. A titanium getter pump (pump speed: 1.8 $m^3 s^{-1}$) and a ion pump (pump speed: 0.2 $m^3 s^{-1}$) evacuate each main chamber unit and each side track line chamber unit. The properties of extreme high vacuum were evaluated with an extractor gauge and a quadrupole mass spectrometer with the separation of an ion source and a quardrupole analyzer. Each main chamber unit starts to be pumped with a iron pump after whole the process is baked out with mantle heater system for 862 ksec at 423 K to 473K keeping the turbo molecular pumping system assisted by rotary pumps in operation.

Substrate Delivery

Transport system to be used in XHV should generate no particle because particles are large sources of outgassing. It is essential to use no sliding components to keep the system XHV. A magnetic levitation transport system can meet the demand as it employs no sliding motion so that XHV may be kept during transport. We adopted two types of magnetic levitation transport, one is electromagnetic levitation transport and the other is superconducting levitation transport.

The electromagnetic levitation transport system is used for the main track line because of easy line extension and quick start of transport operation. Figure 2 (a) shows the schematic of the electromagnetic transport. A stator unit over the main track chamber has the electromagnets to levitate a carrier by magnetic attractive force, the linear synchronous motor to drive, and the position sensors as well as gap sensors with electromagnets to stabilize the levitating carrier. The electromagnets below the stators can control the levitation gap of the carrier about 2 mm between the stators and the carrier. The carrier in the chamber is completely shielded by the type 304L stainless steel sheet for the decrease in outgassing. The running carrier can be stopped within the error of 0.5 mm even in transporting a sample holder at the top speed of 5 cm s^{-1}.

Superconducting levitation transport system is used for the sidetrack because of the easy positioning stability and toughness against mechanical shock. Figure 2 (b) shows the schematic of the superconducting levitation transport. The transport consists of the sidetrack chamber, the cooler filled with He gas coolant which can be cooled by a freezer at the back of the chamber and the carrier rod with a sample holder fork at the head. Three oxide discs with 2 inches in diameter of $YBa_2Cu_3O_7$-X superconductor driven by a long bolt rod in the cooler are cooled down below Tc of 90K and cause the superconducting effect of pinning and the diamagnetism on the three discs of SmCo attached to

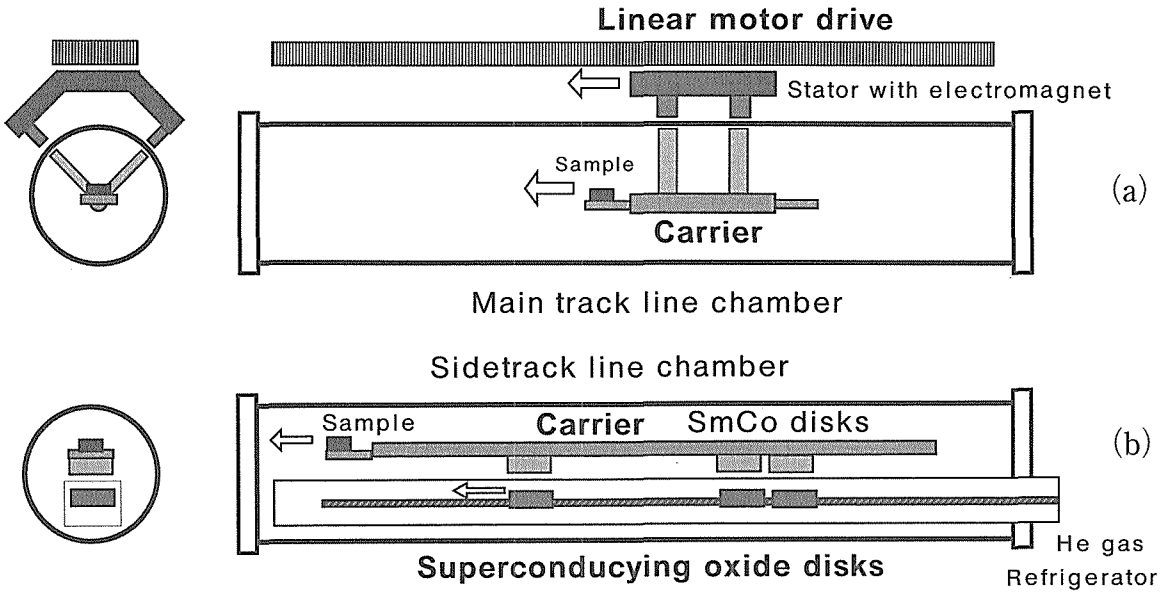

Fig. 2 The schematic of the electromagnetic levitation transport (a) and superconducting levitation transport (b).

the bottom of the carrier. It takes more than 6 hours to cool the transport system down to Tc from room temperature. The pinning effect is strong enough to stably levitate the carrier with a certain gap through the cooler wall and to drag the carrier accurately without any stabilizer nor any sensor. The carrier can transport a sample at the top speed of 10 cm s^{-1}.

A sample is delivered from the carrier of the sidetrack line to the carrier of the main track line by lifting up and down hoist system in the interface chamber. The hoist system also delivers a sample holder between main track line units. Vacuum pressure of 9 x 10^{-10} Pa was obtained in the a main track line chamber but the pressure increased up to 2.0x 10^{-9} Pa after opening the gate valve between the main track and the connected instrument chamber. A sample could be transferred from a hoist stage in the connecting chamber for the sidetrack chamber to the other in the main chamber within the pressure change of 10^{-11} Pa. This indicates that the current system is successful in the transport in near XHV pressure range but that additional improvement of the system is required for the stable transport in XHV.

The first phase is completed on the project to construct the XHV integrated process with levitation transports and the second will start in order to offer the integrated process for the fabrication of advanced substrate and nano scale material with quantum properties on the substrate.

Keywords : integrated process, six instruments, extreme high vacuum, in situ substrate delivery, levitation transport

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❒ Development of a hip joint simulator

K. Nakazawa, M. Sumita, N. Maruyama and A. Yamamoto
Biomaterials Research Team

A hip joint simulator has been developed to evaluate wear of artificial hip joints. In conventional hip simulator that had been developed so far, the loading axis of hip joint is in general aligned vertical. When the femoral head is located on top and the socket is located on bottom, heavy wear particles like metallic wear debris tend to remain onto the socket. When the socket is located on top, air is sometimes entrapped accidentally into the lubricating fluid between articulating surfaces. There is a strong suspicion that the wear characteristics of the hip joint are influenced and hence a long-term evaluation test is difficult to perform by the existence of these air and wear debris.

In the newly developed simulator shown in Photo. 1, the loading axis is aligned horizontal to avoid above suspicion. This simulator is designed to simulate the motions, loads and environment experienced by hip joint during the walking process, and to endure for a long-term test. Motions and loads are applied by programmable servohydraulic actuators. Up to six hip joints can be tested at a time. Each hip joint specimen has an environmental chamber in which lubricating fluid is filled to assure full immersion of the joint components in the fluid. Using this simulator, the effects of temperature and viscosity of the fluid on the wear of hip joint was studied. As the test specimens, popular hip joints that are composed of corrosion resistant alloy head and ultra-high molecular weight polyethyrene (UHMWPE) socket were used. The lubricating fluid was 0.9% saline maintained at 37°C or 40°C (viscosity ca. 1cp). To raise fluid viscosity, Dextran was added to the fluid. The maximum range of the swing motion was 50°, and the maximum load was 300kgf. The walking cycle speed was 1Hz. Under these conditions, a continuous test run during nine weeks was performed, which corresponds to three years' living time with conventional walking. The weight loss of UHMWPE socket was largest in 37°C saline (viscosity 1cp), then in 40°C saline (viscosity 1cp), and smallest in 37°C saline (viscosity 20cp). The wear volume of UHMWPE decreased with the increase in fluid temperature and viscosity. The temperature dependence of wear was contrary to a generally accepted concept. This result suggests that the test conditions should be selected properly for the valid evaluation of hip joint. It is expected that this simulator will contribute to the establishment of standard evaluation test method.

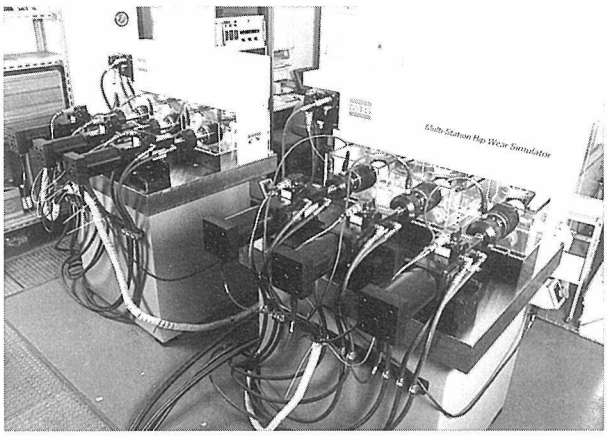

Photo 1

Keywords: artificial hip joint, long-term evaluation test, wear, UHMWPE, lubricating fluid, viscosity, test temperature

❒ A List of Research Program

1. Effects of Pressure on the Physical Properties of Magnetic Materials
2. Characterization and physical properties of metal oxides by infrared and Raman spectroscopy
3. Two Dimensional Photoelectron Spectroscopic Studies on Surface Structures and Properties
4. Investigation on Synthesis and Characterization of Metal Complexes with High Electron-spin Multiplicity
5. Study on the Improvement of Analytical Technologies for Metallic Materials
6. Effect of High Magnetic Field on Phase Transformations in Fe-based Alloys and its Application to Structure Control
7. Effects of High Magnetic Field on Solid/Solid Phase Transformations
8. Diffusion in Ordered Alloys and Preparation of Composition Gradiented Materials
9. Hydrogen Behaviors at Interfaces in Alloys
10. Research on Electrode Reaction between Metallic Ions and CarbonaceousMaterials / Research on Electrochemical Characteristics of TiAl alloy
11. Energy Conversion Materials Fabricated with Composite Structure
12. Thermal Stability of Intermetallic Compound Matrix Composites Reinforced with Fibers
13. Single crystal growth of new nonlinear-optical materials by means of controlling crystal symmetry
14. Crystal-quality improvement of nonlinear-optical oxides by Floating zone method
15. Establishment of Multidimensional Evaluation of Human Senses for Materials Design
16. Development of Knowledge Database for High-Tc Superconducting Materials
17. Theory of Thermal Reaction on Solid Surfaces
18. A Molecular-Dynamics Study of the Initial Process of Fracture
19. Thermodynamic Analysis of Transition Process from Metastable to Stable Phases
20. Development of Third Generation Nickel-Base Single Crystal Superalloys
21. Development of Integrated System for Distributed Materials Scientific data using Network
22. Development of Virtual Experimental Technologies for Material Design
23. Study on the effective Acquisition Method of Metallurgical Information
24. Thermodynamic Analysis of Formation Processes of Metastable and Stable Phases
25. A Computer-Simulation Study of the Plastic Deformation of Crystals
26. Theoretical study on new phenomena in ultrathin magnetic films and multilayers
27. Strengthening Mechanism of High Strength Composite Materials
28. Development of High Strength Metal Matrix Composites with Excellent Physical Properties by the Advanced Bronze Method
29. Relationship between Fatigue Softening / Hardening Behavior and TEM Structure of Titanium Alloys
30. Cyclic Deformation in a Corrosive Environment
31. High Temperature Strength and Fracture of Reinforced Oxide- and Nitride-Base Ceramics
32. Research on Fundamental Science of Frontier Ceramics
33. High Temperature Mechanical Properties of Particulate Reinforced Titanium-Based Metal Matrix Composites
34. Effect of Cryogenic Temperature and Gas-environment on Deformation and Fracture Behavior
35. NRIM Long-Term Fatigue Data Sheet Project- I
36. Relationships between Fatigue Behavior and TEM Structure of Titanium Alloys
37. Fatigue Strength in a Corrosive Environment
38. Fabrication of Ni3Al-Base Intermetallic Compounds by Unidirectional Solidification
39. Basic Study on Refining of Molten Metal and Controlling of Solidification by Electromagnetic Force

40. Solidification in the Strong Magnetic Field
41. New continuous Steelmaking Process
42. Investigation on Nucleation and Crystal Growth Mechanism under Heterogeneous Ambient Phase
43. Spectroscopic and Electrochemical Investigations of the Metal Complexes with an Unusual Electronic Structure
44. Improvement of Mechanical Properties of Intermetallic Compounds by Crystal Growth Control
45. Study on the Mechanical Properties of Directionally Solidified Intermetallic Compounds
46. Study on combustion Synthesis
47. Study on the Efficiency of Resonance Photoionization Process
48. Oxidation Properties of Sintered TiAl Intermetallic Compound
49. Synthesis and Utilization of Mesoporous Materials
50. Fundamental study on microstructure control by slurry processing
51. Photocatalytic Properties of Ultrafine Particles
52. Control of the Oriented Single Crystals of Refractory Metals through Solid State Processes
53. Selenium(VI) Removal from Wastewater by a Fluidized Bed Process
54. Basic Technology Development of Materials Procession in a Short-Duration Microgravity Environment
55. Solidification Processing For Particle Dispersed Unidirectionally Grown Composites
56. Research and Development of Recyclable Simple-System Alloys
57. Fabrication of Highly Functional Composite Cast Irons
58. Micro Structure Control with Plastic Deformation under Mashy State
59. Brazing Experiment under Microgravity
60. Influence of Surface Composition on Joining of Materials
61. Diffusion Bonding of Stainless Steel and Metals of Group Ⅳ ~ Ⅵ
62. Monitoring and Control of Thermal Plasma
63. Coatings Formation by Powder Deposition Processes
64. Evaluation of the structure and properties of advanced thermal spray coatings
65. Metastable Phase Solidification from Undercooled Liquid by Inducing External Nucleation Seed
66. Characterisation of Laser Induced Plasma and Interaction between Laser and Plasma
67. Application of In-situ Strain Measurement by the Laser Speckle Method
68. Synthesis of Superconducting or Supermagnetic Ultrathin Films by Use of Ion Implantation Technique
69. Formation of Fe16N2 by nitrogen ion implantation into Fe films and post annealing in magnetic field
70. Generalized Rules and Guide for Use of Fatigue Data
71. Modeling and Evaluation of Advanced Materials - A coordinated Interlaboratory Research
72. Long-term Creep-fatigue Properties of 316FR Stainless Steel for Fast Breeder Reactor
73. Remaining Life Prediction for Weldments in FBR based on Creep Damage Evaluation
74. Evaluation of corrosion damage at atomic level
75. Development of metal having environmental cleaning ability by photo catalysis reaction
76. Fundamental Studies of the Microbial Reaction with Inorganic Compounds
77. Effect of the Interfacial Damage on Mechanical Properties for Ti-Based Matrix Composites
78. Fundamental Study for Electromagnetic Evaluation of Materials
79. Effect of Aging Degradation on Localized Corrosion of Structural Materials for Light Water Reactors
80. NRIM Creep Data Sheet Project-V
81. Nanoscopic Materials Damage Evaluation
82. Ductile versus Brittle Behavior of Structural Steels
83. Improvements of High Temperature Properties in Materials for High/Ultra-high Temperature Use
84. Study on Transient Behaviour of Deformation and Fracture at the Elevated Temperature
85. Characterization of Creep-Damaged Microstructure of Stainless Steels by Computer Aided Quantitative Metallography

86. Stability of Tetragonal Phase and its Effect on High-Temperature Mechanical Properties of Zirconia
87. Studies on Evaluation of Durability of Organic Coatings for Construction Materials
88. Development of Porous Ceramics Impregnated with Ionic Conductive Materials
89. Relationship between Fatigue Crack Propagation and Cyclic Deformation of Small Specimens
90. Evaluation of Fatigue Strength of Welded Joints in Synthetic Sea Water by the New Testing Method
91. Chemical Analysis of Organotin in Marine Environmental Samples
92. Using Experimental and Theoretical Depth Resolution Functions for High Accuracy Depth Profiling
93. Development of High-Tc Oxide Superconducting Wires and Tapes
94. Study on Anomalous Magneto-Resistance for Bi-2212/Ag Composite Superconducting Tape
95. Characterization and Application of Superconducting Thin Films Synthesized by Atomic layer-by-layer and Epitaxial Growth Methods
96. Study on Atomic Scale Engineering of High Performance Films
97. In situ TEM Observation and Structural Analysis of High Tc Superconductors for Fusion Reactor at Low Temperatures
98. Computational Analysis of Mechanical Properties and Structural Design of Materials Taking Microstructures into Account for Nuclear Power Plants
99. Real-Time Investigation on Surface Reactions and Defect Growth Processes under Irradiation
100. Study on the Deformation and Fracture of Materials under Irradiation
101. Influence of Nuclear Transmutations on Low Activation Structural Materials for Fusion Reactor Application
102. Research on Utilization Technique of "Data-Free-Way" system for Nuclear Materials
103. Synthesis and Characterization of Isotopically Controlled Materials
104. High Performance Materials for Severe Environments-I
105. Mesoscopic Structure Control and Properties of Intermetallic Compounds
106. Heterogeneous Structure Design for High Plasticity and Wide Applicability of Simple System Alloys
107. Development of Advanced Shape Memory Thin Films by Sputtering
108. Fabrication and Fundamental Studies on the Materials with Mesoscopic and Aperiodic Structure
109. Studies on Viscoelasticity and Structure of Supercooled Liquid in Amorphous Alloys
110. Atom probe microanalysis of advanced metallic materials
111. Research and Development of High Temperature Ti2AINb Titanium Aluminide-Based Composites
112. Microstructure Control and Properties of Intermetallic Compounds in Ti-Al System
113. Magneto-optical Study with The NRIM Hybrid Magnet
114. Studies on the Highly Correlated Electron Systems under Multiple Extreme Conditions
115. Development of Basic Superconducting Technology for High Field Micro-SMES
116. Crystal symmetry design for nonlinear-optical crystals
117. Research and Development of High-Performance Light Alloys for Hydrogen Storage
118. Development of Particles Assembly Technology for Integration of Functions
119. Fundamental Research on Flexible Structure and Machine Systems Using Advanced Materials
120. Creation of Multi-Functional Materials by Assembling Elemental Components
121. Development of Advanced Shape Memory Thin Films by Sputtering
122. Fundamental Study on Development of Biointrgrated Materials
123. Investigation on the basic structural materials in the next era
124. Environmental Life Cycle Analysis of Metals
125. Study on the Processing and Assessment of Ecomaterials
126. Formation of amorphous alloys and their physical properties
127. Development of 1 GHz NMR Spectrometer
128. Stability of Superconducting Materials

129. Development of High Strength/High Conductivity Materials for High Field Magnets

130. Essential Technology Development and Design for New Water-Cooled Magnet

131. Microstructure and Electromagnetic-Characteristics Studies on V3Si Multifilamentary Superconductors

132. Development of Magnetic Separation Control System

133. Developments of high strength oxide superconductors for high magnetic field application

134. Evaluation of oxide superconductor wires

135. Basic Research for the Control of Chemical Reactions by High Magnetic Field

136. Study of regenerator for ultra-low temperatures

137. Processing and Development of Isotopically Controlled Materials(ICM)

138. Analysis/Evaluation of Atomic Scale Compositional Change in Materials Due to the Radiation Damage

139. Characterization and Control of the Optoelectronic Properties of Small Crystalline Materials with Electron Probe Analysis

140. Joint research on the structure and properties of implanted and precipitated semiconductor nanocrystals

141. Study on Detection and Evaluation of Radiation Damage in Extreme Particle Fields

142. Development of analytical techniques for characterization of nuclear materials using new generation synchrotron x-rays

143. Development of Fundamental Technologies for Excited Neutral Beams

144. Advanced characterization of micro and nano meter scale structure of materials by brilliant synchrotron x-rays at the SPring-8

145. Surface analysis database

146. Self-control of surface composition of thin film and its application to field emitter

147. The Evaluation and Control of Material Self-organization

148. Fabrication and Characterization of Compond Semiconductor Nanometer Structures

149. Materials with Atomic Scale Structures(COE Project)

150. Study on Melting of Refractory Metals by Cold Crucible Levitation Melting Method

151. Fabrication of particles dispersed materials through controlling of solidification interfaces

152. Random Fatigue of Notched Plates

153. Mechanical Characterization of Thin Layered Structures

154. Study on Strengthening of Ferrite Matrix Steels for Welded Structures

155. Advanced Ultra-High-Strength Steels over 1500 MPa class

156. Strategic Research on Advanced Ferritic Steels for 650℃ USC Boilers

157. Enhanced Durability of Structural Steels in Marine Enviroment

Research Program

Characterization/Properties

1 Effects of Pressure on the Physical Properties of Magnetic Materials

A. Matsushita, Materials Physics Division
[April 1996 to March 1999]

We have been pursuing a program in high pressure physics, centering around the magnetic and transport properties of rare-earth intermetallic compounds and high-Tc superconductors. One of the current research interests focuses on the superconductivity of $Pr_1Ba_2Cu_3O_{7-\delta}$ (Pr123) which does not exhibit superconductivity in usual synthesis conditions. We have measured the structure and the pressure dependence of Tc for superconductor Pr123 and have compared them with those of non-superconductor Pr123. Our program also encompasses developing high pressure techniques. Present efforts in this area are directed toward constructing a diamond-anvil X-ray diffraction system equipped with a monochromator. We are planning to perform structure analysis with Maximum Entropy Method (MEM) using this system.

Keywords : high pressure, magnetic materials, high-Tc superconductor

2 Characterization and physical properties of metal oxides by infrared and Raman spectroscopy

T. Hirata, Material Physics Div.
[April 1995 to Maerch 1998.]

Because phonons play a significant role in many phenomena of solid state physics, we have been concerned with infrared and Raman spectroscopic studies of meal oxides, for understanding their physical properties with reference to phonons. The following topics should be addressed. The octahedral distortions in the mixed oxide system are under study, focusing on the changes in frequency, linewidth and intensity of optical phonons upon substitution of other metal cations with different ion radius and/or valence for constituent parts.

We are also involved in structural phase transitions in metal oxides; the tempertaure dependence of infrared and Raman spectra for metal oxides that undrergo structural phase transitions, provides important information to undrerstand the mechanism of phase transitions. For example, the temperature dependence of the Raman-active B_{1g} and A_{1g} modes in superconducting YNi_2B_2C tells about an electron-phonon coupling based on whether there is any superconductivity-induced change of the modes near a superconducting transition temperature T_c.

Keywors : phonons, infrared/Raman spectroscopy, metal oxides, octahedral distortions, phase transitions, superconductivity.

3 Two Dimensional Photoelectron Spectroscopic Studies on Surface Structures and Properties

M. Shimoda, Materials Physics Division
[April 1995 to March 1997]

Sulfur(S) terminated GaAs (001) surface is the fundamental stage for droplet epitaxy, which is a potential technique to fabricate nano-scale structures on the GaAs surface. In order to establish this technique, the mechanism of the droplet growth must be understood. Correct knowledge of the surface structure are essential for this purpose. So far, STM studies have revealed that a (2x6) reconstruction is dominant for this surface and that the adsorbed S atoms form S-S dimers. In order to investigate the structure of the surface S layer, we have observed S 2s X-ray photoelectron diffraction (XPD) pattern and Ga $L_3M_{45}M_{45}$ and As $L_3M_{45}M_{45}$ Auger electron diffraction (AED) patterns[1]. No forward scattering peak was found in the XPD pattern of S 2s emission, indicating that adsorbed S atoms form a single layer. AED patterns were compared to calculated ones with a single scattering cluster model. The S-S bond length and the interlayer distance between the S and Ga layer are estimated to be 0.24 nm and 0.12 nm, respectively.

[1] M.Shimoda, S.Tsukamoto and N.Koguchi, "Photoelectron and Auger Electron Diffraction Studies of Sulfur-Terminated GaAs (001)-(2x6) Surface", Surface Science. *395* (1998) 75.

Keywords : photoelectron diffraction, Auger electron diffraction, GaAs, sulfur-termination

4 Investigation on Synthesis and Characterization of Metal Complexes with High Electron-spin Multiplicity

H. Isago, Materials Physics Division

Fabrication of molecular devices, where one molecule functions as one device, is one of the most challenging projects in current and future science. In a number of biological systems, such as enzymes, which can be considered as well-designed molecular devices, unusual electronic structures have often been observed in their reaction centers where some metal ions play an important role. In recent years, phthalocyanines and their metal compounds (MPcs), which are industrially important organic dyes, have attracted much attention in the field of electronics, nonlinear optics, and photosensitization in electron-transfer reaction. They are also of interest from the view point of the similarity in their molecular and electronic structures to those of reaction centers of enzymes.

We have studied some MPcs as candidates for such materials. Quite recently, we have isolated antimony (V)-phthalocyanine complex with unusual spectroscopic and electrochemical properties, which might be versatile in the field of non-linear optics, fuel cells, and so on. We have also studied spectroscopic properties of its one-electron-reduced species and have concluded that its ground state was orbitally doubly degenerate unlike the other known MPcs. Electrochemical investigations further supported this conclusion and even suggested that the ground state of its doubly reduced species might be a triplet; if this is true, this species could be a unit of a new type of molecular magnets which can be applied to magneto-optical devices. The latter suggestion, however, could not be confirmed due to chemical instability of the doubly reduced species. Efforts are being made in our laboratory to stabilize doubly reduced species of this type of complexes. Our main purposes in this project are to make compounds, which generate chemically stable, doubly reduced species and to verify the above speculations.

Keywords : phthalocyanine, antimony, electrochemistry, spectroscopy, spin-multiplicity

5 Study on the Improvement of Analytical Technologies for Metallic Materials

T. Kobayashi, Materials Physics Division
[April 1997 to March 2000]

Systematic study on the improvement of various kinds of analytical technologies has been carried out to promote the research and development of new metallic materials. The aim of this study is to increase the adaptable range of samples and elements, and also to improve the detection limits and precision in the instrumental analytical methods.

(1) Direct Analyses

① For the glow discharge mass spectrometry (GD-MS), the study on the effects of preliminary discharge and pretreatment of sample surface on the relative sensitivity factors (RSF) of N and O has been carried out. Accurate analyses of nitrogen and oxygen will be possible by using the RSF.

② In order to determine trace amounts of each analyte in metallic materials by using the graphite furnace atomic absorption spectrometry (GF-AAS), direct atomization of the sample solution prepared only by the acid decomposition has been studied.

(2) Separation Analyses

① The inductively coupled plasma mass spectrometry (ICP-MS) has been applied after extracting by micellar liquid chromatgraphy.

② High sensitive simultaneous determination procedure of trace impurity elements in high purity metallic materials has been established by using the inductively coupled plasma atomic emission spectrometry (ICP-AES).

③ In order to utilize the high sensitivity and the rapidity of the total reflection X-ray spectrophotometry (TR-XRF) in the analyses of solution samples, the mounting method of solution samples on a silicon wafer has been applied.

(3) Microarea Analyses

① In order to reveal the segregation of small amount of each analyte in metallic materials a technique of computer-image processing has been applied to improve the resolution of concentration in the electron-probe microanalyzer (EPMA) X-ray images.

Keywords : GD-MS, GF-AAS, ICP-AES, ICP-MS, EPMA

6 Effect of High Magnetic Field on Phase Transformations in Fe-based Alloys and its Application to Structure Control

T. Kikuchi , 4th Research Group
[April 1995 to March 1998]

Magnetic field is one of the important external physical quantities that affect properties of materials and has been the subject of interest and research for more than a century not only in pure physics but also in materials science. Especially, recent development of the instruments producing extremely high magnetic fields seems to give us completely new means to explore various properties in materials. In our research institute, we have recently succeeded in developing new experimental facilities that are able to produce one of the world-records in both pulse and static magnetic fields. Among various research projects using these facilities, an investigation of effects of ultra high magnetic fields on martensitic transformation has been just

initiated . Effects of ultra high magnetic field of both pulse and steady types on martensitic transformation have been studied, using Fe-Ni-Mn, Fe-Ni-Mn-C alloys with the supplemental tests on Fe-Ni-C and Fe-Ni-Co-Ti alloys. It is found that the martensitic transformation is induced at room temperature not only athermally but also isothermally either by pulse magnetic field or by steady magnetic field for Fe-Ni-Mn and Fe-Ni-Mn-C alloys. This fact is in sharp contrast with that no martensite is induced by ultra high magnetic field for Fe-Ni-C and Fe-Ni-Co-Ti alloys.

Keywords : isothermal martensitic transformation, athermal martensitic transformation, steady high magnetic field, pulse magnetic field, room temperature

Relatd papers

Athermal and Isothermal Martensitic Transformations Induced at Room Temperature by Ultra High Magnetic Field, S. Kajiwara, T. Kikuchi, H. Pal, T. Asano, M. Kosuge, M. Yuyama, K. Inoue and H.Wada, J. Phys. France Ⅳ 7(1997)C5-377

7 Effects of High Magnetic Field on Solid/Solid Phase Transformations

H.Ohtsuka, High Magnetic Research Station
[April 1996 to March 1997]

Effects of high magnetic field on solid/solid phase transformation behaviors and microstructures have been studied in Fe-based alloys.

I. Effects of High Magnetic Field on Martensitic Transformation

I-1. Magnetic parameters of Fe-Ni-C alloys, relevant to the study of martensitic transformation have been quantified. The composition dependence of average magnetic moments and Curie temperature in BCC and FCC phases are expressed by simple polynomial functions. The available data show that a simple Brillouin-Langevin function is inadequate to describe the temperature dependency of magnetic moment of FCC Fe-Ni-C alloys. The application of two-gamma state model should be taken into consideration.

I-2. Dimensions of fully grown martensite plates in Fe-27Ni-0.8C and Fe-31Ni-0.4C (wt%) alloys formed at 4.2K under applied magnetic field (10T) and tensile stress (220MPa) have been studied. It is demonstrated that the formation sequence of the plates has a strong effect on the radial growth and aspect ratio. The aspect ratio of martensite plates formed at 4.2K was measured for the first time. The measured aspect ratio of the plates in the Fe-27Ni-0.8C alloy agrees very well with an earlier prediction based on the aspect ratio measurements in the temperature range of 193 to 77K in an Fe-23.2Ni-2.9Mn alloy.

II. Magnetic Aging of an Fe-23.3Ni-9.4Al Alloy

Aging of an Fe-23.3Ni-9.4Al(wt%) has been carried our both in the presence of a magnetic field of 9T and without a magnetic field at 673, 773, 873 and 973K for 5 hr. The saturation magnetic moment of the samples were measured at 200, 100, 50 and 5K and extrapolated to 0K. Except for aging at 773K, the average magnetic moment of the samples aged under magnetic field is smaller than those aged without a magnetic field. The difference in magnetic moments is attributed to the difference in kinetics of ordering/decomposition due to the magnetic field.

Keywords : martensitic transformation, spinodal decomposition, tensile stress

Relatd papers

Effects of Magnetic Field on Nucleation in Phase Transformations, H.Ohtsuka, Journal of the Japanese Association of Crystal Growth vol.24(1997), p.2-9.

8 Diffusion in Ordered Alloys and Preparation of Composition Gradiented Materials

H. Sasano, 4th Research Group
[April 1995 to March 1997]

One of the purposes of this research program is to make clear diffusion mechanism and phase stability in ordered alloys such as NiAl, Ti_3Al and TiAl . The interdiffusion coefficients in NiAl and Ti_3Al were measured by a chemical transportation technique in a closed tube using transporting medium of aluminum chloride gas . The coefficients were extraordinary low in the composition range between 45 and 51 atomic percent aluminum in NiAl at ll20K and in the range between 23 and 26 atomic percent aluminum in Ti_3Al at ll20K. The results imply the existence of miscibility gaps in these single phase regions.

Another purpose of the program is to develop the fabrication process of composition gradiented materials. The properties of ordered alloys are very sensitive to the composition. If the composition is delicately changed along the longitudinal direction of the material, we could obtain the material possessing unique properties. We tried to control the concentration of titanium in Ti-Ni binary alloys by regulating heating temperature of nickel plate and mixture of titanium and ammonium chloride placed in a closed alumina tube independently. NiTi phase was formed on the surface of the nickel plate.

Keywords : ordered alloy, diffusion, chemical transportation. shape memory alloy

9 Hydrogen Behaviors at Interfaces in Alloys

C. Nishimura, 4th Research Group
[April 1995 to March 1998]

Hydrogen absorption, desorption, diffusion, and trapping behaviors in alloys and intermetallics have been investigated for the following two aims: (1) to obtain a guide to improve performances of the materials for hydrogen-energy applications and (2) to obtain the fundamental data to clarify the mechanism of environmental embrittlement in $L1_2$-ordered intermetallics.

Thermal desorption spectroscopy (TDS) has been performed for fully hydrogenated $Mg_{0.9}Ni_{0.1}$ and $Mg_{0.77}Ni_{0.23}$. In particular, effects of air exposure were investigated. Samples without air exposure began to desorb hydrogen at room temperature and exhibited desorption peaks above 453K. The peaks were shifted to higher temperatures after the air exposure. Air-exposed sample of hypo-eutectic $Mg_{0.9}Ni_{0.1}$, which contained primary crystals of Mg, was affected remarkably by oxidation; the peak temperature for decomposition of MgH_2 was raised by as much as 130 degrees. On the other hand, air-exposed sample of hyper-eutectic $Mg_{0.77}Ni_{0.23}$ showed good resistance to oxidation; the peak temperature for decomposition of MgH_2 in eutectic was raised by only 50 degrees. Hydrogen from MgH_2 phase in eutectic is suggested to be desorbed through adjacent Mg_2Ni phase in the eutectic.

Hydrogen permeation behaviors have been investigated for Mg-1.54Al-0.65Zn-0.05Mn mass % alloy. In particular, effects of Y deposition were investigated. Y deposited membrane of the Mg alloy exhibited two times higher hydrogen-permeability than the alloy membrane without Y deposition. Y deposition was demonstrated to be effective to modify the surface of the Mg alloy, suggesting it is effective to enhance the hydrogen desorption rate of Mg-based alloys for hydrogen storage.

Effects of ordered state was investigated on environmental embrittlement in $L1_2$-ordered $(Co,Fe)_3V$ alloy. Disordered $(Co,Fe)_3V$ alloy showed no indication of moisture-induced environmental embrittlement, whereas the same alloy in $L1_2$-ordered state showed typical environmental embrittlement. Ordered state was suggested to have a critical effect on hydrogen segregation behaviors, affecting grain-boundary cohesion.

This research was performed in collaboration with institute of industrial science, the University of Tokyo.

Keywords : hydrogen, intermetallics, interface, diffusion, trapping

Related Papers

Effects of Ordered State on Environmental Embrittlement in $(Co,Fe)_3V$. C. Nishimura and C. T. Liu, Scr. Mater. 35(1996) 1441-1447.

10 Research on Electrode Reaction between Metallic Ions and Carbonaceous Materials / Research on Electrochemical Characteristics of TiAl alloy

I. Tomizuka, Physical Properties Division
[April 1994 to March 1997]

A. Electrode reaction between carbonaceous materials and metallic ions.

The following three researches have been completed. They have been applied for publication. They are to be presented at the American Carbon Conference in 1997.

a) Characterization of carbon fibers by electrochemical impedance spectrometry(EIS).

EIS was observed in sulfuric acid at a cathodic potential and at an anodic potential for 4 series of carbon fibers (stretched, low-temperature-heated, ex-lignin, and activated) comprising 8 species. For ordinary fibers, the charge transfer resistance and the electrical capacitance calculated from the arc which was considered to come from the reaction at the electrode surface laid at 0 - 1 k Ω cm^2 and 1-10 Fcm^{-2}. The activated carbon fiber, however, showed much smaller resistance and much larger capacitance.

b) Electrical-double-layer capacitance and chronological variation of sodium content of ex-lignin carbon fibers.

Ex-lignin carbon fibers have electrical capacitance larger than the ordinary carbon fibers. They contains sodium whose amount depends on their temperature of heat-treatment. The electrical capacitance was assessed in a potential range of -100mV through +1500mV (vs. Ag/AgCl electrode) for an ex-lignin fiber which was believed to be prepared at 1500℃ and contained 140wtppm of sodium, and for another ex-lignin fiber which was believed to be prepared at 1000 ℃and contained 4.8wt% of sodium. Chronological variation of sodium content was observed for the latter fiber.

c) Elimination of copper from its aqueous solution by an activated carbon fiber and other carbon fibers

The activated carbon fiber was soaked in an acidic solution containing copper ion. Then cathodic potential was applied to the fiber and the variation of residual concentration of copper was observed. The rate of decrease was proportional to the residual concentration itself. The not-activated carbon-fibers with a degree of graphitization comparable with that of ACF did

not eliminate copper ion at all. All the carbon fibers with a higher degree of graphitization eliminated copper ion. The residual concentration decreased by a considerable amount immediately after the soaking and then at a lower rate in the following period. Their lower rates were almost the same to that of ACF. As the initial decrease was relatively small for ACF, the performance of ACF in a given period was smaller than that of the other fibers.

B. Electrochemical characteristics of TiAl-based alloys.

It was already found before by X-ray diffractomtery and thermal desorption analysis that two TiAl-hydrides were formed when TiAl-based alloys were kept at a cathodic potential or at an anodic potential preceded by soaking under a cathodic potential. Last year a SEM-experiment was performed to study where these hydrides are. The results showed that the hexagonal hydride was in the outermost layer and that the face-centered tetragonal hydride was in the layer beneath it.

Keywords : electrode reaction, metallic ion, carbonaceous material, TiAl-based alloy

11 Energy Conversion Materials Fabricated with Composite Structure

I.A. Nishida, 4th Research Group
[April 1996 to March 2002]

Thermoelectric (TE) materials have been widely used for the purpose of direct conversion of thermal energy to electric power without noise and cooling system. Recently, the thermoelectric generators are mainly used for the electric source in space, marine and polar regions, and the thermoelectric cooling systems are mainly used for the precise temperature control in the semiconductor processing, and in the use of optical and electronic devices. Therefore, it is important to develop TE materials with high efficiency.

The high efficient TE materials are given by a figure of merit Z with high thermoelectric power α and electric conductivity σ and low thermal conductivity κ . For a given TE materials with homogeneous matrix and dopant concentration, the optimized Z can only be obtained in the relatively narrow temperature range. In other temperature range, the TE conversion efficiency η becomes comparatively small. Since the temperature of the thermocouple legs changes continuously from the top end (high temperature) to the bottom end (low temperature), the large value of η will be obtained if the carrier concentration in the TE material changes continuously along the thermocouple legs, indicating advantage of functionally gradient TE materials. By preparing these materials, the effective value of η is estimated to increase more than double. In addition, Z can be increased by controlling the composition and grain size of the FGM, because the lattice contribution to the thermal conductivity can be decreased by phonon scattering at the lattice defects such as crystal distortion and grain boundary.

An effective maximum power for three stepped FGM of n-type PbTe with different electron concentrations was found to be larger than one for the optimized homogeneous material.

Keywords : energy conversion, thermoelectric materials, composite materials, thermoelectric conversion, thermoelectric cooling system

Related Paper

Highly Efficient Thermoelectric materials in FGM program, I.A. Nishida, Proc. Japan-Russia-Ukraine Intl. Workshop on Energy Conv. Materials (ENCOM 95), Sendai Japan, January (1995): 1-10.

12 Thermal Stability of Intermetallic Compound Matrix Composites Reinforced with Fibers

Y.Shinohara, Physical Properties Division
[April 1993 to March 1997]

Heat resistive materials which can be used above 1373K are essential to developments of the space-plane, the fusion reactor and the high-efficient turbine engine. TiAl intermetallic compounds have higher strength and toughness than metals and ceramics at elevated temperatures. TiAl matrix composites reinforced with fibers are promising for structural materials above 1373K.

Reinforcements for TiAl matrix are SiC, B and W fibers. B and W fibers are tougher than SiC fibers, while are more reactive with TiAl. If good protection layers are developed, B and W fibers can be applied for TiAl matrix. When the protection layers have insufficient shear stress, roughening of fiber surface is effective in transferring stress from fibers to matrix. In this study, the following subjects have been investigated to develop good protection layers for TiAl matrix and to improve stress transfer from fibers to matrix:

Formation of protection layers and optimization of layer thickness

B fibers were coated with Ti by using PVD method, and were heat treated to form TiB_2 layers on the surface. The fiber strength was deteriorated with the layer thickness. It is due to the heterogeneous formation of TiB_2 and the pits formed on fiber surfaces. The thin layer of less than 1um is desirable to prevent fiber strength from deterioration.

W fibers were coated with BN and ZrO_2 by slurry method, and TiAl matrix composites were reinforced with the coated fibers. Both the protection layers 1-3 μ m thick showed sufficient performance for suppressing the interfacial reaction.

Effect of surface morphology on the strength of W fiber

The surface of W fiber was successfully roughened by oxidizing method. It is obvious that the optimized roughness can keep the original fiber strength and sufficiently transfer stress from fibers to matrix.

Key words : TiAl matrix composite, B fiber, W fiber, protection layer, BN, TiB_2

13 Single crystal growth of new nonlinear-optical materials by means of controlling crystal symmetry

H.Kimura, Physical Properties Division *
* Present: 4th Research Group
[April 1996 to March 1997]

Single crystals for nonlinear-optical materials are important for frequency- modulation devices. It is usually required for this purpose that single crystal should have a center of crystal asymmetry. However, it is generally difficult to grow single crystals having the center of crystal asymmetry even if new materials are discovered. It would be possible to obtain the center of crystal asymmetry part in single crystals (having the center of crystal symmetry) by means of the substitution of constituent elements for another elements in order to form the center of asymmetry part surroundings of substituent elements in micro scale. In this work, single crystal growth of these materials having the center of crystal asymmetry in part are tried by the Czochralski method from their melts and the crystal quality is improved by means of controlling the crystal growth conditions. Furthermore, the optical property of the crystals will be evaluated using the single crystals grown. In this year, BaB_2O_4, where Al or Ga substituted for B, was synthesized as polycrystalline shape. Their nonlinear-optical properties, such as SHG (Second Harmonic Generation) intensity, were investigated. The substituted crystals had larger SHG intensity than BaB_2O_4.

This research was performed in collaboration with the Institute for Materials Research, Tohoku University.

Keywords : frequency-modulation, center of crystal asymmetry, substitution

Related papers

H.Kimura, T.Numazawa and M.Sato, "Melt supercooling behavior and crystal growth of Ba $(B_{1-x}M_x)_2O_4$ (M: Al or Ga)", J.Cryst. Growth 174 (1997) 308-312.

H.Kimura, K.Ishioka, M.Sato, "Estimation of SHG properties on $Ba(B_{1-x}M_x)_2O_4$ (M:Al or Ga)", J.Mater.Sci.Lett. 16 (1997) 5743-5747.

14 Crystal-quality improvement of nonlinear-optical oxides by Floating zone method

H.Kimura, Physical Properties Division *
* Present: 4th Research Group
[April 1996 to March 1997]

Single crystals of low-temperature (β) phase in BaB_2O_4 (β -BBO) are useful for ultra- violet nonlinear-optical devices such as frequency-modulation devices. Single crystals of β-BBO have been previously grown by the Czochralski (Cz) method from nominally pure and supercooled melts. Since the steep temperature gradient is required to grow β-BBO crystals, it is useful to grow by the Floating zone (Fz) method instead of the Cz method. However, it is difficult to keep the BBO molten zone because of an instability caused by a small viscosity and a small surface tension of the melts. In this study, the viscosity of BBO melts, where M (M: Al or Ga, BBMO) substituted for B, was investigated by the rotator method to obtain basic data required to the crystal growth by the Fz method. Then, surface tension was measured by the capillary rise method. Furthermore, the molten zone stability was studied by the hanging melt drop method.

The viscosity and the surface tension of BBMO melts were increased with the increase of M content. Temperature dependence of the viscosity was followed the Arrhenius type equation. According to the values of the viscosity and the surface tension of BBMO melts, it is possible to grow BBMO single crystals by the Fz method.

A part of this work was performed under the Visiting Researcher's Program of the Laboratory for Development Research of Advanced Materials, "Viscosity Change on Oxide Melt by Addition of Group III Element", Institute for Materials Research, Tohoku University.

Keywords : viscosity, surface tension, Floating zone method

Related paper

H.Kimura, M.Sato, K.Shimamura and T.Fukuda,"Viscosity and surface tension change in BaB_2O_4 melt by substitution of Al or Ga for B", J. Mater.Sci.Lett., 16(1997), 911-913.

15 Establishment of Multidimensional Evaluation of Human Senses for Materials Design

Y.Kurihara, Materials Design Division
[April 1994 to March 1997]

Recently, there are increasing demands for materials developments from new aspects such as environment consciousness, amenity, and so on. When we investigate such new needs, most of information is provided mainly by language and human senses. The target of this study is to construct a quantitative evaluation method, which can incorporate sensory information into materials design.

