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Mechanical Strength Enhancement of the Internal Matrix Reinforced Nb_3Sn Multifilamentary Wires Using Cu-Sn-In Ternary Bronze Alloy Matrices by HIP Processing

Y. Hishinuma, H. Oguro, H. Noto, H. Yata, H. Taniguchi, S. Awaji and A. Kikuchi

***Abstract*—We are approaching to high mechanical strength of bronze processed Nb_3Sn wire by internal matrix reinforcement using Cu-Sn-In ternary alloy. The In solute element remained homogeneously in the wire matrix after Nb_3Sn synthesis and contributed to transform (Cu, In) solid solutions. (Cu, In) solid solution became as reinforce material of Nb_3Sn phase and acted to improve the mechanical strength of the Nb_3Sn wire. In this study, we investigated the effect of Hot Isostatic Press (HIP) treatment to the further mechanical strength improvement of the internal matrix reinforced Nb_3Sn wire. Tensile stress and strain sensitivities of transport critical current (I_c) property were enhanced by HIP processing because of the drastic reduction of micro-sized vacancies into the transformed (Cu, In) matrix. We found the HIP treatment contributes to mechanical strength enhancement of the internal matrix reinforced Nb_3Sn wire.**

***Index Terms*—Nb_3Sn, internal matrix reinforcement, ternary bronze matrix, mechanical strength, HIP treatment**

I. INTRODUCTION

ITER project is one of the path-ways to realize nuclear fusion energy generation, and this project is also still in progress. In ITER project, high current capacity Cable in-Conduit (CIC) conductors for the Toroidal magnetic field and Central Solenoid coils were made by the twisted bundles of many Nb_3Sn wires. Through the ITER procurement activities, mechanical strength issues in Nb_3Sn wire as the CIC conductor for beyond ITER were pointed out [1]. Especially, transport I_c degradation by mechanical strain applying due to the electromagnetic force is one of the key research issues to be solved for a future high magnetic field superconducting magnet beyond ITER [1]. With regard to mechanical strength improvement of Nb_3Sn wire, various reinforcement methods providing Al_2O_3 dispersion strengthened copper or CuNb alloy to the outside of Nb_3Sn wire have been investigated [2, 3].

We proposed an internal matrix reinforcement method by solid solution strengthening of the wire matrix [4-6]. The principle of the internal matrix reinforcement method is that the wire matrix is changed from the conventional Cu-Sn binary alloy to the Cu-Sn based ternary alloy containing Zn or In elements. The wire matrices of Cu-Sn-Zn or Cu-Sn-In ternary alloys were transformed to homogeneous and harder (Cu, Zn) or (Cu, In) solid solution after Nb_3Sn phase synthesis heat treatment, and these solid solutions would act as a protective material for Nb_3Sn phase [4, 6]. Furthermore, we confirmed that mechanical strength and intrinsic strain sensitivity in I_c property were improved compared with the conventional bronze processed Nb_3Sn wire [7-9].

For the further mechanical strength improvement, we noticed that the Hot Isostatic Pressing (HIP) after Nb_3Sn synthesis heat treatment. Previously, T. Fukutake et al. reported that strain dependence of transport I_c property in bronze and Internal Tin processed Nb_3Sn wire was improved by HIP treatment [10, 11]. Furthermore, C. Barth also mentioned that the irreversible tensile strain of transport I_c property in Nb_3Sn wire was improved by HIP processing [12].

In this study, the internal matrix reinforced Nb_3Sn wires after heat treatment were performed to HIP treatment and the comparisons of transport I_c behavior under tensile deformation with and without HIP processing were evaluated.

II. SAMPLE PREPARATION AND EVALUATIONS

A. Sample preparation

Two kinds of the internal matrix reinforced Nb_3Sn multifilamentary precursor wires were prepared. The sample code and specification of wire samples is indicated to Table.1.

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The difference between the two kinds of internal matrix reinforced Nb_3Sn wires was the composition of the ternary bronze alloy as the wire matrix, which were Cu-10wt%Sn-5wt%In-0.3wt%Ti (Sample code: 5.0In) and Cu-14wt%Sn-2wt%In-0.3wt%Ti (Sample code: 2.0In). These 2.0In and 5.0In precursor wires were carried out the two step Nb_3Sn phase formation treatment, which is 550°C-100 hrs + 650°C-100hrs in Ar atmosphere.

After that, HIP treatment performed to wire samples at 650°C-2hrs under 200MPa in Ar atmosphere using NIFS-HIP system. Temperature and pressure profile during HIP treatment is shown in Fig. 1. The pressure in the HIP system was elevated using a two-stage Ar gas compressor and then maintained stably at 200MPa.