For this purpose, we inquired of 81 persons from 20 to 50 years old about the relationship between human sense and technical terms on materials properties such as mechanical, chemical and physical ones using a relationship matrix. Three characterlstrc vectors representing "warmth "cleanliness" and "magnificence" were extracted from multivariate statistical analysis on collected sensory data. From the analysis, it is concluded that people understand and express materials properties in comparison with function and properties of human beings. In other words, most of materials represent the functions of human beings.

As the next, stage of this study, we have constructed a database which correlates various kinds of materials properties with functions of human beings by a relationship matrix. This relationship matrix will be applied to find need for new materials and effective uses of materials properties.

Keywords : human senses, materials properties, relationship matrix, multivariate statistical analysis, function of human beings

Related Paper

An Application of Human Sense to Materials Development : Y.Kurihara, T.Kaneko, M.Fujita, k.Hosimoto and Chiaki Tanaka, Symposium on Sensory. Inspection, (1995): 93-98.

An Application of Quality Function Deployment to Establishment of Material Needs Finding System : Y.Kurihara, T.Kaneko, M.Fujita, K.Hosimoto and C.Tanaka, Symposium on Sensory Inspection, (1995): 215-220.

An Application of Human Sense to Materials Design : T.Kaneko, Y.Kurihara, M.Fujita, K.Hosimoto and C.Tanaka, CAMP-ISIJ, vol.8, (1995): 1619.

16 Development of Knowledge Database for High-Tc Superconducting Materials

Y.Asada, Computational Materials Science Division
[April 1995 to March 2000]

We have already developed numerical database for high-Tc oxide superconductors. It is already opened publicly via network. All the data in it are extracted from the papers reported in journals. The papers contain many useful information as well as numerical data. The information is written in text style in abstract, summary or conclusion. It is desirable that researchers being employed in designing new products and exploring new materials retrieve the information from database and apply it to their purposes.

In this project we study how to store the information and how to construct knowledgebase for high-Tc superconductors. For this purpose we introduce 'matisse' and 'REX' as a main software. These are object-oriented database management system (ODBMS). We are developing the data acquisition system with semantic network. Thesaurus, especially semantic thesaurus, dictionary is very important for this purpose.

We developed the Tc-prediction system for a series of Y123 by using Neural Network method. Neural Network was constructed using the data obtained by retrieving from the numerical database "SUPERCON". The system predicts that Ca in Y-site have a valuable effect for high-Tc materials and Tc becomes higher than that of YBa2Cu3O7 if we choose the best in Ca- and O-contents.

Keyword : oxide Superconductors, database, knowledgebase, net work,

Related Papers

Database Development in Assistance of New Superconducting Materials: Y.Asada, E.Nakada: J.Jpn.Soc.Information and Knowledge(in Japanese),5(1995)57.

Prediction of Tc for YBa2Cu3Oz Doped with Ca Using Neural Network, Y.Asada, E.Nakada, S.Matsumoto, H.Uesaka, J.Superconductivity 10(1997)23-26.

17 Theory of Thermal Reaction on Solid Surfaces

T. Ohno, Computational Materials Science Division
[April 1994 to March 2000]

The thermal reactions on solid surfaces play important roles in various surface processes including epitaxial growth, etching, and catalysis. The purpose of this work is to clarify theoretically the thermal reaction processes on several solid surface systems such as semiconductor surfaces and metal surfaces. The analy-

sis is based on the density-functional electronic theory within local-density approximation, which provides a powerful tool to determine the dynamical behavior as well as the stable atomic arrangement of solid surfaces.

The structural stability and optimum atomic arrangement of the Ga-rich GaAs(001)-(4x2) reconstructed surface has been theoretically investigated. This work shows that the stable Ga-rich GaAs(001) surface consists of two Ga dimers on the top layer and another Ga dimer at the third layer and that the calculated surface charge distributions are in good agreement with the observed scanning tunneling microscopy images.

We have investigated the adsorption of chlorine molecules on the reconstructed GaAs(001) surfaces. Chlorine is one of the most important halogens used in the semiconductor etching processes. The stable geometries of the chlorinated GaAs(001) surfaces have been determined theoretically, which are consistent with recent temperature programmed desorption measurements.

Recently, we have proposed a carrier compensation mechanism in Se δ -doped GaAs from first-principles calculations. The formation of Ga vacancies near Se atoms occurs for high Se concentrations, which induces the clustering of Se atoms. The carrier compensation is suggested to result from the formation of clusters consisting of three Se atoms and one Ga vacancy.

Keywords : structural stability, chemisorption, reaction, solid surface

Related Papers

T. Ohno, Energetics of As Dimers on GaAs(001) As-Rich Surfaces, Phys. Rev. Lett. 70 (1993): 631-634.

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J. Nara, T. Sasaki, and T. Ohno, Theoretical Investigation on δ -doping of Se atoms in GaAs, Appl. Phys. Lett. 70 (1997): 3534-3536.

18 A Molecular-Dynamics Study of the Initial Process of Fracture

K. Kusunoki, Computational Materials Science Division

[April 1 994 to March 1997]

Using molecular-dynamics simulations, the present study aims to elucidate how the microscopic forces and displacements of atoms develop into the macroscopic ones. It also aims at an atomistic description of the macroscopic concepts such as crack, stress and strain.

So far we have developed a simulator by which we can make both tensile and compression tests of Cu and Fe single crystals. With use of the simulator, we investigated into the mode-1 fracture process of elemental crystals to ascertain whether the so-called structure-property relationship, that stands on the ductile/brittle nature of real elemental fcc and bcc crystals, can also stands in the present systems composed of atoms with appropriate ineratomic potentials, an EAM and modified Lermard-Jones potentials for Cu, and an EAM and Johnson's ones for α -Fe. In our molecular-dynamics simulation, we apply two types of spatial conditions; the one is no periodical boundary for all directions and the other is periodical boundaries set along the two directions perpendicular to the tensile axis of a specimen. In case the periodical boundary condition is applied, every fracture takes place either by a cleavage or by the combination of voids generated along a specific atom plane that is normal to the tensile axis. In contrast, when no periodical boundary condition is applied, are there observed two types of fracture modes; one is of the ductile behavior for both Cu at any temperature and α -Fe at room temperature or high, and the other is of the brittle one for α -Fe at lower temperatures. These results show that, despite the type of interatomic potentials, the structure-property relationship, one of the fundamental mechanical properties of real crystalline materials, is properly attained for systems with no periodical boundary condition in the molecular-dynamics simulation of tensile tests.

Keywords : molecular dynamics, fracture, atomistic models, crack

Related Papers

Volume-expansion-induced lattice instability and solid-state amorphization, K. Kusunoki, Phys. Rev. B53, 16 (1996).

19 Thermodynamic Analysis of Transition Process from Metastable to Stable Phases

H.Onodera, Computational Materials Science Division

[April 1994 to March 1997]

The crystallization process of sputter deposited Ti48Al52 amorphous alloy was studied by high resolution transmission electron microscopy (HRTEM) and

X-ray diffraction. An unknown phase having spherical morphology precipitates in amorphous matrix in the early stage of crystallization. Electron diffraction patterns from this phase can be identified by the primitive cubic or tetragonal structures with the lattice parameter of a(=c)=0.69nm. Small-angle X-ray scattering profiles of amorphous Ti-60at%Al alloy suggested the existence of concentration and/or density fluctuations in the sample. The formation of the unknown phase seems to be closely related to these fluctuations.

Liquid-amorphous transition process of Ti-Al alloys were investigated by using molecular dynamics techniques with the Embedded Atom Method. The simulation could reproduce the experimentally observed concentration range (40-85at%Al) for amorphization. Structure analyses of the amorphous alloys indicate that the basic building blocks for the amorphous phases of Ti-Al system should be icosahedral clusters with Al-atom in their center.

Keywords : metastable phase, amorphous, Small-angle X-ray scattering, concentration and/or density fluctuations, molecular dynamics

Related papers

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Coating of HfC on Tungsten Powder by Fluidized Bed CVD, T.Itagaki, J. Japan Inst. Metals, Vol.59(1995), p.1157.

Molecular Dynamics Study on Amorphization of Ti-Al Alloys, M.Shimono and H.Onodera, Proc. of CMSMD'96, Japan, (1996), p.215.

Crystallization of Sputter Deposited Amorphous Ti-52at%Al Alloy, M.Ohnuma, T.Abe and H.Onodera, MRS Symposium Proc. Series, Vol.400, (1996), p.209.

20 Development of Third Generation Nickel-Base Single Crystal Superalloys

T. Yamagata, Computational Materials Science Division

[April 1994 to March 1997]

Single crystal (SC) superalloys with balanced hot corrosion resistance and high temperature creep strengths were designed by using NRIM Alloy Design Program (ADP) with help of Cluster Variation Method (CVM) computer program based on statistical thermodynamics. The design condition was as follows; alloy density to be less than 8.9, solution heat treatment window to be larger than 40 °C, lattice misfit to be negative at high temperatures but not beyond certain extent, creep rupture lives both at 900°C-392MPa and at 1040 °C-137MPa to be longer than 1000hrs, etc.

Alloys with multi-chemical compositions, e.g., TMS-75 and TMS-80, having 3wt% Cr, 5wt% Re, etc, were selected as the bests through the programs and were cast to SC bars. The SC bars were successfully solution heat treated by a simple two-step heating at 1300 °C for 2 hrs and at 1320 °C for 5hrs, followed by a two-step aging treatment, first at 1150 °C for 4hrs and second at 870 °C for 20hrs.

Creep tests at temperatures ranging from 900 to 1150 °C showed that the alloys have good balance of creep strengths over this temperature range compared with other Re containing alloys like CMSX-10; typical rupture lives at 900 °C-392MPa, 1000 °C-245MPa and 1100 °C-137MPa being 1000hrs, 330hrs and 220hrs, respectively. A laboratory scale hot corrosion test using Na_2SO_4-25%NaCl salt mixture at 900 °C showed that the alloy has a very good hot corrosion resistance as well, especially when Ir is added (TMS-80).

Design and development of further superior SC superalloys are being conducted.

Keyword : single crystal, superalloy, alloy design, creep strength, hot corrosion resistance

Related Papers

The influence of Co on creep deformation anisotropy in Ni-base single crystal superalloys at intermediate temperatures, H. Murakami, Y. Yamagata and H. Harada, Materials Science & Engineering A, May 1997, p.54 - 58.

Design of a high rhenium containing single crystal superalloy with balanced intermediate and high temperature creep strengths, T. Kobayashi, Y. Koizumi, S. Nakazawa, H.Harada and T. Yamagata, the 4th International Charles Parsons Turbine Conference, 4-6 Nov. 1997, Newcastle, UK (The Institute of Materials).

21 Development of Integrated System for Distributed Materials Scientific data using Network

M. Nihei and M.Sakamoto, Computational Materials Science Division

[Apr. 1995 - Mar. 1999]

Researchs with a view of developing new evaluation technologies of materials strength through the utilization of the materials scientific databases are being conducted. The empirical systematization approach based on the combining the scientific and empirical understanding for material strength was successful for the development of the fatigue life prediction proce-

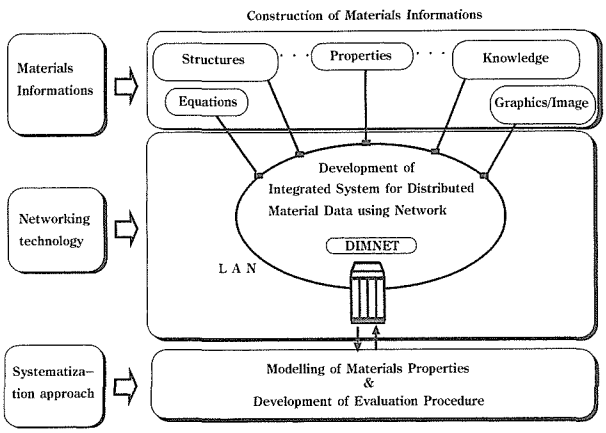
dure in many heat treated structural steels,that is,the procedure to make the new empirical combinations among characterized items of materials among the extracted data set from the factual database.

To apply this systematization approach for other materials properties and to use more efficiently other materials informations, the prototype of new integrated system utilizing a networking environment and treating many different type of materials scientific database, which are on many different sites binding by a network system such an INTERNET, is being developed.

The prototype system for the integration, DIMNET(now only in japanese version), was developed and will be soon accessible for some limited internet users through WWW browser such as NETSCAPE.

The systematization approach using this system is also being conducted for the time-dependent creep rupture properties for heat resisted steels.

Keywords : materials properties prediction, database, networking



22 Development of Virtual Experimental Technologies for Material Design

M.Nihei, Computational Material Science Division
[April 1995 to March 1998]

The coordinated research program of the Science and Technology Agency (STA) to develop the virtual experimental technologies for materials design is being carried out from April 1995. Over twenty researchers participate in this research program. This research program aimes to promote the computational materials science of Japan and to develop the new material design technology through the realization of a virtual experimental system using the computer technologies such as supercomputings, databases and networking technologies.

This research program consists of four parts, the first principal computational approach, meso-scopic material structure prediction using the statistical thermo-dynamics and/or monte-carlo calculation, modeling and simulation of material properties such as strength and development of a virtual experimental system by the integration of computer technologies.

In this research program, NRIM is conducting the following three research projects.

1.Calculation of atom transfer on a material surface by Ab Initio molecular dynamics(Car-Parinello method).

This research aims to develop the Ab Initio computing method based on the density functional theory to analize physical phenomena such as chemical response on the solid surface and thermal atom transfer such as diffusion.

In this research, the force acting on the atom is simulated by the first principle calculation.

2.Material design of heat-resistant alloys by statistical thermo-dynamics calculation.

In this research, the statistical thermo-dynamics calculation method is being improved for the heat-resistant alloys design. The Cluster Variation Method(CVM) using a phenomenological interatomic potential (L-J potential) is employed for the calculation of the equilibrium state in multi-components Ni-base superalloys and new type Ir-base ultra superalloys for a structural use up to 2000℃.

3.Modeling and simulation for the prediction of material strength.

This aims to develop the modelling and/or simulation method for strength,fracture and damage process of materials. The molecular dynamics computing method is being applied for nano-scopic materials evaluation such as the nano-indentation for ultra-thin atomic layers and the combination method using the solid-state mechanics and the fact database is also developed. In this research, the virtual consultant expert system is also constructed by integrating the computer and software technology for a use of material design researchers.

Keywords : computational simulation, modeling, virtual experimental system

23 Study on the effective Acquisition Method of Metallurgical Information

K.Hoshimoto, Computational Materials Science Division
[April 1997 to March 1999]

In order to establish a system for materials design by using a large scale knowledgebase, an information base system to support construction and exploration of an information space in metallurgy, especially in the domain of superalloys, was studied. Information about materials development is, in most cases, obtained em-

pirically, and expressed by means of natural language. A dictionary and a thesaurus of technical terms is being edited as well as the information model of metallurgy, so as to analyse natural language texts automatically. A system which interprets technical papers on metallurgy and constructs information space semi-automatically, is being constructed. The system will offer interactively the information useful to materials development to designers.

Keywords : materials design, natural language, large scale knowledgebase, thesaurus

Related papers

Intelligent Support for construction and Exploration of Advanced Technological Information Space from Technical Papers in Metallurgy, T.Matsuo. T. Nishida and K.Hoshimoto, Journal of Japan Society for Artificial Intelligence, 12(1997), 68-77,

24 Thermodynamic Analysis of Formation Processes of Metastable and Stable Phases

H.Onodera, Computational Materials Science Division

[April 1996 to March 1999]

A wide range of microstructural control can be realized through metastable structures using the transition process from amorphous state to crystalline state. For the effective utilization of these metastable phases to improve mechanical properties of materials, it is necessary to reveal the transition process from metastable to stable phases on the heat treatment. The target of this study is to construct a kinetic model which can describe the transition processes quantitatively.

In the previous study, we have found the formation of metastable α phase in preference to the stable γ phase in the crystallization process of amorphous Ti-Al alloys. Furthermore, small-angle X-ray scattering profiles of amorphous Ti-60at%Al alloy suggested the existence of concentration and/or density fluctuations in the sample. The formation of the unknown phase is considered to be closely related to these fluctuations. Thus, in the present study, the transition process of sputter deposited Ti-Al amorphous alloy is studied by X-ray diffraction, small angle neutron and X-ray scattering and high resolution transmission electron microscopy in view of the correlation between the nucleation site of crystalline phase and the structural and/or compositional fluctuation in the amorphous structure. At the same time, the amorphous-to-crystal transition is studied by using molecular dynamics techniques for the understanding of the transition mechanism in the atomistic scale.

Keywords : amorphous, small angle X-ray scattering, molecular dynamics, amorphous-to- crystal transition, structural and/or density fluctuation

Related paper:

Crystallization of Sputter Deposited Amorphous Ti-52at%Al, T.Abe, S.Akiyama and H.Onodera, ISIJ Int., Vol. 34(1994), p.429.

Molecular Dynamics Study on Amorphization of Ti-Al Alloys, M.Shimono and H.Onodera, Proc. of CMSMD'96, Japan, (1996), p.215.

Crystallization of Sputter Deposited Amorphous Ti-52at%Al Alloy, M.Ohnuma, T.Abe and H.Onodera, MRS Symposium Proc. Series, Vol.400, (1996), p.209.

25 A Computer-Simulation Study of the Plastic Deformation of Crystals

K. Kusunoki, Computational Materials Science Division

[April 1 997 to March 2000]

Using computer simulations, the present study aims to elucidate how the microscopic forces and displacements of atoms develope into a macroscopic plastic deformation. It also aims at an atomistic description of the macroscopic concepts such as stress, strain and slip.

In the precedent study we made a simulator by which we carried out molecular-dynamics simulations on the initial process of fracture of systems with relatively simple structures such as single crystals of fcc and bcc. What was obtained in that study is that simulations under non periodical boundary condition give us more realistic results (eg. reproduction of the structure-property relationship on the ductile/brittle nature of the elemental crystals of fcc and bcc) over those obtained from calculations done under periodical boundary conditions.

More detailed studies of the effects of impurities and structural defects together with investigations into the effects of temperature and strain rate on the ductile/brittle nature of these elemental crystals will be performed in the present study.

In addition to the above, atomistic processes of the deformation of intermetallics, some of which are at present most promising materials as heat resistant alloys showing anomalous temperature dependence of the flow stress, are also the targets of the present study.

Keywords : plastic deformation, atomistic model, molecular-dynamics

26 Theoretical study on new phenomena in ultrathin magnetic films and multilayers

Xiao Hu, Computational Materials Science Division
[April 1997 to March 1998]

The phenomenon of spin reorientations in ultrathin magnetic films is investigated. A micromagnetic theory is developed which reveals the competition between the in-plane shape anisotropy and the normal surface anisotropy through a finite exchange stiffness. For small surface anisotropy, two continuous transitions in spin orientation are derived as the film thickness is increased: first from the uniform, normal configuration to a nonuniform, canting configuration, and then to the uniform, in-plane configuration. This result is consistent with experimental observations. For large surface anisotropy, it is derived theoretically that only the first spin reorientation occurs and the nonuniform, canting configuration remains stable even at large thickness limit. We have obtained analytic expressions for the phase boundaries. The relation between the discrete model and the continuum theory has been clarified by scaling argument, and as the result the sufficiency of the continuum approach in ultrathin films has been established.

We have investigated the effects of the surface anisotropy on the reversal process of magnetization in thin films and small dots, by means of both micromagnetic theory and computer simulation. This issue is important for realizing ultrahigh-density recording, and also of theoretical interest about the treatment of surface effects. In order to treat the surface effect directly, we consider in our micromagnetic formalism the magnetization configuration nonuniform in the direction perpendicular to the surface, while presuming the uniformity of the total system parallel to the surface. As a perpendicular magnetic field is applied to a system of perpendicular surface anisotropy and its strength being increased, the reversal processes of magnetization by the micromagnetic theory are as following: abrupt jumps of magnetization at the coercive field, provided the surface anisotropy is strong, and the thickness is small; coherent rotation of magnetization, if the surface anisotropy and the thickness are moderate; nucleation of opposite magnetization, incoherent rotations, as the surface anisotropy is week, and the thickness is large. The dominating process of magnetization reversal can be summarized into a "phase diagram" with two scaling parameters, involving the film thickness, the surface, and volume anisotropy including shape anisotropy from dipole interactions, and the stiffness constant. The coercivity of ultrathin magnetic films decreases with the square of the reciprocal of the thickness. This thickness dependence of coercivity, coinciding well with experimental observations, is understood from the equation for magnetization configuration in the micromagnetic theory. Magnetization reversal processes under in-plane external fields have also been investigated, and the coercivity decreases with the reciprocal of thickness.

Keyword : ultrathin magnetic film, spin reorientation, micromagnetism

Related Papers

Magnetization reversal and coercive force in ultrathin films with perpendicular surface anisotropy: micromagnetic theory, X. Hu, Phys. Rev. B Vol. 55(1997), p.8382.

Micromagnetic study on ultrathin magnetic films, X.Hu and Y.Kawazoe.

27 Strengthening Mechanism of High Strength Composite Materials

Dr.H.G.Suzuki, Mechanical Properties Division
[April 1996 to March 1998]

Strengthening mechanism of high strength high conductive Cu-15%Cr and Cu-15%Cr-5%Sn composites has been examined by transmission electron microscope(TEM).

1) By the analysis of Burgers vector, it was found that two conjugated <111>/{110} slip systems operated for the deformation of Cr crystals in Cu-15%Cr alloy.
2) Dislocations in Cr crystals recover dynamically and then work hardening due to cold rolling is very small.
3) The factors controlling the strengthening of the composite are (a) lamellar spacing of Cr fibers, followed by Hall-Petch relation, (b) dislocation densities in the Cu matrix, which depend on the degree of cold rolling.
4) Cu matrix shows a dynamical recovery or recrystallization during cold rolling and cell formation occurs after 50% of reduction in Cu-15%Cr alloy.
5) Work hardening rate becomes very large in Cu-15%Cr-5%Sn because of solution hardening of Cu matrix by Sn. Deformation band is formed in Cu matrix and no dynamical recovery occurs.

28 Development of High Strength Metal Matrix Composites with Excellent Physical Properties by the Advanced Bronze Method

Dr.H.G.Suzuki, Mechanical Properties Division
[April 1994 to March 1997]

The studies of the strengthening mechanism and process optimization have been conducted for Cu-15%Cr binary and Cu-15%Cr-C, Zr ternary alloys.

1) Optimum amount of Cr as a second phase former is located at around 15 to 18wt% and the cold drawability becomes poor at higher contents and the strength decreases at lower content than this range.
2) The strength is controlled by ① lamellar spacing of Cr phase, and ② awork hardening of Cu matrix by cold drawing. Finer spacing gives higher strength. Finest spacing attained by cold drawing is 0.5 Mm in binary alloy.
3) The precipitation of Cr in Cu matrix does contribute to the increase of electrical conductivity although very little contribution to the precipitation hardening.
4) The strengthening by the addition of small amount of C or Zr: The addition of 100 to 200ppm of C or 0.1 to 0.2wt% of Zr accelerates dendritic Cr into more needle like shape. This is considered to be due to the acceleration of supercooling during solidification.
5) Precipitation strengthening by Zr addition: The secondary hardening due to the precipitation of Cr and Zr becomes very high at around 700K and results in the increase of electrical conductivity by the addition of 0.1 5wt%Zr.
6) By optimizing the manufacturing process, a new alloy Cu-15wt%Cr-0.15wt%Zr with the tensile strength 1150MPa and electrical conductivity of 70%IACS was developed.
7) Scale up mill trial: Two alloys, Cu-15wt%Cr and Cu-15wt%Cr-0.15wt%Zr were melted by 150kg capacity vacuum induction melting and extruded to rod and finally cold rolled into wire. The Cu-15wt%Cr binary alloy was succeeded to get a sound ingot having a homogeneous distribution of Cr through the ingot and mechanical properties showed almost the same value with tiny lab-scale 2kg ingot.

29 Relationship between Fatigue Softening / Hardening Behavior and TEM Structure of Titanium Alloys

T.Kainuma, Mechanical Properties Division
[April 1994 to March 1997]

The change of the width or the area of the hysteresis loop during the fatigue testing of materials at constant stress amplitude is taken usually as a measure of dynamical changes in mechanical properties. By testing at stress amplitudes higher than the yield stress, fatigue hardening only takes place in alloys as well as pure metals (decrease in the width of hysteresis loop). While at stress amplitudes lower than the yield stress, fatigue softening occurs. Details of fatigue softening behavior have been reported for the materials such as aluminum (1), copper (2), silver and iron (3). The aim of this work is to reveal the mechanisms of fatigue softening and hardening behaviors of titanium alloys in connection with the characteristics of crystal structures, using transmission electron microscope (TEM).

1. Fatigue softening and hardening behaviors

We determined at first the specimen shape most suitable for tension-compression fatigue testing, then examined the crystal structure dependence of fatigue softening and hardening behaviors using Ti-3A1-8V-6Cr-4Mo-4Zr alloy (β -type titanium alloy. Ti-3-8-6-4-4), pure titanium (α -type) and Ti-6A1-4V alloy ($\alpha + \beta$ type). We also examined the stress ratio (R=-1, R=0.1) dependence of fatigue softening and fatigue hardening behaviors using β -type titanium alloy, pure titanium (α - type) and ($\alpha + \beta$)-type titanium alloy. The experiments for the fatigue softening and hardening phenomena were carried out on work-hardened and annealed materials, respectively. Following are the main results. (1) In fatigue testing of annealed materials, fatigue hardening behavior of α -type pure titanium was more remarkable than other-type titanium alloys. (2) In fatigue testing of cold-worked materials, fatigue softening behavior of β -type titanium alloy and α -type pure titanium took place more remarkably than $\alpha + \beta$ -type titanium alloy.

2. Relation between fatigue behavior and electron microscopic structure

The TEM observation has been aimed to reveal the mechanisms of fatigue softening and hardening in the α -type pure titanium and β -type titanium alloy. At low and intermediate cyclic stress ranges (stress ratio R=-1) in annealed materials, dislocation loops, loop patch (dislocation loop cluster) and network of dislocation cells were observed. At larger cyclic stress levels, equiaxed and elongated subgrains appeared. In cold-worked materials TEM structure exhibited high density tang leddislocation structures. On the other hand, TEM structure of fatigue softening changed equiaxed and elongated subgrains with low dislocation density.

Keywords : Ti-3-8-6-4-4, pure titanium, Ti-6A1-4V, TEM structure, fatigue softening, fatigue hardening,

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30 Cyclic Deformation in a Corrosive Environment

R. Hamano, Mechanical Properties Division
[April 1994 to March 1997]

The growth of fatigue crack nucleated along the slip bands of PSBS or PLBS Occurs over two successive propagation stages - Stage I (shear mode) and Stage 11 (normal mode). The past data on the effect of environments on crack growth in Stage 11 have been reported in previous works. Comparatively little attention has been paid to the effect of hydrogen-related environments on Stage I crack propagation and the transition of crack growth from Stage I to Stage II.

We present experimental results suggesting that the early transition from Stage I to Stage 11 in a hydrogen environment is a potential index for hydrogen-related embrittlement and discuss whether the early transition of the stages of fatigue crack growth is caused by the localized deformation or the decohesion at a notch root, carrying out fatigue tests on specimens of iron single crystal with the specimen axis of [013] and precipitation hardened aluminum polycrystalline alloy in dry Ar gas and in saturated water vapor or in 3.5% NaCl aqueous solution.

Fatigue tests were performed on a closed loop servo-hydraulic machine, using a sinusoidal wave and a load ratio of 0.1. The testing temperature was 295K. A frequency of 15Hz in dry Ar gas and a frequency of IHZ in dry Ar gas and 3.5% NaCl aqueous solution were employed. After fatigue testing, fracture surfaces were observed with scanning electron microscope (SEM).

The observation of the early transition from Stage I to Stage II and the appearance of striations on the fracture surfaces in saturated water vapor suggested that secondary slip plays an Important role in the transition of fatigue crack growth and the fatigue crack grows by the operation of more than two slip systems leading to macroscopically brittle (but transcrystalline) fracture surface bisecting active slip planes. It seems reasonable to propose that the dominant, hydrogen-related fatigue cracking is hydrogen-assisted strain localization ahead of notch root and requires subsequent crack propagation in two more slip systems to explain the suppression of Stage I growth.

Keywords : Stage I fatigue crack, Stage 11 fatigue crack

31 High Temperature Strength and Fracture of Reinforced Oxide- and Nitride-Base Ceramics

K. Hiraga, Mechanical Properties Division
[April 1995 to March 1997]

Though polycrystalline ceramic materials are brittle at room temperature, most of them show plasticity at high temperatures under the aid of grain boundary sliding accommodated by matter transport through or across the boundaries. The localized failure of such accommodation process causes cavitation damage at multiple grain junctions or phase boundaries. The basic information on such deformation and damage processes, which are quite different from those at room temperature, is important when the materials are used at high temperatures. From this point of view, this study is conducted to examine the role of second phases in the elemental process of high temperature deformation and fracture. Special attention is placed on the constitutive behavior, dynamic microstructural changes during deformation and cavitation behavior at relatively low tensile stresses (σ /E-10^{-4}), at temperatures higher than 1500 K.

Recent quantitative examination on the cavity size distribution has revealed the cavitation behavior to be different between fine grained alumina- and zirconina-base materials. In the former, the evolution of damage volume arises mainly from the active cavity nucleation and the growth of isolated cavities tends to be limited in sizes smaller than 3 μ m. In contrast, the nucleation of cavities in the latter is inactive and they continue to grow into sizes of 10-30 μ m without coalescence. It indicates that the damage accumulation is strongly controlled by cavity nucleation in the former and by growth in the latter. The quantitative examination has also gives us the information on the effects of the shape of second phases on cavitation behavior.

Keywords : creep, superplasticity, intergranular cavitation

32 Research on Fundamental Science of Frontier Ceramics

K. Hiraga, Mechanical Properties Division, and Y. Sakka, Chemical Processing Division
[April 1995 to March 1998]

This study aims at obtaining systematic bases for the microstructural design of advanced oxide-base ceramics for high temperature applications. For this purpose, we have promoted tight cooperation between two research gropes concentrating on highly qualified material synthesis by colloidal processing and high temperature fracture mechanisms in ceramics, respectively.

In the research on synthesis, we have systematically studied the relationships between the surface charge and the dispersion of fine oxide particles in electrolytic liquids. We have also examined the influence of the particle dispersion in colloidal suspension on the following process consisting of consolidation and sintering. The obtained information has given us both theoretical and practical bases for synthesizing advanced materials such as monolithic r-ZrO_2 and CuO-doped t-ZrO_2 with grain sizes less than 0.1 μ m, SiO_2-doped t-ZrO_2 with uniform grain boundary modification, and ZrO_2-dispersed alumina with matrix grain sizes smaller than 0.5 μ m.

In the research on mechanical properties, we have studied the elemental process of high temperature deformation and fracture of fine-grained oxides. The quantitative examination of cavity size distribution have revealed the cavitation behavior governing tensile ductility of fine grained oxides. The study has also included the examination on dynamic structural changes in grain boundaries. The obtained information has brought about theoretical bases for the microstructural design.

The recent topic of this work is the first attainment of substantial superplasticity in an alumina-base material. On the basis of the research described above, we have synthesized a ZrO_2-dispersed alumina with a matrix grain size of 0.45 μ m. In this material, the severe cavitation which occurs in conventional materials to limit tensile ductility less than 140% is strongly suppressed, and accordingly the ductility reaches 340-550 % at 1673-1773 K.

Keywords : oxides, colloidal processing, high temperature mechanical properties

33 High Temperature Mechanical Properties of Particulate Reinforced Titanium-Based Metal Matrix Composites

M.Hagiwara, Mechanical Properties Division
[April 1994 to March 1997]

Titanium alloys are attractive materials for aircraft and automobile applications due to their high strength/weight ratio. However,the service temperature is limited to 600℃ due to a degradation of tensile strength, creep resistance, thermal stability and environmental resistance. Moreover,Ti alloys exhibit lower stiffness and poor abrasion-related properties than nickel-based alloys. The dispersion of ceramic particulate such as TiB into Ti matrix is expected to overcome these drawbacks. These particulate metal matrix composites (MMC's) have isotropic properties and can be processed more cheaply using the conventional near net shape methods. However, mechanical property data available for these types of MMC's are still lacking.

This research program was aimed to produce Ti-based particulate MMC's using the advanced powder metallurgy (P/M) processes such as blended elemental (BE) and rapid solidification techniques. During the last three years, we have produced Ti-based MMC's such as P/M Ti-6Al-2Sn-4Zr-2Mo/5-20TiB, Ti-6Al-1.7Fe-0.1Si/10TiB, Ti_3Al/10TiB and Ti/TiC. These MMC's showed superior high temperature tensile and creep properties, Young's modulus and high cycle fatigue strength compared with those for the matrix alloy. Special attention has been paid to improve the high cycle fatigue strength of the Ti-based MMC's by the proprietary new BE technique, in which sintered preforms were quenched into water from above the beta transus temperature and subsequently annealed in the two phase region. Three main factors such as the increase in tensile strength, increase in Young's modulus and matrix microstructure refinement were found to contribute to the increase in high cycle fatigue strength.

Keywords : *particulate-reinforced, titanium, MMC's, mechanical properties, powder metallurgy*

Related Papers

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34 Effect of Cryogenic Temperature and Gas-environment on Deformation and Fracture Behavior

T. Ogata and T. Yuri, Mechanical Properties Division
[April 1996 to March 1999]

To promote the application of clean energy and space technology, there is a project of constructing large scale facilities to transport and store liquid hydrogen. It is very important to evaluate the mechanical properties of the materials including weld metals to keep the reliability of large scale structures used at cryogenic temperatures and in hydrogen gas environment. It is required to comprehend the mechanical properties such as continuous data of fracture toughness from room temperature to liquid helium temperature, and the clarification of effects of hydrogen-gas environment at low temperatures on deformation and fracture behavior of structural materials for the design of structure and se-

lection of materials. Especially, hydrogen embrittlement at low temperatures in the large scale structures of stainless steels is an important subject. In this study, we are going to establish the testing technique of tensile, impact properties, and fracture toughness to evaluate the effects of low temperature and gas environment from room temperature to liquid helium temperature, and investigate the effects of microstructure, grain size, δ -ferrite, and hydrogen gas environment on tensile, impact, fatigue properties, and fracture toughness of austenitic stainless steels from room temperature to cryogenic temperature. In 1996, we have designed a testing instrumets which enable to control temperature around 4 K to 20 K in the tensile test and the Charpy impact test . We also evaluated the tensile properties, the fracture toughness, and fatigue properties of SUS 304L and SUS 316L weld metal. Fracture toughness decreased with the decrease of temperature and increase of δ -ferrite. Fatigue strength at 10^6 cycle of SUS 304L weld at 77 K or 4 K was about 55% of that of base metal.

Keyword : structural material, stainless steel, cryogenic temperature, tensile properties, fatigue properties, hydrogen embrittlement

35 NRIM Long-Term Fatigue Data Sheet Project- I

H. Irie, Mechanical Properties Division
[April 1997 to March 2002]

The project aims at the establishment of long term standard reference data on the fundamental fatigue properties of Japanese engineering materials most commonly used for machines and structures under fatigue conditions.

Since 1975, NRIM has established standard data base of conventional fatigue properties of various metals and their welded joints and published 83 data sheets and 16 technical documents. In the documents, fatigue fact data of various metals, their relationships with basic mechanical properties such as elasticity, the fracture mechanism and so on were summarized and described under fundamental investigations on fatigue phenomena.

Recently the life of machines and constructions has been strongly required to be elongated from the ecological and economical points of view. According to the classical theory of fatigue, many of steels have their fatigue limit and there is no problem for any longer time if they will be used below the fatigue limit. However it has been pointed out that the fatigue limit decreases again for longer use, for instance more than 1010 cycles. Then we will start again the production of long term standard reference data even with some technical difficulties.

In this new project, (1)fatigue data of high strength steels for machine at more than 10^{10} cycles, (2)fatigue data of steels for high temperature use from low to high cycle fatigue regions under controlled strain, (3)fatigue data of titanium alloys for up to long term use and (4)fatigue data of welded joints for big constructions considering residual welding stress will be produced and published as data sheets. In addition, in each category of data production basic researches on fracture mechanism of metal due to fatigue from the micro and macro scopic points of view and development of new evaluation methods will be carried out.

Keywords : fatigue properties, standard reference data, steel, aluminum alloy, titanium alloy

36 Relationship between Fatigue Behavior and TEM Structure of Titanium Alloys

T. Kainuma, Mechanical Properties Division
[April 1997 to March 1999]

The dislocation arrangement introduced in aluminum, copper and iron by fatigue has been studied by a number of workers using TEM techniques (1-5). There is general agreement that fatigue moderate stress results in a widespread formation of subgrains. Segall (1) reported that no subgrain formation was observed, if the stress was sufficiently low but that the dislocation existed in patches with a rather large concentration of dislocation loops being present.

The change of the width or the area of the hysteresis loop during the fatigue testing of materials at constant stress amplitude is taken usually as a measure of dynamical changes in mechanical properties. By testing at stress amplitudes higher than the yield stress of annealed materials, fatigue hardening only takes place in alloys as well as pure metals (decrease in the width of hysteresis loop). While at stress amplitudes lower than the yield stress of cold-worked materials, fatigue softening occurs. Details of fatigue softening behavior have been reported for the materials such as aluminum (2, 3), copper (4) and iron (5).

The aim of this work is to reveal the mechanisms of fatigue softening and hardening behavior of titanium alloys (hcp, bcc structure), fcc metals (copper, nickel) and bcc metals (iron, molybdenum) in connection with the characteristics of crystal structures, using transmission electron microscope (TEM).

1.Relation between fatigue behavior and electron microscopic structure

We examined the crystal structure dependence of fatigue softening and hardening behavior using Ti-3Al-8V-6Cr-4Mo-4Zr alloy (β -type titanium alloy, Ti-3-

8-6-4-4), pure titanium (hcp), fcc metal (pure copper) and bcc metals (iron, molybdenum). We also examined the stress retio (R=-1, R=0. 1) dependence of fatigue softening and fatigue hardening behavior using pure titanium (hcp), β -type titanium alloy (bcc), fcc metal (pure copper) and bcc metals (iron, molybdenum). The experiments for the fatigue softening and hardening phenomena were carried out on work-hardened and annealed materials, respectively.

The present work is to reveal the mechanisms of fatigue softening and hardening behavior of titanium alloys (hcp, bcc structure), fcc metals (cupper) and bcc metals (iron, molybdenum) in connection with the characteristics of crystal structures using transmission electron microscope (TEM).

2. Stress ratio dependence of fatigue behavior of titanium alloys

The aim of this work is to reveal the relationship between fatigue softening and aging temperature of various β -type titanium alloys, and to reveal the relationship between stress ratio of fatigue behavior and TEM structure.

Keywords : pure titanium, β -type titanium alloy, iron, molybdenum, cupper,TEM structure fatigue softening, fatigue hardening, stress ratio dependence

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37 Fatigue Strength in a Corrosive Environment

R. Hamano, Mechanical Properties Division
[April 1997 to March 2000]

Fatigue crack is nucleated along persistent slip bands persistent Luders bands (PLBs), or at grain boundaries where slip bands impinge. The nucleated fatigue crack propagates over two successive stages-stage I (shear mode) and stage II (normal mode).

To improve the resistance to corrosion fatigue cracking which is sensitive to microstructures, we must clarify first; the mechanisms of how corrosive environments assist fatigue damages in the processes such as microstructure-sensitive stage I crack initiation and second: transition of fatigue crack growth from stage I to stage II. We presented experimental results that the transition of fatigue crack growth from stage I to stage 11 is the potential index for the hydrogen-related embrittlement.

In the paper, microstructures immune to environmental damage were examined using a precipitate-hardened steel, based on the concept of the previous paper. That is, at stage I, fatigue crack initiates and advances along the intense shear bands. The triaxial stress field at the tip of the stage I fatigue crack is more enhanced by the inhomogeneous slip deformation of the material. Hydrogen diffused into the triaxial stress field accelerated the transition of fatigue cracking from stage I to stage II, leading to premature failure. To improve the resistance to the fatigue cracking of high-strength materials in a hydrogen-related environment, it is suggested that the slip deformation of materials is not concentrated in a particular slip band.

The precipitate-strengthened alloys are known to have a tendency for slip channeling when the stress is high enough for dislocations to cut through the particles. Slip channeling increase the tendency for planar slip and initiate the fatigue crack at stage I along shear slip bands.

In the present study, the age-hardenable steels of Nitralloy N added the element of carbon (other main alloying elements ; aluminum, nickel, chromium, and molybdenum) are strengthened with the coexistence of the precipitates of carbide and intermetallic compound were used. The coexistence of the coherent precipitates for strengthening the matrix and incoherent precipitates for preventing slip channeling can be expected to improve fatigue life in a corrosive environment of a 3.5 pct NaC1 aqueous solution.

The effects of the coexistence of coherent and incoherent precipitates with the matrix on ductility and fatigue lives in air or in a 3.5% NaCl aqueous solution were studied, to find a method for improving fatigue lives of metallic materials in a corrosive environment.

Keywords : stage I and II crack initiation, environmental damage, fatigue life

38 Fabrication of Ni_3Al-Base Intermetallic Compounds by Unidirectional Solidification

T.Hirano, Mechanical Properties Division

[April 1997 to March 1999]

So far, we found that unidirectional solidification using a floating zone (FZ) method can improve the ductility of polycrystalline Ni_3Al without addition of alloying elements such as boron. Also, we could successfully grow high quality single crystals of stoichiometric Ni_3Al which has been considered to be impossible to grow. The results indicate that the FZ method is a powerful technique not only to fabricate ductile intermetallic compounds but also to provide us single crystals necessary for fundamental study of deformation mechanism.

The objective of this study is twofold. First is to improve high-temperature strength of Ni_3Al. Since the brittleness of Ni_3Al is almost overcome as mentioned above, one of the remaining problems is lack of high-temperature strength. Our target is Ni_3Al-base intermetallic compounds strengthened by alloying or second phase precipitation. We apply the FZ method to fabrication of theses compounds. Second is to study the deformation mechanism of Ni_3Al using single crystals grown by the FZ method. The effect of stoichiometry on the deformation is focused. It is because in our preliminary study we found some interesting deformation behaviors which are characteristic of binary stoichiometric Ni_3Al.

Keywords : unidirectional solidification, floating zone method, Ni_3Al, mechanical properties, deformation mechanism

Related papers

Stress response by the strain-rate change in binary, stoichiometric single crystal Ni_3Al, M.Demura and T.Hirano, Phil. Mag. Lett.,75(1997), 143-148.

High-Temperature Yield Strength of Binary Stoichiometric and Al-rich Ni_3Al Single Crystals, D.V.Golberg, M.Demura, and T.Hirano, Proc. of 2nd International Symposium on Structural Intermetallics, 1997.9, Champion.

39 Basic Study on Refining of Molten Metal and Controlling of Solidification by Electromagnetic Force

A. Fukuzawa, Chemical Processing Division
[April 1994 to March 1997]

A cold crucible type levitation melting method using high frequency electric power is known as a non-contacting melting method. Therefore, this melting method is advantageous for melting of high purity metals, chemically reactive metals and refractory metals.

In these several years, we have made various kinds of trials using the cold crucible type non-contacting induction furnace and we have obtained many fundamental results concerning the cold crucible.

Another advantage of this method is that it has the ability of separating inclusions from the molten metal, because magnitude of electromagnetic force working toward the molten metal is different from that of inclusions due to the difference in physical properties between metal and inclusions. Therefore, the optimum levitating conditions for separating inclusions from molten metal, such as the shapes of the cold crucible and high frequency coil and the electric output power, have been sought to obtain materials with excellent quality. The study on separating mechanism and separating rate of oxide inclusions from molten titanium were carried out, and we succeeded in reducing oxygen content of molten titanium by the addition of metallic cerium. The experimental result shows that the cerium oxide inclusions are separated from molten titanium, and are caught into calcium fluoride flux.

Keywords : cold crucible, levitation melting, electromagnetic force

40 Solidification in the Strong Magnetic Field

A. Fukuzawa, Materials Creation Research Station
[April 1995 to March 1998]

In the dendritic crystal growth, there exists Seebeck current between the top and base of dendrite caused by the temperature difference. The current is very weak, however, in the strong magnetic field it would be possible to induce Lorentz force in the solidification front, and make the flow in the liquid metal trapped in the dendrite region. From this effect, it can be expected to make a homogeneous composition ingot from the surface to the center of the ingot with finer grain size. With above mentioned idea the research work will be carried out by using a 10 Tesla superconducting magnet. Last year, some minor troubles such as mismatch of heater transformer regulator to Ta heater, change of thermocouple of W-Re to Pt-Rh and design of crucible itself were improved. In this fiscal year, investigation on the solidification behavior of Sn and Al alloys will be continued, and Mn-Sn and related alloys will be melted to survey the effect of magnetic anisotropy of primary crystallized phase. The effect of magnetic field on the grain growth will be also examined.

Keywords : dendrite growth, strong magnet, Seebeck effect, Lorentz force

41 New continuous Steelmaking Process

A. FUKUZAWA Materials Creation Research Station

[April 1996 to March 1998]

Today, basic oxygen steelmaking furnace(BOF) is almost only one process, which converts pig iron, produced in the blast furnace to steel. Among many kinds of processes in the steel industry, BOF is the only one process, which keeps batch type operation process. Over a quota century ago, various types of continuous steelmaking processes were proposed and examined by using tons of hot metal. However, none of them were realized directly in industrial scale, since it was in the midst of developing era of BOF. However, an idea or a technology of continuous steelmaking is succeeded now in the continuous desiliconization or continuous dephosphorization process in front of blast furnace. However, an expectation to the continuous steelmaking still exists because the demands for higher productivity and lower manpower are becoming stronger in these days.

Here, a new continuous steelmaking process is proposed to solve these problems. The characteristics of the new one is in one trough type oxygen bottom blowing furnace, that is, by setting blowing nozzles at certain interval in the bottom of the furnace steelmaking reactions can be divided by the bubble curtain without changing the furnace. This will bring the reduction of heat loss and manpower, and simplification of layout. This year water model experiments have been carried out to simulate the mixing characteristics in the bath. As a consequence it became clear that bath depth is one of the key factors to control bath conditions, that is mixing and vibration.

Keywords : continuous steelmaking, steelmaking, oxygen bottom blowing, trough type furnace

42 Investigation on Nucleation and Crystal Growth Mechanism under Heterogeneous Ambient Phase

T. Fujii, Materials Processing Division

[April 1994 to March 1997]

The purpose of the present study is to obtain the fundamental knowledge of the crystal formation process in the circumstance in which other solid phase or foreign particles are included. Among others, the following two subjects were dealt with:

1) Nucleation and crystal growth in peritectic system.
2) Nucleation in solid state reaction and exaggerated grain growth.

For the first subject, we intend to observed in situ the crystal growth process in $Sr(NO_3)_2(H_2O$ binary peritectic system using optical microscope. It has been known that above and below the peritectic temperature T_p (29.3℃, $Sr(NO_3)_2$ phase and $Sr(NO_3)_2 \cdot 4H_2O$ phase are equilibrium with the liquid in this system, respectively; it is generally stated that at the peritectic temperature the upper solid phase reacts with the liquid to induce the lower solid phase. It was found that, however, when the liquid with the composition of peritectic point, namely the intersecting point of two liquidus lines, was maintained at a temperature below Tp, both solid phases could grow simultaneously in the liquid. It can be explained that in such condition, the liquid is supersaturated for both solid phases. It was also found that the growth rate of the lower solid phase was much larger than that of the upper solid phase and the difference in the growth rate between two phases increased with the decrease of the temperature. It follows that the upper solid phase was surrounded by the lower solid phase simply due to the difference in growth rate, which resulted in a 'peritectic texture'.

Growth mechanism of $Ba(NO_3)_2$ {111} and {100} faces grown in aqueous solutions was investigated by ex situ atomic force microscopy (AFM). $Ba(NO_3)_2$ is an isomorph of $Sr(NO_3)_2$ and shows the similar growth feature to that of $Sr(NO_3)_2$. On both faces, spiral layers created by dislocations and isolated islands formed by two-dimensional (2D) nucleation were found. The thinnest growth layers observed on {111} and {100} faces were d111 (4.7 A and d200 (4.1 A in height, respectively, regardless of step formation mechanism whcih agrees with the elementary steps expected in Donnay-Harker and Hartman-Perdok theories. It was also found that spiral layers composed of single, double and triple elementary layers arised on {111} faces and those with double layers formed on {100} faces depending on the Burgers vectors of the central dislocations.

In the second subject, we have succeeded in establishing a new technology to develop commercial scale refractory single crystals from hot-sorked materials doped with a certain amount of oxides as well as corbides by means of exaggerated grain growth in solid state.

Keywords : nucleation, crystal growth, peritectic reaction, secondary recrystallization, exaggerated grain growth

43 Spectroscopic and Electrochemical Investigations of the Metal Complexes with an Unusual Electronic Structure

H. Isago, Chemical Processing Division

Fabrication of molecular devices, where one molecule functions as one device, is one of the most chal-

lenging projects in current and future science. In a number of biological systems, such as enzymes, which can be considered as well-designed molecular devices, unusual electronic structures have often been observed in their reaction centers where some metal ions play an important role. In recent years, phthalocyanines and their metal compounds (MPcs), which are industrially important organic dyes, have attracted much attention in the field of electronics, nonlinear optics, and photosensitization in electron-transfer reaction. They are also of interest from the viewpoint of the similarity in their molecular and electronic structures to those of reaction centers of enzymes.

We have studied MPcs of group-15 elements, of which the electronic structure could be interesting. Quite recently, we have isolated antimony(V)-phthalocyanine for the first time, which showed the smallest HOMO-LUMO transition (Q-band) energy and the smallest phthalocyanine-centered reduction potentials of the known MPcs. The Q-band appeared in the near-infra-red region (13.8×10^3 cm^{-1}) and the first ring-reduction occurred at -0.22 V vs. ferrocenium$^+$/ferrocene, of which the values were extraordinarily small. These characteristics might be useful in the field of non-linear optics, fuel cells, and so on. Furthermore, we have also studied spectroscopic properties of electrochemically-generated one-electron-reduced species of this complex and have concluded that its ground state was orbitally doubly degenerate. Electrochemical investigations further supported this conclusion and even suggested that the ground state of its doubly reduced species might be a triplet; if this is true, this species could be versatile as a unit of a new type of molecular magnets. The latter speculation, however, could not be confirmed due to the chemical instability of the doubly reduced species. Efforts are being made in our laboratory to stabilize doubly reduced species of this type of complexes.

Keywords ; phthalocyanine, antimony, electrochemistry, spectroscopy

44 Improvement of Mechanical Properties of Intermetallic Compounds by Crystal Growth Control

T.Hirano, Chemical Processing Division
[April 1992 to March 1997]

The objective of this study is to improve the mechanical properties of intermetallic compounds by crystal growth. We have found that unidirectional solidification using a floating zone method remarkably enhances the room-temperature ductility of Ni_3Al without addition of alloying elements such as boron. We call this method FZ-UDS. Stoichiometric Ni_3Al exhibits more than 60% tensile elongation at room temperature. It is also found that FZ-UDS can enhance even the ductility of Al-rich Ni_3Al which can not be ductilized by the alloying method, indicating that FZ-UDS is a new promising method to improve brittle intermetallic compounds.

In this study three subjects were stressed. First, crystal growth technique was developed in detail. Columnar-grained structure which was closely related to the large ductility was obtained by selecting the growth rate, depending on the deviation from the stoichiometry. Second, the solidified columnar structure is characterized, paying attention to the grain boundary structure and chemistry. It was found that this structure consists of low energy grain boundaries. Third, the mechanical properties and deformation behaviors were studied.

Keywords : unidirectional solidification, floating zone method, Ni_3Al, room-temperature ductility

Related papers

Effects of Unidirectional Solidification Conditions on the Microstructure and Tensile Properties of Ni_3Al, T.Mawari and T.Hirano, Intermetallics 3(1994), 23-33.

Stress response by the strain-rate change in binary, stoichiometric single crystal Ni_3Al, M.Demura and T.Hirano, Phil. Mag. Lett.,75(1997), 143-148 .

Effect of alloy stoichiometry on grain boundary chemistry and fracture behavior of directionally solidified Ni_3Al, E.P.Geroge, M.Imai, and T.Hirano, Intermetallics, 5 (1997), 425.

45 Study on the Mechanical Properties of Directionally Solidified Intermetallic Compounds

T.Hirano, M.Imai, M.Demura, T.Mawari, E.P.George, and C.T.Liu*,Chemical Processing Division, * Oak Ridge National Laboratory*
[April 1996 to March 1997]

Directionally solidified Ni_3Al is quite ductile unlike conventionally cast Ni_3Al. It seems to be because the grain boundaries mostly consist of low angle and low Σ -type which can be considered to be crack-resistant. Another probable reason is grain boundary segregation of Al and beneficial impurity elements. The objective of this study is to elucidate the influence of boundary type on the grain boundary fracture strength and grain boundary chemistry.

The starting materials are stoichiometric Ni_3Al which were unidirectionally grown by a floating zone method. Bicrystalline tensile specimens were prepared by cold rolling and recrystallization of these materials.

The specimens were fractured in tension in a scanning Auger microprobe and the Auger analyses were performed on the fractured surface. Since the fracture occurred completely at the grain boundary and the fracture surface was clean and flat, we could perform the Auger analyses with high accuracy. It was found that the Al/Ni peak height ratio varied from grain boundary to grain boundary, indicating that Al segregation depended on boundary type. We still continue this experiment to obtain a comprehensive relationship between the Al segregation and boundary type.

Regarding the boundary fracture strength, we had a crack problem at the boundary when we cut the specimens, which caused premature fracture below yield stress. Now we have solved this problem and hopefully we will be able to obtain good data.

Keywords : Ni_3Al, grain boundary type, grain boundary segregation, boundary fracture strength, bicrystalline tensile specimens

Related papers

Intrinsic and Extrinsic Factors Affecting the Ductility and Fracture of Ni_3Al, E.P.Geroge, C.T.Liu, D.P.Pope, and T.Hirano : in Proc. of 4th International Conference on High-Temperature Intermetallics, April, 1997.

Effect of alloy stoichiometry on grain boundary chemistry and fracture behavior of directionally solidified Ni_3Al, E.P.Geroge, M.Imai, and T.Hirano: Intermetallics, 5 (1997), 425.

46 Study on combustion Synthesis

Y. Kaieda, Process Control Division
[April 1996 to March 1999]

The fundamental study to reveal the reactions in combustion synthesis is carried out. The propagation of the reaction front and the synthesis process of the materials synthesized through the reaction are also studied. Investigation by the thermal analysis with rising temperature at constant speed and/or in alternating speed is carried out to reveal the conditions for the initiation of the reaction, the propagation and the synthesis. The influence of pressure and convection on the phenomena in the reaction process of the system containing gaseous phase or liquid phase is studied using a high gaseous pressure apparatus.

The selection of the combinations of elements, which is focused in the present study, will be investigated. The system of the combination that might exhibit the effect of convection and pressure during the reaction and synthesis process is selected considering the system that performs the effect of liquid and gaseous phase. The system of elements, in which the safety during the experiment is assured, is selected.

Most intermetallic compounds are conventionally produced by the processes including high frequency induction vacuum melting and casting.

It is difficult to control accurately the chemical components of intermetallic compounds produced by the conventional process. Homogeneous intermetallic compound is produced by the industrial process including a combustion synthesis method, which is a newly developed manufacturing process in this institute. The chemical components, the impurities industrially produced by the process are revealed. These properties are vitally important when the combustion synthesis method is applied to an industrial mass production process for producing intermetallic compounds.

Keywords : combustion synthesis,

47 Study on the Efficiency of Resonance Photoionization Process

Y.Ogawa, Materials Processing Division
[April 1995 to March 1998]

Laser resonance photoionization continues to be popular in many research fields and the process may find a wide variety of applications. In this process, atoms are selectively pumped from their ground state to an intermediate excited state and subsequently the excited atoms are ionized by direct photoionization.

The ions generated are then extracted from a photoionization induced plasma by an externally applied electric field. One of the important factors in evaluating the process performance is the overall ion yields, which is restricted by two conditions: small bound-free transition cross-section in the ionization scheme and space charge limitation in the plasma.

The objective of this study is to improve the overall efficiency of the resonance photoionization process. The reaserch is composed of the following two subjects.

1. Investigation for autoionizing levels or highly excited Rydberg levels to achieve the efficient photoionization of atoms via bound-bound transitions.
2. Measurement of plasma characteristics to elucidate the ion extraction behavior from the plasma.

Keywords : resonance photoionization, photoionization induced plasma, auto-ionizing level, Rydberg level, plasma characteristic.

Related Papers

Laser Material Purification of Neodymium, Y. Ogawa et al., J. Jpn. Inst. Met. 55(1991):545-552.

48 Oxidation Properties of Sintered TiAl Intermetallic Compound

Y. Muramatsu, Material Creation Research Station, Frontier Research Center for Structural Materials
[April 1996 to March 1998]

This study aims at improving the oxidation resistance of sintered TiAl intermetallic compound with niobium addition. For this purpose,sintered TiAl alloys containing niobium of 1 to 5 at% were prepared by conventional sintering. Some of the sintered alloys were further densified by HIPing. Sintered and HIPed alloys were subjected to oxidation tests which were conducted in an $N_{2\text{-}20}$vol%O_2 atmosphere at1220K. The oxidation behaviors of sintered and HIPed alloys were compared. The effect of niobium addition on oxidation resistance was examined. The scale structure and the mechanism of oxidation resistance were also examined.

The oxidation behaviors of these alloys differed from each other. The sintered alloys showed larger mass gains and earlier spalling in the oxidation stage. The niobium addition made it possible to reduce mass gain and to delay spalling. The HIPed alloys containing 4 and 5at%Nb showed excellent oxidation resistance over an oxidation time of 1800ks. EPMA analyses clarified that the scale formed on the sintered alloy of 5 at%Nb consisted of five or six layers in the following arrangement from outside,

TiO_2/Al_2O_3(continuous)/TiO_2+Al_2O_3(reticular)/
TiN+Ti_2AlN/Nb-rich+Al_2O_3(acicular)+Ti_3Al/Ti_3Al.

The scales of the HIPed alloys, without niobium and with 5 at%Nb, were found to have similar arrangements. In these alloys, although the TiN+Ti_2AlN layer could not be detected by EPMA analysis, those nitrides were confirmed by X-ray analysis. Investigations on the mechanism of oxidation resistance suggested that the difference of oxidation behavior between sintered and HIPed alloys was caused by residual pores and the structural difference of their scales, and that the improvement of oxidation resistance with niobium addition was due to the suppression of TiO_2 growth and the promotion of dense and continuous Al_2O_3 formation.

Keywords : TiAl intermetallic compound, powder metallurgy, oxidation behavior, niobium addition, scale structure

Related Papers

Effect of Niobium Addition on Oxidation Resistance of TiAl Intermetallic Compound Prepared by Powder Metallurgy,Y.Muramatsu, K.Honma, A.Miyazaki and K.Funami, J.Japan Inst.Metals 60(1996),1116-1124.

49 Synthesis and Utilization of Mesoporous Materials

Y. Sakka, Materials Processing Division
[April 1996 to March 1999]

Two types of porous materials are used for fabricating nanocomposite, heterogeneous phases, hierarchically structured materials, etc. One is synthesized by consolidating fine powders through a colloidal processing. The other is microporous materials with layered perovskite structures, which are synthesized by soft chemical processing i.e. a sol-gel or a hydrothermal processing.

The colloidal processing is a useful tool for preparing mesoporous materials with desired pore size distribution. An important factor is to control the interparticle interaction in liquid. Porous alumina or zirconia body with a narrow pore size distribution, which is determined by a mercury porosimetry, is produced by slip cast of dispersed suspension followed by low temperature sintering. Infiltration of metal solutions into the porous materials and sintering result in the advanced materials.

Intercalation or ion-exchange reaction in host materials (antimony oxide, vanadium oxide, etc.) results in a novel electronic material which is not synthesized by heating at high temperatures. Vanadium oxide-organic composite is synthesized by a hydrothermal method. The lithium insertion characteristics are examined. The reaction is influenced by the micro-pore size of the host-material. Powder and film of amorphous antimonic acid are prepared by a direct reaction of $Sb(O\text{-}n\text{-}C_3H_7)_3$ with H_2O_2 aqueous solution. Effect of the water vapor pressure and micropore structure on the proton conductivity is studied.

Keywords : soft chemistry, micro-pore, colloid, vanadium oxide, antimonic acid

Related Papers

Water Sorption-Desorption Characteristics of Proton-Conducting Antimonic Acids with Cubic and Monoclinic Structures, Y. Sakka, K. Sodeyama, T. Uchikoshi, K. Ozawa and M. Amano, J. Am. Ceram. Soc. **79**(1996):1677-80.

Preparation and Electrical Conductivity Measurement of Amorphous Antimonic Acid, K. Ozawa, Y. Sakka and M. Amano, J. Ceram. Soc. Japan **104** (1996):756-59.

50 Fundamental study on microstructure control by slurry processing

Y. Sakka, Materials Processing Division
[April 1996 to March 1997]

In the processing of advanced materials by sintering, using fine powders is advantageous for reducing the sintering temperature and resulting in fine-grained microstructures. However, fine particles, submicrometer-sized or less, tend to agglomerate due to van der Waals attractive forces. Slurry processing provides part of the solution to this problem. In the slurry, van der Waals attractive forces can be counteracted by repulsive forces that can be established between particles due to either electrostatic and/or steric interactions.

Well-dispersed slurry of Al_2O_3,Si_3N_4 and/or ZrO_2 particles is prepared by pH adjustment or adding appropriate amount of polyelectrolyte. In case of the slurry with high solids content, green body with a narrow pore size distribution is produced by a slip cast. When the solids content is low, however, long range segregation occurs due to the differences of mass and particle size. In the system of Al_2O_3 (particle size 0.2 μm) and ZrO_2 (0.08 μm), the solids content where such segregation phenomenon takes place is below 20%, and in the system of Si_3N_4 (0.4 μm) and Al_2O_3 (0.2 and 0.8 μm) that is below 30%. Using the slurries with an appropriate amount of solids, various types of advanced materials, such as functionally graded material, hierarchical structure material, nanophase material, etc., might be produced.

Keywords : colloidal processing, zirconium oxide, aluminum oxide , dispersion, slurry

Related Papers

Pressure Filtration of Alumina Fine Particles, T. Uchikoshi, Y. Sakka and K. Ozawa, 5th WORLD CONGRESS OF CHEMICAL ENGINEERING IV (1996):1007-12.

Colloidal Processing for Fine Particles of Al_2O_3-15 vol% ZrO_2 System, T. S. Suzuki, Y. Sakka and K. Hiraga, J. Japan Soc. Powder and Powder Met. 44 (1997):356-61.

51 Photocatalytic Properties of Ultrafine Particles

S. Ohno, Materials Processing Division
[April 1996 to March 1998]

It is well known that the ultrafine particles (UFP) have many excellent physical and chemical properties. In particular, UFP of oxide semiconductors is a very promising material as a photocatalyzer.

In this study, titanium oxide (TiO_2) UFP synthesizes by a RF-plasma CVD technique. Then the photocatalytic property of the TiO_2 UFP characterizes by the photodecomposition of water.

When coarse TiO_2 powder (rutile or anatase) is used as a starting material, the crystal structure of TiO_2 UFP synthesized by the RF-plasma CVD is composed of binary phases of rutile and anatase regardless of the crystal structure type of starting material. The composition of rutile and anatase phases is influenced by the crystal structure of the starting material and the concentration of reaction gas (O_2). Those TiO_2 UFP has a high photocatalytic activity of 2-10 times as compared with that of commercial TiO_2 UFP for a photocatalyzer.

Keywords : ultrafine particle, arc plasma, RF-plasma, TiO_2, photocatalytic property

52 Control of the Oriented Single Crystals of Refractory Metals through Solid State Processes

T. Fujii, Materials Processing Division
[April 1997 to March 1999]

Molybdenum and tungsten, b.c.c refractory metals, have many advantage such as high melting points, useful elevated temperature strength, high thermal and electrical conductivity, low thermal expansion and good resistance to liquid metal corrosion. Thus, at the present, molybdenum, tungsten and their alloys are widely utilized in the fields of electrical and electronic industries, high temperature vacuum and chemical industries. However, such refractory metals undergo a severe loss of ductility after recrystallization, welding or heavy neutron irradiation. It is generally accepted that such a problem is due to intergranular embrittlement. This is the greatest weak point of these metals.

Recently, NRIM (National Research Institute for Metals) has succeeded in establishing a new technology to develop commercial scale molybdenum and tungsten single crystal and their multilayer crystals from hot-rolled sheet doped with a certain amount of oxides by means of secondary recrystallizaiton. However, no research and development for preparation of the single crystal with desired orentaion by a solid state process have been carried out.

Thus, the main purpose of this study is to develop a new technology for preparation of the oriented single crystals of refractory metals such as a molybdenum or a tungsten through solid state processes.

The crystal formation processes in liquid phase, particularly in peritectic system will be studied in the

next subject as well. In the previous study, it was found that both high and low temperature solid phases grow in the liquid at the temperature range in which lower solid phase is stable and these two phases show the large difference in growth rate. We intend to clarify such growth process of metastable phase and the difference in growth kinetic between two solid phases in peritectic system.

Keywords : Oriented single crystal, refractory metal, solid state process

53 Selenium(VI) Removal from Wastewater by a Fluidized Bed Process

K. Goto, Materials Processing Division
[April 1997 to March 1999]

The Japanese government office enacted in 1994 that the effluent water quality standard for selenium was 0.1mg/l. This was an important problem in the copper refineries, because they discharge a large amount of effluent containing the selenium ions higher than the standard. A provisional standard of 20mg/l has been applied to the industries, and the moratorium will finish within a few years. In order to clear the general standard, various methods were examined and investigations were carried out. According to their efforts, it becomes clear that Se(IV) can be removed but Se(VI) cannot. In the laboratory, Se(VI) can be removed by the reduction and the coprecipitation with ferrous and ferric ions. The cost is, however, too expensive and the method cannot be applied to the wastewater treatment from the practical viewpoint.

The aim of this study is to develop a new method to remove the selenium ions. We propose a new fluidized bed process, which involved reduction of Se(VI) to Se(IV) or to metallic selenium. The research is carried out to verify that Se(VI) is reduced by the fluidized bed process, and to decide the optimum conditions of the process.

Keywords : selenium, removal, wastewater, fluidized bed, selenate

54 Basic Technology Development of Materials Procession in a Short-Duration Microgravity Environment

1. A study on double combustion synthesis apparatus

Y. Kaieda, Chemical Processing Division
[April 1995 to March 1997]

The main purpose of the study is synthesizing high performance intermetallic compounds using short time micro-gravity environment. The micro-gravity is achieved by dropping the experimental apparatus.

Combustion synthesis is a production process to produce chemical compounds from elemental powders in a short time using the heat of formation of component elements. The short time double combustion synthesis apparatus was developed in order to conduct the experiment of combustion synthesis in micro-gravity. The experiments were carried out using the same combustion synthesis apparatus in order to compare the 1G and micro G results exactly. The velocities of propagation of combustion synthesis were measured by two sets of W-Re thermocouple.

Keywords : combustion synthesis, Intermetallic Compounds

2. A study on the synthesis of high temperature superconductors

H. Kitaguchi, 1st Research Group
[April 1995 to March 1997]

One of the purposes of this study is to establish the basic knowledges related to the solidification phenomena of high temperature oxide superconductors, such as phase diagram, reactivity and wettability with other materials. Another purpose is to establish the technology for high temperature experiment in a short-duration microgravity environment.

Melt-solidification process was performed for Bi-Sr-Ca-Cu-O high temperature superconductor in the aircraft and the effect of microgravity on the microstructure formation was studied. The experiment under extremely high gravity was also carried out on the ground in order to clear the gravity effect. We found the gravity has influence on the alignment of Bi-2212 crystal in the melt and on the distribution of impurity phases. The effect of undercooling on the formation of metastable intermetallic compound superconductors $Y_1Pd_2B_2C_1$ was also studied.

Keywords : high temperature superconductors, Bi-Sr-Ca-Cu-O, aircraft experiment

55 Solidification Processing For Particle Dispersed Unidirectionally Grown Composites

A.Sato, Advanced Materials Processing Division
[April 1994 to March 1997]

The crystalline structure relates greatly to properties of metallic materials. Properties of metallic materials are affected by fine crystal grain structures, unidi-

rectional solidification crystal structures or mono-crystal structures. On the other hand, the strength and the rigidity of metallic materials are improved by dispersed fine ceramic particles. The purpose of this study is to analyze the behavior of ceramic particles at the solidification interface of the composite material solidifying unidirectionally. In this study, we hope that the experiment should be performed simply as much as possible. Therefore, we decided to use Aluminum as a matrix metal and alumina as a ceramic particle. A planetary ball mill (for mechanical alloying) was used to mix the matrix aluminum powder and the alumina powder. We selected the aluminum powder because its melting point is low and because its handling is very easy. Physical and chemical properties on alumina powder are also easy to obtain. Samples of four kinds of addition of alumina, 0.2, 0.5, 1.0, and 5.0vol%, were prepared. The mixed powders of aluminum and alumina was pressed in a metallic mold. The pressed powders was sintered and then swaged to make the density high. The swaged samples were used for the unidirectional solidification experiments. We used two kinds of solidification rates of 3.3×10^{-6}m/s and 6.6×10^{-6}m/s under the constant temperature gradient (4.7K/mm). The effect of the quantity of alumina addition and the solidification rate was not recognized in the sample of 1.0vol%, and particles added was pushed during solidification, and the engulfment of alumina particles by solid phase could not found. The sample of 5.0vol% alumina was not melted uniformly due to both alumina on surface of aluminum powders and the alumina added.

Keywords : solidification, unidirectional solidification, composite, particle dispersion

56 Research and Development of Recyclable Simple-System Alloys

A. Sato, Frontier Research Center for Structural Materials

[April 1996 to March 2001]

Because of the various additives, recycling of the metallic materials used for the parts of automobiles and other aspects remains a question awaiting to solve. Nowadays, the recyclabiliy of materials should be fulfilled in the development of new structural materials. In the present research, four kinds of metallic materials and processing methods for improving recyclability are concerned.

(1) High damping Mn20Cu5Ni2Fe alloy is developed and shows superior damping capacity and workability. A directly furnace cooling process is used to place the alloy in the high damping condition, and extensive increases in damping capacity have been observed in the vicinity of 40℃. The higher damping capacities are ascribed to the existence of antiferromagnetic and FCT structured Mn-enriched matrix in the alloy. Slower cooling after solution treatment is effective to promote the critical transition temperature, hence higher damping capacity is sustained in a wide temperature range.

(2) Fe-Fe composite material is fabricated by sintering iron powder and reinforcing SCIFER fiber, made severely drawn steel wire, with ordinary powder metallurgy technique. SCIFER fiber has the cold worked microstructure and a tensile strength of 400kg/mm^2, however the characters may deteriorate when heat treatment is conducted above 450 ℃. In the present work, SCIFER fiber and iron powder are primarily modified with ultra-fine iron particles by a wet dispersion method and sintering of the mixture can be realized only at 400℃ after compressing. It is observed with bending experiment that the strength of the sintered Fe-Fe composite exceeds 30kg/mm^2.

(3) Alloying and refinement of crystal grains are usually attempted to increase the strength of aluminum alloys. However, the addition of grain refiners and alloy elements degrades recyclability of the alloys. In the present work, a strong stirring casting process on the basis of the rheocasting method, and an ultrasonic vibration process used for solidification have been successfully developed for the casting of aluminum alloys. It is confirmed that fine microstructure and superior mechanical properties could be reached in many aluminum casting alloys with these processes.

(4) The improvement of chip-disposability for metals and alloys is important for the automation of machining processes and the utilization of unmanned machine tools. The Chip-disposability and cutting resistance during tuning are examined for some low alloy steels with the microstructure of ferrite and martensite. It is shown that the chip-disposability can be remarkably improved when the machined steels are involved with martensite microstructure, as compared with the spheroidized or normalized steels. For steels which have martensite microstructure and hardness of HV200 ~ 350, chips in the high cutting speed region are observed being broken below one curl.

Keywords : high damping alloys, Ultrafine Fe particles, Strong stirring casting, Ultrasonic vibration casting, Chip-disposability, martensite microstructure

57 Fabrication of Highly Functional Composite Cast Irons

A. Sato, Frontier Research Center for Structural Materials
[April 1996 to March 1999]

The present study, as one theme in the "Living and Social Basis Project" jointly researched in some institutes, universities and corporations, was started in 1996. Fabrication of ceramics particles-dispersed cast iron is being tried at NRIM, with a compocasting technique used to produce composite materials by adding various particles into the solid-liquid co-existing slurry. Simultaneously, a CVD coating method is attempted to the particles and iron powder to improve the wettability with molten cast iron and the dispersion state of the particles. Alumina, silica and silicon carbide are possibly used as dispersion particles in cast iron. Although alumina is the most promising one, the unsatisfactory wettability of alumina with cast iron makes it necessary to coat with some other compounds such as silica, silicon carbide, to improve the wettability. Therefore, a chemical vapor deposition technique in fluidized bed is applied to make coating layers on alumina particles. The coating of silica on alumina substrate and on the cast iron powder is proceeded in a gaseous mixture of Tetramethylsilane and water. The deposited silica layer is an amorphous silica. The amorphous silica solitary is stable up to 1393K, however, when the particles are mixed with calcium hydroxide, the amorphous silica layers are found to crystallize into α -quartz crystals at 1300K, and to crystobalite at 1500K. Finally, the mechanical properties of the fabricated composite materials are characterized, intending to qualifying the materials to different applications. The compocasting technique, usually used for alloys with lower melting temperatures, has been improved and applied to the creation of cast irons, through solving some processing problems and designing suitable apparatus.

Keywords : Cast iron, Compocasting, Alumina particles, Stirring, CVD, SiO_2

58 Micro Structure Control with Plastic Deformation under Mashy State

T. Dendo, Advanced Materials Processing Division
[April 1996 to March 1998]

Processing under semi-solid or semi-molten state i.e. mashy state has attracted the attention as an exotic technique for refinement of the micro structure and/or for creation of new composite materials. In the procedure of the above processing, plastic working is mostly inevitable. Plastic working under mashy state in which solid and liquid phases coexist, however, induces unfavorable problems such as segregation due to the flow of the liquid component and/or difficulty of the shape retention during handling of work piece.

In order to solve the above problems, a new metal working process is proposed, in which the semi-molten metal is inserted in a solid hollow metal and worked plastically together with the hollow metal that suppresses the flow of the liquid component and retains the shape of mashy metal. The work piece fabricated through this process is consequently clad in the hollow metal. Experimental studies are carrying out to grasp the comprehensive feasibility of this process.

On the other hand, the preferred orientation in the crystalline introduced by plastic deformation under mashy state is explored so as to aim at developing new method of texture control.

Keywords : mashy state, plastic working, clad metal, preferred orientation

59 Brazing Experiment under Microgravity

K. Sasabe, Frontier Structural Materials Research Center
[April 1996 to Mach 1998]

The brazing experiment under microgravity was carried out. The purpose of this experiment was to understand capillary penetration phenomena under microgravity to establish a guideline for the decision of brazing process parameters in space environment. The specimens were sleeve joints of stainless steel tubes of the thickness of 3 mm. The filler alloy used was Ag-Cu-Li alloy. The specimens were classified into 6 types by the combinations of the width of joint gap, the position of filler alloy and the temperature gradient. The specimens were heated up to the brazing temperature and cooled down to the solidus temperature under microgravity during parabolic flight of the small rocket, TR-1A#5 by the NASDA.

The test results proved that our theoretical prediction method for capillary gap penetration by the thermal conductivity theory and the surface tension theory was reasonable one. Furthermore, it was cleared that the control of temperature distribution is very important for brazing under microgravity.

This experiment was performed as a part of "Space Utilization Frontiers Joint Research Projects".

60 Influence of Surface Composition on Joining of Materials

K. Sasabe, Joining and Interface Research Station
[April 1995 to March 1998]

Ion bombardment has several effects on a joining surface; not only cleaning, but ion implantation and surface roughening. It is not clear yet that the relationship between each effect and joint properties. On the other hand, hydroxyl radical and thermal oxide on the surface accelerate the joining process of silicon. The bonding mechanism is not clear yet.

The aim of this study, in joining of metals, is to investigate; i) the relationship between each effect and joint properties, ii) effect of gases on a joining surface. The aim, in joining of silicon, is to investigate; i) the effect of hydroxyl radical and thermal oxide on a surface, ii) the difference in bonding mechanism between metals and silicon.

I. Joining of metals

In this study, we carry out the diffusion joining using a clean surface with little defects. We investigate the effect of an adsorptive on joining properties.

II. Joining of silicon

In order to clarify parameters that affect mechanical and electric properties of joint, we investigate the relationship between surface adsorptives and joint properties using a clean surface in an ultra high vacuum atmosphere. It is expected that oxygen in material affects disappearance of surface adsorptives, so we also investigate the relationship between behavior of surface adsorptives and joint properties of CZ silicon in different oxygen concentration.

keywords : joining, adsorptive, silicon

61 Diffusion Bonding of Stainless Steel and Metals of Group Ⅳ～Ⅵ

T. Kasugai, Materials Processing Div.

[April 1996 to March 1999]

The joinability of the diffusion bonding of stainless steel and metals of group Ⅳ (Ti, Zr, and Hf), the formation of metallic compounds in the bonding zone of this steel and metals, and diffusion mechanism were investigated in connection with the periodic table.

The materials used in the experiment were industrial pure metals and JIS SUS304L stainless steel. The bondings were carried out using a high frequency induction heating device with 400kHz. The temperature of bonding was used between 923～1373K and bonding pressure was 5～10MPa. The bonding was made in vacuum of $3 \sim 6 \times 10^{-3}$ Pa.

An irregular diffusion was observed in all of SUS304L/metals of group Ⅳ bonding joints, where chromium was condensed into the diffusion bonding interface and at the same distance nickel was diluted. This up-hill diffusion of Chromium was caused by the formation of Fe-Cr phase containing a small amount of Nickel. Moreover, the up-hill diffusion of Chromium was observed in the bonding interfaces in 80Ni-20Cr alloy/metals of group Ⅳ. Products of these diffusion bonding interfaces are under investigation. It is considered that the up-hill diffusion phenomena of Chromium ia observed commonly in SUS304L/metals of group Ⅳ and Ni-Cr alloy/metals of group Ⅳ diffusion bonding interfaces.

Keywords : Diffusion bonding, Stainless Steel, Group Ⅳ metals

62 Monitoring and Control of Thermal Plasma

K. Hiraoka, Advanced Materials Processing Division

[April 1996 to March 1999]

The characteristics of the laser plasma induced in laser welding were investigated by spectroscopic measurement and probe measurement. Furthermore, the stability of bead formation in arc welding was discussed from the damped oscillation characteristics of molten pool metal. A new method for producing a large and stably induced laser plasma has been proposed to characterize the plasma. It is attained with a good combination of the laser beam and an arc. To analyze the plasma in welding, the characteristics of this stable plasma were investigated systematically by some measurements. The distribution of the electron density was evaluated by measuring the infrared radiation from the plasma. The electron temperature distribution was calculated numerically from the value of the electron density on an assumption that the plasma was in local thermodynamic equilibrium. The incident and transmitted laser powers were measured using a power probe. The plasma energy was evaluated from both value. From the experimental data of the above measurements, it was confirmed that the plasma was maintained by inverse Bremsstrahlung.

A monitoring method of the bead formation on a thin plate has been developed. In this method, the heat transfer in the molten pool and the damped oscillation of the pool can be detected during an arc welding with a pulsed current. The principle of the detecting method was proposed in 1974 (Japanese Patent No. 876269, U.S. Patent No. 3988567, British Patent No. 1493524), and the detecting method was advanced to apply to a new welding monitoring system. In the developed system, the depth in a partially penetrating weld was roughly estimated from the waveform and phase differences between the pulsed arc current and the heat radiation detected from the back side pool. In addition, the back bead sizes such as back bead width and reinforcement height in fully penetrating weld were esti-

mated by analyzing the waveform of the heat radiation detected from the back side pool corresponding to the damped oscillation of the molten metal.

Keywords : laser, plasma, electron density, plasma temperature, arc, welding, molten metal characteristics, damped oscillation

Related papers;

1. Plasma Structures in Ar-H_2 Mixed Gas Tungsten Arcs Determined by Some Spectroscopic Measurements, Hiraoka K., Quarterly J. JWS, 15(1997)2, 259-266 (in Japanese)
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4. Formation of Stably Induced Laser Plasma and Its Characteristics, Tsukamoto S., Hiraoka K., Asai Y., Irie H., Yoshino M. & Shida T., Proc. 6th Int. Cong. JWS, 1 (1996) 197-202.
5. Bead Formation and Characteristics of Heat Transfer and Damped Oscillation, Okada A., Hiraoka K., Nakamura H. & Yamamoto H., Quarterly J. JWS, 14(1996)3 538-545 (in Japanese)

63 Coatings Formation by Powder Deposition Processes

S. Kuroda, Advanced Materials Processing Division
[Apr. 1995 - Mar. 1997]

Coatings with thickness over 50 μ m are highly demanded in various industries. The most successful process in this category has been a family of thermal spray processes. Thermally sprayed coatings have been used mostly for surface protection but recent applications include more functional areas such as biomedical implant, electric heaters and radiators, and catalytic cleaners. Our group has worked on plasma spray in the past 10 years to clarify the mechanism of coating formation.

In this research, special emphasis was placed to clarify the bonding between sprayed splats, which plays a key role in the development of the microstructure of sprayed deposits. Effects of the substrate temperature, spraying environment, the velocity and temperature of particles before impact on the inter-lamellar bonding were studied systematically.

Another point of interest has been to develop a new process to deposit thick coatings with improved properties, such as less porosity. Finer powders than those usually used for thermal spray are electrified and deposited onto a substrate. A possibility to consolidate the deposit by a CO_2 laser is under investigation.

Keywords : thick coatings, spray deposition

Related papers

The Quenching Stress in Thermally Sprayed Coatings, S. Kuroda and T. W. Clyne, Thin Solid Films, 200(1991) : 49-66.

Quenching stress in plasma sprayed coatings and its correlation with the deposit microstructure, S. Kuroda, T. Dendo, and S. Kitahara, J. Thermal Spray Technol. 4(1)(1995) : 75-84.

64 Evaluation of the structure and properties of advanced thermal spray coatings

S. Kuroda, Advanced Materials Processing Division
[Apr. 1996 - Mar. 1997]

Advanced thermal spray processes such as plasma spray and high velocity oxy-fuel (HVOF) thermal spray are gaining an increasing importance in various industries because they can form thick protective coatings of a wide range of materials. However, there remain deficiencies in scientific understanding of the coatings formation phenomena as well as the characterization and evaluation of thermal sprayed coatings. This program is to enhance international collaboration to resolve the situation and enhance the technological foundation of the surface modification technology. Prof. C. Berndt and S. Sampath of State University of New York (SUNY) at Stony Brook visited Japan and gave lectures on protection of infrastructures, wear and friction, and failure mechanisms of thermal sprayed coatings at NRIM, Japan Institute of Metals annual meeting, and a special symposium organized by the Surface Finishing Society and the Japan Thermal Spray Society.

From NRIM three researchers visited SUNY, gave lectures on plasma diagnostics, formation of FGM coatings by the twin torch method developed by NRIM, and residual stress in thermal sprayed coatings. We also conducted thermal cycle test on TiC-Mo coatings with two-layer and gradient structure by a burner rig installed at SUNY. Further cooperation in the fields of shared interests is now considered. This program was supported by the Bilateral International Joint Research by Special Coordination Funds Promoting for Science and Technology.

Keywords : plasma spray, high velocity oxy-fuel ther-

mal spray, gradient coatings

Related papers

Surface Modification by Thermal Spray Processes, C.C. Berndt, Japan Institute of Metals 1997 Spring Meeting, A special lecture by distinguished foreign scholars.

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65 Metastable Phase Solidification from Undercooled Liquid by Inducing External Nucleation Seed

S. Tsukamoto, Advanced Materials Processing Division
[April 1993 to March 1997]

Metastable metallic materials produced from deeply undercooled melts can provide some interesting properties. The aim of the investigation is to elucidate the role of nucleation during the solidification of undercooled melts and to develop a new technique for producing metastable bulk materials using an electromagnetic levitation melting and an external seeding.

Phase selection in the solidification of deeply undercooled Ni-Cr alloys were investigated using the electromagnetic levitation method. Double recalescence behaviours were observed for hypoeutectic alloys Ni-52vol% Cr and Ni-53.4vol% Cr. Triple recalescence behaviour was also observed for the first time which indicates a three stage solidification process for Ni-53.4vol% Cr alloy. From the recalescence temperatures which coincide with the critical temperatures of the local composition in the system, it was deduced that the metastable BCC α phase formation preceded the equilibrium FCC γ phase formation in the solidification of deeply undercooled hypoeutectic alloys. The nucleation and growth kinetics for the competing phases was calculated with recent models. The theoretical calculation revealed that the preferential metastable BCC α phase formation is due to its favourable nucleation kinetics.

The external nucleation seeding was also attempted to control the phase selection of deeply undercooled melts where the equilibrium phase is preferable to the metastable phase for nucleation kinetics. Type 316 stainless steel (equilibrium solidification phase : BCC ferrite) was undercooled to the pre-set temperature in the levitation coil. Then, the seeding was carried out using a Fe-50vol% Ni alloy of FCC structure, the lattice parameter of which is similar to that of the metastable γ phase of Type 316. The microstructure revealed that the metastable FCC γ phase solidification took place at the first stage of the solidification due to epitaxial growth from the nucleation seed.

From above experimental results, it is clear that the nucleation process can be controlled by the external nucleation seed and metastable materials can be formed using the new technique we proposed.

This research was performed in collaboration with Research Development Corporation of Japan and University of Cambridge.

Keywords : solidification, undercooling, metastable phase, electromagnetic levitation, Ni-Cr alloy, double recalescence, stainless steel, external nucleation seed

Related paper

Metastable Phase Solidification in Electron Beam Welding of Dissimilar Stainless Steels, S. Tsukamoto, H. Harada and H.K.D.H. Bhadeshia (Unv. of Cambridge), Mater. Sci. Eng., A178 (1994) 189-194

Metastable Alloy Phase Formation from Undercooled Steel and Ti-Al Melts, S. Tsukamoto and O. Umezawa, Mater. Sci. Eng, A223 (1997) 99-113.

Theoretial Calculation of Nucleation Temperature and the Undercooling Behaviors of Fe-Cr Alloys Studied with the Electromagnetic Levitation Method, X.Zhang, S. Tsukamoto, Metall. Mater. Trans., 30A (1999) 1827-1833.

66 Characterisation of Laser Induced Plasma and Interaction between Laser and Plasma

S. Tsukamoto, Advanced Materials Processing Division
[April 1996 to March 1997]

One of main problems of the laser processing is the presence of a laser induced plasma which absorbs or scatters the laser beam and leads to a reduction in heat input or power density supplied into the materials. In the present study, we propose a new method for producing a large and stable laser plasma to elucidate the role of the plasma in laser processings. The plasma can be formed by irradiating the focused laser beam onto DC discharge simultaneously with flowing an assist gas through the coaxial nozzle. The plasma is very stable enough to analyse some characteristics.

From the experimental data of the transmittance, local electron density and temperature, it was confirmed

that the absorption of CO_2 laser was caused by inverse Bremsstrahlung. This means we can calculate the absorption loss of the laser energy in the laser processing using inverse Bremsstrahlung theory. For example, if the Ar plasma of 5 mm in length is formed during the laser welding, the absorption loss is estimated to 24 %.

The plasma was maintained at the location where the absorption energy was balanced with gas cooling. A critical value of the power density for maintaining the plasma exists depending on the plasma composition. Ar plasma was formed in a wide range of the power density, whereas the formation of Ar-He plasma was limited only in a small range of high power density due to a large ionisation energy of He.

This research was performed in collaboration with Ibaraki University.

Keywords : CO_2 laser, laser induced plasma, characterisation, electron temperature, electron density, absorption, inverse Bremsstrahlung

Related Papers

Characteristics of Stably Induced Laser Plasma, S. Tsukamoto, K. Hiraoka, Y. Asai, H. Irie, M. Yoshino & T. Shida, Proc. ICALEO'96, 81 (1996) B76-B85.

Formation of Stably induced Laser Plasma and Its Characteristics, S. Tsukamoto, K. Hiraoka, Y. Asai, H. Irie, M. Yoshino & T. Shida, Proc. 6th International Welding Symposium of JWS, 2 (1996) 197-202.