B. Transport I_c evaluations under tensile deformation with and without HIP processing

Transport I_c measurement under uniaxial tensile deformation was carried out using 18 T superconducting magnet with 55 mm bore and the I_c measurement probe with uniaxial tensile deformation mechanism in Institute Material Research, Tohoku University. Before the wire sample mounting to the I_c measurement probe, two strain gages were attached to surface of wire sample. Transport I_c measurement system with tensile deformation mechanism and sample mounting were already reported [13].

The I_c probe after sample mounting was inserted into 18 T superconducting magnet, and transport I_c measurement with tensile deformation was performed at 4.2 K and 15 T of external magnetic field. The tensile strain was defined to the average value of two attached strain gages. A tensile load, which is measured by 1 kN load cell, was applied by the load lever moving. The tensile stress was estimated by the tensile load value divided by the cross-sectional area including the stabilized copper of the wire sample. Transport I_c measurement was carried out using a DC four-probe method, and the I_c criterion was determined by 1 μV/cm electric field generation.

TABLE I
THE SAMPLE CODE AND WIRE SPECIFICATION OF THE INTERNAL MATRIX REINFORCED NB_3SN MULTIFILAMENTARY WIRES IN THIS STUDY

Sample code	2.0In			5.0In		
Wire matrix	Cu-14Sn-2In-0.3Ti			Cu-10Sn-5In-0.3Ti		
Matrix composition	Sn	In	Ti	Sn	In	Ti
(Sn, In and Ti: wt%)	14.05	1.99	0.35	10.17	5.06	0.31
Number of Nb fila.	7771			7771		
Diameter of Nb fila.	3.4 μm			3.4 μm		
Barrier material	Nb			Nb		
Final wire diameter	0.9 mm			0.9 mm		
Cu ratio	1.30			1.30		

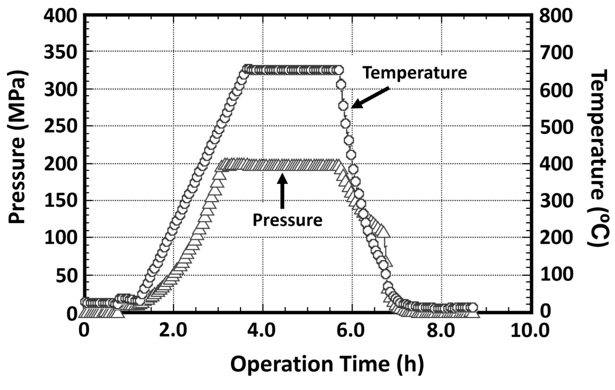

Fig. 1. Temperature and the pressure profiles during HIP processing in this study.

III. RESULTS AND DISCUSSIONS

A. Transport I_c behavior under tensile deformation of the 2.0In wire sample with and without HIP processing

The comparisons of transport I_c behavior at 4.2 K and 15 T as a function of the uniaxial tensile stress and strain between the 2.0In wire samples with and without HIP processing is shown in Fig. 2. Here, each normalized I_c values shown in Fig. 2 corresponded to the I_c values loaded to some tensile stress (σ) or strain (ε) divided by the maximum I_c value ($I_c$max), and these normalized $I_c\sigma/I_c$max or $I_c\varepsilon/I_c$max values indicate the I_c degradation rate by the uniaxial tensile stress and strain. In this study, the uniaxial tensile stress and strain shown in $I_c$max value are also defined as the peak stress and strain, respectively.

$I_c$max value of the 2.0In wire sample was roughly increased from 93 A to 113 A by the HIP processing. Normalized I_c values loaded to zero tensile stress and strain ($I_c\sigma_0/I_c$max and $I_c\varepsilon_0/I_c$max) were decreased by the HIP processing. These suggested that the high pressure of 200 MPa by HIP process effectively applied to the inside of the the 2.0In wire sample

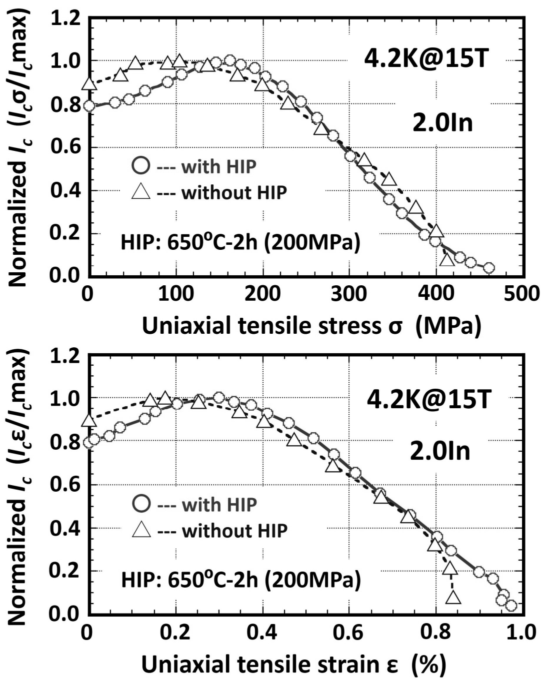

Fig. 2. The comparisons of transport I_c behavior at 4.2 K and 15 T as a function of the applied uniaxial tensile stress and strain between the 2.0In wire samples with and without HIP processing.