67 Application of In-situ Strain Measurement by the Laser Speckle Method

Y.Muramatau, Advanced Materials Processing Division
[April 1996 to March 1 999]

Non-contact strain measurement using the laser speckle method is characterized by its high-responsibility and high-accuracy. We already proved in the previous research that it was possible to do the in-situ strain measurement almost all period in a thermal cycle even if the measuring point was in a high temperature range.

In this year, we examined the strain behaviors in several parts of heating line applied on rectangular thin stainless plate, for examples, the starting point, center or the crater. It was clarified that the restraint in each part of heating line strongly affects the behaviors. Strains during phase-transformation should be also affected by the restraint and it is significant to predict the residual stress distribution.

A debonding of the sprayed coating films from the substrate materials by the thermal impact was also examined in our research. Many strain curves in the surface of the material which were already detected by the laser speckle method were compared with those in coated films sprayed on the same substrate materials (Stainless thin plate). Ceramic coated and metal coated thermal sprayed specimens were prepared for the purpose. The local heating with some degree of heat inputs were applied on the specimens. The method could detect the strain behaviors in the sprayed film clearly especially the strains just before the debonding of the films from the interface. In case that the debonding did not exist in appearance, we could estimate the peeling detecting a time-strain curve in the film and comparing with that of the substrate plate.

Keywords : laser speckle method, dynamic strain, in-situ strain measurement, high temperature, austenitic stainless steel, plasma spraying, debonding, coating film

68 Synthesis of Superconducting or Supermagnetic Ultrathin Films by Use of Ion Implantation Technique

K.Saito, 1st Research Group
[April 1996 to March 1999]

BiSrCaCuO ultrathin films with thicknesses below 100 Å have been synthesized on (001) MgO substrates by using single-target magnetron sputtering and by optimizing the heat-treatment conditions. Remarkably high values of the zero-resistance transition temperature, Tc,o, 106K,88K and 84K were obtained for 70 Å ,40 Å and 20 Å thick films, respectively. A 10 Å thick ultrathin film was almost insulating. Cross-sectional high resolution transmission electron microscopy revealed that the minimum thickness for superconductivity to occur was about 20 Å , corresponding to the half-unit cell thickness of the Bi system layered structure. Low-quality ultrathin films with Tc,o below 70 K were improved significantly by means of ion implantation combined with postannealing at relatively low temperatures.

In order to synthesize α" -$Fe_{16}N_2$ nitride with giant magnetization,(001)-oriented 500 Å thick ultrathin films were implanted with nitrogen ions at cryogenic temperatures below 25K. α'-martensite phase with a volume fraction of 90% and Fe_3N were formed at low temperatures. X-ray half-width of the α'-line was broader than that for specimen implanted at room temperature. Coating of copper or gold films on the implanted surfaces reduced escaping of nitrogens and increased the volume fraction of α"-$Fe_{16}N_2$.

Keywords : ultrathin films, BiSrCaCuO, $Fe_{16}N_2$, superconductor, giant magnetization,

Related papers

Synthesis of Superconducting BiSrCaCuO Ultrathin Films of Thicknesses below 10 nm, M.Kaise and K.Saito, J.Japan Inst. Metals,1997, in press.

Synthesis of α"-$Fe_{16}N_2$ Nitride by means of Nitrogen Ion Implantation into Iron Thin Films, H.Shinno, M.Uehara and K.Saito, J.Mater.Sci.32 (1997):2255-61.

69 Formation of $Fe_{16}N_2$ by nitrogen ion implantation into Fe films and post annealing in magnetic field

H. Shinno, 1st Research Group
[April 1995 to March 1998]

$Fe_{16}N_2$ has been studied by many researchers since the saturation magnetization of the material has been reported to be as large as 2.8 to 3.0 T, and is much larger than that of Fe (2.2 T). However, further investigations are necessary in order to utilize $Fe_{16}N_2$ for practical applications, because it is difficult to fabricate materials with large volume fraction of $Fe_{16}N_2$.

We have implanted nitrogen ions into 50 nm thick Fe films. The volume fractions of α'-martensite of the implanted films were more than 90%. $Fe_{16}N_2$ was formed during the annealing of these films, but volume fractions of $Fe_{16}N_2$ were not larger than 36%. To increase volume fraction of $Fe_{16}N_2$, it is necessary to increase annealing temperature and duration, however the temperature should be lower than about 473 K to avoid decomposition of $Fe_{16}N_2$, and the duration is limited by escape of nitrogen from the surface.

To increase diffusivity of nitrogen atoms without increasing temperature, the ion implanted Fe films were annealed in magnetic field. Since the crystal structure of ferromagnetic material slightly deforms in magnetic field, diffusivity of nitrogen will also change in magnetic field. Experiments were performed using films implanted with 50 and 25 keV N_2^+ ions to doses of 5×10^{20} and 5×10^{20} ions/m^2, respectively. The surface of the specimen was coated with a 60nm thick Cu film to prevent escape of nitrogen. The magnetic field strength was 1.3T and the direction was parallel to the film surface. The annealing temperature was 443K and the duration was 3.6 ks. Volume fractions of $Fe_{16}N_2$ were estimated from X-ray diffraction patterns after the annealing. No distinct differences were found between the X-ray diffraction patterns of samples annealed in magnetic field and those of samples annealed without magnetic field.

Keywords : $Fe_{16}N_2$, ion implantation, annealing in magnetic field

70 Generalized Rules and Guide for Use of Fatigue Data

S.Nishijima, Center for Materials Science under Extreme Envronments
[April 1995 to March 1997[

NRIM has been conducting a series of project researches since 1975 to establish standard reference data on fundamental fatigue properties of typical Japanease materials for machines and structures. The program of work has been accomplished in March 1995 by the publication of 83 Data Sheets.

The present research aims at the presentation of generalized rules found in differnt fatigue properties that can be explained from view point of fundamental mechanisms of fatigue and, therefore, that are most important for adequate use of those fatigue data. It is, in other words, to offer a guide for understanding the change in properties of different materials and for applying the data to engineering requirements. Selected

Table 1 NRIM Fatigue Data Sheet Technical Document

No.	Subjects	Issued
1	Fundamental Fatigue Properties for Carbon, Cr and Cr-Mo Steels	1981
2	Fatigue Strength for Welded Joints of High Strength Steels	1983
3	Crack Growth in Arc-Welded Butt-Joints of High Strength Steels	1984
4	Strain Rate Effect in Low Cycle Fatigue of Alloys at High Temps.	1985
5	Fundamental Fatigue Properties of JIS Steels for Machine Structures	1989
6	High Cycle Fatigue of Alloy Steels at Elevated Temperatures	1990
7	High Cycle Fatigue Properties of Carburized Steels	1992
8	Fatigue Crack Propagation for Welded Joints of Structural Steels	1995
9	High Cycle Fatigue Properties of Hard Steels	1995
11	Low Cycle Fatigue Properties of Engineering Materials for High Temperature Components	1996
12	Fatigue Properties of Aluminum Alloys for Welded Structures	1996
13	Stress Ratio Effect on Fatigue Life of Butt Welded Steels for Pressure Vessels	1996
14	Fatigue Properties of Carbon and Low Alloy Steels	1996
15	Elastic moduli of Steels	1997
16	Intermediate Temperature Fatigue Test Method for Butt-Welded Steel Joints and Fatigue Properties	1997
17	Index Properties for Fatigue of Engineering Alloys	1997

data from related Data Sheets are collectively analyzed to be fit to theoretical models, for some cases, or additional experiments are performed to verify theories, for the other cases.

The effort to show the generalized rule on fatigue property data actually been continued in the previous Fatigue Data Sheet Project. The outputs have been published in Japanease as Fatigue Data Sheet Technical Documents as listed in Table 1.

Keywords : fatigue, steel and alloys, NRIM fatigue data sheets

71 Modeling and Evaluation of Advanced Materials - A coordinated Interlaboratory Research

S. Nishijima, Director of Center for Materials Science under Extreme Environments
[April 1992 to March 1996]

This work aimed at the development of models describing the behavior and properties of also advanced materials of high performance. It delete covers also the establishment of rationalized test methods for advanced materials, as a part of the international research cooperation known as Versailles Project on Advanced Materials and Standards (VAMAS).

(1) Intermetallic compounds

The effect of γ grain size and α_2 precipitates on creep rupture properties was studied at 1023-1373K using Ti-49at.%Al and Ti-51at.%Al which were extruded and heat treated. Ti-49at.%Al with a large γ grain size and the α_2 precipitates at grain boundaries is expected to exhibit good creep rupture properties as well as good room temperature ductility.

The fatigue strength of TiAl varied according to the volume fraction of the γ phase in the lamellar structure. A fatigue crack initiated inside of an isolated γ grain for the specimeus with lower γ volume fractions and the fatigue strength could be estimated from a simple rule- of- mixtures. When the volume fraction was higher, a fatigue crack initiated at the γ grain boundaries and the strength was independent of the volume fraction.

(2) Metal matrix composites (MMC)

The fatigue properties and mechanisms at room temperature were discussed for SiC/Ti-15-3 MMC. The fatigue crack initiation and propagation processed as follows: (i) initiation at the reaction layer, (ii) interfacial debonding between the reaction layer and outer carbon layer, (iii) cracking the outer carbon layer, (iv) interfacial debonding between the outer carbon layer and SiC fiber (v) SiC fiber cracking, (vi) matrix cracking. It is suggested that the reaction layer between the outer carbon layer and matrix promotes crack initiation because it is very brittle. For new composites coated with ductile materials such as Cu to avoid the reaction layer formation, the interfacial crack initiation was not observed. The crack propagation rate was much lower than delete when the fibers were not coated.

(3) Plane woven fabric continuous SiC fiber-reinforced SiC matrix composite (SiC/SiC)

The tensile properties and notch sensitivity of SiC/SiC composites have been studied at room temperature. The gross fracture stress of notched specimens showed notch insensitivity. The tensile stress-strain curves showed non-linear behavior associated with progressive matrix cracking and fiber pullout. The increase of a permanent strain caused the stress concentration between notches to be lower. Fracture mechanisms at elevated temperatures were also studied by flexure test. In order to improve mechanical properties of SiC/SiC, it is necessary to prepare fibers with surfaces which to have high resistance to the reaction between the matrix and oxygen as well as matrices with a smaller amount of voids.

(4) Development of Materials Characterization Technique with an X-ray Microbeam

A high-resolution X-ray tomographic technique has been developed with particular attention for use in advanced materials research. Images of element distribution in objects can be obtained from absorption contrast using two monochromatic x-ray beams which straddle its absorption edge. In this project we have attempted to utilize polychromatic beams of conventional x-ray generators other than monochromatic beams obtained with SR and monochromator, with radioisotopes or with fluorescence from secondary targets. We have extended this critical absorption technique to polychromatic x-ray with filter modulation. The elemental information was extracted by subtracting two tomographs obtained with different filters each other and also quasi-monochromatic features were provided by subtracting two radiographs with different filters each other before reconstruction process.

(5) International Project VAMAS

There are 16 Task Working Areas (TWA). NRIM offers international chairpersons for 3 TWAs respectively on superconducting materials, cryogenic structural materials and materials properties databases. Leading members are also from NRIM for 5 TWAs: method of evaluating MMC, method of creep crack growth test, method of low cycle fatigue test, method of surface chemical analysis, and method of mechanical evaluation of very thin films.

The second domestic round robin test on the critical current, Ic measurement method of Ag-sheathed Bi oxide superconductors was implemented with 6 participants. COV's of Ic among participants were approximately 5 %, and much reduced compared with those at the first round robin. The reduction of COV's

was accomplished by clarifying Ic degradation mechanisms due to thermal cycling and improving the specimen mounting method on G-10 substrate.

A series of international interlaboratory comparisons of both compression & shear tests for composite material G-10CR and tensile tests for solder were performed to promote the prestandardization program on material properties tests of GFRP composite materials and alloys at liquid helium temperature. The scatter of the compression strength decreased by the limitation of specimen geometry. The scatter of shear strength was small in the same testing procedure, but the span ratio and the edge shape at the groove in the guillotine type tests influenced delete the obtained shear strength. The amount of scatter of tensile properties of solder was similar to that of high strength materials.

Today computerized materials data systems are commonly developed in various R&D organizations worldwide. We want to share part of the data and knowledge stored on such systems. VAMAS Inventory of data evaluation models and methods have been compiled and will be published soon. Three organizations, NRIM-NMC (Japan), NIST (USA) and NPL (UK) shared the work for steels and alloys, ceramic materials and polymer materials, respectively. In addition to the conventional printed report a computerized version was also developed and put on our WWW server (www.nrim.go.jp:8080/vamas_twa10/).

The round robin tests of creep crack growth tests have been conducted on TiAl intermetallic compounds in Japanese group. The effect of specimen configuration, size, temperature, microstructures of materials and fracture mode on creep crack growth properties was clarified. Based on these results, we have proposed the guideline of standard creep crack growth test method for creep brittle alloys to the VAMAS technical working group.

SiO_2 layer on Si wafer formed by thermal oxidation was sputtered by 2kV argon ion under various sputtering conditions. The sputtering rate at each condition was obtained by the time used to sputter away SiO_2 layer. It is concluded that the sputtering rate is proportional to ion beam current. It is recommended to control sputtering rate by ion beam current. We also developed the software named "Common Data Processing System" to share these spectral data.

Tensile test method at room temperature for short fiber reinforced aluminum alloy matrix composites was proposed to ISO Technical Trend Assessment and discussed now in the advanced countries. Tensile tests at high temperatures were also performed and the result was discussed to clear the reason for large scattering of data between the participants and decided to retry to test under the same conditions among participants.

Keywords : advanced materials, property evaluation, modeling, VAMAS project, interlaboratory testing, TiAl, metal matrix composite, SiC/SiC composite, X-ray tomograph

Related papers:

H. Kitaguchi, K. Itoh, H. Wada and K. Togano, "Standardization of transport critical current measurement for HTSC; Study on reproducibility of the measurement", (to be presented at ICMC, Portland, August 1997, and) to be published in Adv. Cryog. Engin. Mater. 44(1998)

T. Ogata and D. Evans, "VAMAS Tests of Structural Materials on Aluminum Alloy and Composite Material at Cryogenic Temperatures", Advances in Cryogenic Engineering. Vol.42, p. 277(1996)

M. Tabuchi, A. Fuji, A. T. Yokobori, Jr. and K. Kubo, "Creep Crack Growth Test and Evaluation Method for High Temperature Brittle Alloys", Proc. of the 42th Japan National Symposium on Strength, Fracture and Fatigue, (1997), 37-44.

M. Yoshitake and K. Yoshihara, "Compositional and Chemical State Analysis of Layered Intermetallic Compounds by Line Scanning of Etched Crater with Scanning XPS", J. Surf. Anal., 3, 478(1997).

72 Long-term Creep-fatigue Properties of 316FR Stainless Steel for Fast Breeder Reactor

K.Yamaguchi, Failure Physics Division
[April 1995 to March 2000]

The 316FR stainless steel has been developed a candidate material for fast breeder reactor of twenty first century. For the structural design of components of the reactor,a simple and accurate analysis method for evaluating the low cycle fatigue life of the 316FR steel is required, because the main conponents of the reactor are subjected to cyclic thermal stresses. One of the objects in this research is to apply a parametric analysis method for time-dependent low cycle fatigue life data of the 316FR steels. This mehtod was developed by the authors and was successful to represent the time-dependence of the fatigue life for many kinds of conventional engineering heat resisting materials at the strain rate up to 10^{-5}/sec. Now the fatigue tests are running at 500, 550 and 600Å under the strain rates from10^{-2}/sec to 10^{-6}/sec.

Second object is to evaluate creep rupture and creep-fatigue properties for modified 316FR steels which is now developing by a steel maker and fabricators. So the testing data would be useful to the materials design. In the first half of this research the effects of the chemical composition such as C, N, Mn, P, Cr, Ni , Mo, Si and microstructure such as grain size on the

high temperature strength properties are examined. In the second half a development of prediction method of the creep-fatigue life is aimed by using the monotonic data such as creep rupture ductility.

This research is performed in collaboration with Mitubishi Heavy Industry Ltd., Nippon Steel Corp. and The Japan Atomic Power Co.

KeyWords : low cycle fatigue, fast breeder reactor, 316FR stainless steel

Related papers

Parameter Analysis for Time-Dependent Low-Cycle Fatigue Life, K.Yamaguchi and K.Kobayashi, Trans.ASME, J.Eng.Mater.Tech.,116(1994):479-482.

73 Remaining Life Prediction for Weldments in FBR based on Creep Damage Evaluation.

Y. Monma, Failure Physics Division
[April 1996 to March 2001]

Weldments in fast breeder reactor(FBR) such as pressure vessels and pipes for primary circuit are required to be used for long term under creep condition. The main purposes of this study are (1) to develop techniques quantifying the time-dependent, cumulative creep damage in the weldments for FBR, and to simulate the failure process of the weldments by finite element method(FEM) using a new model based on the continuum mechanics and high temperature failure physics. From these techniques, it is expected that the accuracy for the assessment of structural integrity of FBR is improved in the regular service mode as well as in the emergency mode. Further, through these, basis for the remaining life prediction techniques in the middle and end life periods of FBR will be established, which leads to the improvement of structural integrity of FBR.

The materials used are typical structural materials for FBR, 304 and 316 stainless steels, and a modified 9Cr-1Mo ferritic steel. The large scale welded joints (thickness:50mm) of each material are fabricated and creep tests on the specimens of base metal,of weld metal and of welded joint cut from the large scale welded joints are conducted.

The results obtained in this year are as follows;
(1) Using the crept 304 steel, the relations among ultrasonic echo response, change in density and creep properties were investigated. Positive correlations were recognized between creep rate and energy loss of ultrasonic propagation and between change in density and change in the ultrasonic velocity.
(2) In the large scale welded joints of 316 steel thick plates deposited using 16Cr-8Ni-2Mo wire, creep failure under higher applied stresses occurred at the base metal part. However, the failure under lower applied stresses occurred at the weld metal part.
(3) TEM micrographs showed that dislocaion density in the region near the surface of weld metal part in the welded joint is extremely lower than that in the region at the middle of the weld metal part (middle of the thickness). The fact indicates that creep tests of the large scale welded joints are needed and also that to understand the creep deformation behavior of the real structure, measurements of strain distribution in the through-thickness direction are needed.

Key words : creep damage, welded joint, stainless steel, microstructure, FEM

Related Paper

Creep Rupture Behavior and Creep Strain Distribution of Welded Joints of 304 Stainless Steel Thick Plates, H. Hongo, M. Yamazaki, T. Watanabe, J. Kinugawa and Y. Monma, J. Soc. Material Science JAPAN,45(1996): 1328-1333.

74 Evaluation of corrosion damage at atomic level

H. Masuda, Frontier structural material center
[April 1996 to March 1998]

This year the mechanism of indoor corrosion was studied by Kelvin force method (KFM). The indoor corrosion is believed to be occurred above the humidity of 60 %. We have done in-situ observation of proceeding of corrosion on pure iron at the humidity of 40 % by KFM. The simultaneous observations of topography and potential distribution showed (Figure 1) that corrosion on iron progressed even at the humidity of 40 % and the potential of corroding part is more positive than other part. The mechanism of indoor corrosion was found to be obeyed Evans' model.

Keywords : Corrosion, KFM, Potential

Related paper

Analysis of Corrosion Reaction on Metal Surface by KFM: H.Masuda, Hyomen Kagaku, Vol.18, No.2 (1997) 72-78

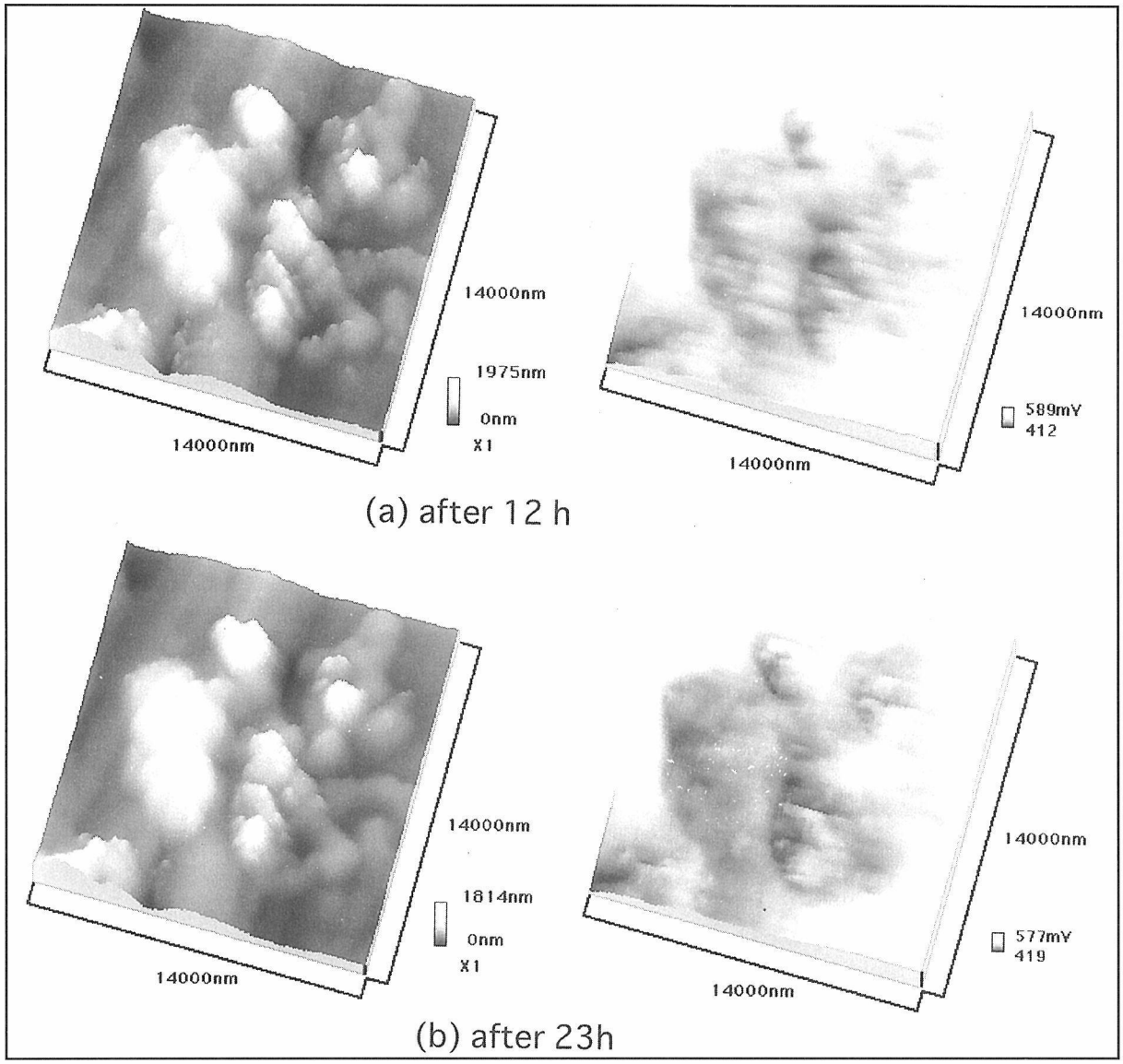

Fig. 1 topography and potential distribution on pure iron

75 Development of metal having environmental cleaning ability by photo catalysis reaction

H. Masuda, Frontier structural material center
[April 1996 to March 1998]

The small water droplets which adhere the material surface cause corrosion and photo catalytic reaction. So it is very important to study the behavior of water droplet. We used a graphite to obtain the atomically flat and clean surface and study the behavior of water droplet of nano meter scale by atomic force microscope (AFM) with non-contacting mode. A lot of nano scale water droplets were observed on the surface freshly created by peeling then pouring pure water and sprayed with cleaner gas (Figure 1). The water film was formed and the water droplets moved when the surface was slowly scanned under applying certain bias. Multi-layer water films and water droplets were observed on the surface freshly created then pouring pure water. These

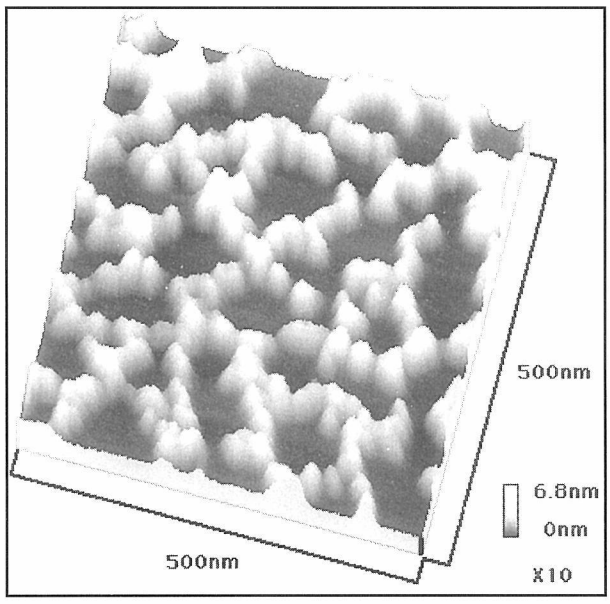

Fig. 1 Adsoped water on newly created graphite surface

water film and droplets show the different behavior of ordinary water droplets.

Keywords : water cluster, non-contact AFM

Related paper

Direct Observation of Water Droplet on Graphite by Non-contacting AFM: H. Masuda, J. Japan Inst. Metals, Vol. 61, No.2 (1997) 140-144

76 Fundamental Studies of the Microbial Reaction with Inorganic Compounds

A. Aoki, Mechanical Property Division
[April 1996 to March 1998]

Certain species of microorganism accelerate the aqueous oxidation of metals and inorganic compounds. These are called chemolithotrophic bacteria and have played an important role in mining to extract metal valuables from ore. Much effort has been done to clarify and control the potential usefulness of microbiology in this field. However, the interfacial process of the interaction between the microorganism and the inorganic compound is still not sufficiently understood.

To clarify this ambiguous point, as a first step, we are investigating the characteristic of the microbial oxidation of the inorganic compounds. Thiobaccilus ferroxidans, a typical microorganism of the chemolithotrophic type, is applied in this study. The results of our previous study showed the significant catalytic influence of the microorganism in the oxidation process of iron sulfide(FeS_2 and $Fe_{1-x}S$). With the purpose of obtaining further information for the characteristic of the microbial reaction, we are investigating the nature of the solid phase formed on the sulfide surface by using XRD, FTIR and SEM. As to $Fe_{1-x}S$, the oxidation products were jarosite $KFe_3(SO_4)_2(OH)_6$, schwertmannite $Fe_8O_8(OH)_6SO_4$ and elemental sulfur; however, in the chemical reaction these were oxyhydroxide FeOOH and elemental sulfur. By using SEM, the process of the oxidation was morphologically investigated and the difference of the oxidation mechanism was also clarified between the microbial and chemical reaction.

Furthermore, the corroded surface of the sulfide crystal will be investigated to understand the details of the early stage of the microbial reaction using SEM, EDX and AFM.

Keywords : chemolithotrophic bacteria, Thiobaccilus ferroxidans, interfacial process, redox potential, XRD, SEM, FTIR

77 Effect of the Interfacial Damage on Mechanical Properties for Ti-Based Matrix Composites

C. Masuda, Interface and Joining Division
[April 1996 to March 1999]

Silicon-carbide (SiC) fiber reinforced titanium alloy matrix composites are attractive for structural applications such as gas turbine engines, because of their high specific modulus and strength; and good stability at high temperature. There are many reports on the fabrication methods and mechanical properties of those composites. It is well known that the major problem during processing is fiber degradation due to the unavoidable nature of active reaction behavior between fiber and matrix, that is, the reaction layer thickness was related to the degradation of mechanical properties. The effect of the reaction layer thickness on the mechanical properties could not be deduced by the micromechanics, because the mechanical properties of reaction layer could not be obtained.

(I) Fatigue crack initiation and propagation mechanisms tested at room temperature are as follows; (1) initiation at the reaction layer, (2) interfacial debonding between the reaction layer and outer carbon layer, (3) cracking the outer carbon layer, (4) interfacial debonding between outer carbon layer and SiC fiber, (5) SiC fiber cracking, and (6) matrix cracking. Fatigue mechanisms tested at high temperature in vacuum was nearly the same as those tested at room temperature. The life duration for the formation of the debonding between outer carbon layer and SiC fiber was longer at high temperature than that at room temperature at the same stress level. It is suggested that the fatigue life at high temperature is higher than that at room temperature due to the formation of debonding.

(II) As revealed experimentally, the interface region plays an important role in damage initiation and propagation. To understanding this role, it is necessary to model and analyze the mechanical behavior of the interfacial region. Analytical and numerical results are obtained regarding 3 types of damage interaction between interface bonding/debonding and a crack in fiber-reinforced metals, namely, a circumferencial crack approaching a fiber-matrix interface, crack deflection and penetration at an interphase, 3-dimensional plasticity growth around a crack tip and interface. These results are summarized as follows: (1) bonding interface may hinder or accelerate the growth of an approaching crack depending on the combinations of fiber and matrix material properties, (2) properties and thicknesses of the interphase are very important to crack deflection and penetration conditions at interphase interfaces, (3) plasticity growth is likely to occur preferentially around an interface because of thermal stresses induced during composite processing, and this com-

plicates debonding conditions.

78 Fundamental Study for Electromagnetic Evaluation of Materials

I.Uetake, Failure Physics Division
[April 1994 to March 1997]

Flaws in structural materials remarkably decrease the materials strength. The detection of surface flaws and material degradation is essential to insure safety of important structures such as pressure vessels in a reactor and other large constructions. In this study fundamental phenomena regarding electromagnetic evaluation method were investigated.

With regard to alternating magnetic flux leakage testing method, we studied some suitable magnetization frequencies for flaws detection. The suitable frequency is found to depend on a flaw dimension. A high frequency is suitable for a shallow flaw while a low frequency for a deep flaw. This result is due to the phenomenon of skin effect of magnetic flux. The frequency dependency is obtained quantitatively. When a flaw depth is less than 0.5mm, suitable frequencies were found to be in the range of 5-20kHz, while a frequency between 2-5kHz is proper for a flaw of about 1mm in depth. As for a flaw more than 2mm in depth, the suitable frequency should be less than 2kHz. It is also revealed that the skin depth induced by a suitable magnetization frequency for examination reaches an extent of about 30% of the flaw depth.

Keywords : non-destructive evaluation, materials evaluation, electromagnetic method, magnetic flux leakage testing, surface flaw

79 Effect of Aging Degradation on Localized Corrosion of Structural Materials for Light Water Reactors

Y.Katada, Joining and Interface Research Station
[April 1996 to March 2000]

Almost thirty years have passed since nuclear power plants started for commercial operation in Japan. It is important, therefore, to secure the integrity of structural materials for long-term aged nuclear power plants.

As one of the nuclear safety researches the objectives of this research are to investigate the interaction between aged structural materials and environmentally assisted cracking (EAC) such as stress corrosion cracking and corrosion fatigue in high temperature water, and to restrain the enlargement of local damage in weld joints by using a laser technique.

1.Interaction between aged materials and EAC

In the case of evaluation of SCC susceptibility for hardened materials due to degradation, a slow strain rate test (SSRT) which has been commonly used may not be proper due to the inner stress of the strained materials being increased rapidly. In this study as an alternative method for SSRT a constant loading rate test (CLRT) was investigated and compared with data obtained by SSRT method. Material used in the study was sensitized stainless steel SUS304. As a result of comparison between the two methods in terms of stress-strain curve, two both data were quite consistent, which imply that the CLRT method was promising method for SCC susceptibility of hardened materials.

2.Restraint of enlargement of local damage by laser technique

Weld joints of structural components including weld metal and heat affected zone (HAZ) are considered to be more sensitive than base metal for aging degradation because of the discontinuous micro-structure, residual stress, segregation and so on. In this year, an electrochemical approach was conducted by using a micro vibration electrode of 10 μ m in diameter in order to investigate the local corrosion of weld joint of SUS304 steel in 3% NaCl solution. As a result it was found that the maximum potential gradient was observed at a few millimeters apart from the weld bond, where sensitized region was formed and corrosion pits were observed. Accordingly this electrochemical approach seemed to be a promising technique to evaluate the corrosion behavior of weld joints. The behavior of helium bubbles yielded at the melting spots of a specimen surface treated by laser beam was also investigated. It was found that the helium bubbles revealed under the condition of the amount of doped helium more than 20 appm.

Keywords : aging degradation, localized corrosion, light water reactor, weld joints, laser beam

80 NRIM Creep Data Sheet Project-V

H. Irie, Mechanical Properties Division
[April 1996 to March 2001]

The major objectives of the NRIM Creep Data Sheet Project are to accumulate long-term creep and rupture data up to more than 10^5 h data for heat resistant steels and alloys, which are produced in Japan, and to publish the data as a series of NRIM Data Sheets. This project has been continued since 1966 and the series of NRIM Data Sheets have been distributed not only in Japan but also in abroad. In fiscal year 1996, we published three sheets listed in Table.

In parallel with the testing and publication program,

Table Summary of NRIM Data Sheets in fiscal year 1996

Materials	Number	Issued
9Cr-1Mo-V-Nb steel tubes for boilers and heat exchangers and steel plates for boilers and pressure vessels	43	September 30,1996
Relaxation properties of 1Cr-0.5Mo-0. 25V and 12Cr-1Mo-1W-0.25V bolting steel for high temperature service	44	September 30,1996
Normalized and tempered 2.25Cr-1Mo Steel plates for boiler and pressure Vessels(SCMV 4 NT)	11B	March 31, 1997

we have made researches on long-term creep and rupture behavior of heat-resistant steels and alloys.

On the basis of inherent creep strength concept, for development of superior steels with respect to long-term creep strength it is much more important to obtain stable microstructure for long time rather than short term strength and to elongate the period when the strength goes down to the inherent one.

An analysis of creep behaviors of three kinds of heat treated 2.25Cr-1Mo ferritic steels with annealing, normalizing and tempering and quenching and tempered were carried out using the modified θ method and observation of microstructure. The creep rupture strength under high stress and short period strongly depended upon the heat treated structure. On the other hand dependency of strength on structure under low stress and long period was not considerable because of recovery of the microstructure.

Relationship between abnormal creep phenomenon and change in microstructure of SUS 316 steel was investigated. In the past, it was considered the abnormal creep behavior was due to formation of oxides in cracks. It was made clear that nitrogen in steel strongly effected this phenomenon through the fact that the σ phases in grain boundaries partly changed to nitrides and that nitrogen affected precipitated phases in grain.

Keywords : long-term creep, creep rupture, stress relaxation, microstructural evolution

81 Nanoscopic Materials Damage Evaluation

S. Matsuoka, Frontier Research Center for Structural Materials, Strength and Life Evaluation Research Station
[April 1994 to March 1996]

It is important to examine the materials damage in the vicinity of crack tip or grain boundary to improve the reliability of structural materials. Recently, the damage in small-size structural components has also become a subject of much concern as seen from an example of the stress or electro-migration for aluminum thin-film conductors in the technology of integrated circuits.

This research has been conducted to develop the materials damage evaluation technique in the nanoscopic region. First, topographic images of high magnification were obtained for various fracture surfaces and thin-film structure in semiconductor devices, using a scanning tunneling microscope (STM) and atomic force microscope (AFM). Secondly, a nano-indentation hardness tester has been developed which is based on AFM. Because of the imaging ability of the AFM, it is much easier to choose where a nano-indentation hardness test will be conducted and to measure size of indentations. Using this system, mechanical properties of various materials such as gold, steel, tungsten, molybdenum, silicon, and glass were investigated in less than 100 nm regions. Lastly, a finite element method (FEM) and molecular dynamics (MD) simulation was conducted for the nano-indentation process and agreed well with the experiments. It is concluded that both experimental and computational technique will be useful for a further work in this field.

Keywords : materials damage, nano-indentation, STM, AFM, FEM

82 Ductile versus Brittle Behavior of Structural Steels

S. Matsuoka, Frontier Research Center for Structural Materials, Strength and Life Evaluation Research Station
[April 1996 to March 1998]

Many steel structures were damaged on Hanshin-Awaji great earthquake of January 17, 1995. One typical example was the buckling of steel columns in highways and another was the brittle fracture of steel box columns in high-rise buildings. Such a difference in damage modes is closely related to the ductile versus brittle behavior of structural steels.

The collaborative project aimed at investigating the

damage of steel structures has been carried out by using a large-scale test bench in National Research Institute for Earth Science and Disaster Prevention. We have contributed to the project by the fracture analysis. That is, the fracture surfaces of large T-type specimens of SM490B steel have been examined by a scanning electron microscope. In parallel to this, we have been developing an advanced fracture analysis by scanning tunneling microscope and atomic force microscope.

Keywords : steels, earthquake, ductile fracture, brittle fracture, STM, AFM

83 Improvements of High Temperature Properties in Materials for High/Ultra-high Temperature Use

T.Tanabe, Environmental Performance Division
[April 1996 to March 1999]

1. Materials for high temperature use.

The purpose of the study in this year is to investigate the effect of microstructures on the creep rupture properties of Ti-49at.%Al at the temperatures ranging from 1023 to 1373K in a high purity helium environment. The results were partly compard with those of a Ni-based superalloy, Inconel 617.

Hot extruded Ti-48.8at.%Al bars with 18mm diameter from a skull melted ingot were used in this investigation. They were heat treated in α , $\alpha + \gamma$ or γ region to obtain 7 kinds of microstructures ; (1) fully lamellar structure(colony size:435 μ m), (2) γ grains containing α_2 platelet in each grain(γ grain size:150 μ m),(3) γ grains with α_2 at their boundries(γ grain size:50 μ m), (4)-(7)equiaxed γ grains with diameter of 60,140,220 and 350 μ m. Round tensile creep specimens having the gauge section of 6mm diameter and 25mm length were machined from the heat treated bars. Creep rupture tests of 7 kinds of specimens were conducted under constant load condition at 1273K, while at 1023,1173 and 1373,only 2 kinds of specimens with γ grain sizes of 60 and 350 μ m were tested. The tests were carried out in a high purity helium gas environment(O_2<2vpm, dew point<200K).

The results obtained from the creep rupture tests of the TiAl alloy were as follows;

1)Creep rupture properties of the alloy depend on its microstructures.

The equiaxed coarse-grained material is stronger than the fine grained ones.

2)The alloy has good ruture ductility due to dynamic recrystallization occurring during the tertiary creep stage.

3)Rupture strength of the alloy wit γ grain size of 350 μ m are comparable to those of a Ni-based superalloy, Inconel 617 at 1023-1373K.

2.Mterials for ultra-high temperature use.

The purpose of the study in this year is to investigate how the thermophysical and thermomechanical properties of graphite as plasma facing materials in fusion reactor devices are improved by replacing some part of graphite with TiC and TiB_2. A variety of graphite powders containing different fractions of Ti powders and B powders was synthesized by powder metallurgical process and hot-pressed uniaxially in vacuum at 2273K under the stress of 50MPa to produce disks with 30-60mm diameter. The obtained disks were all composed only of graphite, TiC and TiB_2. They then cut to the disks with about 5-10mm thickness, which were used as thermal shock test specimens. The coupons of 3x5x36mm^3 for 3 point bend test were also machined from the disks.

The thermal shock tests were performed using an electron beam thermal shock apparatus. The beam diameter was 13mm and the maximum heat duration was 13 sec. In order to analyze the phenomena including erosion, vaporization and so on, the specimen current and temperature were continuously monitored during the test. Further, 3 point bending tests were carried out at room temperature.

The results obtained up to now were as follows;

1) The synthesized graphites were composed of graphite, TiC and TiB2 only.
2) There existed incubation time for erosin(Tie) in graphite containing TiC and TiB2. Tie increased with increase of TiC contents.
3) Graphite containing 45wt%TiC and 5wt%TiB_2 showed the best thermal shock resistance among them and is superior to IG110U,a commercial base isotropic graphite. Its bend strength is almost the same as that of IG110U.

key words : intermetallic compound, TiAl, creep, plasma facing material, thermal shock resitance.

Related papers.

Effect of Microstructure on the Creep Rupture Properties of Ti-49at.%Al Alloy, I. Mutoh, Y. Kawano, T. Tanabe and M. Nakamura, Proc.1996 Annual meeting of JSME/MMD,(1996):31-32

Thermal Shock Resistance of Graphites Containing TiC and TiB_2, M. Fujitsuka, I. Mutoh, T. Tanabe and T. Shikama, Proc. 13th Korea-Japan Seminar on New Ceramics,(1996):299-303

84 Study on Transient Behaviour of Deformation and Fracture at the Elevated Temperature

F. Abe, Strength and Life Evaluation Research Station

[April 1994 to March 1997]

Complex transient behaviour which does not obey classical form, is observed on both creep deformation and creep crack growth of heat resistant steels and alloys at the elevated temperature. Creep deformation and creep crack growth are influenced by the changes in microstructure of materials. This study aims at giving a role of microstructural change in the transient behaviour at the elevated temperature.

Complex creep deformation which reveals multiple local minima in creep rate is observed for engineering heat resistant steels. Complex creep deformation of SUS316 stainless steel is related to both changes in carbide precipitation strengthening effect and precipitation of Laves phase. Complex creep deformation of carbon steel are annihilated by the ageing prior to creep test.

In order to develop an accurate creep crack growth evaluation method, creep crack growth properties of several heat resisting materials have been investigated. The estimation method of transient creep deformation due to crack growth was discussed by using the large CT specimen for Cr-Mo steel. The evaluation method of crack growth life was indicated by taking the microstructural change into account for creep brittle Ni-base superalloys. The creep crack growth rate could be predicted more accurately by taking the creep fracture mechanism and creep damage density ahead of the crack tip into account.

Keywords : creep deformation, inherent creep strength, creep crack growth, microstructural change

Related Papers

Creep Deformation Assessment of Aged Steel based on Inherent Creep Strength Concept, K. Kimura, H. Kushima, F. Abe and K. Yagi, Proc. Int. Symp. Materials Ageing and Component Life Extension, 1(1995): 459-468.

Effect of Thermal Ageing on the Complex Creep Deformation Behaviour of Carbon Steel and It's Inherent Creep Strength, K. Kimura, H. Kushima, F. Abe and K. Yagi, Tetsu-to-Hagane (J. Iron Steel Inst.), 82(1996): 713-718 (in Japanese).

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The Influence of Fracture Mechanisms on The Creep Crack Growth Behavior of 316 Stainless Steel.

K. Yagi, M. Tabuchi, K. Kubo, J. J. Kim and Y. H. Huh, Engineering Fracture Mechanics (in press).

85 Characterization of Creep-Damaged Microstructure of Stainless Steels by Computer Aided Quantitative Metallography

Fujio Abe, Strength and Life Evaluation Research Station

[April 1995 to March 1998]

In the NRIN Creep Data Sheet Project, we have published the long-term creep and rupture data containing 10^5 h data for principal heat resisting steels and alloys. We are now making a plan publishing the Microstructure Data Sheets for type 304, 316, 321 and 347 stainless steels, as a parallel series of the Creep Data Sheets. The Microstructure Data Sheets will contain not only the microstructural changes during creep but also relating data such as time-temperature-precipitation diagrams, distributions of precipitates and creep voids, and creep damage parameters. The purpose of the present research is to characterize the creep-damaged microstructure for the stainless steels. The obtained results will be incorporated in the Microstructure Data Sheets.

Penning discharge micro-sputtering techniques using Ar ions were applied for etching a variety of heat resisting steels and alloys ; 1Cr-1Mo-0.25V ferritic and 316H austenitic steels and Ni-base Inconel 713C. All the materials exhibited clear and reproducible microstructures at the sputtering conditions of 1 kV anode voltage and 10 mA discharge current, irrespective of different chemical compositions and different history of the materials. The grain boundaries, creep voids and precipitates such as carbides and σ - phase were characterized using optical and scanning electron microscopes. It is concluded that the present method requires less skill and the resultant microstructures were insensitive to sputtering conditions.

Keywords : creep-damaged microstructure, computer aided metallography, stainless steels

Related papers

The Characterization of Microstructures of Stainless Steels for Evaluation of Long-Term Creep Properties, H. Tanaka, M. Murata, F. Abe and K. Yagi, Proc. 15th International CODATA Conference, (1996): 163-64.

A New Preparation Techniques of Surface to Microstructural Examination of Heat Resisting Steels and Alloys by Using Penning Discharge Micro-Sputtering, H. Tanaka, E. Nishikawa, F. Abe, K. Yagi and T. Sugita, Tetsu-to-Hagane, 83 (1997): 263-68.

86 Stability of Tetragonal Phase and its Effect on High-

Temperature Mechanical Properties of Zirconia

Fujio Abe, Strength and Life Evaluation Research Station

[April 1996 to March 1999]

Transformation-toughened zirconia (ZrO_2) base ceramics have been of much interest in recent years, because of the considerable potential of these ceramics in structural applications. The author and co-workers already showed for a ZrO2-9.7mol%MgO that the tetragonal (t) to monoclinic (m) phase transformation occurred in the two distinct stages at 1000-1200 K and 400-700 K on cooling. In this research, the origin of the low-temperature stage at 400-700 K was examined in detail by means of dilatometry and transmission electron microscopy. The low-temperature stage was caused by martensitic transformation of the ellipsoidal particles of the t-phase in the cubic phase matrix. During cyclic heating and cooling, the formation of microcracks at the interface between the t-phase particles and matrix as well as the diffusion-controlled coarsening of the t-phase particles occurred, which accelerated the transformation in the low-temperature stage on cooling and increased the transformation temperature.

Several kinds of ideas on toughness improvement in zirconia base ceramics have been proposed. In this research, the effect of volume fraction of the m-phase on fracture toughness KIC and hardness has been investigated for ZrO_2-9.7mol%MgO and ZrO_2-2mol%Y_2O_3. The volume fraction of the m-phase was changed from 0 to 0.987 for the ZrO_2- 9.7mol%MgO by various heat treatments. The KIC and hardness were measured by indentation method over the temperature range between room temperature and 1173 K. In the ZrO_2- 9.7mol%MgO, the KIC increased with increasing the volume fraction of the m-phase, while the hardness decreased. The KIC was inversely proportional to the hardness similar as that of metallic materials. The ZrO_2-2mol%Y_2O_3, consisting of 100% t-phase, scarcely showed the cracking at the end of indentation, indicating higher KIC.

Keywords : partially stabilized zirconia, transformation, fracture toughness

Related papers :

Tetragonal to monoclinic transformation and microstructural evolution in ZrO2-9.7mol% MgO during cyclic heating and cooling, F. Abe, S. Muneki and K. Yagi, J. Mat. Sci., 32 (1997): 513-22.

Relationship between hardness and fracture toughness of a 9.7mol% MgO partially stabilized zirconia alloy, S. Muneki, F. Abe and H. Irie, Proc. 13th Korea-Japan Seminar on New Ceramics, November 20-22, 1996, Pohang, Korea, pp.216-20.

Effect of specimen size on ductile to brittle transition behavior of martensitic 9Cr steels after various heat treatments, F. Abe and H. Kayano, J. Nucl. Mater., 232 (1996): 44-51.

87 Studies on Evaluation of Durability of Organic Coatings for Construction Materials.

Toshiaki Kodama, Team of Director of Special Research

[April 1996 to March 1999]

Facing the transition of Japan toward a more mature and aged society in the next century to come, it has become commonly accepted that infrastructures be used for prolonged period time with the minimum expense of maintenance cost. Along this line we started a new research on the durability of organic coatings used for the protection of infrastructures. In the research included are environmental factors influencing the durability of polymers, mechanism of adherence of paint to metal, detection of structural changes of polymers in the degradation process and monitoring of metallic corrosion at metal / paint interface.

In order to evaluate outdoor degradation of paint films, exposure tests using a variety of paints coated on aluminum plates were carried out at three exposure sites in Japan and two sites in Thailand. Aluminum plate treated in a phosphate-chromate solution was used as a substrate material; It was selected for the purpose of avoiding the effect of metallic corrosion. Environmental factors such as average temperature, relative humidity and a cumulative of ultraviolet (UV) irradiation were measured monthly. A standard polyethylene film was exposed on the same exposure rack. The generation of carbonyl in polyethylene was detected by infrared spectroscopy (FTIR) after exposure of one month, and it was used as a parameter indicative of UV intensity and environmental aggressiveness to polymers. For evaluating the degradation of polymer coatings we used several techniques such as gloss retention (optical method), contact angle measurement, and detection of structural changes by means of FTIR. Close correlation was observed among data of different techniques. Observed data of paint films were plotted and evaluated as a function of carbonyl index. Among paint coatings studied, degradation progressed in the order of epoxy > alkyd > polyurethane > fluorocarbon, while red pigments showed better resistance than white ones.

Keywords : durability of organic coating, environmental factors

88 Development of Porous Ceramics Impregnated with Ionic Conductive Materials

H. Nakamura, Team of Diretor of Special Research
[April 1995 to March 1998]

Recently, research works on solid high ionic conductor have been mainly focused on oxygen ion conductor such as ZrO_2, proton conductorsuch as $SrCeO_3$ and sodium ion conductor such as β -Al_2O_3.

One of the reasons why no other solid electrolytes have been developed as high ionic conductor is that cracks caused by grain growth or voids along grain boundaries tend to initiate in the sintered materials prepared for the solid electrolyte.

The objective of this study is to develop a new type of solid electrolyte in which grain boundaries or voids are extremely decreased by impregnation of liquid compounds (M_2O-B_2O_3- M_2SO_4, M= Li,Na,K)into the porous oxides.Subsequently,its electrical properties will be evaluated by the measurement of electrical conductivity, the investigation of polarizing behavior and the determination of charge carrier.Furthermore,the thermal characteristics will also be evaluated using various thermal anlysis methods.

Keywords : ionic conductor, solid electrolyte, charge carrier, electrical conductivity

89 Relationship between Fatigue Crack Propagation and Cyclic Deformation of Small Specimens

A.Ohta, Environmental Performance Division
[April 1994 to March 1997]

On the higher stress ratio condition, the fatigue crack propagation properties become unique among different stress ratios, because the fatigue crack closure does not occur. In these conditions, the effective range of stress intensity factor coincide with the range of applied stress intensity factor. It is defined that the fatigue crack propagation properties obtained at these conditions are fundamental fatigue crack propagation properties.

The fundamental fatigue crack propagation properties have been obtained for steels, aluminum alloy and titanium alloy by the two sets of maximum load hold tests in which the minimum load increased accompanied by the crack extension.

The fundamental fatigue crack propagation properties become superior with the increase of the Young's modulus when they are plotted on log da/dn versus log Δ K. The distance along log Δ K is proportional to the Young's modulus.

Therefore, the da/dn are repotted on log da/dn versus log Δ K/E. It is confirmed that the relationship between fatigue crack propagation rate and the strain intensity factor become unique in spite of the material having different Young's modulus. This means that the fatigue crack propagation properties of material is controlled by the strain range

Keywords : fatigue crack, fatigue properties, cyclic deformation

90 Evaluation of Fatigue Strength of Welded Joints in Synthetic Sea Water by the New Testing Method

A.Ohta, N.Suzuki and Y.Maeda, Environmental Performance Division
[April 1995 to March 1998]

The fatigue strength of box weld in synthetic sea water has been investigated at the condition where the maximum stress has kept to be the yield strength of material while the minimum stress changed with the stress range. This test condition is considered to simulate the high tensile residual stress near the box weld in large beam specimens. The fatigue tests have been performed in 40℃ synthetic sea water in which the air bubbles were blown in.

Large beams are selected as specimen which contains yield strength magnitude tensile residual stress. The stress ratio for the beam test is 0.1.

The test frequency is 0.17 Hz. The fatigue crack initiation life is automatically detected by using the crack opening displacement signal from eddy current detector. The reduction of stress range at the detection of fatigue crack initiation as the 5 percent drop of the crack opening displacement amplitude from the initial value of it creates the clear beach mark on the fracture surface.

It is confirmed in the short fatigue life region that the results of both tests give the similar life. That is, the evaluation of fatigue life by the $\sigma_{max} = \sigma_y$ tests on small specimen is equivalent to the fatigue strength of welded beams which really contain yield strength magnitude tensile residual stress.

Keywords : fatigue welded Joint, yield strength, steel

Related Paper

Fatigue Strength Evaluation of Welded Joints Considering High Tensile Residual Stresses, A.Ohta, Y.Maeda, T.Mawari, S.Nishijilna and H.Nakamura, Int.J.Fatigue 8(1986)147-150.

91 Chemical Analysis of Organotin in Marine Environ-

mental Samples

H.Okochi, Team of Director of Special Research
[April 1991 to March 1996]

The optimum conditions for the solid phase extraction (SPE) of organotin compounds (OTC) extracts from sediments or shellfishes have been systematically investigated. As for the acidity, the recoveries of TBT and TPT were better in 0.12 and 0.24 M HCl, while those of DBT and DPT were better in more acidic solutions, such as 1.20 and 1.44 M. Accordingly, a two steps SPE procedure was established. The recovery of OTC decreased with the increase of the drying time by Ar flow in SPE. At evaporating procedure, in the case of 100 % MeOH the recoveries remarkably lowered while 85 % MeOH gave better recoveries. The temperature control at 35℃ is very important at the evaporating step under Ar flow.

The optimum conditions for extracting OTC from sediments have been investigated. When extracting with MeOH, the addition of HCl was not adequate for TBT, while it gave better recoveries for DBT. Accordingly, a two steps extracting procedure was selected. The extracting procedure of OTC from sediments is as follows ; Take 0.5 g or 1.0 g of a sediment(dry) into an extracting vessel. Add 20 ml of MeOH and shake for 60min and then centrifuge for 45 min using a high speed cooling centrifuge (-5℃, 1200 rpm). Then add 20 ml of MeOH and 2 ml of 12 M HCl and repeat the same procedure. The recovery tests were TBT 97 %, TPT 97 %, DBT 84 % and DPT 93 %.

The SPE of TBT and TPT in shellfish samples has been also established. The interference of the lipid in OTC extracts could be removed without a clean-up procedure by changing it to micelle with TDS and SPE at pH 10 (0.25. The recoveries of TBT and TPT were ca.96 - 97 %. The NIES CRM No.11 "fish tissue" was analyzed by the established procedure, that is, SPE / MLC / ICP-MS. The determined values of TBT and DBT showed good agreement with the certified reference values.

Atomic absorption spectrometry (AAS) of OTC was comparatively studied among FAAS, hydride generation AAS and GF-AAS without changing to inorganic tin. In GF-AAS the stabilized temperature platform furnace technique was applied.

Keywords : ICP-MS, organotin, solid phase extraction, GF-AAS, sea water, sediments, shellfish

92 Using Experimental and Theoretical Depth Resolution Functions for High Accuracy Depth Profiling

S. Hofmann, Special Research
[January 1996 to March 1999]

In order to determine the composition of thin films with a precision of the order of one atomic monolayer, depth profiling by ion sputtering in combination with a surface analysis method has to be optimized. The deviation between the in depth distribution of composition and the measured profile is usually characterized by a certain depth resolution z (16%-84%), which describes the apparent broadening of an atomically sharp interface [1,2]. If information about the concentration distribution within this length z is required, the relation responsible for this blurring, i.e. the depth resolution function, has to be known. The exact knowledge of the depth resolution function is the key to a correct transformation of the measured sputter profile into the original in-depth distribution of composition with monolayer accuracy. Recently, a theoretical description of the depth resolution function useful for both SIMS and AES profiling was developed starting with a simple theoretical model for the mixing influence and extending it to include surface roughness and information depth (called MRI-model from Mixing - Roughness - Information Depth [3]). These three fundamental parameters, given by the mixing length w, Gaussian roughness and exponential information decay length also include the observed shift of the measured interface with respect to the original interface [4]. High accuracy profile reconstruction can be achieved by careful experimental determination and theoretical modelling of the depth resolution function. A test of the extent of nonlinear behaviour with increasing concentration is proposed by comparing the shape of the depth profile of a monolayer (delta layer) (AlAs in GaAs) with that of a differentiated profile at the interface of GaAs/AlAs.

According to our design, a reference sample was fabricated by Sony Research Laboratories with the following structure: (in atomic monolayers): 41 GaAs/1 AlAs/43 GaAs/36 AlAs/GaAs.../Si(111), or in nm units (1ML=0.28 nm): 11.5/0.28/12.1/10.1/... It was depth profiled in a Fisons Microlab 300 F instrument using 0.5 keV Ar+ ions at 68 deg. incidence angle and recording the Auger peak-to-peak-heights of the high energy Al signal emitted from the rotating sample surface.

Tests of the validity of the above concepts and of the occurence of nonlinear behaviour with respect to concentration were carried out with the above described layer structure containing one monolayer of AlAs and another AlAs layer of about 36 monolayers, both surrounded by about 40 monolayers of GaAs. The results for the high energy Al peak sputtering profile were compared with the theoretically determined depth resolution function by the MRI-model [5] and show good agreement with the parameters atomic mixing length

w= 1.0 nm, surface roughness =0.6 nm and Auger electron escape depth = 1.16 nm. The obtained accuracy is about 1-2 monolayers. Some discrepancies, particularly on the second interface, require more studies. Further improvements will be made by using lower ion energy and more heavy ions (Xe) and lower electron escape depths to obtain one monolayer accuracy or below. With this accuracy, other effects like original interface width or differences in concentration distribution can be easily and quantitatively detected. This will greatly enhance our capabilities in characterization of thin films and interfaces with atomic monolayer accuracy, which is an important contribution to our COE-project on nanostructures.

Keywords : Depth Profiling, Depth Resolution, Thin Films, Auger Electron Spectroscopy, Ion-Solid Interaction, Atomic Monolayer

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93 Development of High-Tc Oxide Superconducting Wires and Tapes

H. Kumakura, 1st Research Group
[April 1995 to March 1999]

Bi-based oxide superconductors are very promising for high-field superconducting magnets. In the case of $Bi_2Sr_2CaCu_2Ox$(Bi-2212), well grain oriented microstructure is easily obtained by slow solidification from a temperature of ~ 880℃, where the Bi-2212 composition is in a partially molten state. This grain alignment significantly increases the critical current density, Jc, in magnetic fields. We have developed Bi-2212 superconducting tapes with high Jc by applying this melt-solidification method to Bi-2212/Ag composites prepared by a coating method. Oxygen partial pressure, PO_2, during the melt-solidification is one of key parameters that influence the microstructure and superconducting properties of Bi-2212/Ag tapes. Tc and c-axis parameter decreased with increasing PO_2, indicating that the samples were in the over-doped state. The heat treatment temperature which gave highest Jc increased with increasing PO_2. This was explained by the increase of the solidification temperature of Bi-2212 with PO_2. Both the resistive Jc and inter-grain Jc estimated from the dc magnetization increased with increasing PO_2 when the samples were melt-solidified under an optimum condition for each PO_2. Amplitude dependence of ac susceptibility suggested that the coupling of Bi-2212 grains was improved with increasing PO_2. Irreversibility field B_{irr} below ~ 60K was also enhanced with increasing PO_2. Low temperature annealing under high PO_2 after the melt-solidification also increased the oxygen content in Bi-2212 and hence B_{irr}. However, the annealing did not change the amplitude dependence of ac susceptibility, and transport Jc at 4.2K was almost independent of the annealing. The results indicate that the increase of Jc at 4.2K with increasing PO_2 during melt-solidification is primarily attributed to the improvement of the grain coupling.

Keywords : Bi-based oxide, oxygen partial pressure, grain coupling

Related Paper

Effect of high oxygen partial pressure heat treatment on the superconducting properties of $Bi_2Sr_2CaCu_2O_x$/Ag tapes, H. Kumakura, H. Kitaguchi, K. Togano and N. Sugiyama, J. Appl. Phys. 80(1996)5162-5168.

94 Study on Anomalous Magneto-Resistance for Bi-2212/Ag Composite Superconducting Tape

H. Kitaguchi, H. Kumakura, H. Fujii and K. Togano, 1st Research Group, 2nd Sub-Group
[National Research Institute for Metals: April 1996 to March 1997]

This study is performed to give a clear explanation for the anomalous dependence of magnetoresistance of Bi-2212/Ag composite tape superconductor on the direction of magnetic fields. The author found that the tape shows anomalous distribution of electric potential in magnetic fields perpendicular to the tape surface. This distribution can not be explained in terms of the usual current transfer from silver to Bi-2212. In order to establish a model which gives a clear explanation, I-V characteristics and the distribution of electric potential were measured for Bi-2212/Ag composite tape in magnetic fields of different directions. An anomalous distribution of electric potential was observed especially in the magnetic fields perpendicular to the tape surface. Along the current direction in the vicinity of both ends of superconducting oxide layer, large voltage was measured. However, zero voltage was monitored in the middle area of samples. Large voltage was also measured across the current direction around both ends.

These voltages are proportional to the value of applied current below Ic and to the magnetic field component perpendicular to the tape surface. In order to give an explanation to this anomalous distribution of electric potential, we establish a model based on the Hall effect in silver substrate and current transfer from silver to superconductor. This model explains experimental results well.

Anomalous Current Distribution in Bi-2212/Ag Composite Superconducting Tape caused by Hall Effect, H. Kitaguchi, H. Kumakura, K. Togano : Cryogenics 36, 351-357(96)

Keywords : superconductor, current transfer, Hall effect, magnetoresistance

95 Characterization and Application of Superconducting Thin Films Synthesized by Atomic layer-by-layer and Epitaxial Growth Methods

T. Hatano, 1st Research Group
[April 1995 to March 2000]

In order to keep better surface morphology and in-plane orientation, thin films of $Bi_2Sr_2CaCu_2O_y$(2212) and $Bi_2Sr_2Ca_2Cu_3O_y$(2223) were deposited by pulsed laser ablation onto MgO(100) and $LaAlO_3$(100) single crystal substrates at temperature about 50-100K lower than the decomposition point of 2212 or 2223 phases. To get the effect of *ex-situ* annealing on the *in-plane* orientation and T_c, the films were accurately post-annealed in oxygen potential controlled furnace. The results showed that the T_c of the annealing films reached to 75-80K depending on the *in-situ* substrate temperature before annealing. The annealed 2223 films showed homogeneous intergrowth mixing of 2212 phase by 10-20% and showed the T_c at 100-105K.

We found that the *in-plane* orientation of the 2212 films on MgO(100) substrates depends on the substrate temperature during deposition. The film deposited at higher substrate temperature have either 2212[100]// MgO[100] $\pm 11^\circ$ and 2212[100]//MgO[110] relation or 2212[100]//MgO[100] $\pm 11^\circ$. While the film deposited at lower substrate temperature showed only the 2212[100]//MgO[100] $\pm 11^\circ$ epitaxial relation. After annealing, the *in-plane* direction completely rotated by 45°, and only the 2212[110]//MgO[100] epitaxial relation became observable. On the other hand, from the films deposited on $LaAlO_3$ substrate, only the strong 2212[100]//$LaAlO_3$[100] *in-plane* epitaxial relation was observed independent of the substrate temperature and post-annealing.

Keywords : high-T_c superconductor, thin film, annealing, pulsed laser ablation, high-T_c thin film, $Bi_2Sr_2Ca_{(n-1)}Cu_nO_y$, intergrowth, lattice mismatch, surface morphology, *in-plane* orientation

96 Study on Atomic Scale Engineering of High Performance Films

T. Hatano, 1st Research Group
[April 1994 to March 1997]

The atomic scale engineering has been developed in the semiconductor field. The high performance materials are designed based on the prediction of quantum mechanics which rules the properties of materials in this dimension. The aim of this study is to extend this technique to the other materials such as high T_c superconducting oxides and molecular crystals. However, in these materials, the crystal structure is much more complicated and especially the molecules show a quite different characteristics in course of the film synthesis. The molecular beam epitaxy technique under ultra high vacuum and the electron diffraction technique are not the most suitable technique for these materials. To solve these problems, synthesis technique having higher selectivity and reactivity is necessary and other probe should be installed instead of the electron beam. As a new fabrication technique for these materials, we introduce a reactive deposition technique equipped with x-ray analysis system in the total reflection region.

The total reflection x-ray analysis technique was applied to the artificially layered $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ films prepared by sequential sputter deposition. In the $\theta/2\theta$ diffraction spectrum at the grazing incidence, the oscillatory pattern can be observed which is due to the interference of the reflected x-ray from the surface and interface of the film. Such patterns are also observed between the neighboring (002) Bragg peaks. The number of oscillation exactly correspond to the number of units in the film. The thickness of the films can be analyzed in the scale of one atomic layer by this analysis. The atomically layer-by-layer synthesis can be established by this analysis. The superstructure of $Bi_2Sr_2Ca_{n-1}$-Cu_nO_{2n+4} system was successfully synthesized by applying these results to the deposition conditions. The total reflection x-ray analysis technique can be extended in another two ways. One is the in-plane x-ray diffraction which reveals the surface structure and the texture of the films. The other is the fluorescent x-ray analysis of the surface. The composition of the film surface can be analyzed.

Keyword : thin films, atomic scale characterization, x-ray total reflection

Related papers

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Structure and properties of $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ films prepared by sequential sputter deposition, T. Hatano and K. Nakamura, in "Bismuth based High Temperature Super-conductors" ed. H. Maeda and K. Togano, Marcel Dekker, New York, 1996, pp545.

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97 In situ TEM Observation and Structural Analysis of High Tc Superconductors for Fusion Reactor at Low Temperatures

T. Kimoto, 1st Research Group
[April 1994 to March 1999]

Few principles for the development of high Tc superconductors for fusion reactors have been found because the mechanism of their superconduction has not been understood so far. The image resolution limit of a transmitted electron microscope (TEM) is extremely low at low temperature because of specimen drift due to the unstable temperature control and specimen vibration due to the evaporation of liquid helium. The first objective of our research is to improve the resolution limit of TEM imaging at low temperatures in order to observe O and Cu atoms in high Tc superconductors near or above Tc where abnormal lattice distortion occurs which could reveal the mechanisms of their superconduction. The other objective is to develop the method to examine the long-range order (LRO) parameter in the microscopic region of high Tc superconductors and to develop the analytical method to measure the abnormal lattice distortion from the convergent beam electron diffraction (CBED) pattern.

It was found that a short-time exposed imaging improved TEM resolution at low temperatures to some extent, but that the reduction of S/N ratio due to the short-time exposure makes the image unclear. In 1996, we developed for the first time a new imaging system for TEM to solve this problem as a first step by combining the high-speed CCD camera system, whose minimum exposure time is 50 nano sec), developed in 1994 and the computer system (composed of the computer ; sun SPARCstation20) for image processing developed in 1995. By using this system, we are able to take short-time exposed TEM images sequentially at a speed of 5 images/sec. The pixel number of the CCD device is 1018×1000 and its dynamic range is 10 bit, and maximum number of images (2.1MB memory) which can be input sequentially into the main memory of the computer is about 160.

When the specimen drifts or vibrates at low temperatures, there are image-shifts between the short-time exposed TEM images which are taken sequentially with the developed new imaging system. The second main achievement in 1996 is to develop a software for image processing, which calculates the distance and direction of the image-shift between images, shift these images to be adjust with each other and finally add these shifted images to get a clearer image by increasing S/N ratio. The effect of this special image processing, which was called "shift and addition of images", was verified by taking lattice images from a drifting or vibrating (Bi, Pb)$_2Sr_2Ca_2Cu_3O_{10}$ (Bi2223) specimen at low temperatures. It was verified that we can take a clear lattice image from the drifting Bi2223 specimen by using the new imaging system and the developed image processing of "shift and addition of images". It was also verified that a clear lattice image was taken from the vibrating specimen by using them, if the vibration of the specimen is not hard.

In 1996, we started to develop a new type of electron source by using a CW laser, which could produces high intensity electron beam for TEM. For this purpose, we constructed the device to produce a CW Ar ion laser and developed a vacuum chamber to examine the fundamental technical problem such as conversing of a laser beam, cooling of the area irradiated by a laser beam with the Peltier cooling method, and so on. We also developed the method to determine a long-rang order parameter of high Tc superconductors and started to develop the method to measure an abnormal lattice distortion from the convergent beam electron diffraction (CBED) method.

98 Computational Analysis of Mechanical Properties and Structural Design of Materials Taking Microstructures into Account for Nuclear Power Plants.

H. Shiraishi, 2nd Research Group
[April 1994 to March 1999]

Damages such as void and helium bubble are formed by neutron irradiation and mechanical properties are degraded for materials used in reactor core. Large deformation finite element method program was produced for evaluation of so-called grain boundary helium bubble embrittlement in previous year and the effects of bubble size, bubble density and work hardening exponent were surveyed. The extension of this program to more general void lattice is being done and the effect of void lattice aspect ratio is reported in the present paper. The calculational procedures and results were reported elsewhere.[1,2]

The aspect ratio is defined as Ly/Lx where Ly and Lx mean the y and x direction void interval. Here, Lx is fixed to be 1.0 μ m. With decrease of void aspect ratio from 5.0 to 0.2, total elongation moderately decreased from 20.24% to 12.11%. The tensile strengths lowered from 590.0Mpa to 567.5Mpa. All curves were coincident with each other till plastic instability points. This result is understandable because it is considered that the mutual interaction between the neighboring two parts becomes smaller and each part behaves more independently as the aspect ratio approaches to 0.0.

At first, a deformation band grew in upper slant direction. In the example of aspect ratio 1.0 and conventional strain of 12.22%, strain concentration was limited fairly small region around void. The plastic instability was attained at the value of 13.41% for conventional strain and 584.3Mpa for tensile strength. When the conventional strain was 14.91%, the strain concentration area expanded along the line between two voids. The strong strain localized narrow band was formed in about 20 degree inclined direction to x axis in which the equivalent logarithmic strain was more than 2.0. This formation of strain localized band was observed in all aspect ratio values from 5.0 to 0.2. In the grain boundary bubble cases reported previously, the void aspect ratio was from several to several tens and much higher than in the present case. It was clarified that the total elongations reduced from several tens percent to the values slightly over than ten percent by the mechanism of strain localization, irrespective of aspect ratio values. This calculation assumed continuum theory and plane stress condition.

Keywords : FEM simulation, Stress/strain curve, void lattice, strain localization

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99 Real-Time Investigation on Surface Reactions and Defect Growth Processes under Irradiation

M. Kitajima, 2nd research Group
[April 1994 to March 1999]

Reaction dynamics of material surfaces under irradiation of ions, plasma, molecular beams and light has been investigated by developing fast analytical techniques that enable real-time observation of dynamic processes. Resonance enhanced multiphoton ionization (REMPI) measurements have been carried out to obtain the internal-state distributions of GeO molecules desorbing from Ge surfaces in reaction with O_2 beam. We have extended our multiphoton ionizational (MPI) work to the reactive scattering of O_2 on carbon surface. The MPI result gives a clue for further development of the study on surface chemical reaction dynamics. We have also studied on carbon cluster generated with laser-ablation. Real-time ellipsometry is successfully applied to the study of erosion of tungsten with neon plasma. The reflectance on material surface is being measured with femtosecond time-resolution using optical-heterodyne method.

Hydrogen molecule in crystalline silicon is investigated in detail using Raman spectroscopy. Less (no) Raman signal of H_2 in microcrystalline (amorphous) silicon suggests that H_2 is trapped in non-defective, well-ordered crystalline silicon. Annealing experiments are also carried out to examine the thermal stability of hydrogen molecule in silicon.

Keywords : irradiation, surface reaction dynamics, growth process of surface defect , real-time observation

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100 Study on the Deformation and Fracture of Materials under Irradiation

J. Nagakawa, 2nd Research Group
[April 1993 to March 1998]

Changes in microstructure and mechanical properties induced by the irradiation with energetic particles are critically important for the structural materials of nuclear application. They significantly influence the endurance of structural components. In this research project, deformation and fracture of materials, as well

as the point defect kinetics and microstructure development, under irradiation have been studied experimentally and theoretically. The following is one of the results obtained during the fiscal year 1996.

The radiation-induced swelling (equiaxial expansion of the material) caused by high energy neutrons has been recognized as one of the most serious problems for nuclear reactor materials. It has been generally considered that the cold working suppresses the swelling. Recently, however, it was reported that the cold working did not always reduce the swelling of Fe-Cr-Ni alloys irradiated in the fast reactor at 793-873 K but it rather increased swelling at higher temperatures (> about 800 K). 180 keV proton irradiation to 10 dpa at temperatures 773 to 873 K was carried out for Fe-15Cr-20Ni alloy preinjected with 10 appm in both solution-annealed (SA) and 20% cold-worked (CW) conditions. There was no significant difference in dislocation microstructure between SA and CW specimens at 773-873 K. No radiation-induced precipitate was observed in both. This indicates that the dislocations induced by cold-working have been fully recovered. Void microstructure was also similar in both specimens at 773 K, while larger and fewer voids were observed in CW specimens at 823-873 K. The effect of cold working on void microstructure appears to be less important at lower temperature (773 K) where the preinjected He atoms are more stable. The amount of swelling was similar in both SA and CW specimens at all the irradiation temperatures. The cold working seems to be less effective in controlling the swelling as compared with the preinjected He at least in the present experimental condition.

Keywords : irradiation, radiation damage, point defects, deformation

101 Influence of Nuclear Transmutations on Low Activation Structural Materials for Fusion Reactor Application

N. Yamamoto, 2nd Research Group
[April 1996 to March 2001]

In order to contribute to the development of irradiation - resistant structural materials with low - activation characteristics for future nuclear fusion systems, such as prototype and DEMO reactors, the influence of transmutational elements on the integrity of those materials has been investigated by using ion irradiation with a cyclotron. The particular concern is paid to estimate effects of gas elements (helium and hydrogen) on mechanical properties, since these species are liable to induce severe grain boundary embtittlement.

For this purpose, a high vacuum creep testing device for post irradiaion experiments was introduced to NRIM cyclotron facility and the methodology of testing has been established through tests on unirradiated samples. In the meanwhile, preliminary modeling of the phenomenon has been proceeded by utilizing data previously obtained on a variety of helium implanted Fe-Ni-Cr austenitic steels and alloys throughout wide ranges of experimental parameters (applied stress, helium concentration and helium implantation rate). The comparison between helium bubble size distribution at grain boundaries and morphology of creep ruptured surfaces proved a very good agreement between the pore spacing on intergranularly fractured faces and the average distance of grain boundary bubbles with over critcal sizes. This fact indicates that the intercrystalline fracture of helium containing materials would be most probably triggered by unstable growth of intergranular bubbles exceeding the critical size. In the next step, this model will be critically inspected and refined through data from low activation ferritic steels which will be examined this fiscal year.

Keywords : helium embrittlement, hydrogen embrittlement, ferritic steel, vanadium alloy

Related Paper

Bubble Microstructure and its Correlation to Creep Rupture Properties in Austenitic Alloys Implanted with Helium, N. Yamamoto, J. Nagakawa, Y. Murase and H. Shiraishi, Proc. 4th Japan-China Symp. on Materials for Advanced Energy Systems and Fission and Fusion Engineering, Sapporo, 1996, p. p. 161-166.

102 Research on Utilization Technique of "Data-Free-Way" system for Nuclear Materials

M. Fujita, 2nd Research group
[April 1995 to March 2001]

Material information system with a huge database and an effective computer aided tools are required for the alloy design or selection of advanced nuclear materials. However, there are limitations in storing numerous material properties into the database and developing a variety of tools in one research institute. Thus, under corporation among National Research Institute for Metals (NRIM), Japan Atomic Energy Research Institute (JAERI), and Power Reactor and Nuclear Furl Development Corporation (PNC) with different special fields, the pilot distributed database system for development of nuclear materials called " Data-Free-Way" was built since 1990. The project on the second stage was started in 1995 under the collaboration among NRIM, JAERI, PNC, and Japan Science and Technology Corporation (JST). The aim of

this project is to store the data in the database and to develop useful computer utilized tool for data analysis and simulation codes for various phenomena under irradiation environments.

The method of the connection among the database of each organization in "Data-Free-Way" system changed from private line to the Internet. Using tools used in the Internet, users can easily refer to any necessary information by accessing to the four databases with WWW browser without taking account of the database site in which the required data exit.

The trials of the remote experiment using the technique used in "Data-Free-Way" system are carried out among NRIM, JST, and Michigan State University (MSU) on the test line of 45MB wide band. We have succeeded in accessing from NRIM server to JST and MSU client and transferring real time movies for various micro-structure in several alloys. The results suggest that both researches among the countries will share the material information and the results of the experiment on the real time base and discuss about the results simultaneously.

Keyword : Data-Free-Way, distributed database, advanced nuclear materials, data share, remote experiment

103 Synthesis and Characterization of Isotopically Controlled Materials

T.Noda, Nuclear Materials Group
[April 1997 to March 2001]

Materials composed of isotopically selected elements are expected to indicate improved physical and chemical properties as well as to solve the essential problems in nuclear properties such as induced activity, He embrittlement, and compositional change caused by reactions with energetic particles. The objectives of the program are (1) R&D of the isotopically controlled infrared laser utilized for isotope concentration covering a wide wavenumber range, (2) synthesis and characterization of isotopically controlled single crystals such as B, Si and SiC etc., (3)development of processes for ceramics and their composites with advanced nuclear and mechanical properties, and (4) computational simulation to evaluate and design the isotopically controlled materials for nuclear applications.

An isotopically controlled pulse CO_2 laser system is being developed. The operating gas is changed from natural $^{12}C^{16}O_2$ to isotopic CO_2 such as $^{12}C^{16}O^{18}O$ and $^{12}C^{18}O_2$. A mixture of CO_2, He and N_2 gases is circulated through a catalyst to recover CO_2 from CO.

The isotopically enriched silicon flake was produced by plasma CVD at 623-723K from SiF_4 and Si_2F_6 gas after isotope separation. Single crystals of isotopically controlled silicon, SiC and boron are being grown using FZ and a sublimation process. The crystallography of the crystals are characterized and physical properties are examined.

Chemical vapor infiltration process to obtain SiC composites with a high purity and a stability against heating and neutron or ion irradiation is being developed using crystalline SiC fiber preforms.

The simulation code, IRAC, calculating transmutation was improved by combining with a neutron transport calculation code and introducing multi-step reactions to predict more precisely the transmutation of materials.

Keywords : isotopically controlled materials, physical properties, isotope separation, crystal growth, Silicon, Silicon carbide, Boron

Related papers

Enrichment of Silicon Isotopes by Infrared Laser Irradiation, H.Suzuki, H.Araki and T.Noda, J.Japan Inst. Metals, 61,(1997)145-152.

Si Isotope Separation for Low Activation, T.Noda, H.Suzuki,H.Araki and M.Okada, J. Nucl. Mater., 233-237, (1996)1496-1499.

Evaluation of Transmutation and Low Induced Radioactivity and Requirements for Candidate Structural Materials, T. Noda, J. Nucl. Mater., 233-237, (1996)1475-1480.

104 High Performance Materials for Severe Environments-I (Microstructure and Properties of Intermetallic Compounds with High Specific Strength)

M. Nakamura, 3rd Research Group
[April 1990 - March 1997]

Light-weight, heat-resisting intermetallic compound TiAl is a candidate for a structural use in severe environments i. e. a space plane etc., and the knowledge of various properties of TiAl base alloys is required for a practical use in such environments. In this research program, the microstructure control and the mechanical properties are systematically studied for TiAl base and the fundamental methods to control microstructure which gives the optimum properties for a practical use to materials are discussed.

A very fine lamellar structure composed of α(α_2) and γ phase plates was observed distant from the prior α grain boundaries as well as the massively transformed γ phase near the grain boundaries in the water quenched Ti-48Al alloy which was heated in the α phase field for a long period of time and had large

prior α grains. We studied the microstructure change with heat treatment after quenching for the very fine lamellar structure. When it was aged in a temperature range of 900 to 1100℃at a rapid heating rate in a salt bath, the very fine lamellae changed to fine γ grains, of which region grew into the fine lamellar region from the boundaries between the massively transformed region and the fine lamellar regions. The boundaries between the massively transformed region and the fine γ grain region were hardly distinguished, and the latter region contained various crystal defects like dislocation, antiphase domain boundary and planar defect as well as the former region. From these experimental results, we suggested the following process as a formation process of the massively transformed γ region.

$\alpha \rightarrow (\alpha(\alpha_2) + \gamma)$ lamellar structure $\rightarrow$ massively transformed γ

The microstructure of the Ti-48at%Al alloy quenched from a high temperature α phase field was analyzed using high-resolution electron microscopy. We, in particular, discussed the microstructure of the antiphase domain boundaries (APB) formed in the massively transformed γ region in detail. We observed that both sides of APB consisting of thin rotated domain (RD) exhibited lattice distortion besides the antiphase relationship. This distortion was concluded to be caused by the difference between the lattice constants in the $L1_0$ tetragonal structure of TiAl. We also observed a simple APB, which did not consist of RD, and it is suggested that the thin RD is formed from the simple APB by short range diffusion of the constituent elements.

TiAl base alloys containing Sb from 3 to 6 at.% were tested in tension at 1100℃. The cast alloys contained the Sb-rich phase particulates, and exhibited the tensile strength as low as 120MPa. The cast alloys hardly had elongation even at 1100℃. Thus, the alloys were unidirectionally solidified using a floating zone furnace. The fibrous Sb-rich phase in the central part of the directional solidified specimen was obtained, and the directionally solidified alloys were HIPed at 1200℃. The HIPed alloys containing 4 at% Sb exhibited the tensile strength of higher than 240 MPa, and the elongation of above 10%.

The oxidation behavior at 900℃ and tensile properties at room temperature were studied for aluminized TiAl base alloys. The surface coating by aluminizing decreased the elongation at room temperature, although it improved oxidation resistance at 900℃ remarkably.

keyword : intermetallic compound, TiAl, microstructure control, high-resolution electron microscopy, Sb, high temperature strength, oxidation resistance

Related papers:

T. Kumagai, E. Abe, M. Takeyama and M. Nakamura, "Reaction Process of $\alpha \rightarrow \gamma$ Massively Transformation in Ti-Rich TiAl Alloy", Mat. Res. Soc. Symp. Proc., Vol. 364, pp. 181-186, (1995).

E. Abe and M. Nakamura, "The structure of antiphase domain boundaries in massively transformed γ -TiAl studied by high-resolution electron microscopy", Philo. Mag. Lett., vol. 75, pp. 65-73 (1997).

K. Hashimoto, M. Nobuki, H. Doi, E. Abe and M. Nakamura, "High Temperature Strength and Room temperature Ductility of TiAl Base Alloys with Antimony", Proc. Int. Symp. on Gamma Titanium Aluminides, ed. by Y-W. Kim, R. Wagner, and M. Yamaguchi, TMS, pp. 761-770, (1995).

105 Mesoscopic Structure Control and Properties of Intermetallic Compounds

M. Nobuki, 3rd Research Group

[April 1996 to March 1997]

Fine grain structure consisting of α_2, γ and B2 type intermetallic phases in Ti-Al-Cr cast alloys were prepared by a thermomechanical processing. The phase equilibria of these alloys were investigated by EPMA after heat treatment over a long period of time. The chemical composition was shifted to Ti-rich side from the conventional equilibrium phase diagram in which three phases of α_2, γ and B2 type intermetallic compounds coexist in the same volume fraction at 1270K.

Some preliminary works are being carried out for an $L1_2$ type Al_3Ti base alloy which was indust-skull melted and casted to a graphite mold. In the cast ingot of about 11kg, some macro cracks and complicated dendritic structure containing granular or plate like precipitates were observed. To change the microstructure, the cast material was multi- step forged isothermally, Then the microstructure exhibited the matrix consisted of equiaxed grains of about 70 μ m in diameter and a slight amount of cavities and granular or plate like precipitates of less than 10 μ m.

$L1_2$ type Al_3Ti base quaternary alloys Ti-Al-9mol%(Cr,Mn)-X(Ag,Ga,Sn,Pd,Ta,W) were prepared by arc melting and the brittleness of these alloys was examined by stamping with a die at room temperature. As a result, three kinds of alloys did not crack.

A, single crystal-like sample of Al_3Ti base alloy containing 8mol% Cr was prepared by unidirectionaly solidification with an optical zone refining device. Small amounts of cavities and Al-rich and Ti-rich phases were observed in the alloy. The Ti-rich phases were observed to be coherent with the matrix.

Fine α_2/γ lamellar samples of a TiAl base alloy were prepared by rapidly quenching. The fine lamellar structure changes to a fine γ grain structure by the

subsequent aging. The γ grains tend to show a preferred orientation in each lamellar colony under particular aging conditions. These results suggest that the fine γ grained sample showing the preferred orientation can be prepared by the heat treatments (rapidly quenching and aging) for the cast materials controlled by the unidirectional solidification method.

We have investigated a crystallization process of a Ti-48at.%Al amorphous thin film using high-resolution electron microscopy. In the early stage of crystallization during isothermal aging at 800K, a new phase was found to be formed as well as the disordered α phase.

EDS analysis showed that significant compositional differences do not exist between the phases, suggesting that the early stage of crystallization has been progressed with being composition invariant.

Keywords : intermetallic compounds, microstructure, phase transformation, processing, mechanical properties, TEM analysis

106 Heterogeneous Structure Design for High Plasticity and Wide Applicability of Simple System Alloys

K. Nagai, 3rd Research Group (now Frontier Research Center for Structural Materials)
[April 1996 to March 1997]

Mechanical properties can be generally controlled by changing microstructures. In the present study, a high density and fine dispersion of second phase in the matrix is designed using a new thermo-mechanical processing on Al-Si cast alloys.

When the second phase is undeformable, the plastic work causes the cracking of the second phase. If the plastic work is too much, the growth of the second phase crack leads to the sample fracture. With a soft matrix, a certain amount of plastic work only breaks the second phases. Succeeding annealing can heal the microcracks or voids and soften the matrix again. Several times combination of the plastic work and annealing makes the high density and fine dispersion of the second phases.

The Al-Si alloy is a typical cast material. The heavy cold-work introduces sever cracking in the eutectic or hyper-eutectic Al-Si cast materials. Therefore, the materials are well-known as undeformable materials. However, the matrix is a ductile Al-rich solution, and undeformable particles, i.e. large primary Si crystals and needle-like small eutectic Si crystals, are dispersed in the matrix.

By a given amount of cold-working, the primary Si crystals crack into a few pieces and decrease the size, and the eutectic Si crystals also break. The voids formed by the cracking are healed up by an annealing. A series of these treatments results in a fine microstructure. The mean size of large Si particles is decreased from 50 μ m to less than 10 μ m. The sample with refined structure shows the excellent plasticity as a 99% reduction in section area is performed both in cold sheet rolling and in wire drawing. Both the tensile strength and the elongation are higher than twice those of the as-cast material.

The similar idea is applicable to the Al-Si alloys with impurity elements such as Fe and Cu.

O.Umezawa and K. Nagai, Trans. Mater. Res. Soc. Jap, 20(1996),p.20.

K. Nagai and O. Umezawa, Japanese Contract 8-152424(1996).

keyword : cold-working, fine microstructure, high plasticity, high strength

107 Development of Advanced Shape Memory Thin Films by Sputtering

A. Ishida, 3 rd Research Group
[April 1996]

Since 1993, we have been successful in showing that sputter-deposited thin films of Ti-Ni posssess perfect shape memory effect, perfect superelasticity and two way shape memory effect. However, these phenomena take place in elastic deformation region. For practical use of shape memory thin films, it is also important to clarify the mechanical properties such as yield stress and elongation. In this study we therefore applied stress above yield stress to the thin films and measure their stress-strain curves.

For the measurement of stress-strain curves, Ti-Ni thin films with two kinds of compositions, Ti-50at.%Ni and Ti-51.5at.%Ni, were prepared by sputtering. After sputtering Ti-50at.%Ni thin films were annealed at 773K for 1h to form a Ti-Ni single- phase structure, while Ti-51.5at.%Ni thin films were solution-treated at 973K for 1h and age-treated at 773K for 1h to produce distribution of fine Ti_3Ni_4 precipitates in Ti-Ni grains. The measurement of stress-strain curves was carried out at a constant strain rate of 6.7×10^{-5} s^{-1} at temperatures between 223K and 473K. The specimen size was 400 μ m $\times$ 5mm (gauge portion) $\times$ 6 μ m (film thickness).

The tensile tests revealed that a Ti-51.5at.%Ni thin film exhibits a yield stress of 1.5GPa. This value is larger than that in bulk specimens, being attributable to a small grain size in the thin film. On the other hand, a Ti-51.5at.%Ni thin film was found to exhibit a yield stress of 1GPa and elongation of about 40%. This elongation is almost comparable to that in bulk specimens. These results show that the sputter-deposited thin films

possess good mechanical properties as well as good shape memory effect, superelasticity and two-way shape memory effect, and suggest that sputter-deposited thin films can be used as microactuators.

Keywords : micromachine, shape memory, Ti-Ni, Sputtering

Related paper

Stress-strain curves of sputtered thin films of Ti-Ni; A. Ishida, A. Takei and M. Sato, Thin Solid Films, 281(1996): 337-339.