TABLE II
THE SUMMARY OF MAJOR MECHANICAL PROPERTIES IN THE 2.0IN WIRE SAMPLE WITH AND WITHOUT HIP PROCESSING IN THIS STUDY

2.0In sample	Without HIP	With HIP	Increment (%)
Maximum I_c (A)	93.33	113.33	+ 21.42
Fracture stress (MPa)	412.30	474.24	+ 15.02
Fracture strain (%)	0.8385	0.9626	+ 14.80
Peak stress (MPa)	104.30	162.79	+ 56.08
Peak strain (%)	0.1760	0.2986	+ 69.66

TABLE III
THE SUMMARY OF MAJOR MECHANICAL PROPERTIES IN THE 5.0IN WIRE SAMPLE WITH AND WITHOUT HIP PROCESSING IN THIS STUDY

5.0In sample	Without HIP	With HIP	Increment (%)
Maximum I_c (A)	64.60	63.19	- 2.17
Fracture stress (MPa)	564.84	578.83	+ 2.48
Fracture strain (%)	0.9320	1.1668	+ 25.19
Peak stress (MPa)	265.89	308.29	+ 15.95
Peak strain (%)	0.3052	0.3600	+ 17.96

and increased the residual stress and strain. $I_c\sigma/I_c$max and $I_c\varepsilon/I_c$max values improved by the applying tensile deformation. These would be caused by the release of the residual stress and strain into the wire. The peak stress and strain of the 2.0In wire sample with HIP processing were obtained to be approximately 162 MPa and 0.299 %, and these values were much higher than those of the sample without HIP. The fracture stress and strain were estimated to be approximately 474 MPa and 0.963 %, and then they were improved roughly 15 % by the HIP processing. The summary of major mechanical properties in the 2.0In samples with and without HIP processing is indicated to Table. 2. We found that mechanical strength of the 2.0In wire sample was enhanced by the HIP processing.

B. Transport I_c behavior under tensile deformation of the 5.0In wire sample with and without HIP processing

Similar to Fig. 2, the comparisons of transport I_c behavior at 4.2 K and 15 T as a function of the uniaxial tensile stress and strain between the 5.0In wire samples with and without HIP processing is shown in Fig. 3. Similar to the 2.0In wire sample, normalized $I_c\sigma_0/I_c$max and $I_c\varepsilon_0/I_c$max were decreased by the HIP processing. This suggested that the high pressure of 200 MPa by HIP process effectively also applied to the inside of the 5.0In wire sample and increased the residual stress and strain. The peak stress and strain of the 5.0In wire sample with HIP processing were obtained to be approximately 308 MPa and 0.3600 %, and these values were much higher than those of the sample without HIP. The fracture stress and strain were estimated to be approximately 578 MPa and 1.1668 %. The summary of major mechanical properties in the 5.0In wire sample with and without HIP processing is indicated to Table. 3. We found that mechanical strength of the 5.0In wire sample was also enhanced by the HIP processing.

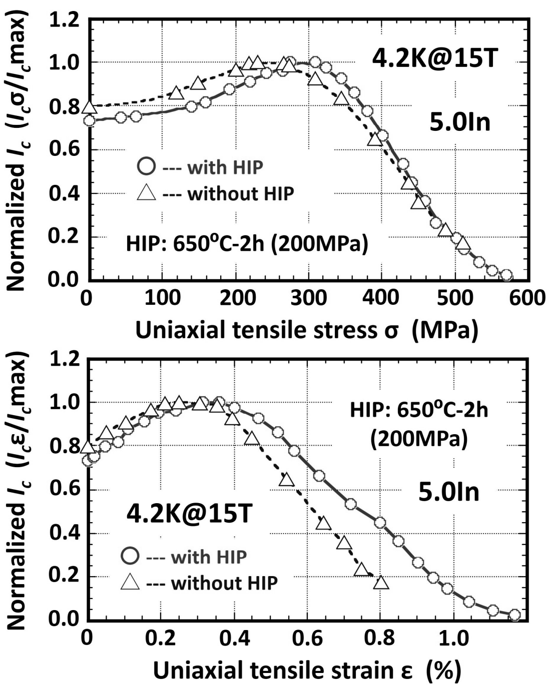

Fig. 3. The comparisons of transport I_c behavior at 4.2 K and 15 T as a function of the applied uniaxial tensile stress and strain between the 5.0In wire samples with and without HIP processing.