108 Fabrication and Fundamental Studies on the Materials with Mesoscopic and Aperiodic Structure

A.P. Tsai, 3rd Research Group
[April 1996 to March 1997]

A texture consisting of nanoscale Pb-particles homogeneously dispersed within an amorphous matrix has been obtained in a melt-quenched alloy with composition A175Cu15V10-20wt%Pb. High resolution transmission electron microscopic observations revealed that the nanoscale Pb particles were isolated within the amorphous matrix. The nanoscale Pb particles showed broader superconducting transition under an applied field of 200e and smaller critical field (H,) at 5K, in comparison with those in bulk Pb. The amorphous matrix could be converted into the icosahedral quasicrystal phase with the same composition by heating to 720K. X-ray and transmission electron microscopic examinations revealed that the particles had spherical shape in the specimens within amorphous as well as with the quasicrystalline matrix. The melting behavior of these particles was investigated using a differential scanning calorimeter, and the results obtained thereby indicated that the melting points of the Pb-particles embedded in the matrix material were lower than the equilibrium melting point.

This work was partly performed in collaboration with Professor K. Chattopadhyay Indian Science of Institute.

Keywords : amorphous phase, nanoscale, lead particles

109 Studies on Viscoelasticity and Structure of Supercooled Liquid in Amorphous Alloys

A.P. Tsai, 3rd Research Group
[April 1996 to March 1997]

Structure of $Pt_{60}Ni_{15}P_{25}$ amorphous alloy on supercooled-liquid states have been studied from a bulk sample by means of small angle neutron scattering in real time in isothermal state. In-situ measurements of small angle neutron scattering from a bulk amorphous alloy $Pt_{60}Ni_{15}P_{25}$ Were carried out in the momentum transfer range of $0.008<Q<0.08$ Al as a function of time during isothermal annealing at the temperature of 483K which was just below the glass transition temperature ,T_g. (T_g=488K) The small angle neutron scattering intensities initially increase with time and reach saturation values. The small angle neutron scattering intensity of the glass cooled down to room temperature after the annealing increases at the every momentum transfer value observed. The small angle neutron scattering intensities follow a Q^{-4} dependence and have no peak structure in the momentum transfer range observed. There is no trace of phase separation in nanoscale evidenced in the present study. To realize the inherent nature of the glass transition for the amorphous alloy a complemental study on viscoelasticity is in progress.

Keywords : supercooled liquid, small angle neutron scattering

110 Atom probe microanalysis of advanced metallic materials

K. Hono, Materials Physics Division
[April 1996 to March 2001]

Properties of metallic materials are controlled by microstructures. Conventional structural materials such as steels, aluminum alloys, intermetallics and superalloys are all strengthened by controlling microstructures making use of solid→solid phase transformations. The scale of the microstructures of these materials are becoming smaller and smaller, and many microstuructures in modern metallic materials are in a subnanometer scale. Thus, microstructural characterizations of metallic materials in less than a nanometer scale resolution is essential for understanding the mechanisms of various novel properties in materials. This project aims to obtain better understandings to the underlying mechanisms of mechanical and magnetic properties of metallic materials by characterizing microstructures of advanced materials. For this purpose, we employ the atom probe field ion microscope (APFIM) technique which is capable of analyzing local chemical compositions of alloying elements with an atomic resolution. Unlike other analytical techniques, it can detect all elements including light atoms such as Li, Be, B, C, N and O which often make key roles in controlling materials properties. By making use of a three dimensional atom probe (3DAP), it is possible to map out elemen-

tal distributions in alloys in a three dimensional real space with almost an atomic resolution. For complementary microstructural characterization, conventional transmission electron microscopy (TEM) and high resolution electron microscopy (HREM) techniques are also employed. Our research interests include nanocrystalline magnetic materials (Fig. 1), magnetic thin films, amorphous and nanocomposite alloys, microstructures and phase transformations in steels and aluminum alloys and other industrial metallic materials.

Part of this project is conducted in collaboration with the Frontier Structural Materials Center. This project is partly supported by New Energy and Industrial Technology Development Organization (NEDO) International Joint Research Grant. The most up-to-date reports of this project can be found in the following WWW site:

http://inaba.nrim.go.jp/apfim

Keywords : atom probe, APFIM, nanostructure, microstructure, phase transformations

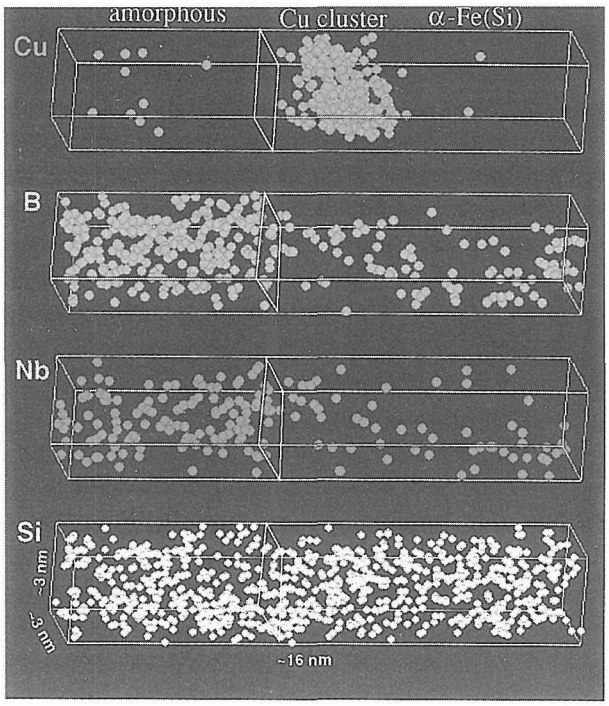

Fig. 1 Three dimensional atom probe elemental map near a Cu precipitate in a rapidly solidified Fe-13.5Si-9B-3Nb-1Cu alloy (FINEMET) annealed at 550 ℃ for 10 min. This data show nanocrystalline α -Fe particle is adjacent to a Cu precipitate which is believed to work as a heterogeneous nucleation site for the α -Fe primary crystal.

111 Research and Development of High Temperature Ti_2AlNb Titanium Aluminide-Based Composites

M.Hagiwara, Third Research Group
[April 1997 to March 2001]

Titanium alloys and titanium aluminides are of great technological interest because of their low density and high temperature strength. One main drawback of these titanium-related materials for structural application is that the service temperature is limited to 760 ℃ due to a degradation of tensile strength, creep resistance, thermal stability and environmental resistance. Moreover, they exhibit lower stiffness and abration-related properties than nickel-based alloy. However, for future aircraft design, there are still demands for advanced lightweight alloys having superior combinations of mechanical properties in the temperature range up to 1,000 ℃.

The fabrication of titanium-based metal matrix composites (MMC's) reinforced with ceramic particulate creates considerable potential for improvement in various properties. These particulate MMC's have isotropic characteristics and can be processed more cheaply using the conventional net shape technologies.

Recently, a new class of titanium aluminide based on the ordered orthorhombic phase Ti_2AlNb has been developed. Because of their attractive combinations of room temperature ductility and elevated temperature strength compared to Ti_3Al (α_2) and TiAl (γ), Ti_2AlNb can be considered as a potential matrix alloy for particulate composites.

The objective of this research program is to produce Ti_2AlNb-based MMC's reinforced with both relatively large-sized ceramic particulate such as TiC and TiB and very fine oxide particulate such as Er_2O_3 using advanced P/M processes, and creep and fatigue properties will be evaluated with emphasis on relating microstructural characteristics to these high temperature mechanical properties.

Keywords : titanium aluminide, particulate-reinforced, composites, powder metallurgy, mechanical properties

Related Papers

1. Microstructures and Mechanical Properties of Blended Elemental Powder Metallurgy Ti_2AlNb Intermetallics, S.Emura, J.H.Liu, M.Hagiwara, Y.Kawabe and A.Okada, Titanium '95 edited by P.A.Blenkinsop, W.J.Evans and H.M. Flower, The Institute of Materials, (1996): 404-410

112 Microstructure Control and Properties of Intermetallic Compounds in Ti-Al System

M. Nobuki, 3rd Research Group
[April 1997 to March 2001]

Microstructure has been controlled by thermomechanical processing for $L1_2$ type Ti-Al-Cr

cast alloy, and tensile test has been carried out at elevated temperatures. The proof stress of 160 MPa and the fracture strain of 23% have been obtained at 1070K and at a strain rate of I x $10^{-4}s^{-1}$. A severe influence of strain rate on the fracture strain was detected at high temperatures. It will related to the formation of the precipitates and coarsening of the cavities during hot deformation.

To examine the effect of additive element, Mn and Cr, on microstructure of $L1_2$ type $(Al,Mn)_3Ti$ and $(Al,Cr)_3Ti$ alloys, the microstructures of these alloys with changing the additive amount from 9 to 14 at % have been examined by SEM and X-ray diffraction after the heat-treatment of 1450K -604.8ks. A11 of the alloys have exhibited $L1_2$ single phase microstructure. However, fine voids or $A1_2O_3$ precipitates smaller than 1 μ m have been observed in the grain interior of the matrix. The lattice parameter change of the alloys due to Cr addition was larger than those in the alloys with Mn in the range of 9-14 at%.

The structure determination of a new phase which is formed in the early stage of crystallization of Ti-48at.%AI alloy is in progress, using computer simulation of high-resolution structure image and electron diffraction analysis. TEM observation on a feathery-type microstructure in a Cr added TiAl-based alloy is also in progress. Based on the results, the possible α / γ phase transformation mechanisms in the TiAl-based alloy will be discussed.

Keywords : intermetallic compounds, microstructure, phase transformation, processing, mechanical properties, TEM analysis

113 Magneto-optical Study with The NRIM Hybrid Magnet

K.Takehana M.Oshikiri and G.Kido 4th Group

A hybrid magnet system, which consists of a large superconducting magnet and a high power resistive magnet, has been built to generate steady high magnetic fields up to 40 T at National Research Institute for Metals (NRIM) [1]. Recently, the world highest steady field of a 36.5 T, which was calculated from the coil constants obtained by ESR measurements as described below, as recorded in September 1 995. A far-infrared region is quite important for an optical study in a high field range since the Zeeman energy for the field of O - 30 T corresponds to O - 40 cm~1 when the effective g-value equals to 2. We have constructed a Fourier-transform far-infrared spectrometer for using with the hybrid magnet in order to measure the electron spin resonance, the cyclotron resonance and the mechanism of low-energy excitations in high magnetic fields. A lamellargrating interferometer was employed to obtain an interferogram in the low energy region below 40 cm^{-1} instead of the Michelson-type interferometer which is commonly used in the infrared region. The spectrometer was examined by measuring the ESR signals of the spin-Peierls cuprate $CuGeO_3$ with a superconducting magnet and it was found that the effective g-value takes different value for the uniform, the spin-Peierls and the magnetic phases [2]. The spectrometer was also employed to calibrate the coil constants of the hybrid magnet by ESR measurements, since the NMR magnetometer seems not to be effective for our hybrid magnet due to a relatively large ripple and fluctuation (10^{-4}) of our power supply for the resistive magnet, in spite of the usefulness of the NMR magnetometer to determine a exact value of the magnetic field.

An experimental setup for the magneto-optical measurements in far-infrared (FIR) region is shown in Fig.1. A high pressure mercury lamp is used as an FIR light source. Diverging light from the end of a light pipe A is collimated by a large parabolic mirror. The parallel light from the mirror is reflected by the lamellar grating which introduces the optical path difference g = 2d, where d is a displacement of the movable grating from the fixed one. As the maximum displacement is 25 mm in our system, a theoretical resolution of the interferometer is 0.1 cm^{-1}. The light from the grating is focused on the entrance of another pipe B by the same parabolic mirror. The lamellar grating is made of aluminum, and it has a grating constant of 20 mm and a total area of 200 × 200 mm^2. A movable grating is mounted on a translational stage which is driven by a stepping motor. A Si bolometer at 3He temperature and an InSb bolometer at 4.2 K are prepared as a detector of the spectrometer. A light chopper is located on the way of the light pipe A, whose chopping frequency of

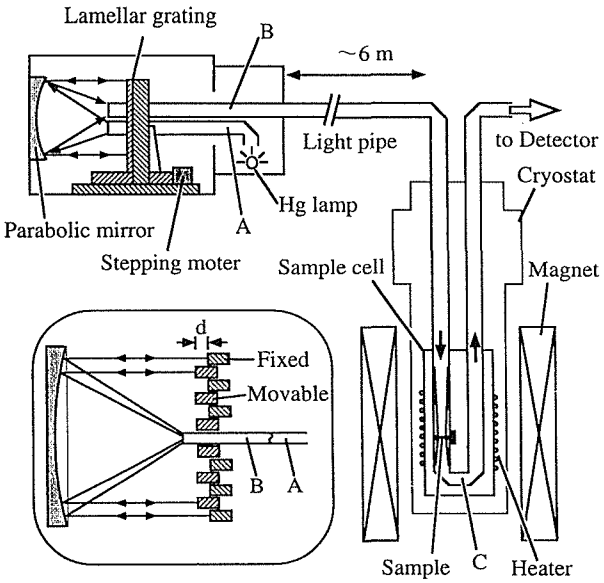

Fig. 1 Experimental setup of the magneto-optical spectrometer. Inset shows a top view of the lamellar grating.

light is about 300 Hz. Output signals of the detector are recorded as a function of the displacement with the same scanning interval. The interferometer is placed about 10 meters from the magnet where the leakage flux density becomes smaller than 5 mT. The chamber of interferometer is made of thick iron plates (12mm) to shield the magnetic flux, because the stepping motor and the high-pressure mercury lamp do not work in the presence of the magnetic field. The FIR light from the interferometer is led to the detector through a sample by a light pipe system (i.d. 10mm). In order to gain the light intensity, light cones are placed near the sample. For the Voigt configuration the sample is placed at the position C, while the figure shows the Faraday configuration. The optical pass is evacuated in order to avoid the absorption due to water vapor.

The FIR spectrometer has been used for the calibration of the field intensity of the NRIM hybrid magnet by the ESR measurements. Fig. 2 shows an experimental configuration of the spectrometer when that is combined with the hybrid magnet (bottom of the figure). The interferometer and the detector can be seen at the upper part of the figure. The magnetic flux density of the hybrid magnet, B_{HM}, is contributed by both the superconducting magnet and the resistive one. Therefor B_{HM} is expressed by using the current flowing in the superconducting part, I_{SM}, and that in the resistive one, I_{WM}, as:

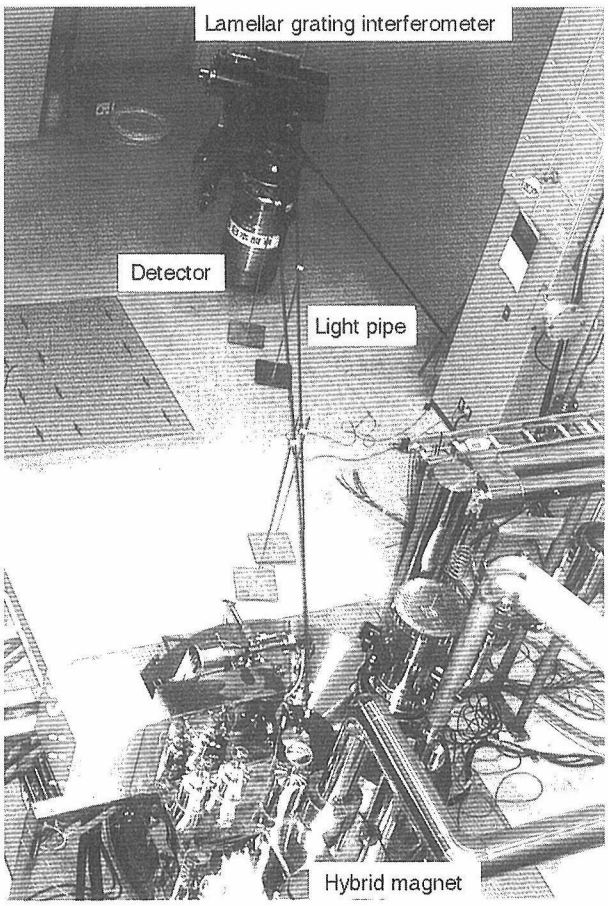

Fig. 2 Configuration of the magneto-optical spectrometer.

$$B_{HM}=C_{SM} \times I_{SM} + C_{WM} \times I_{WM}$$

where C_{SM} Is a coil constant of the superconducting magnet and C_{WM} that of the resistive one. Fig.3 shows the ESR spectra of $Cd_{0.9}Mn_{0.1}Se$, whose effective g value equals to 2 at room temperature, at various fields. The trace with an index of SM (WM) was obtained when only the superconducting part (the resistive part) was excited. From these resonance wavenumber, C_{SM} and C_{WM} was deternrined to be 0.01006 (T / A) and 0.639 (T / kA), respectively. Traces with the index of HM were obtained for the hybrid mode which meant that both the superconducting part and the resistive one were excited simultaneously. The field intensity derived by these ESR absorptions were compared with that by the formula above and these were confirmed to agree with each other quit well with an error less than 0.2 %. As the magnetic field was increased, the broadening of the ESR lines was observed, which is caused by an intrinsic effect of the sample because the ripple and fluctuation of the field is smaller than 0.01 %.

Keywords : magneto-optics, far-infrared, high magnetic field, hybrid magnet

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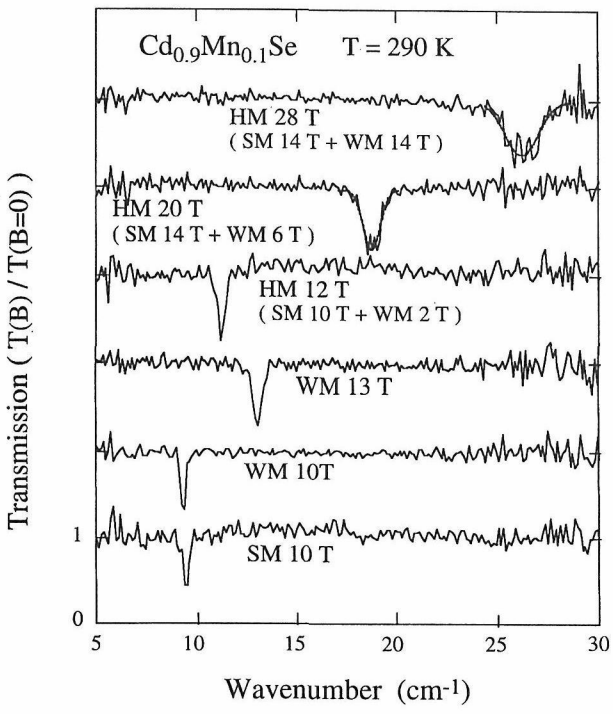

Fig. 3 ESR spectra of Cd0.9Mn0.1Se at various fields of the hybrid magnet.

114 Studies on the Highly Correlated Electron Systems under Multiple Extreme Conditions

H.Aoki, Physical Property Division
[April 1995 to march 2000]

The highly correlated electron systems, such as high Tc oxides , heavy fermion compounds and organic conductors, have been the most important subjects of materials science and have been intensively studied in the past decade. However, many central issues have not been solved yet. A magnetic field is very useful and powerful to study the physical properties and electronic structures of these materials, particularly when it is combined with other experimental environments such as low temperatures and high pressures. NRIM has developed several types of high field magnets, i.e. a hybrid magnet, a large-bore-size magnet, pulsed magnets, high resolution magnets whose capabilities are of the highest class in the world. The purpose of this project is to develop several types of high precision detection systems under low temperatures and high pressures for the high field magnets and is to apply them to the study of the highly correlated electron systems.

So far we have developed detection systems for transport and magnetic measurements under the multiple extreme condition of high pressure, low temperatures and high magnetic fields. Particularly, we have succeeded for the first time in detecting the signals of quantum oscillations from several kinds of highly correlated f electron systems under high pressure. The low temperatures generated by a dilution refrigerator and the sensitive detection using a field modulation method are indispensable to observe the signals. However, it has been believed that the low temperatures is incompatible with the detection method under high pressure. Our success has revealed unconventional changes of electronic structures in these kind of materials under pressure.

Keywords : high magnetic field, low temperature, high pressure, highly correlated electron system

115 Development of Basic Superconducting Technology for High Field Micro-SMES

K. Inoue, 4th Research Group
[April 1997 to March 2002]

Because the stored energy density of SMES (Superconducting Magnetic Energy Storage) is increased rapidly with increasing the magnetic field, the increase of the operation field makes a micro-SMES (a compacted SMES) possible. The operation field is determined by the superconducting properties of commercialized superconducting wires; Nb-Ti in low fields and Nb_3Sn in high fields. Although Nb_3Al, Nb_3Ge, Nb_3Ga, Bi-2212, and Bi-2223 have not yet commercialized, they are the most promising candidate superconducting materials for increasing the operation field of SMES due to their higher H_{c2}(4.2 K)'s than that of Nb_3Sn(4.2 K). Recently we found that the Nb_3Al multifilamentary wire, fabricated through the rapid-heating & rapid-quenching, shows 2-5 times larger J_c in high field than that of the commercialized Nb_3Sn multifilamentary wire, and excellent strain tolerances, which are the most required properties of superconductor for the micro-SMES. In this study, we will investigate on the coil fabrication technique, stabilization, superconducting junction, and ac loss of the Nb_3Al multifilamentary wire. We will also try to fabricate Nb_3Ga and Nb_3Ge multifilamentary wires through the rapid-heating & rapid-quenching process, because they have not only similar superconducting properties but also similar phase diagrams to those of Nb_3Al. Superconducting junction between superconducting wires is one of the basic superconducting technologies required for micro-SMES, because Joule heat loss should be avoided for high efficient operation of the micro-SMES. We will study the superconducting junction process for not only metallic superconductors but also oxide superconductors. Several kinds of superconducting junctions will be tried and evaluated by the performances of persistent-current mode operations for the closed circuits including the junctions.

Keywords : rapid-heating & rapid-quenching, superconducting junction, Nb_3Al

116 Crystal symmetry design for nonlinear-optical crystals

H.Kimura, 4th Research Group
[April 1997 to March 2001]

Nonlinear-optical crystals are important for frequency-modulation devices. Up to now, single crystals were used having a center of crystal asymmetry. However, it is generally difficult to grow single crystals having the center of crystal asymmetry even if new materials with good properties are discovered. It would be possible to obtain single crystals controlling the crystal symmetry in part by means of the crystal symmetry design such as the substitution of constituent elements for another elements in order to form the center of crystal asymmetry part surroundings of substituent elements in micro scale. In this year, we focus on borate crystals, where Al or Ga substituted for B, for synthesizing polycrystalline samples. Their nonlinear-optical prop-

erties such as SHG (Second Harmonic Generation) intensity are investigated. In the next step, single crystal growth of these materials having the center of crystal asymmetry part will be tried by the TSSG (Top Seeded Solution Growth) method from their solutions and the Fz (Floating zone) method from their melts. Furthermore, the optical properties of the crystals will be evaluated using the single crystals grown.

This research is performed in collaboration with the Institute for Materials Research, Tohoku University.

Keywords : frequency-modulation, center of crystal asymmetry, substitution

117 Research and Development of High-Performance Light Alloys for Hydrogen Storage

C. Nishimura, 4th Research Group
[April 1997 to March 2001]

Hydrogen can be regarded as an ideal energy source for the following reasons: (1) it is almost inexhaustible, (2) clean and tender to ecology, (3) easy to be stored and transported. There is no doubt that hydrogen will play an important role as an energy source in the near future after the fossil-energy era. In scope of the large-scale use of hydrogen as an energy source, it is inevitable to establish the technological fundamentals of materials related to hydrogen-energy applications. In this study, it is aimed to give guidelines for developing high-performance light alloys for hydrogen storage, based on Mg or other light metals, which can be applied in automobiles as gas-containers or batteries.

Mg_2Ni, which absorbs hydrogen to form the stable hydride Mg_2NiH_4, has long been regarded as a promising compound for hydrogen storage. High dissociation temperature of the hydride, however, has precluded the practical use of the compound. In this study, alloying elements to lower the dissociation temperature of Mg_2NiH_4 is being searched. Microstructure control is being attempted to improve hydrogen absorption and desorption kinetics for alloy based on Mg-Mg_2Ni system. A new melting furnace has been introduced for fabrication of Mg-based alloys.

There is little data on hydrogen diffusion in alloys for hydrogen storage. In this study, hydrogen diffusion behaviors in the alloys for hydrogen storage are being investigated by means of a hydrogen permeation technique. The emphases are on the diffusion path in alloys composed of multi-phases, and surface effects on hydrogen absorption and desorption. Thermal Desorption Spectroscopy (TDS) is also being performed to investigate the hydrogen desorption behaviors. The obtained fundamental data will be effectively fed back to fabrication of the alloys.

This research is performed in collaboration with Yokohama National University, Science University of Tokyo, and Institute of Industrial Science, the University of Tokyo.

Keywords : hydrogen, hydrogen storage, magnesium, Mg_2Ni, and permeation

118 Development of Particle Assembly Technology for Integration of Functions

N. Shinya, 5th Research Group
[April 1992 to March 1997]

For creation of intelligent materials, technologies for systematization and integration of multiple material functions should be developed in advance. Systematically coordinated multiple functions will lead to manifestation of intelligent functions, such as self-repair, self-diagnosis, feedback and so on, which can respond to environmental conditions.

The particle assembly is a new approach to provide materials with systematically coordinated multiple functions. Since each particle has a primitive function, coordination and systematization of the primitive functions may be realized through three dimensional particle arrangement. Thus, we have been developed three new handling methods for fine particles.

One is a process to produce complex particles by a forced electrification method. The process can be applicable to both metallic and dielectric particles. Complex particles of semiconducting $BaTiO_3$ and metallic In were prepared, and the packed bed of the complex particles showed a good electrical characteristic.

The second is a particle manipulation by a manipulating system developed in this study. Particles were manipulated one by one by a microprobe. The microprobe was applied also to welding of the manipulated particles, and three dimensional arrangement of particles was constructed.

The last one is an arrangement of particles on a substrate using an electron and/or an ion beam. Negatively electrified images and positively electrified images can be formed on the substrates by the electron beam and ion beam, respectively. By dipping the substrates in the suspension of particles, the particles preferentially adhered on the electrified pattern by the electrostatic force, and clear images of particle arrangement were obtained. Different kinds of particles can be arranged at the prescribed positions by repeating the drawing and dipping.

keywords : intelligent materials, multiple functions, particle assembly

119 Fundamental Research on Flexible Structure and Machine Systems Using Advanced Materials

N. Shinya, Intelligent Materials Research Group
[April 1995 to March 1998]

In order to realize flexible structures and machines which have abilities to respond to environmental changes, it is necessary to develop advanced materials provided with the functions of sensors and actuators. For manifestation of the multi-function in advanced materials, utilization of peculiar functions of interfaces in materials is very useful. In this work fabrication and measurement techniques of artificial material interfaces are developed, and properties of various interfaces fabricated artificially are evaluated.

(1) Micro-welding

In addition to the powder particle manipulation, micro-welding techniques using a micro-probe are being developed. After a Au powder particle was deposited at a pre-determined point on a metallic substrate, high voltage of about 10kV was applied between the probe and the substrate. By applying the high voltage, the glow discharge was generated at interface between the Au particle and the substrate. The interface of the particle and the substrate was melted by the glow discharge, and the particle was welded into the substrate.

This micro-welding technique makes it possible to fabricate the artificial interfaces and micro-structures composed of powder particles.

(2) New PTCR material

New PTCR (positive temperature coefficient of resistivity) materials are being developed by controlling interfaces between powder particles. The materials were prepared as follows. (a)Semiconductive $BaTiO_3$ granules and metallic indium powder particles were electrified reciprocally. (b)By mixing the two kinds of particles, $BaTiO_3$ -Indium complex particles were formed. These complex particles showed very good PTCR properties.

Keywords : interface, micro-welding, complex particles, muti-functions

120 Creation of Multi-Functional Materials by Assembling Elemental Components

N. Shinya, 5th Research Group
[April 1997 to March 2002]

One of the most important and attractive research subjects in the material science is a creation of new materials, which show systematically coordinated multiple functions as a mechanical or an electric parts. The materials are called like intelligent materials. Once a mechanical or an electric parts can be replaced by the intelligent material, integration and miniaturization, which are the keywords in the industrial development, are easily advanced. Furthermore, the reliability of the machinery will be improved.

The particle assemblage is proposed as a process for the intelligent materials. New techniques to handle fine particles were developed for the sake of the particle assemblage in the previous study. This research succeeds the previous study, and the aim is to create new materials, which can be replace with a mechanical or electric parts, by applying the developed techniques.

All particles have various primitive functions, such as conductivity, adsorptivity and so on. The components of materials are considered to be particles. Then, a new material can be constructed from particles and a new function can be given to the material by a proper arrangement of various particles so as to manifest the systematical and integrated functions of each particle. That is the idea of the particle assemblage. The particle assemblage has a great advantage that various functional materials can be processed by assembling various particles and that the function of the material can be designed.

Keywords : particle assemblage, multiple functions, intelligent materials

121 Development of Advanced Shape Memory Thin Films by Sputtering

A. Ishida, 5 th Research Group
[April 1997 to March 2001]

Since 1993, we have been successful in showing that sputter-deposited thin films of Ti-Ni possess perfect shape memory effect, perfect superelasticity and two-way shape memory effect. These successes have made shape memory thin films attractive for microactuators and stimulated interest in sputter-deposited thin films of Ti-Ni.

In this research subject, we try to produce high performance shape memory thin films. Such advanced films will be obtained in several ways. One of the ways to improve the shape memory effect is addition of a third element to Ti-Ni alloys. Third elements such as Hf and Pd will raise the martensitic transformation temperature; a high martensitic transformation temperature is keenly demanded for applications in automobiles. However, in the case of thin films, it is difficult to use the effect of the third element because of difficulty in controlling the film composition. Therefore we have to establish the way of controlling the film composition. That is also important even in Ti-Ni binary

alloy thin films. Another interesting approach to improvement of the shape memory effect is to utilize unstable structures formed in sputter-deposited thin films. Ti-rich Ti-Ni thin films after annealing at 773K for 1h were found to contain G.P. zones. This structure cannot be obtained in bulk specimens and gives us the opportunity to improve the shape memory effect. Furthermore, when thin films are deposited in the crystalline state, some unique textures can be obtained by controlling the sputtering conditions. The texture formed by sputtering will be interesting in both fundamental and practical aspects.

Keywords : micromachine, shape memory, Ti-Ni, Sputtering

Related Papers

Microstructure of Ti-48.2at.%Ni Shape Memory Thin Films , A. Ishida, K. Ogawa, M. Sato and S. Miyazaki, Metallurgical and Materials Transactions A, 28A (1997) : 1985-1991

122 Fundamental Study on Development of Biointegrated Materials

M. Sumita, Biomaterials Research Team
[April 1997-March 2001]

Towards 21st century, the aged society progresses, and requirements for affluent quality of life are getting stronger. Numbers of the aged and the physically challenged implanted in their bodies increase. It is important that artificial organs and medical devices exist and function in their bodies without any trouble for many years until they die. Release of metallic ions and debris from materials implanted in body should be avoided, because they always or infrequently cause carcinogenicity,physical deformity enhencement and allergies. Unexpected failure of artificial organs and medical devices implanted also should be avoided, because the reluctant re-operation forces the patient physical and mental pains. Therefore, non toxicity,adhesion to tissue, control in bioreaction caused by foreign bodies, sufficient resistance to corrosion and wear, and long fatigue life are considered as necessary characteristics for the use in vivo conditions.

As metallic materials had the higher strength and the higher toughness than all of the other materials, 316L stainless steel, Co-Cr-Mo alloy, Ti-Al-V alloy etc. have experimentally been used as biomaterials, though those alloys had been developed for other applications. Therefore, capability exists in development of other materials with higher biocompatibility than those being used as biomaterials. The research conducted here is the accumulation of fundamental data to develop new biomaterials, and subsequently development of biointegrated materials.

The content of the present research subject is as follows:

(1) Quantitative evaluation and systematic interpretation of cytotoxicity of biomaterials.
(2) Measurements of cell adhesive force to various materials and observations of cell response to mechanical stimulation using a new system to measure shearing cell adhesive force.
(3) Measurements of corrosion, wear, fatigue, and fretting fatigue propaties of biomaterials under a quasi-biological environment.
(4) Development of biointegrated structural materials.
(5) Reliability tests and standardization of artificial hip joints by a simulator having been developed.
(6) Durability tests of bone plates and artificial hip joints in animal bodies, being carried out by cooperative researches.

Keywords : biomaterials, cytotoxicity, cell adhesive force, durability tests, bio-simulator, in vivo tests

123 Investigation on the basic structural materials in the next era

K.Halada, Ecomaterials Research Team
[April 1996 to March 1997]

In the coming 2lth century, the materials technology will face the new and severe problems and is expected to develop in the following area; 1) further development of the conventional type approaches on creating and manufacturing new materials, 2) new materials and new material technologies which can be harmonized with the global environment to support the sustainable development of mankind, 3) new system which can be correspond to the structural change of industries and economical situations such as the increase of non-skill workers,4) adaptability for the dispersing widely varied request from final user of materials in the living side.

In this investigation, following six fields are projected to response to previous claims and problems;1) easy-recyclable material which have less impurities and can adapt to the coming resources-circulating social system;2) materials which have high performance for processing such as welding, cutting or near-net-shape processing and can be easily processed in the secondary manufacturing processes;3) new processing technology which will bring process-reengineering from the viewpoint of energy-saving, sliming of the system, and expanding the flexibility;4) improvement of the

characteristic performance of the materials, such as multiplying the strength or the life-time of the material;5) increasing the reliability and the safeties in the phase of the assembled and structured elements; and 6) advancement of new functions of materials which have not realized yet now.

Especially for the steel, which have enormous influence to the society of mankind, feasibility of technical break-thorough were investigated by focusing three following elemental fields. In the field of materials-creation, it was confirmed that mesoscopical fining of the microstructure is effective to increasing the strength of steels, and that the solid phase transformation induced by heavy deformation and the fine particles dispersion such as oxide metallurgy or powder metallurgy are expectable technologies. In the field of joining/interface technology, the importance of small area welding technology was emphasized to prevent from the deteriorating the obtained microstructure. For evaluation, the intelligent evaluation method applicable to the high strength/long life steels and the life cycle evaluation method including the phase of assembling, using and recycling are necessary.

This work was a part of the feasibility investigation for R&D of Structural Materials for 2lst Century which has been carried out by gathering the wisdom of NRIM and related researchers.

Keywords : structural material, ultra steel, materials creation, Joining and Interface, Strength and Life Evaluation, 2lth century,

124 Environmental Life Cycle Analysis of Metals

K.Halada, Ecomaterials Research Team
[April 1996 to March 1997]

This work was has been carried out as a cooperative work between Japan and Peoples' Republic China in Bilateral International Joint Research by Special Coordination Funds Promoting for Science and Technology. The counter partner in China was Prof. Wang Tian Min, Institute of Materials Science and Engineering of Lanzhou University.

Materials enormously affect on the global environmental loading. The aim of this study is to establish the common procedure to evaluate the environmental loading of materials world and to prepare the fundamentals of the database for the evaluation method. Especially, China and Japan have the different situation against resources, industries and material-flows in economy. Environmental data are necessary to be exchanged by the common method.

MLCA (Materials Life Cycle Analysis) which was developed in NRIM was used to evaluate the environmental loading of material from cradle (ore mining) to gate (extraction of metals and production of materials). The environmental loading data of materials processing in the two countries having different type of industrial structures were collected and compared. In the case of China, main processes had much room to reduce the environmental loading. In the case of Japan, the environmental loading of transportation was significant

Keywords : environmental loading, processing, resources, materials environmental life-cycle analysis, database

Related papers

K.Halada, "The next stage of Inventory Analysis as a common language", Proc. 2nd Int. Conf. on EcoBalance, 1996 Tsukuba

K.Ijima, K.Halada, K.Yagi,"Environmental loading of steels according to JIS" Zairyou to Process, 1997 spring, Japan Iron Steel Institute [in Japanese]

125 Study on the Processing and Assessment of Ecomaterials

K.Halada, Ecomaterials Research Team
[April 1997 to March 2002]

ECOMATERIALS, which have high recyclability and give less environmental load to the eco-sphere without deteriorating their properties, are expected to be coming materials adapting to the sustainable development. ECOMATERIALS have superior properties in the each following indexes. 1) Expanding the human frontiers, 2) Co-existing with eco-sphere, and 3) Optimizing the amenities: This study is a case study on processing to develop material into ecomaterial.

Preparation of fiber reinforced porous Fe-Fe composite composed from SCIFER (highly drawn iron fiber) and Fe powder was investigated as an attempt of production of ECOMATERIAS by P/M technology. This Fe-Fe composite is expected to have high recyclability because the microstructure of SCIFER reinforces the matrix without using any alien material nor any alloying elements. In order to utilize the cold-worked structure of SCIFER, sintering temperature was limited under 450℃. UFP (ultra-fine powder) of Fe was used for the acceleration of sintering in lower temperatures. SCIFER fiber and Fe-powder were covered and embellished with Fe-UFP by wet dispersion method. Sintering behavior of embellished SCIFER and Fe powder mixture was examined, and raptured microstructures were observed. Fe-Fe composite had 30kg/mm^2 of Fracture resistance strength by sintered

at 400℃, while sintered Fe-powder had 10.3kg/mm^2 by sintered at the same temperature. SEM observation showed that Fe-UFP improved the interconnection of SCIFER fiber and Fe powder at lower temperature.

Adding to the recyclablity and property, energy consumption and other environmental burdens to produce the material ought to be discussed in order to develop the Ecomaterials. MLCA(Materials environmental Life-Cycle analysis) will be applied and modified to use as one of the important materials-data on computerized database.

Keywords : ecomaterials, recyclability, powder processing, materials environmental life-cycle analysis, computerized materials data

Related Papers

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Porous Fe-Fe Composite as an Environment Conscious Materials (ECOMATERIAL), K.Halada, K.Minagawa, S.Ohno,H.Okuyama, N.Itsubo, Advances in Powder Metallurgy & Particulate Materialsl996, (1996)

Environment Load database of Alloys on the Network K.Ijima, K.Halada and K.Yagi, Proceedings of The Second International Conference on EcoBalance (1996)p. 564

126 Formation of amorphous alloys and their physical properties

A.P. Tsai, Aperiodic Materials Research Team
[April 1997 to March 2000]

1. Mechanical Properties of Bulk Amorphous Alloys

Since most amorphous alloys rarely showed a significant glass transition or stability against crystallization, nature of glass transition in amorphous alloys is mostly unknown. Recently, a large number of amorphous alloys revealing clear glass transition and supercooled liquid have been discovered in metallic alloys. Especially, these alloys show high glass formability which allows to produce a bulk amorphous alloy with large size scale. We are studying the preparation of the bulky amorphous alloys and their physical properties. Specifically, we are approaching the studies on the static and dynamic mechanical properties in relation to the deformation and fracture mechanism of bulk amorphous alloys. The dynamic compression measurements are performed. The micro- and macro structures are studied by transmission electron microscopy, scanning electron microscopy and small angle neutron scattering.

2. Growing Single quasicrystals

Current interest of study on quasicrystals has been focussed on the structure and physical properties from a single quasicrystal. This has been achieved in icosahedral Al-Pd-Mn alloy where single grain with a size of centimeter was obtained. Studies on resistivity, magnetic properties and neutron scattering for the single icosahedral quasicrystal are extensively in progress. On the other hand, only a few works concerning to the preparation of single decagonal quasicrystal were reported. The sizes of the single decagonal quasicrystals were about few mm and their quality was not good enough for structural analysis. We have grown a single decagonal quasicrystal with a size of me in an Al-Ni-Co alloy, by floating zone method. The single quasicrystals have been characterized by X-ray Laue back scattering as well as precession camera and verified to have high perfection in structure. To grow a larger single grain is still in trial. The other object in this study is to grow a singe icosahedral quasicrystal in the newly discovered Zn-Mg.-rare earth metals system.

Keywords : bulk amorphous alloy, quasicrystal,

127 Development of 1 GHz NMR Spectrometer

H. Wada, High Magnetic Field Research Station
[April 1995 to March 2001]

When used at 4.2 K, high Tc oxide superconductors (HTS) may be able to generate much higher magnetic fields than low Tc metallic superconductors (LTS). We have initiated a program in which we will develop a 1 GHz superconducting NMR spectrometer including a 23.5 T superconducting magnet. Such a magnet needs HTS coils in combination with LTS coils. In fiscal year 1996, the design work of the total NMR system was carried out, together with the development of high performance metallic superconductors and moderate-size HTS coils.

In our design the whole cryostat consists of outer and inner vessels. The LTS coils are placed in the outer vessel and operated in persisting mode at a field of 21.1 T in a 150 mm diameter bore. The HTS coils placed in the inner vessel should generate an additional field of 2.4 T in a 54 mm room temperature bore. This design makes an easy replacement of the HTS coils possible when required, and enables parallel development of the LTS and the HTS coils. The LTS coils in the outer vessel are cooled with atmospheric superfluid helium at 1.8 K.

Development of high performance LTS is one of the key issues to this program. For compact winding

LTS coil conductors need to have high yield strengths as well as high critical current densities. A critical current density of 103 A/mm^2 was obtained at 1.8 K and at 21 T.

HTS coil development requires a number of technological challenges. We have fabricated several HTS coils with an outer diameter of ca. 130 mm. Among those coils a Bi-2212 coil made of double-pancakes generated an incremental field of 1.8 T in a backup field of 18 T.

Keywords : NMR spectrometer, high field magnet, oxide superconductor

Related Papers

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128 Stability of Superconducting Materials

H. Wada, High Magnetic Field Station
[April 1994 to March 1999]

The main purpose of this study is to define stabilities of superconducting materials under various conditions they may experience when wound to superconducting magnets. Stabilities of new promising superconducting materials for fusion use, such as Nb_3Al, V_3Si and a series of high-temperature oxide superconductors, are examined in terms of temperature, magnetic field intensity/direction and mechanical stress/strain.

In fiscal year 1997 an effect of flat-roll forming on microstructure and the dependence of magnetic field direction on critical currents I_c were studied for the transformed rod-in-tube processed Nb_3Al multifilamentary conductor, indicating excellent strain tolerance and thus being a candidate material for large-scale application. Flat roll forming is an effective way to increase the packing factor of the coil and thereby coil-current-density. The anisotropy in I_c with respect of the magnetic field direction is directly related to the Ic margin and hence the stability of the coil. Supersaturated bcc-solid-solution $Nb(Al)_{ss}$ was so ductile that the as-quenched $Nb(A1)_{ss}$/Nb composite was successfully flattened by cold rolling, without mechanical fracture, to a tape geometry with an overall-aspect-ratio of 15. Flat-roll forming gradually decreased J_c, but the degradation was of the order of a few tens of per cent even at the overall-aspect-ratio of 15. In contrary to the commercially available bronze-processed Nb_3Sn conductor, the anisotropy in I_c was negligibly small for the flattened transformed-Nb_3Al-conductor. One of the possible reasons is the grain boundary pinning which is isotropic with respect to the field direction. Negligible degradation of I_c and isotropic features despite the flattened conductor geometry may suggest the prospect of using transformed Nb_3Al conductor as a compact strand cable for large-scale applications.

Keywords : transformed rod-in-tube Nb_3Al, negligible anisotropy, angular dependence, critical current

129 Development of High Strength/High Conductivity Materials for High Field Magnets

K. Inoue, High Magnetic Field Research Station
[April 1995 to March 1998]

In this study we are developing new high-strength/high conductivity materials for resistive high-field magnets, such as Bitter-type magnets, polyhelix-type magnets, and non-destructive pulsed magnets. Recently we successfully developed a new Cu-Ag microcomposite alloy, which is composed of Cu-based alloy matrix and Ag-based alloy filaments, has a promising combination of high mechanical strength (0.7 - 1.1 GPa) and high electrical conductivity (75 - 83 %IACS) at room temperature. Two or three times intermediate annealing at about 350 - 450℃ during cold-working is very effective to improve not only its mechanical strength but also its electrical conductivity through the deposition of solute elements (Ag in the Cu-based alloy matrix and Cu in the Ag-based alloy filaments). This year we optimized the fabrication process of Cu-Ag microcomposite alloy sheet to obtain wide sheets having high strength as well as high conductivity. Then Cu-24wt%Ag sheets with 350-mm width have been fabricated and evaluated as the Bitter-type conductors. We are planning to use the wide Cu-Ag alloy sheets as the conductors of a Bitter-type inner magnet, which is inserted into a 14 T outer superconducting magnet and used as an insert magnet of a hybrid magnet. The sheets showed tensile strength, 0.2 % yield strength, and elongation of 850 MPa, 700 MPa, and 4.5 %, respectively, at 150℃, which is the highest experienced temperature of Bitter plate in normal operation. Fatigue tests are also performed for the sheets up to 3492 cycles at room temperature. The sheets showed the maximum stress of about 800 MPa at fatigue life of 1000 cycles, which is corresponding to 5 years operation of the hybrid magnet. Using above described data we are now designing and fabricating an insert water-cooled magnet, which can generate magnetic fields up to 35 T in a clear bore of 52 mm.

Keywords : Cu-Ag microcomposite alloy, high

strength/high conductivity, high-field conductor material

Related Papers

Ultra-High Strength, High Conductivity Cu-Ag Alloy Wires, Y. Sakai and H. -J. Schneider-Muntau, Acta Metall. Mater., 45 (1997): 1017-23.

Strength and Conductivity of Cu-Ag Microcomposite, Y. Sakai, T. Asano, K. Inoue, and H. Maeda, Proc. of International Workshop on High Magnetic Fields : Industry; Materials and Technology (1997).

130 Essential Technology Development and Design for New Water-Cooled Magnet

K. Inoue, High Magnetic Field Research Station
[April 1996 to March 1997]

Cu-Ag alloy plate, newly developed at NRIM, is composed of Cu-based alloy matrix and Ag-based alloy microfilaments, and a promising material for resistive high-field magnets due to the useful combination of high mechanical strength and high electrical conductivity. Bitter-type water-cooled magnet is one of the hopeful applications of the Cu-Ag alloy plate. National High Magnetic Field Laboratory (NHMFL) has the most powerful DC power supply and the most powerful water-cooling system for operating water-cooled magnet. In addition there are excellent magnet designers having much experience to fabricate Bitter-magnets in NHMFL. For evaluating the new material as the conductive material in Bitter-type water-cooled magnet, we are performing the present collaboration study with NHMFL.

The last year, we successfully fabricated a 30 T-class water-cooled magnet in this collaboration study. The magnet has been installed and used as a high field magnet in NHMFL. In the collaboration study of this year, we have designed and fabricated a new 30 T-class water-cooled magnet, which will be installed and used as a high field magnet in NRIM. Of course there are several differences in the power supplies and the water-cooling systems in NRIM and NHMFL. Therefore we had to change the magnet design a little at first. According to the design, we fabricated Cu-Ag alloy plates with optimizing the fabrication conditions and sent them to NHMFL. At NHMFL the Cu-Ag alloy plates were processed into Bitter-plates, assembled into a new Bitter-type water-cooled magnet. The test operation of the new magnet is progressing at NHMFL. After the test operation the new magnet will be sent to NRIM, installed and used as a high field magnet in NRIM.

Keywords : Cu-Ag alloy, Bitter-type magnet, water-cooled magnet

Related Papers

Ultra High-Strength, High-Conductivity Cu-Ag Wires, Y. Sakai and H. -J. Schneider-Muntau: Acta Metall. Mater., 45 (1997): 1017-23.

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131 Microstructure and Electromagnetic-Characteristics Studies on V_3Si Multifilamentary Superconductors

T. Takeuchi, High Magnetic Field Station
[April 1 994 to March 1997]

A V_3Si multifilamentary superconductor produced by a modified bronze process is promising for ac applications, since the V_5Si_3 compound layer arranged circumferentially around superconducting filaments (V_3Si) has high resistivity enough to separate the filaments from each other for electromagnetic decoupling. High overall J_c is achieved by reducing a filament-size to -2 um. From a result on the angular dependence of J_c for a flattened multifilamentary conductor, we have explained the enhancement of overall J_c with an increase in the superconductor (S) - normal metal (N) interface density, rather than fine grain structure as a result of completing a diffusion reaction in a short time. As effective pinning centers, there are 3 possible S-N interfaces: V-V_3Si, V_3Si-V_5Si_3, and V_3Si-Cu(Si). The last one is expected to appear when V_5Si_3 phase becomes unstable and disappears at long heat treatments. In the present study, an attempt was made to prepare an unsymmetrical sample in interfacial configuration about the V_3Si-plane and thereby separate the individual contribution of S-N interface pinning. Unsymmetrical samples were prepared by hot pressing a Cu-Si bronze plate on a V plate, and then cold rolling them to a laminated tape. Resulting tapes were heat treated to form compound layers geometrically in the order V/V_3Si/V_5Si_3/Cu(Si). J_c was measured two times at a transverse magnetic field which is parallel or perpendicular to V_3Si-plane, by reversing a current direction. Despite no discrepancy of J_c in reversing the current direction for perpendicular fields, J_c for parallel fields strongly depends on the current direction. This suggests that the elemental pinning force of V-V_3Si interface differs from those of V_3Si-V_5Si_3 and V_3Si-Cu(Si) interfaces. Mechanism of interfacial pinning was explained with the F_p field dependence, proximity effect and irregularity of interface.

Keywords : flux pinning, S-N interface

132 Development of Magnetic Separation Control System

T. Ohara, Strong Magnetic Field Research Station
[April 1995 to March 2000]

Progress in applied superconductivity technologies has resulted in the development of superconducting (sc) magnets with excellent operability. One of the promising application fields is magnetic separation. The primary advantages, those being energy saving, compact size, and increased speed becomes visible only after the system is superconductorized. The full-scale application of this process will also contribute greatly to the preservation of global environments.

We have developed the key system technologies of application of high Tc sc magnets to magnetic separation. We proposed magnetic chromatography (mc) as a new technique for ultra-fine particle separation which separates chemically similar but magnetically dissimilar materials, such as lanthanide and actinide elements. Our mc simulator modeled the transient behavior of 300 Å and 500 Å particles by taking into account the fluid flow field, magnetic field, and particle diffusion. For the range of flow velocity between 1.0×10^{-4} and 1.0×10^{-3} m/s, the separation efficiency increased with decreasing the flow velocity and increasing the ratio of particle magnetic susceptibility. To improve the separation efficiency, it is also very important to extend the channel length; longer channels (4.0m) completely separated two kinds of ultra-fine particles that cannot be separated by shorter channels (i.e., 0.4m). For the smaller particles (300 Å), increasing the applied magnetic field strength improves the separation efficiency. We also simulated other types of mc systems, and found that regardless of flow velocity an mc system with a staggered-wire arrangement always has better separation efficiency than an mc system with a face-to-face wire arrangement.

We conduct this research in collaboration with the Electrotechnical Laboratory, AIST, MITI.

Keywords : ultra-fine particles, high gradient magnetic separation, magnetic chromatography, Computer Simulation, Control Volume Method

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Optimization of a Superconducting Solenoid for High Gradient Magnetic Separation Systems, T.Ohara, K.Kaiho and T.Kiyoshi, IEEE Trans. Magnetics 32(1996) : 5103-05

Computer Simulation of Magnetic Chromatography System for Ultra-Fine Particle Separation, X.Wang, T.Ohara, E.R.Whitby, K.C.Karki, C.H.Winstead, Trans. IEE of Japan 117-B (1997) : 1466-74

133 Developments of High Strength Oxide Superconductors for High Magnetic Field Application

Y.Tanaka, 1st Research Group
[April 1995 to March 1997]

Round cross-sectional wires seem to be suitable to achieve high homogeneity in the superconducting magnets with easiness in winding process. Bi-2212 Ag-sheathed 1-, 7-, 19- and 37- cores multifilamentary wires have been fabricated by the powder-in-tube technique, where each cores are composed of two Ag-tubes of different diameter with a centered Ag-wire and filled with oxide powders between them. A composition of the powder used is $Bi_{2\cdot0}Sr_{2\cdot0}Ca_{0\cdot64}Cu_{1\cdot64}Ox$, which is considerably different from the $Bi_{2\cdot0}Sr_{2\cdot0}Ca_{1\cdot0}Cu_{2\cdot0}Ox$ in order to improve the formation of Bi-2212 phase. Round cross-section multifilamentary wires of 1mm in diameter have been prepared by drawing composites including each numbers of core. Short samples cut from these wires have been heat treated at 885 - 906℃ for 15 minutes followed by slow cooling, 4 - 5 ℃/hr, down to 820℃ and furnace cooling. Microstructures of the cross section of the wires were observed by optical and scanning electron microscope, along with analysis by EDX on the phases and precipitates formed. Critical current, Ic, and transition temperature, Tc, were measured by 4-probe resistive and ac susceptibility method, respectively. In the present study most appropriate heating temperature lies between 900 - 904℃ with the highest Ic value and the sharpest Tc transition. The Ic in general increases with increasing core-number or decreasing the thickness of oxide layers between Ag-tubes and Ag-tube/Ag-center-wire in the core; about 40um, 25um and 18um for 7-, 19- and 37-core wires, respectively. The highest critical current density, Jc for oxide core area, reached to 4.1×10^4 A/cm^2 at 4.2 K, 10 T with overall J_c of 2.1×10^4 A/cm^2 for the 37-core wire heated at 902℃.

This research was performed in collaboration with Sumitomo Heavy Industries, Ltd and Sukegawa Electric Co. Ltd.

Keywords : Bi(Pb)SrCaCuO, Ag sheath, Powder-in-tube, Multifilamentary wires

134 Evaluation of Oxide Superconductor Wires

K. Itoh, High Magnetic Field Research Station
[April 1995 to March 2000]

To realize a magnet made of a high-Tc oxide superconductor, it is indispensable to have a long wire with high quality and homogeneity. We often use short-length specimens cut from the different positions of the whole length of a wire to check the quality and homogeneity. However, such technique does not only waste a wire but also disable to use it for magnet winding.

In this study we develop and establish efficient techniques to measure superconducting properties such as the critical current as function of field, temperature, mechanical strain, etc.. In addition, we develop measurement techniques on long and/or coil shaped specimens, and compare the results with those obtained by the techniques on short -length specimens. Such comparison would be useful for the development of wire fabrication and winding techniques.

This year, we measured the field angle dependence of critical current for Bi-2212 and 2223 silver sheathed high-Tc oxide tapes. We found that except for fields nearly parallel to the tape surface, the field angle dependence of these conductors was dominated by the field component normal to the tape surface. Results obtained should be useful for the design of high-Tc oxide coils, at the end parts of which the normal component of field becomes large and for the evaluation of field irreversibility against coil current.

We also tested various kinds of metallic and oxide superconductors developed for the 1 GHz NMR magnet and the magnet for magnetic separation, and evaluated the test data obtained.

Keywords : oxide superconductor, evaluation, measurement technique, long length

135 Basic Research for the Control of Chemical Reactions by High Magnetic Field

H. Abe and K. Takazawa, High Magnetic Field Research Station
[April 1996]

With the aim of producing new materials in the high magnetic fields more than 10T, we have started to investigate a possibility of controlling chemical reactions by the high magnetic fields. Taking advantage of availability of high magnetic fields in Tsukuba Magnet Laboratories, we are to develop a methodology to measure magnetic field effects on each elementary process in chemical reactions. Such studies hitherto have been performed in the fields up to 2T and have given a clear ground for the interpretations of many aspects of the magnetic field effects on the photochemical reactions in solutions and on the dynamic behavior of electronically excited molecules in the gas phase. The aim of this project is not an extension of such studies to high fields, but exploitation of new research field, "the dynamics of excited molecules in high magnetic fields", which may be a basic research for magnetic field control of chemical reactions.

Two apparatus are used for the following experiments.

(1) Mechanism of photochemical reactions in solutions under 40T class pulsed magnetic field. The reaction is initiated with the excitation of target molecule to the electronically excited state by the third harmonic of a pulsed YAG laser (355nm, 15ns). The creation and annihilation of the resultant reaction intermediate such as triplet state, radical pair, and bi-radical is observed by its transient absorption. The monitor light source is a Xe flash lamp. Trigger timing of the laser and the flash lamp is controlled to synchronize with the maximum field strength of the pulsed magnet.

Using this apparatus, the photoreduction of benzophenone in micellar solutions was investigated under magnetic fields of 0 - 29.6T.[1] Large magnetic field effects were observed for both of the lifetime of radical pairs and the yield of escape radicals.

(2) Dynamic behavior of electronically excited molecules in the gas phase under high magnetic fields up to 10T. Target molecule is excited to a specific energy level located around its predissociation threshold by a tunable pulsed laser. Another tunable pulsed laser is used to detect a resultant dissociation fragment by observing a laser induced fluorescence. Changing time interval between the firing of the lasers, time evolution of the creation and annihilation of the fragment is measured under magnetic fields up to 10T, which is applied by a liq.He free superconducting magnet. Magnetic field effects on the elementary processes of combustion reaction and photo-ionization will be also investigated.

As the first step to the excitation to the highly excited state, the electronic spectra of gaseous NO $X^2\Pi \rightarrow A^2\Sigma$ transition were observed under magnetic fields up to 10T. Large Zeeman effects on the spectra were observed. A quantum mechanical calculation was carried out to obtain the eigen-function of each Zeeman perturbed level and at the result, both transition energies and the intensities of all spectral lines could be completely reproduced.

1) Nishizawa et al. Chem. Phys. Lett. 267(1997)501-506.

Keywords : high magnetic field, chemical reaction, photo-dissociation, photo-ionization, reaction interme-

diate, photoreduction

136 Study of regenerator for ultra-low temperatures

A. Sato, T. Numazawa, F. Matsumoto and H. Nagai, High Magnetic Field Station
[April 1995 to March 1998]

This study is focused on a key component of a new refrigeration system which provides various temperatures lower than I K by the super fluid helium Stirling cycle. Several regenerator materials which have high heat capacity in the temperature range below 2 K are investigated. Generally magnetic materials show specific heat anomaly and magneto-caloric effect related to magnetic phase transition or Shottoky anomaly. In particular, some rare-earth garnets are very useful because of high entropy properties and low transition temperatures. Recently, a large specific anomaly of $Gd_3Ga_5O_{12}$ has been explained by its geometrical frustration; short range order of magnetic interaction. This 'high' entropy is very useful as the heat capacity for refrigeration below I K, but the refrigeration temperature is limited down to 0.8 K because its peak temperature in specific heat is about 0.9 K. Thus we have grown the $(DyGd)_3Ga_5O_{12}$ system to introduce the long range magnetic order in $Gd_3Ga_5O_{12}$ and to be lower the transition temperature. At 250/0 substitution ofthe Dy ion for the Gd ion in $Gd_3Ga_5O_{12}$, a heat capacity of $(Dy_{0.25}Gd_{0.75})_3Ga_5O_{12}$ showed the peak value of 0.6 K and the large heat capacity comparable with $Gd_3Ga_5O_{12}$. We found this method is useful to provide high heat capacity materials for magnetic refrigerants below I K. Optimization of substitution rate is under study.

Keywords ; low temperatures, regenerator, helium

Related Papers

Thermal Properties of $(Dy_xGd_{1-x})_3Ga_5O_{12}$ Garnet Single Crystals

T. Numazawa, H. Kimura, A. Sato, H. Maeda, K. Shimamura and T. Fukuda

Advances in Cryogenic Engineering-Materials, vol.42(1 996) 459

137 Processing and Development of Isotopically Controlled Materials(ICM)

T.Noda, Nuclear Materials Group
[April 1992 to March 1996]

Materials composed of isotopically selected elements realize the essential solution of subjects such as induced activity, He embrittlement, and compositional change caused by reactions with energetic particles. The objectives of the program are (1)R&D of in-situ ICM processing facility(ICMPF) utilizing infrared multi-photon decomposition reaction, (2)search of working materials for isotope separation, (3)development of in-situ synthesis of isotopically controlled Si and SiC etc. and (4)development of ceramics and their composites with advanced properties.

The isotope separation experiment using Si_2F_6 was conducted under the irradiation of pulse infrared laser at 930-985cm^{-1}. SiF_4 with condensed ^{29}Si and ^{30}Si was produced at 944-967cm^{-1} while ^{28}Si was concentrated in the residual Si_2F_6. The natural Si is composed of 92.23% of ^{28}Si, 4.67% of ^{29}Si and 3.10% of ^{30}Si. The optimum separation of each isotope mainly depends on the wavenumber of CO_2 laser. $^{30}SiF_4$ of 43.3% and $^{29}SiF_4$ of 12% at maximum could be continuously obtained with a yield of 4% at 951.20cm^{-1} and 10% at 956.20cm^{-1}, respectively. The concentration of ^{28}Si in the residual Si_2F_6 was made using a long cell, 60mm in diameter and 200mm long. The addition of inert gas such as He, Ar, and Kr to Si_2F_6 was effective to increase the ^{28}Si concentration. The maximum concentration of 99.72% was attained at 952.923cm^{-1} with a yield of 5.4%.

The SiF_4 and Si_2F_6 enriched with ^{28}Si, ^{29}Si or ^{30}Si were transformed to Si flake at 623-723K using a plasma CVD technique.

The simulation code, IRAC, calculating transmutation was improved by combining with a neutron transport calculation code and introducing multi-step reactions to predict more precisely the transmutation of materials including ICM. Chemical vapor infiltration process to obtain SiC composites with a high purity and a stability against heating and neutron or ion irradiation is being developed using crystalline SiC fiber preforms.

Keywords : isotopically controlled materials, ICM processing facility, isotope separation, chemical vapor infiltration

Related papers

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Effect of Neutron Spectra on the Transmutation of First Wall Materials, T. Noda and M.Fujita, J. Nucl. Mater., 233-237, (1996)1491-1495.

138 Analysis/Evaluation of Atomic Scale Compositional Change in Materials Due to the Radiation Damage

K. Furuya, High Resolution Beam Research Station
[April 1995 to March 1999]

Radiation damage in materials is characterized by the atomic displacements associated with the destruction of crystalline structure, transmutation by nuclear reaction and radiation induced solute segregation by the irradiation of energetic particles such as neutrons and ions. In addition to the formation of many types of defect clusters produced by this atomic process and the resultant microstructural changes, enhanced diffusion associated with these defect reactions results in the local change in the concentration of solute atoms, the segregation at grain boundaries and surfaces, and the precipitation of secondary phases. For the basic understanding of radiation damage and compositional changes, an analytical TEM consists of 1 MeV electrons with two ion accelerators, energy dispersive X-ray spectroscopy (EDS) and energy loss spectroscopy (EELS), so-called "SUBNANOTRON", is used in this research. In this year, the reseraches on the advantage of the extensive usage of EDS at higher voltages and on the behavior of defect clusters in Al irradiated by He^+ ion with 15 keV were investigated.

The EDS spectra from Si (100) TEM thin specimen were measured at 400 and 1000 kV at a constant beam current and diameter, and the build-up of C-K(and O-K(was compared in terms of the voltage and time. The vacuum during EDS operation was about $2x10^{-6}$ Pa. At 400 kV, the height of C-K(signal significantly increased with the time over 1000 s, while this build-up has been masked by the background noise at 1000 kV. After subtracting the noise, the intensity of C-K(due to the contamination is very low at 1000 kV. Similar tendency was obtained for O-K(. Several sources of these contaminations have been considered; surface contamination, adsorption from the vacuum and so on. The present results clearly indicated very low contamination rate and precise analysis at higher voltage for EDS.

The radiation induced defects in Al with 15 keV He irradiation have been investigated with the atomic scale imaging at room temperature. After the bombardment of He^+ to a dose of $2.3x10^{20}m^{-2}$, two types of defects were seen in HRETM along [110] projection. One has a planar shape and is located along apparent {111} planes. Another has three dimensional shape with very weak contrast. The former is clearly the stacking fault type loops, probably with an interstitial type, which is similar to that in the case of electron irradiation. The latter has no atomic displacement, so that He bubbles and/or cavities associated with the enhanced migration of vacancies with He atoms. The details in the structural morphology of these defects was compared with the results of image simulation, in which the models of various kinds of defects were proposed. The defect clusters and the local composition change by ion beam irradiation will be investigated as a next step.

Keywords : SUBNANOTRON, EDS, He irradiation, contamination

Related Papers

Practical performance of energy-dispersive X-ray spectroscopy with a high-voltage TEM up to 1000 kV, Kazuo Furuya, Mitsuaki Osaki, Mitsuhiro Awaji and Tetsuya Saito, J. Electron Microsc. 45 (1996) 285

High Resolution Transmission Electron Microscopy of Defect Clusters in Aluminum during electron and Ion Irradiation at Room Temperature, Kazuo Furuya, Min Piao, Nobuhiro Ishikawa and Tetsuya Saito, Mat. Res. Soc. Symp. Proc. 439 (1997) 331

139 Characterization and Control of the Optoelectronic Properties of Small Crystalline Materials with Electron Probe Analysis

Kazuo Furuya, High Resolution Beam Research Station
[April 1995 to March 2000]

It is possible to control the specific optoelectronic properties of very small crystalline materials by enclosing the crystals with a different kind of materials, or by terminating the conducting electrons inside particles. This type of small structures are feasible by burying nanometer-sized crystals into materials and covering the surface of one material with other material. We are trying to create those small "hetero structures" and to characterize their structures.

Porous silicon is chosen for one example of non-equilibrium nanocrystals embedded in the bulk matrix. A highly porous silicon (PS) made by anodization is known as a material with efficient visible photoluminescence (PL) at room temperature. Up to now, the models based on quantum size effects, quantum size/luminescence centers, surface compounds, etc. have been proposed to explain the efficiency and spectrum of PL. However, the PL mechanism is not clear yet. PL properties are reported to vary with light exposure or thermal oxidation. Aging phenomena at room temperature are very important for PL mechanism and applications, but have many points still unclarified.

In this year, the work on the aging behavior of PL in PS has been continued from last year. The correlation between PL properties and chemical states during room temperature aging of PS over a 192-days time

span has been investigated in detail to clarify the origin of PL. The p-type Si wafers with 5-10 Ω cm resistivity were used to fabricate light-emitting PS layers. The PS layers were formed by anodization in a 50%HF-ethanol (1:2) solution at room temperature at different current densities of 10-40 mA/cm^2. PL spectra were measured at room temperature by using a nitrogen laser (337 nm) as an excitation source. The PS samples exhibited red PL peaking at around 650 nm. While the PL intensity increased with aging time, the Si-H and SiH_2 bands in infrared spectra decreased. The Si-O bond content increased with time and correlated closely with the PL intensity. The PL lifetime increased during aging (3 to 37 μs). Transmission electron microscopy indicated the presence of Si nanoparticles and amorphous substance in PS layer.

Moreover, the aging behavior of high resistivity PS was compared to that of low resistivity PS. Immediately after preparation, high resistivity PS showed weaker red PL than low resistivity PS, whereas after 2 months aging the former showed stronger PL than the latter. The substrate resistivity may affect the aging, that is, air oxidation of PS layer as well as the microstructure, thus causing a change in PL intensity. These results suggest that the PL from PS may be ascribed to the cooperation of quantum confinement effect inside Si nanoparticles and oxygen-related effect outside the particles.

Keywords : small crystalline materials, surface terminated or buried particles, porous silicon (PS), photoluminescence (PL)

Related Papers

"Aging behavior of photoluminescence in porous silicon", Y. Fukuda, K. Furuya, N. Ishikawa, and T. Saito, submitted to J. Appl. Phys.

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"Correlation between Photoluminescence and Local Chemical States of Porous Silicon by Means of Electron Loss Spectroscopy", M. Song, Y. Fukuda and K. Furuya, J. Surf. Anal., 3 (1997) 446

140 Joint Research on the Structure and Properties of Implanted and Precipitated Semiconductor Nanocrystals

K. Furuya. High Resolution Beam Research Station
[April 1996 to March 1997]

Understanding of the behavior of implanted atoms which make nonequilibrium phase is interesting because of the physical point of view as well as the application point of view. For example, in the case of semiconductor atom-clusters precipitated as crystals in metals and semiconductors, the inhomogeneity of atom structures and of electron state density results in the unique-properties of materials. In this study, the investigation of the microstructures of implanted ions into the crystalline materials at an atomic scale has been performed as a joint research with Argonne National Laboratory (ANL).

The assignment of this collaboration work is as follows. The 6H-SiC, SiO_2 and Al TEM specimens for transmission electron microscopy (TEM) were prepared in NRIM by electrochemical polishing and ion milling. Then Xe^+ and Ge^+ were implanted into the specimens by using the NEC-650kV accelerator in Tandem-Facility at ANL with accelerating voltage of 25-35 kV. TEM and high-resolution electron microscopy (HRTEM) were done with SUBNANOTRON (JEM-ARM-1000) at NRIM.

Ion implantation of semiconductors at room temperature generally results in amorphization at about 10^{18} ions/m^2. Therefore, the temperature in this experiment was raised to 1123 K during implantation. A selected area diffraction pattern of the specimen of SiC implanted with Ge^+ indicated clear spots of crystal for 6H structure. A TEM micrograph showed a small precipitates with a high density. However, the detailed structural observation of such materials requires precise determination of experimental conditions for HRTEM. Xe nanocrystals embedded in Al matrix were used for this purpose. Based on the theory of image formation in TEM with changing defocus values and specimen tilt, the highest contrast of a Xe nanocrystal without any disturbance of Al lattice fringes were achieved by changing the defocus value to - 76 nm and tilting the specimen to about 3 dgree from [110] zone axis. Similar technique is now planned to apply for Ge nanocrystals in SiC and SiO_2.

Keywords : precipitation of nanocrystals, TEM, HRTEM, specimen tilt, defocus value

141 5. Study on Detection and Evaluation of Radiation Damage in Extreme Particle Fields

N. Kishimoto, High Resolution Beam Research Station
[April 1992 - March 1999]

Hybrid particle fields of ion and photon exert strong interactions with materials and are potent not only to detect elementary processes but to explore novel properties of materials. Especially if both high energy and

high density of ion and photon are attained, unexplored non-equilibrium effects will be expected, by virtue of their contrasting effects of momentum, energy, excitation modes. The extreme particle field is also an important aspect for practical environments of high energy devices, such as fusion reactors, MHD generators etc. The main purpose of this research program is to detect and evaluate non-equilibrium processes of materials in the extreme-particle-fields(EPF), associated with radiation damage.

This year, the program concentrated on system integration of the EPF generator, especially on Phase-III, i.e., construction of a switching magnet, beam-lines I and II. Also, beam-material interactions in the low-energy/high-current ion fields were studied by use of the injector beam-line. To attain mA-class beams at the target position, the important were not only the intense-ion production but also the efficient beam transport. To meet the high beam power, the relevant parts were also armed with graphite and effectively cooled at 6 kW. One of the irradiation chambers was equipped with twin turbo-molecular pumps, a high-power Faraday cup and the beam-profiler.

In-situ DLTS experiments were conducted using a C-meter and a cryostat, integrated within a beam-line of NRIM cyclotron. The in-situ method enables us to study real-time evolution of deep energy levels induced by radiation damage. Also, defect recovery below room temperature can be properly evaluated, since the measurement is carried out without exposing to either atmosphere or room temperature. Experimental results confirmed that V_0 and V_2 centers were dominant and proportional to the proton fluence. Meta-stable deep levels of carbon-defect complex also formed and showed a temperature-sensitive variation in the post-irradiation annealing. Importance of the in-situ measurements was thus demonstrated.

Keywords : extreme particle field, high current, negative ion source, in-situ measurement

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142 Development of analytical techniques for characterization of nuclear materials using new generation synchrotron x-rays

K. Sakurai, High Resolution Beam Research Station
[April 1994 to March 1999]

The SPring-8, which is being constructed at Harima science city, generated the first synchrotron light in March, 1997, and the facility will be opened in coming autumn. One of the most important advantages of the SPring-8 is the use of undulator radiation in X-ray region, of which photon density is 104-106 times higher than that of conventional synchrotron light source. Because of such high brilliance, trace or very small quantity of materials will become a target of the characterization. Sensitivity enhancement will enable the detection of the faint signals. Another benefit is availability of high energy X-rays, typically, 40-100 keV. High intensity and energy tunability, which any other source has not been provided, will make it possible to inspect the inside of metallic materials with high precision. Both features of new synchrotron radiation are very attractive from a view of materials characterization.

(1) Nanometer scale measurement of rough surface by diffuse scatter at grazing incidence (Use of collimated strong X-rays)

Grazing incidence X-ray techniques are powerful tools for the surface and interface study. Besides the specular reflection which is widely used for the evaluation of the polished surface and thin films, the diffuse scattering is significant to analyze the physical shape of the surface and interface. In the present program, we have developed a prototype instrument for measuring diffuse scattering at grazing incidence. It works well with a laboratory scale source, and glass surface has been studied by this reflectometer. We are continuing further instrumentation for the beamline at the SPring-8.

(2) Development of a cryostat for high resolution detector (Use of high energy X-rays)

A superconductor tunneling junction (STJ) detector is promising to realize chemical characterization of heavy metals, because of 3-10 times better energy resolution compared with conventional Si(Li) detectors. A He3 cryostat was developed for the detector. It was confirmed that lower than 400 mK is achieved and maintained for longer than 24 h. A detector test will be carried out in near future.

Keywords : synchrotron radiation, instrumentation, surface and interface analysis, materials characterization

Related Papers

Trace chemical characterization of liquid drop by fluorescence detection of absorption edge shifts using total reflection support, K. Sakurai, A. Iida and H. Shintani, J. Phys IV (France) 7, C2-713 (1997)

143 Development of Fundamental Technologies for Excited Neutral Beams

Y. Yamauchi, Neutral Beam Unit, High Resolution Beam Research Facilities
[April 1996 to March 2003]

The electronic states of surfaces play significant roles in chemical reactions and crystal growths on surfaces of materials. The excited neutral beams are promising probes to get information from exactly outmost surfaces rather than electron or photon beams. Fundamental technologies to generate excited neutral beams are being developed.

In spite of the fact that photoelectron spectra or electron-excited secondary electron spectra include the information on the outmost layer, photons and electrons penetrate through the outmost layer and reach to more deeper layers. Those spectra contain the information on deeper layers where the ejected electrons are generated. Thus some ambiguity remains whether any features of spectra are originated from the outmost layer or the deeper layers. On the other hand excited neutrals also may release their internal energy to the surface electrons. The slow neutrals with a kinetic energy of several tens meV are reflected by the potential which comes from the surface atoms and never reach to the deeper layers. The ejected electrons pick up only the information on the electronic state outside of the top-most layer. Therefore slow excited neutrals are essentially sensitive to the electronic state of adsorbed molecules or states spatially extended wave function from the surface to the vacuum. Further the spin selected neutral beams enable us to understand surfaces more precisely.

The present project includes two kinds of technologies, i. e., generating techniques for excited neutral beams and detecting techniques for secondary particles ejected from irradiated surfaces. As regarding to the beam forming, a beam system creating high density neutral beams under a clean vacuum environment, exciting atoms by electron impacts, removing ions and high Rydberg atoms, selecting the velocities, and polarizing the electron spins of atoms will be developed. Means to measure the energy angular distribution and the spin polarization of ejected low energy electrons will also be investigated.

To concretely design a neutral beam system, a preliminary study using a prototype device has been conducted for beam generation and exitation. A He-atom beam effused through a capillary plate was exited with an electron shower. The beam characteristics were measured by a time-of-flight(TOF) method. The TOF spectra showed that the arrival sequence at the detector is light emission, ions, fast neutrals, and slow metastable atoms. An intensity of 10^{10} metastables/sec·sr was achieved with this type of effusive beam source.

Keywords : neutral beam, metastable atom, excited neutral, surface

144 Advanced characterization of micro and nano meter scale structure of materials by brilliant synchrotron x-rays at the SPring-8

K. Sakurai, High Resolution Beam Research Station
[April 1997 to March 2002]

The present research program aims to establish new techniques for analysis with super precision and high resolution to evaluate micro and nano meter scale structure of materials. We are involved in the construction of the physico-chemical analysis beamline at the SPring-8, which is a new brilliant synchrotron facility and is expected to be opened in October, 1997. Although almost the first 1 year will be construction and commissioning phase, the following research program will be carried out in the following year.

(1) Micro imaging of trace metals
(2) Characterization of nano meter scale structure of thin films
(3) Evaluation of micro damage of materials

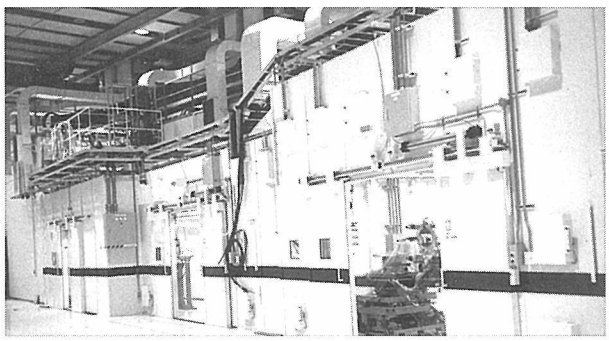

Photo 1 The beamline 39XU (physico-chemical analysis) at the SPring-8.

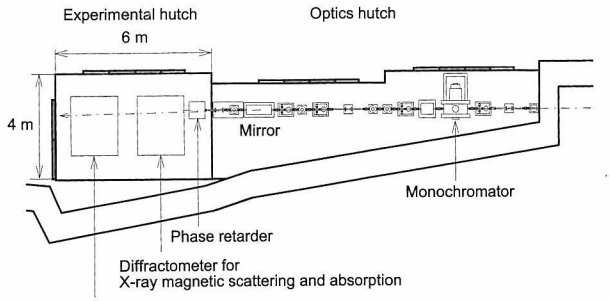

Fig.1 Plan view of the beamline. The instruments will be installed in 'the experimental hutch'.

145 Surface analysis database

K.Yoshihara, Extreme High Vacuum Station
[April 1995 to March 1997]

As a part of the international collaborative reset of VAMAS, we have developed a PC-based system, Common Data Processing System(COMPRO), which integrates the spectral and physical data, peak position database, GUI query and analysis system. COMPRO Version 5.0 runs on Windows95, and is now distributed through Internet (http://sekimori.nrim.go.jp). This version can convert spectral data of ASCII format to the VAMAS Standard Data Transfer Format (ISO DIS 14976), and has an easy access interface to a Internet spectral database provided by the Surface Analysis Society of Japan(SASJ). COMPRO also has calibration systems for energy and intensity scales, and every body can easily calibrate his/her analyzer. In 1994 the Science and Technology Agency (STA) of Japanese Government launched a project to interconnect networks under various ministries and agencies. As a site of this network we are implementing a network-oriented database for surface chemical analysis such as AES and XPS spectra. SASJ is responsible to provide the spectral data, and controls its quality. Internet spectral data now has about 2,000 spectra of metals, semiconductors and ceramics. The file structure of spectral data is based on ISO NP 14976 and ISO DIS 14975, and is fully compatible with VAMAS Standard Data Transfer Format. Because this file structure can carry the information on specimens, calibration and data-processing, we could construct a GUI searching system for Internet database. One can choose searching items (ex: host materials, in-situ preparations, keywords, techniques, instruments, operators and etc.) by GUI. The searching results are displayed on a client's PC, and a target spectrum can be downloaded to a client's PC through Internet. In future we hope all computers of the surface analysis machines can be connected to Internet system so that every surface analyst worldwide can share the spectral data and the common data processing software to identify the surface chemistry of new materials.

Database for Surface Analysis, M.Yoshitake and K.Yoshihara, J. Korean Vac. Soc. 5(1996):p57-60

Keywords : AES, XPS, database, Internet

146 Self-control of surface composition of thin film and its application to field emitter

M.Yoshitake, Extreme High Vacuum Station
[April 1995 to March 2000]

When deposited metal film on substrate was heated in a vacuum, it was observed that substrate element diffused to the surface of film. The surface concentration of segregant was always constant. Even if the segregated layer was removed by argon ion sputtering, the segregated layer of the saturated concentration was formed again by re-heating. Therefore, the surface composition of the segregated layer on metal film is expected to have a self-controlling property.

It is well known that the surface adsorption of metal changes the work function of a solid surface. Therefore, it is expected that the work function changes by the surface segregation. The work function varies with a coverage of adsorbed materials. However, if we use the self-controlling property of segregation behavior on metal film, the stable work function on the surface, i.e., stable field emission current can be expected and we can produce a field emitter of high performance. It has been shown by authors that Nb film with Ti surface segregation works as a getter for residual gas in ultra high vacuum, in which environment a field emitter functions.

In this study, the stability of surface composition will be quantitatively analyzed in the wide range of temperature, and the change of work function by segregation will be measured. Until now, the recovering rate of the surface composition during Ar ion sputtering in case of Ti segregation on Nb thin film was measured. At first, ion sputtering rate was precisely calibrated quantitatively. Then, the dynamic balance of Ti brought by the removal rate by ion sputtering and the supply rate by diffusion from substrate was used to determine the rate of Ti supply at various temperatures. The relation between the amount of adsorbed contaminant and the segregation behavior was also examined in case of Cu segregation on Ti film. The instrument has been modified so that work function can be measured under specimen heating.

Keywords : surface segregation, saturated surface composition, self-composition control, work function, field emitter

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147 The Evaluation and Control of Material Self-organization

M.Tosa. Extreme High Vacuum Station
[April 1996 to March 1999]

Segregated hexagonal boron nitride layer (h-BN) on metals or alloys doped with born and nitrogen have the c (0001) crystal plane parallel to the base substrate surface. The binding force between the c planes is much smaller than that of other planes and the friction force on the c plane surface will be small in case of the horizontal force due to the easy sliding of the planes. Such property as the small adsorption force of segregated h-BN layer surface will be applied to the self levitating probe system which can scan the solid surface very stably at the nano gap by means of van der Waals' force without friction force.

This h-BN compound layer can be prepared on the surface of co-sputtered film substrate with BN and alloy targets, but the BN surface segregation so far requires in situ vacuum annealing just after film preparation and lacks the perfect surface coverage and plane orientation. XHV surface modification apparatus was developed for the preparation of self organization by the stress control in the film aiming at the full surface coverage and perfect plane orientation of h-BN segregation by means of the control of stress in the film. The apparatus has helicon sputtering source for film preparation, which can separates BN target source and alloy source, can control wide range sputtering yield with little damage on the prepared film surface by sputtering plasma field.

Hexagonal boron nitride can be successfully formed on the surface of copper substrate without any in situ annealing different. From the stainless steel case which requires annealing though the h-BN coverage is still about sixty percentage of the substrate. The distribution of h-BN is uniform with auger scanning prove analysis with the probe diameter of less than 1 μ m. This could be the first step to fabricate the h-BN film by means of self organization with stress control of sputtered film.

Keyword : self-organization, surface segregation, co-sputtered film, vacuum tribology, hexagonal boron nitride

148 Fabrication and Characterization of Compond Semiconductor Nanometer Structures

N.Koguchi, Extreme High Vacuum Research Station
[April 1994 to March 1999]

Predictions of enhanced electron mobility device and advanced semiconductor laser with highly-monochromized and low threshold current density caused by quantum size effect have been made for application of semiconductor nm-structures, especially so-called quantum dot structures. It is necessary to fabricate numerous epitaxial semiconductor microcrystals with homogeneous size below 10nm and density above 10^{12} cm^{-2}.

In order to fabricate these structures, we are now researching three methods; (1) self-organized formation by using Droplet Epitaxy, (2) Droplet Epitaxy combined with selective deposition of Ga droplets on a substrate, and (3) microfabrication using metal or semiconductor fine particles deposited on semiconductor quantum wells.

Size and distribution of GaAs microcrystals fabricated by using Droplet Epitaxy correspond to those of Ga droplets deposited on an S-terminated substrate. We have observed the S-terminated GaAs(001) with (2x6) surface reconstruction by STM and found out this reconstruction originating from the cell which contains five S-S adatom dimers and one missing dimer. Size and distribution of Ga droplets were measured as functions of Ga flux and substrate temperature. The selective deposition of arraied Ga droplets using finely focused Ga ion beam was performed on S-terminated GaAs substrate. InAs microcrystals fabricated by Droplet Epitaxy on GaAs/AlGaAs quantum wells were used as etching mask to fabricate vertically coupled quantum dots.

We are trying to investigate quantum size effect of these samples by using photoluminescence and/or cthodeluminescence measurements.

Keywords : quantum dots, compound semiconductor, droplet epitaxy

149 Materials with Atomic Scale Structures(COE Project)

M.Okada, Director-General
[April 1995 to March 2000]

Under its 5-year plan implemented in 1995, National Research Institute for Metals (NRIM) is conducting researches on the creation of advanced materials with atomic-scale structures exhibiting quantum phenomena. This research is promoted as one of the Center of Excellence Development Program (COE Project) of the

Japanese Government, and is financed with the special coordination fund from the Science and Technology Agency (STA).

The main part of our COE Project is progressed at the Center for Advanced Physical Fields(APF) of NRIM, equipped with some stations which provide extreme experimental environments such as high magnetic fields, high resolution beams and extreme high vacuum. The project is in fact designed to make use of the advantages of the these facilities in researches on advanced materials.

The COE Project is roughly divided into two parts. One is "Improvement of techniques to establish extreme physical fields". The techniques provided by the Center for APF are important to carry out research on the creation of materials with atomic scale structure. We call this research "COE supporting research". The other one is "Creation and physical properties of atomic scale structure materials". We call this research "COE core research". Atomic scale structure materials are categorized into quantum dot, quantum wire and thin film. Therefore, we divided "COE core research" into 6 sub projects, which cover quantum dot, quantum wire and thin films. We have created new nano dimension crystals, fabricated single atomic width lines which exhibited electron transport properties, and found interesting quantum phenomena in the high magnetic fields.

Keywords ; advanced physical fields, atomic scale structures, quantum phenomena

150 Study on Melting of Refractory Metals by Cold Crucible Levitation Melting Method

A. Fukuzawa, Materials Creation Research Station
[April 1997 to March 1999]

A cold crucible type levitation melting method using high frequency electric power is known as a non-contacting melting method. Therefore, from the point of view of high pure material creation, this melting method is advantageous for melting of high purity metals, chemically reactive metals and refractory metals. As for the refractory metals, there have never been methods for melting them by using crucible made of common refractories.

The purpose of this study is the creation of molten refractory metals and its alloys by the cold crucible levitation method and the origination of homogeneous solidified structures that have been never gotten by the usual melting methods for refractory metals for instance, electron beam remelting, plasma arc remelting.

As the most of refractory metals have not only high melting point but also high density and high thermoconductivity, it is considered that levitation of refractory metals and its alloys is not easy. So the optimum levitating conditions such as the shape of the cold crucible, high frequency coil and the electric output power are to be examined, and we are gong to design and manufacture trial of the cold crucible devices for this purpose.

Keywords : cold crucible, levitation melting, refractory metal, electromagnetic force

151 Fabrication of particles dispersed materials through controlling of solidification interfaces

A. Sato, Frontier Research Center for Structural Materials
[April 1997 to March 2000]

Metal matrix composites shows higher mechanical properties than the currently available cast materials by the conventional alloying. Powder dispersed materials are one of MMCs and have higher strength and higher wear resistance. The mechanical properties of powder dispersed materials are influenced by the distribution state and the size of the powder.

In this research, the relationship between the distribution and the condition of solidification is investigated. Unidirectional solidification experiments are carried out and the behavior of particle at solidifying interface is investigated.

Powders of pure aluminum and alumina are mixed mechanically in stainless steel pots set on a planetary ball-mill, and pressed and sintered. After sintering, this sample is swaged into a bar with 10 mm diameter. This bar is melted and solidified unidirectionally. Then the microscopic observation is carried out to investigate the behavior of particles at the solidifying interface and the final particle distribution state.

Transparent organic materials are used for model experiment, because metal is not transparent and particle behavior is not directly observed in liquid metal. Succinonitrile is selected as transparent organic material, because its roughness of solid surface is almost same as that of metals. Therefore the shape of solid / liquid interface become planar or cellar or dendritic like metals. The mechanism of engulfment or entrapment of particle is known.

Keywords : solidification, solidification processing, unidirectional solidification, composite, particle dispersion

152 Random Fatigue of Notched Plates

A. Ohta, Strength and Life Evaluation Research Sta-

tion

[April 1997 to March 1998]

The fatigue strength of SM570 steel was investigated at the condition where the maximum stress was kept to be the yield strength of material while the minimum stress changed randomly. This test condition is considered to avoid the complex residual stress inducement at the notch root during variation of stress.

Four types of specimen were used. That is, the stress concentration factors of side notched strips were 1.0, 2.0, 3.0 and 4.0.

The random loading was that corresponding to a Rayleigh distribution of stress ranges.

The applicability of Minner's rule is being examined.

Keywords : fatigue, stress concentration, yield strength, steel

Related Papers

Fatigue Strength of Butt-Welded Joints under Constant Maximum Stress and Random Minimum Stress Conditions, A.Ohta, Y.Maeda and N.Suzuki, Fatigue Fract. Engng. Mater. Struct. 19(1996) 265-275.

Fatigue Crack Propagation in Welded Joints under Random Loadings in the Splash Zone, A.Ohta, Y. Maeda S.Machida and H.Yoshinari, Weld. World 35(1995) 391-394.