C. The comparisons of the HIP effect on transport I_c behavior under tensile deformation between the 2.0In and 5.0In samples

As indicated in Tables. 2 and 3, the major mechanical property factors were improved by the HIP treatment, and it contributed to the mechanical strength enhancement of the internal matrix reinforced Nb_3Sn wire. However, difference in the HIP effect was observed between the different compositions of the wire matrix. This was caused by the difference in the microstructure of the wire matrix before HIP treatment. X-ray Diffraction (XRD) patterns in wire matrix region of the 5.0In and 2.0In wire samples between before and after Nb_3Sn synthesis heat treatment using micro-focused XRD system is shown in Fig. 4.

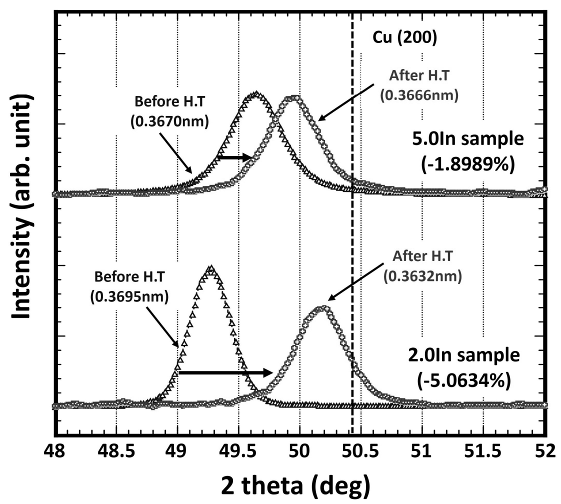

Fig. 4. The comparisons of X-ray Diffraction (XRD) patterns in wire matrix region of the 5.0In and 2.0In wire samples between before and after Nb_3Sn synthesis heat treatment using micro-focused XRD system.

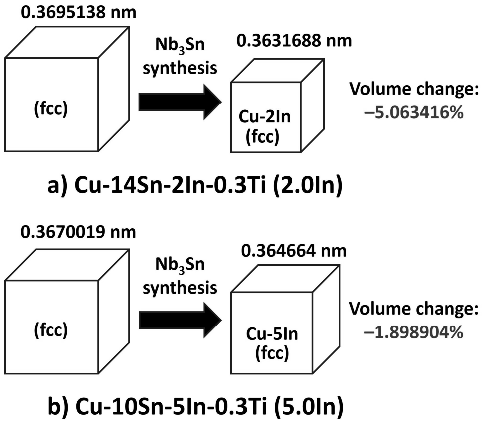

Fig. 5. The volume change of the wire matrix in the 2.0In and 5.0In wire samples between before and after Nb_3Sn synthesis heat treatment

The X-rays were focused to a diameter of 100 μm by the collimator and irradiated only to the wire matrix region. In previous studies, we already confirmed that the wire matrix after Nb_3Sn synthesis heat treatment has transformed to a homogeneous (Cu, In) solid solution with an initial In element composition [6-9].

As shown in Fig. 5, both the Cu-Sn-In ternary bronze alloy and the (Cu, In) solid solution have the Face-Centered Cubic structure, the volume change of the wire matrix before and after the Nb_3Sn formation could be calculated based on the change of the lattice parameter in the wire matrix estimated by XRD. In the 2.0In wire sample, where the HIP effect was significant, the lattice constant changed from 0.3695 nm to 0.3632 nm and the volume decreased by approximately 5 %. In the 5.0In wire sample, the lattice constant changed from 0.3670 nm to 0.3666 nm, resulting in a volume decrease by approximately 2 %. The decrease in volume in the wire matrix was one of the factors of the void formation in the wire matrix, and these voids would become the origin point of mechanical fracture. The 2.0In wire sample have formed more voids in the wire matrix after Nb_3Sn formation compared with the 5.0In wire sample. The HIP treatment increased the mechanical strength by removing many voids. In references [10-12], the HIP treatment improved the mechanical strength properties in critical current properties and concluded that the void reduction was the main reason for this improvement.

In other words, HIP treatment of the internal matrix reinforced Nb_3Sn wire using a ternary bronze alloy acted more effectively to the larger volume change of the wire matrix during Nb_3Sn synthesis heat treatment.

IV. Conclusions

The peak stress and strain after HIP processing were clearly improved. HIP treatment was one of the effective processes to improve mechanical strength of the internal matrix reinforced Nb_3Sn wire. Because the voids into the wire matrix after Nb_3Sn formation formed by the volume change of the wire matrix were reduced by the HIP process. And the HIP effect had appeared more effectively to the wire with larger volume change of the wire matrix due to the Nb_3Sn phase formation.

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