153 Mechanical Characterization of Thin Layered Structures

S. Matsuoka, Frontier Research Center for Structural Materials, Strength and Life Evaluation Research Station

[April 1997 to March 2000]

Depth-sensing indentation techniques have been developed to investigate the mechanical properties such as hardness, Young's modulus and yield stress in nano scale. Using the techniques, the international collaborative research on "Mechanical properties of films and coatings" started in 1996 as Task Working Area 22 of VAMAS projects.

The purpose of the study on "Mechanical Characterization of Thin Layered Structures" is to develop the nanoindentation techniques for thin films and to contribute to the VAMAS projects. Fig. 1 show the primary results. The indentation was conducted on aluminum thin films with thicknesses of 0.3, 1.0 and 3.0 μm on sapphire substrates, using an ultra-micro hardness tester we have developed. Relationships between maximum load, P_{max}, and maximum penetration depth,

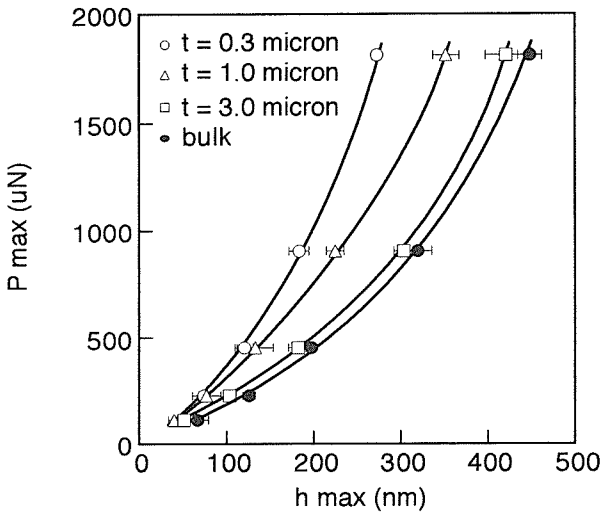

Fig. 1 Relationships between maximum indentation force and maximum penetration depth for Al thin films.

h max, for three aluminum films and bulk aluminum were almost coincident each other when h_{max} was smaller than 100 nm. This suggest that the resonable results of indentation can be obtained even for 0.3-μm-thick aluminum thin film by our AFM ultra-micro hardness tester.

Keywords : Depth-sensing nanoindentation, Hardness, Young's modulus, Yield stress, Thin films, VAMAS project

154 Study on Strengthening of Ferrite Matrix Steels for Welded Structures

K.Nagai, Frontier Research Center for Structural Materials

[April 1997 to March 2002]

Building steel is one of major mass-produced items in the steel market. Easier recyclability, more efficient and less skilled welding and enhanced mechanical properties will be required for the building steels in the next century to construct sustainable infrastructure. However, present high strength steels of 600-800 MPa in tensile strength contain the alloying elements undesirable for recycle, and their welded joints show very poor fatigue strength, i.e. only one-tenth of tensile strength.

Low strength C-Si-Mn ferritic steels are potential alternatives, since they are desirable for recycle and easy to weld. To overcome the low strength, in the present study, new thermomechanical processing will be developed to refine the ferrite grain size into 1 μm and double the tensile strength from 400 MPa to 800 MPa. The process parameters to be optimized are forming modes, deformation temperature, deformation rate, amount of deformation, dwelling temperature and time,

start temperature to cool , and cooling rate. The microstructure to be processed is also varied.

High speed and no defect welding has to be developed coincident with preserving the ultra-fine microstructure from its heat in the heat-affected-zone (HAZ). Hence, the feedback controlling of the plasma generation in a high-power CO_2 laser one-pass welding and the arc pulses in a multi-layer arc welding will be elucidated.

In order to keep the ultra-fine grains in the HAZ on welding process, the function of finely-dispersed particles like oxides will be investigated in terms of pinning sites for grain growth as well as nucleation sites for austenite to ferrite transformation.

Welded specimen large enough to simulate the conditions for service will be subjected to mechanical tests with a large capacity of power to evaluate fatigue and fracture toughness properties.

Computer simulation of the microstructure evolution in the steels studied and the deformation and fracture behavior of welded points will be also studied in the present study.

Keywords : ferritic steels for welded structures, thermomechanical treatment, grain refinement, welding process, heat-affected-zone, residual stress, welded joint, tensile strength, fatigue strength, simulation

155 Advanced Ultra-High-Strength Steels over 1500 MPa class

S. Matsuoka, Frontier Research Center for Structural Materials, Strength and Life Evaluation Research Station

[April 1997 to March 2001]

Demands for ultra-high-strength steel with tensile strengths in excess of 1500 MPa are increasing in such fields as use of high strength bolts as a new joining technology in building construction and as reducing the weight of automobile parts and of main cables of long-span suspension bridges. The key of the exploitation does not lie in the achievement of high strengths exceeding 1500 MPa but in the preservation of high confidence for delayed fracture and fatigue.

As advanced ultra high strength steels we are developing new martensitic steels with effective hydrogen trap sites and carbide-free boundaries for high delayed fracture resistance and ultra-low carbon high nitrogen martensitic steels for high fatigue strength. Atomic scale analyses of fine precipitates and interphase boundaries by AP-FIM and nanoscopic observation of deformation and fracture structures by STM and AFM are being used in this study, which allow us to clarify the mechanisms of delayed fracture and fatigue and to propose new concepts of material design. Efforts will also be directed for standardizing the evaluation of delayed fracture resistance and constructing database of 10^{10} cycles fatigue properties for the purpose of guaranteeing the long term reliability.

Keywords : martensitic steel, delayed fracture, fatigue, AP-FIM, SPM

156 Strategic Research on Advanced Ferritic Steels for 650 ℃ USC Boilers (R&D of Structural Materials for 21st Century)

Fujio Abe, Strength and Life Evaluation Research Station

[April 1997 to March 2002]

There are strong environmental and ecomonic pressures to increase the thermal efficiency of coal fired electric power plants, leading to recent increases in steam temperature and pressure. The thermal efficiency is evaluated to increase to 43.8% for the ultra-supercritical (USC) steam conditions of 650℃ and 350 atm from 38.9% for the present supercritical steam conditions of 538℃ and 246 atm. The enhanced thermal efficiency results in lower carbon dioxide emissions and lower fuel cost. The environmental reason is increasingly quoted as the driving force for the development of higher efficiency plants. For construction of large USC plant suitable for cyclic operation, it is necessary to develop advanced ferritic heat resisting steels, because austenitic steels are highly susceptible to thermal fatigue resulting from their low thermal conductivity and high thermal expansion. The major limiting factors to an increase in steam temperature above 620℃ for thick section boiler components such as main steam pipe and header are the availability of ferritic steels with sufficient creep strength and sufficient resistance to steam oxidation. The purpose of the present research is to investigate strategically the advanced ferritic heat resisting steels for application to thick section boiler components of USC plant at 650℃ and 350 atm.

The project consists of five sub-themes.

Sub-theme 1 - Alloy design and materials creation of high-Cr advanced ferritic steels
Sub-theme 2 - High-temperature mechanical properties of welded joints
Sub-theme 3 - Resistance to steam oxidation
Sub-theme 4 - Creep strength properties
Sub-theme 5 - Fatigue properties

In the Sub-theme 1, alloy designing of high-Cr ferritic steels toward a stabilization of tempered martensitic microstructure for long time at high temperature is carried out using thermodynamic codes such as

Thermo-Calc and DICTRA. Optimization of the distributions of lath subgrains and carbides in the tempered martensite is tried by multiple mode TMCP. In the Sub-theme 2, optimization of HAZ microstructure as well as the creep damage process and creep crack growth behavior in welded joints are examined for high-Cr ferritic steels. In the Sub-themes 3 to 5, relating to materials evaluation, reliable methodology for accelerated evaluation based on short term tests and prediction of long term properties will be established.

Keywords : ferritic heat resisting steel, ultra-supercritical power plant, alloy designing, creep, steam oxidation, fatigue, welded joint

157 Enhanced Durability of Structural Steels in Marine Environment

Toshiaki Kodama, Director of Special Research
[April 1997 to March 2001]

The construction of new steel structures is expanding from densely populated coastal zones to marine and offshore environments in such forms as huge infrastructures and oil well platforms. Marine environment, however, gives very aggressive conditions to metals and steels. Although the effectiveness of low-alloy weathering steels is well established in rural, urban and industrial atmospheres, they show poor performance in marine environment. Even stainless steels suffer in the presence of chloride ion from various types of localized corrosion such as stress corrosion cracking, pitting and crevice corrosion. The present study covers the development of low- and high-alloy steels and coating technology resistant to marine corrosion, basic researches using modern microprobe technology and construction of corrosion database.

In marine environment, however, rust on conventional weathering steel does not stabilize itself. In advance to the alloy design, it is thought inevitable to collect basic data on the long-term stabilization of rust formed in marine environment. Bearing this line in mind and for clarifying the stabilization process of rust we will start new exposure program of marine corrosion using model steels of simplified compositions of primary additive elements of weathering steels. The program is carried out in cooperation with outside organizations. The data obtained will be compiled in the form of atmospheric corrosion database, which is expected to contribute for designing new weathering steels. In parallel with data work, detailed analysis of actual and synthetic rust will be carried out, where microprobe technology will be effectively applied.

For the development of high alloy steels we focus the target on the increased addition of nitrogen and simultaneous removal of detrimental elements such as S, P and C in stainless steels. Nitrogen in stainless steels is effective in not only improving corrosion resistance but in reducing cost by saving resources of rare metals such as Cr and Mo. For massive addition of nitrogen pressurized melting with simultaneous refining is to be employed.

Among various methods of coating technology we have chosen spray coating for the protection of steels against marine corrosion partly because in NRIM we have accumulation of plasma spray technology and partly because spray coating is deemed unavoidable for the on-site repair of aged structures. For the formation of barrier coating consisting of corrosion resistant alloys we will develop high velocity oxy-fuel thermal gas spray technique.

Keywords : weathering steel, stainless steel, marine environment, spray coating, scull melting

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❐ NRIM Publications (Apr.1996 to Mar.1997)

1. Bulletin of National Research Institute for Metals, in Japanese.
   No.19(Mar.1997)
2. Annual Report of National Research Institute for Metals, in Japanese.
   for fiscal year of 1995(Mar.1997)
3. Kinzaigiken News' in Japanese. No.4to12(1996)and No.1to3(1997).
4. NRIM Research Activities. in English (Mar.1997)
5. Material Strength Data Sheet, in English.
   NRIM Creep Data Sheet,
   No.43(Sep.1996) No.11B and 44(Mar.1997)
   NRIM Material Strength Data Sheet Technical Document. in Japanese No.15(Mar.1997)
6. Guide to National Research Institute for Metals. in Japanese and in English. for fiscal year of 1996

International Cooperation

Apr 1996 to Mar. 1997

❒ International Collaboration Theme

Australia

1. Studies on conductor fabrication Processes of high-TCP BiSrCaCuO superconductors(University of Wollongong)
2. A comparison of high energy density beam and arc welding technique for joining advanced materials of both the metal-matrix composite and intermetallic compound types(CSIRO)
3. Process technology and characterization of advanced metal matrix Composites(CSIRO)

China

1. Investigation of high temperature Titanium alloy for application over 600℃(Northwest Institute for Non-Ferrous Metal Research)
2. Studies on structural control and superconducting properties of high Temperature superconductors (Institute of Metal Research. Chinese Academy of Science)
3. Fundamental study on the improvement of superconductivity for high-Tc oxides(Northwest Institute for Non-Ferrous Metal Research)
4. Studies of local corrosion damage of corrosion resistant alloy in high temperature aqueous solution(Shanghai Jiaotong University)
5. Study on the improvement of brittleness of Ni_3Al and NiAl by unidirectional solidification(Institute of Metal Research, Chinese Academy of Science)
6. Environmental life cycle analysis of materials (Lanzhou University)
7. Study of TiAl base alloy(Northwest Institute for Non-Ferrous Metal Research)
8. Fundamental research on TiA1 base intermetallic compounds(University of Science and Technology, Beijing)
9. Studies on crevice corrosion of low alloy pressure vessel steels in high temperature aqueous solution(Institute of Corrosion and Protection of Metals)

France

1. Superconducting and cryogenic magnetic materials (Service National des Champus Intenses, CNRS)
2. Mechanical properties such as fatigue and fracture for short fiber reinforced metal matrix composites (INSA Lyon, ENSMP, CNRS)
3. Physical properties of oxide superconductors and highly correlated electron systems(Service National des Champs Intenses, CNRS)
4. Design of advanced materials(CNRS)

Germany

1. High peformance superconducting Materials (Forschungs zentrum Karlsruhe)
2. Information and documentation panel numerical data (Heinrich fachinformations Zentrum Karlsruhe)
3. Designing of new continuous refining process(Haus der Techink e.v. Essen)

India

1. Studies on fabrication processes for superconducting oxide conductors(National Physical Laboratory)
2. Atmospheric corrosion studies in relation to the effect of microenvironment on metals and alloys during early stages of exposure(National Metallurgical Laboratory)
3. Studies of quasi-crystalline based composites(Indian Institute of Science)

Italy

1. Superconducting properties of advanced superconductors in time-varying magnetic fields(CISE SPA, Technologia Innovative Thermophysics & Cryogenics Sec.)
2. Intercomparison of methods and materials for strain measurements at cryogenic temperatures(Instituto di metrogia "G. Colonnetti" -C.N.R)
3. Metallic materials(Instituto per la Technologia dei Materiali Metalici Non Tradizionzil)

Korea

1. Development of the aluminum base intermetallic compounds for structural application at high temperature(Korea Institute of Machinery and Materials)
2. Performance characterization of materials at high temperature(Korea Research Institute of Standards and Science)
3. Development of metallic superconducting materials(Korea Research Institute of Standard Science)
4. Thermoelectrics research for advanced intermatallic compounds(Rapidly Solidified Materials Research Center)
5. Evaluation of the high temperature properties for titanium-based particulate Composites(Korea Advanced Institute of Science and Technology)
6. Studies on the fabrication of B_i-2223 superconduct-

ing wire and its application(Korea Institute of Machinery and Metals)

Poland

1. Magnetization and magnet-optics in ddiluted magnetic semiconductors in high magnetic fields(Polish Academy of Science)

Russia

1. Research on materials property data modeling (Institute Inorganic Chemistry, Academy of Sciences)
2. Water purification through adsorptive dispersed electrochemical system (Frumkin Institute of Electrochemistry)
3. Eco-balance analysis in life cycle of materials (Russian Research Center for standardization, Information and Certification of Materials)
4. Study on electron transport through atomic scale tunnel junctions(Novosibirsk State University)

Sweden

1. Fabrication and characterization of semiconductor quantum dots(Lund University)

U. K.

1. Prediction technology of life and renraining life of huge structures under service condition and its application to design(The Welding Institute)

U. S. A.

1. Research and development on systems and materials for magnetic refrigeration(Francis Bitter National Magnet Laboratory, MIT)
2. Databases on high temperature superconducting materials(National Institute of Standards and Technology)
3. Studies of high-strength/high-conductive materials and their application to high-field magnets(Francis Bitter National Magnet Laboratory, MIT)
4. Fundamental studies on the conductor fabrication of high temperature oxide superconductors(National High Magnetic Field Laboratory)
5. Measurement and evaluation methods for superconducting properties(National Institute of Standards and Technology)
6. Developments and applications of extremely high-field magnets and magnet systems(National High Magnetic Field Laboratory)
7. Study of nano-composites magnetic materials for cryogenics(National Institute of Standards and Technology)
8. Joint Research on the "in-situ" analysis/evaluation of atomic and microstructural changes in materials (Argonne National Laboratory)
9. Fundamental studies of vortex state in high Tc superconductors(Argonne National Laboratory)
10. High pressure research on strongly correlated electron systems(University of California)
11. Effect of high magnetic field on solid/solid phase transformations(Northwestern University)
12. Studies on mechanisms of nanoscale microstructural evolution in advance metallic materials (University of Virginia)
13. Study on the mechanical properties of directionally solidified intermetallic compounds(Oak Ridge National Laboratory)
14. Evaluation of thick coatings formed by advanced thermal spray processes (New York State University)
15. Research on superalloys(Oak Ridge National Laboratory)

❒ List of Visiting Foreign Researchers

*STA Fellows

Country and Name	Affiliation	Term	Research Subject
Australia			
Xuezhi Zhang*	Ceramic Fuel Cells Limited	1996. 5.24 - 1998. 5.23	Nucleation process control of undeocooled Fe-Cr-Ni & Cr-Ni alloys
Yuan Chang Guo	Supercinducting Electronic materials Center	1996. 5.27 - 1996. 5.31	Development of silver-sheathed Bi-oxide superconducting wires
Roland Grossinger	Vienna University	1997. 2.10 - 1997. 2.23	Magnetic materials development
Bulgaria			
Krassimi r Nedialkov Stoev*	Institute on Nuclear Research and Nuclear Energy, Bulgarian Academy of Sciences	1997. 1. 6 - 1997. 4. 5	Characterization of thin films using X-ray total reflection
Brazil			
Eduardo Albertin	Institute for Technological Ressearch of the State of Sao Paulo	1997. 3. 3 - 1997. 4.10	Directional Solidification Technology
Reiko Sato	Vienna University	1997. 2.10 - 1997. 2.23	Nano-magnetic materials development
Camada			
Dancan Rogers*	University of Toronto	1996.12. 2 - 1998.12. 1	Fabncatlon of atomic scale tunnel junction with a scanning tunneling microscope
China			
Liu Yufu	Japan Science and Technology Corporation	1996. 4. 1 - 1999. 3.31	Experimental and analytical study on thermal-fatigue of SiC fiber-reinforced Titanium matrix composites
Fuxing YIN	Japan Science and Technology Corporation	1996.10. 1 - 1999. 9.30	Characterization of the damping mechanism In MuCu based damping alloys and development of advanced damping materials
Geng Tu	Japan Science and Technology Corporation	1996.10. 1 - 1999. 9.30	Molecular dynamics study on the embritllement of Alcausedby grain boundary segregation of impurities
Yu Jiakang	Institute of Corrosion and Protection of Metals	1996.10.21 - 1997. 3.31	A Microscopic Study on Initiation of Corrosion Pit by using AFM in Aqueous Solution
Xu Xuelian	Shanghai Nuclear Engineering Research and Design Institute	1996.11. 4 - 1997. 3.31	Low Cycle Fatigue Behavior of SUS316 Stainless Steel in High Temperature Water
Xu Jincheng	Institute of Materials Science and Engineering of Lanzhou University	1996.11.16 - 1996.12.13	Environmental Life Cycle Analysis of Metals
Tang XingGui	Beijing Institute of Nuclear Engineering	1996.12.20 - 1997. 3.31	Fundamental study of atomic structural analysis of radiation damage for nuclear materials under electron and ion irradiation
Wang Tian Hin	Institute of Materials Science and Engineering of Lanzhou University	1997. 1.10 - 1997. 2. 1	Environmental Life Cycle Analysis of materials
France			
Claude Landron	Centre National De La Recherche Scientifique Centre de Recherche sur la Physique des Hautes Temperature	1997. 1.21 - 1997. 3.30	Structural determination of High temperature materials by X-ray spectroscopic techniques
Gerard Guenin		1997. 2.17 - 1997. 3. 1	
Bernard Chenevier*	Laboratoire des Materiaux et du Genie Physlque, ENSPG, CNRS	1997. 2.24 - 1997. 3.23	Study on superconducting properties and microstructure for YBCO films prepared with modified CVD and sputtering process
Stephane Odasso	National research Institute of Crystallography	1997. 3. 3 - 1997. 3.13	Pd ad sorption on Si(100) surface using synchrotron radiation
Germany			
Frank If. Zimmermann	Department of Physics & Astronomy Rutgers Univ.	1996.12. 7 - 1996.12.28	Experimental and theoretical study on surface chemical reaction dynamics
Hnas. J Steffen	Munchen Institute of Technology	1996.12.15 - 1996.12.22	Research on the depth profiling analysis technique
Christoph Buchal	Research center of Joelich GmbH	1996.10. 3 - 1996.10.11	Optical Materials and devices fabricated by ion implantation
Richard Berndt	Aachen University	1996. 8.20 - 1996. 8.29	Atomic-resolution photon scanning tunneling microscopy from Au(110) and fullerene molecue

Country and Name	Affiliation	Term	Research Subject
Andreas Erb	Geveva University	1997. 2.23 - 1997. 2.27	Sample preparation of highly correlated electron systems
Gerd Schoen	Karsluhe university	1997. 3. 9 - 1997. 3.15	Theory of single electron transistor and coherent tunneling
Horst Vehoff	Saalandes University	1997. 3.24 - 1997. 3.30	Material Science and Methods
Italy			
Sergio Zannella	CISE Spa-Segrate, Italy	1997. 3. 2 - 1997. 3. 9	Studies on measuring system of superconductivity
India			
Gunadhor Singh Okram*	Center of Nuclear Science, India	1996.12. 2 - 1998.12. 1	Magnetic properties of Oxide Superconductors
Japan			
Yoichi Matsumiya	Brown University	1997. 3.24 - 1997. 3.29	Studies on measuring system for super conductivity
Korea			
Chae Deok Lee	Korea Research Institute for Standards	1996. 5.13 - 1996. 8. 8	Fabrication of Compound Semiconductor Quantum Dots
Ja-Yong Koo	Korea Research Institute for Standards	1996. 5.13 - 1996. 8. 8	Nano-indentation for the elastic deformation region of metals
Lim Cha-Yong	Korea Institute of Machinery and Materials	1996.10.21 - 1997.10.20	Study on Recyclable Materials Design for Aluminum Alloys and Steels
Chang Soo KIH*	Korea Research Institute of Standards and Science	1996.11.18 - 1996.12.17	A study of texture control of Bi2212/Ag high-Tc superconducting composite tapes
Han Beom-suck	Korea Automotive Technology Institute (KATECH)	1996.11.28 - 1997.11.27	Experimental Studies on Fabrication of Partially Composite Materials by
Oh Yun-Jeong	Department of Physics, Cheju National Univ.	1997. 1.16 - 1997. 2.28	Specimen preparation and FIB microfablication of $TiSi_2$ and $CoSi_2$ thin film
Choi Yi Sik	School of Materials Science & Engineering, Seoul National LJniv.	1997. 1.16 - 1997. 2.28	1/Microstructure of coldworked cast alloys 2/Rapid and/or undercooled solidification in Al-Si-X alloys
Park Kyu Ho	Department of Materials Science & Engineering, Korea Advanced Institute of Sci. and Tech.	1997. 1.16 - 1997. 2.28	Observation by TEM of microstructural changes by spinodal decomposition in Fe-Ni(Mu)-Al-C Alloys
Joo Sung Min	Dept. of Metallurgical Engineering Chonnam National University	1995. 1.16 - 1997. 2.28	Combustion Synthesis(SHS) of Intermetallic Compounds
Hyo-Sook Lee	Korea Institute of Geology Mining and Materials	1997. 2. 3 - 1997. 2.15	Studies on Chemical Stabilities of Iron-Nitride Magnetic Fluid
Kyung Youl Min	Memory Lab. , Hundai Electric Co.	1997. 2.12 - 1997. 2.15	Ion beam Assisted surface modification on BN film
Kyung Sub Lee	Sungkyunkwan University	1997. 3.12 - 1997. 3.18	Study on the mechanical and chemical properties of segregated hexagonal BN layer
Dong Lak Kim	Korea Basic Science Inst.	1997. 3. 3 - 1997. 3.12	Development of detection systems in high magnetic fields
Poland			
Grzegorz Karczewski	Institute of Physics, Polish Academy	1997. 2.18 - 1997. 3. 3	Semimagnetic semiconducting materials
Russia			
Yurii Michailovich Gufan*	Institute of Theoretical Physics Russian Academy of Science	1996.11. 8 - 1996.12.23	A Study of relation between cation ordering and superconducting state in oxide superconducting films
Igor Yu Sokolov	Tronto University	1996. 8.20 - 1996. 8.24	Theoretical investigation of electron in tunnel barrier
Slovakia			
Milan Polak	Institute of Electritical Engineering Slovak Academy of Science	1997. 2.18 - 1997. 3. 3	Characterization of interfaces on the atomic monolayer scale
Spain			
Hang Ping Miao*	Liniversity of Zaragoza, Spain	1997. 2.17 - 1998. 8.16	Effect of Secondary Phase on the Transport Property and Uniformity in BSCCO/Ag Composite Superconducting Wires and Tapes
Switzerland			
Stanislav Veprek	Technical LIniversity of Munich	1997. 3. 7 - 1997. 3.27	Study on plasma chemistry and nanocrystalline composites
LJrs Ramsperger*	Institut fur Mikrostrukturforschung, ETH	1997. 3.17 - 1999. 3.16	Fabrication of atomically thin metal wire with a scanning tunneling microscope

Country and Name	Affiliation	Term	Research Subject
United Kingdam			
Howard Graham Read*	Japan Science and Technology Corporation	1996. 4. 1 - 1997. 2.28	Studies of microstructures in nanostructured metallic materials
Christopher John Haworth*	University of Bristol, Low temperature physics, specializing in superconductivity	1996. 4. 1 - 1997. 3.31	Electronic Properties of Low Dime nsinal and Mesoscopic Systems in High Magnetic
Christopher Hole*	Clarendon laboratory University of Oxford	1996. 8. 8 - 1997. 4. 7	Research on high-magnetic field properties of superconducting wires
Ian J.Davies	National Aerospace Laboratory	1999. 9. 1 - 1997. 9.30	Observation of microstructures for ceramic matrix composites using a transmission electron microscop
Cameron W. McLeod	Center for Analytical Sciences, Department of Earth Sciences, The University of Sheffield	1996. 9. 2 - 1996. 9. 5	Hicrocolumn Field Sampling for Determination and Speciation of Organotin in Seawater
Glen D. Woods	Center for Analytical Sciences, Department of Earth Sciences, The University of Sheffield	1996. 9. 2 - 1996. 9. 5	Hicrocolumn Field Sampling for Determination and Speciation of Organotin in Seawater
Robert John Stephens on*	University of Cambridge	1997. 1. 6 - 1997. 4. 5	Measurement at optical property of atomic scale tunnel junction with a scanning near-field optical microscope
Patrick S. Grant	Oxford University	1997. 1.10 - 1997. 1.19	Development of higher perfonnance of energy conversion materials by Research on Strong magnetic field
Harry Jones	Oxford University	1996.12. 9 - 1996.12.19	Research on Strong magnetic field
Stephen Olof Hill	National High Field Magnet Laboratory	1996.12.15 - 1996.12.17	Quantum Hall effect in organic conductors
John Singleton	Oxford University	1997. 1. 6 - 1997. 1.17	Composite Fermion
Philip Gee	Oxford University	1997. 1. 6 - 1997. 1.10	Composite Fermion
Mark Edward Welland	Cambridge University	1997. 3. 5 - 1997. 3.11	Photon emission from nanometer scale structure and organic molecules
Laurence Marks	Northwestern University	1997. 3.23 - 1997. 3.31	TEM study of reconstructed surface in UHV condition
Steve Donnelly	University of Salford	1997. 3. 2 - 1997. 3.14	In-situ study of ion implanted 3D-nanocrystals under irradiation functionally graded structures
Ukraine			
Alexander V. Kra jnikov	Institute of Materials Science Problems National Acadcmy of Science of Ukraine	1996.11. 4 - 1996.11.24	Study for non-destructive control of intergranular embrittlement in advanced high temperature materials based on simulation of impurity segregation
United States			
Reynolds, William T, J r.	University of Virginia	1996. 8. 1 - 1997. 7.31	The influence of interfacial chemistry on Precipitate Growth and Stability
Xuan-Chau Kou	Technical University of Vienna	1996. 8.22 - 1996.10.20	Study of magnetic anisotropy in rare-earth compounds
Scott J. Welton	National High Magnetic Field Laboratory	1996.11. 4 - 1996.11.17	Development of Essential Technology and Designing New Water Cooled Magnet
Mark D. Bird	National High Magnetic Field Laboratory	1996.11.17 - 1996.11.24	Development of high-performance resistive magnets
Gregory Blues Olson	Northwestern University	1997. 1.26 - 1997. 2. 8	Effects of high magnetic field on phase transformations
Chen Ho Sou	Lucent Technologies, Bell Laboratories	1997. 1.28 - 1997. 2.26	Mechanical properties of ultra high strength Al-based nano-meso scopic materials
Easo P. George	Oak Ridge National Laboratory	1997. 2. 6 - 1997. 3. 3	Study on the Mechanical Properties of Directionally Solidified Intermetallic Compounds
Gautam Chosh	Northwestern University	1997. 2.16 - 1997. 3. 8	Effects of high magnetic field on solid/solid phase transformations
Huub Weijers	National High Magnetic Field Laboratory	1997. 2.24 - 1997. 3.17	Evaluation of Bi-based oxide superconducting tapes and coils
Charls W. Allen	Argonne National Laboratory	1997. 3. 2 - 1997. 3.22	Research on the structure and properties of implanted and precipitated semiconductor nanocrystals
Charls W. Allen	Argonne National Laboratory	1997. 3. 2 - 1997. 3.22	Research on the structure and properties of implanted and precipitated semiconductor nanocrystals
Robert C. Birtcher	Argonne National Labo ratory	1997. 3. 5 - 1997. 3.25	Research on the structure and properties of implanted and precipitated semiconductor nanocrystals

Country and Name	Affiliation	Term	Research Subject
James If. Howe	Virginia University	1997. 3. 8 - 1997. 3.16	Subnanoscale microstructural characterizations of advanced metallic materials
William C. Jhonson	Virginia University	1997. 3. 8 - 1997. 3. 16	Subnanoscale microstructural characterizations of advanced metallic materials
Robert S. Feigelson	Center of Materials Research, Stanford University	1997. 3.10 - 1997. 3.20	Crystal-quality improvement of nonlinear-optical oxides by Floating zone method
Christopher C. Berndt	University of New York	1997. 3.20 - 1997. 3.29	Evaluation of thick coatings formed by advanced thermal spray processes
Sanjay Sampath	University of New York	1995. 3.20 - 1997. 3.29	Evaluation of thick coatings formed by advanced thermal spray processes
James Brooks	National High Field Magnet Laboratory	1996.12. 5 - 1999.12.30	Fermi Surface of Organic conductors

❒ List of Visitors

T:Tsukuba Site M:Meguro

Nationality and Name	Affiliation	Site	Date
U.S.A			
Mr.Rick Savercool his party	SafetyManager,GA	T	Jun.1996
Jean-Louis Desplat his party	US Department of Energy	T	Oct.1996
Oles Romacky	International Science and Technology	T	Nov.1996
China			
Zheng Yi Fu	Systhesis and Processing, Wuhan Univ.	T	Oct.1996
Prof.Fang Xiangwei	Shanghai Research Institute of Materials	T	Dec.1996
Korea			
Prof.Wang Tianmin	Lonzhou Univ.	T	Jan.1997
Kim Kwan Hyu	Chonnam National Univ.	T	Jan.1997
Germany			
M.Alaya His party	Univ.ofKarlsruhe	T	Aug.1996
France			
Boussa Djaffar	CNRS,LMA	T	Oct.1996
Dr.Gera1dBastard	Laboratorie de Physique de la Matiere Condensee de l'recoleNormale Superieure	T	Dec.1996
U.K.			
Prof.Richard Donovan Kenway	The University of Edinburgh	T	Nov.1996
Switzerland			
Stephan Bieri	The Board of the Swiss Federal Institute of Technology	T	Feb.1997
Indonesia			
Mr.Soehartono SOEDARGO	UREAUSCIENCE&TECHNOLOGY	T	Nov.1996
Australia			
Jim I.M. Low	Curtin Univ. of Technology	T	Oct.1996
Ms.Rosiyn JORGENSEN Her party	The Australian National University	T	Nov.1996
Canada			
Dr.CliveWillis	National Research Council Canada	T	Sep.1996
Basil R.Marple	National Research Council Canada	T	Oct.1996
Austria			
Heinz E. Pettermann	Univ. of Copenhagen Vienna University of Technology	T	Oct.1996
Israel			
UriAdomon	Ben-Gurion Univ.	T	Oct.1996
Finland			
Michael M.Gasik His party	Helsinki University of Technology	T	Oct.1996
Denmark			
Preden J. moller	Univ. of Copenhagen	T	Oct.1996
Czecho			
Vladimir ClHAL	Technical Univ. of Ostrava	T	Jan.1997

❒ Brief Introduction of STA Fellowship Program

In response to growing calls from the international community for greater international cooperation the STA Fellowship program was established to provide foreign researchers with opportunities to conduct research at Japanese national laboratories and public corporations which are not part of the university system.

The program is managed by the Japan Science and Technology Corporation (JST) Which is one of the key organizations for implementing policies of the Science and Technology Agency(STA) in cooperation with the Japan International Science and Technology Exchange Center(JISTEO. Fellowship qualifications are as follows :

1. Possession of Ph D. or equivalent qualifications.
2. Long term fellowship is less than 35 years of age. Short term fellowship is no limitation of age.
3. Good health to do research-work and live in Japan
4. Skillful language of Japanese or English words.

The tenure is from 6 months to 2 years(long term Fellowships), or 1-3 months (short term Fellowships).

JST provides fellowships with a round-trip airline ticket, a monthly living expenses, a family allowance, an initial international moving allowance, accommodations(1cng term fellowships), accommodations allowance (short term fellowships).

And to the long term fellowships it is paid the travel cost in Japan related to research activities but it is not applied to the short-term fellowships. Research expenses will be paid to the host institutes.

Further information can be obtained at JISTEC:

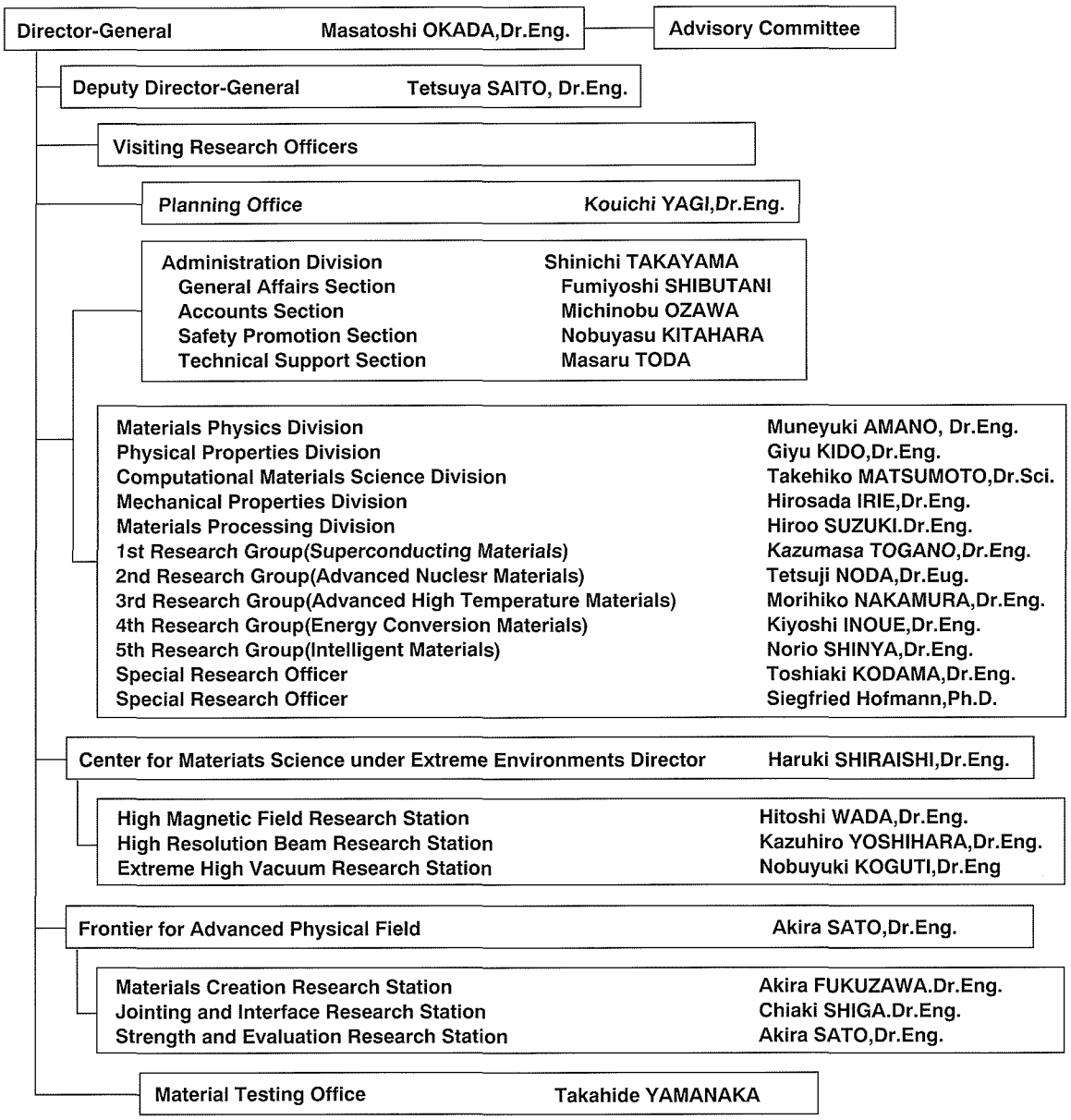
2-20-5, Takezono, Tsukuba City, Ibaraki Pref. 305, Japan.

Phone +81-298-53-8250

Fax +81-298-53-8260

Organization of NRIM

❒ Organization



❒ Budget and Personnel in Fiscal Year of 1997

Budget	
Research and facilities	7,074
Personnel expenses	3,758
Total	10,832

unit: million yen

Personnel	
Administrative staffs	90 (7)
Researchers	323 (7)
Total	413 (14)

Number in parenthesis: Material Testing Office

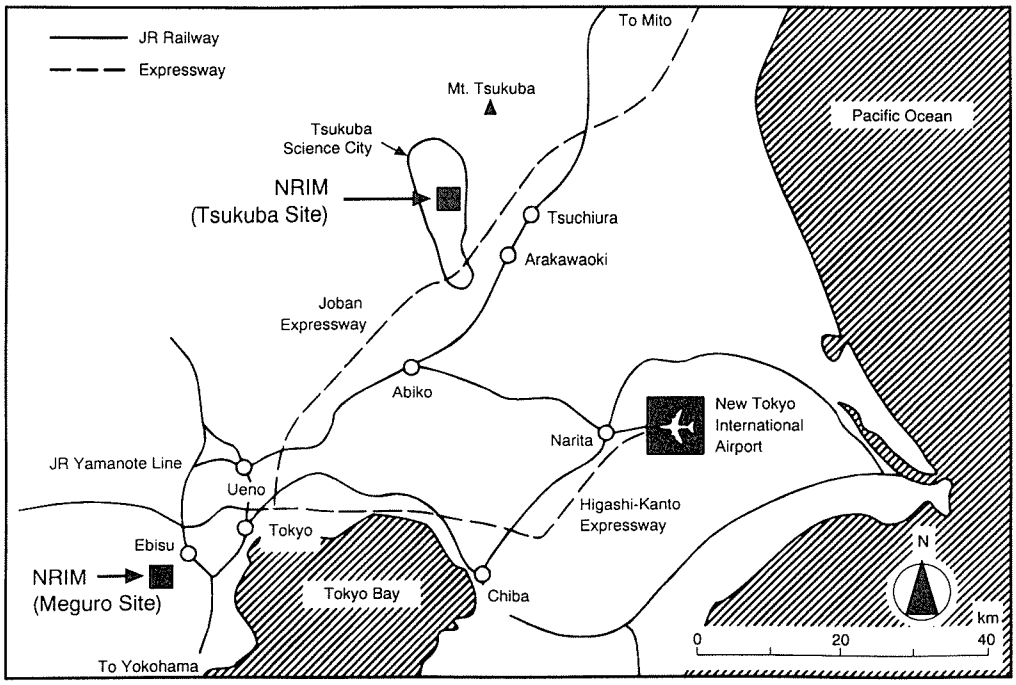
How to get to NRIM

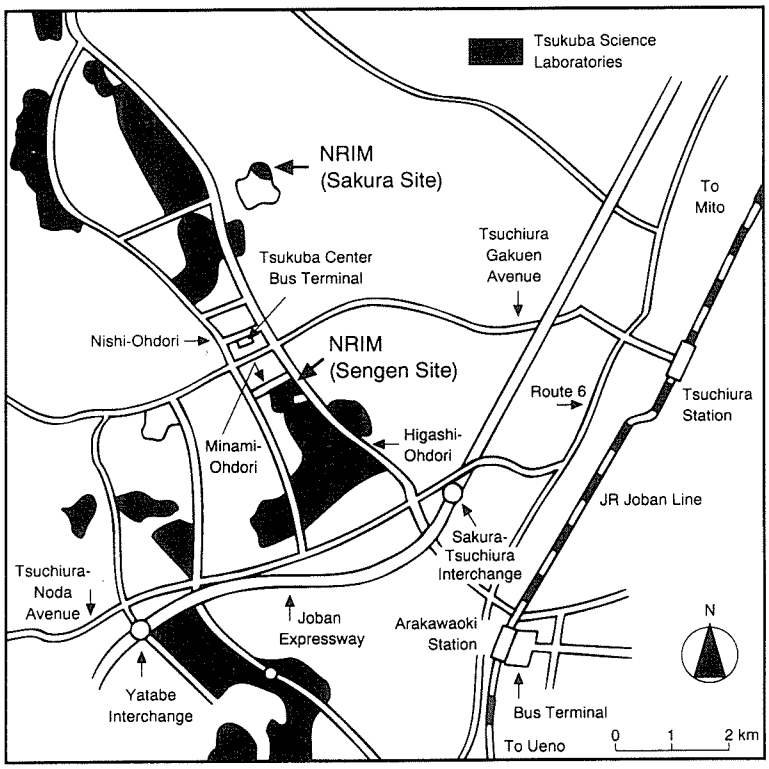
To NRIM Tsukuba Site
1-2-1 Sengen, Tsukuba-shi, Ibaraki 305
Phone +81-298-53-1000, Fax +81-298-53-1005

Narita Airport
↓ by JR Narita Express train approxmately 60 minutes
Tokyo Station
Yaesu Minami Exit
↓ by Joban Highway Bus for Tsukuba Center approximately 70 minutes
Tsukuba Center
Bus Terminal
↓ by taxi approximately 5 minutes, or on foot approximately 15 minutes
NRIM

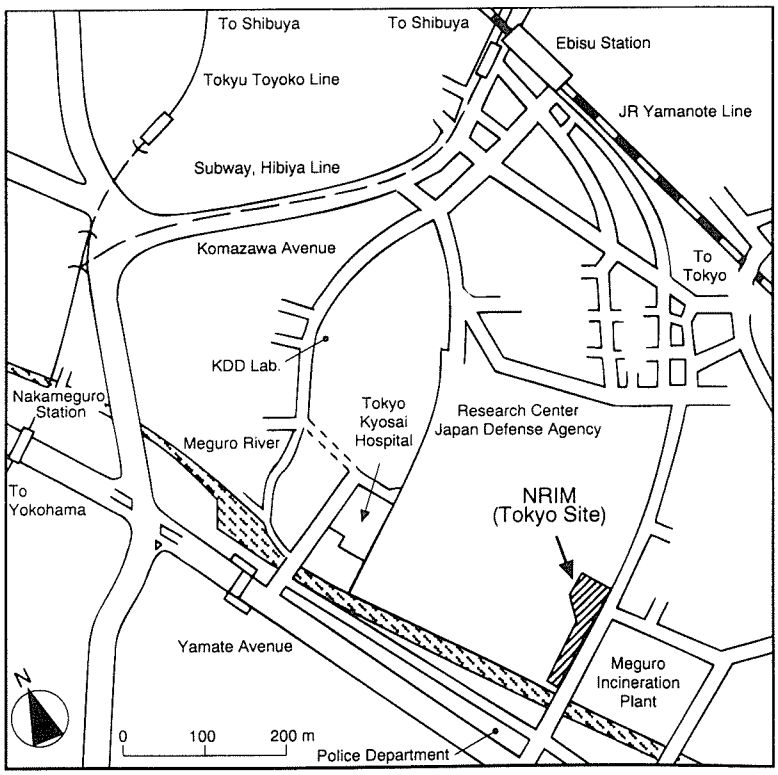
To NRIM Meguro Site
2-2-54 Nakameguro, Meguro-ku, Tokyo 153
Phone +81-3-3719-2271, Fax +81-3-3719-2177

Narita Airport
↓ by JR Narita Express train approxmately 60 minutes
Tokyo Station
↓ by JR Yamanote Line train approximately 25 minutes
Ebisu Station
↓ by taxi approximately 5 minutes, or on foot approximately 15 minutes
NRIM





Tsukuba Site



Tokyo Site

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