

Insights into Creep Properties and Microstructure of Gr. 91 Steels with Varied Impurity Contents

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The creep properties and microstructures of two types of Gr. 91 steel with different impurity (Sb, As, Sn) contents were investigated. The steel with higher impurity concentrations (Steel 1) ruptured in slightly shorter time than the steel with lower impurity concentrations (Steel 2). The reduction of area for Steel 1 was slightly lower than that for Steel 2 due to higher density of creep voids in Steel 1. However, no clear correlation was observed between impurity contents and either elongation or creep intolerance parameter (λ). Microstructural observation revealed that the effect of impurities on the initial microstructure was considered negligible. During creep, the crystallographic relationship between common rotation axes and rotation angles at block/packet boundaries gradually disappeared. Among block boundaries, $\langle 011 \rangle 53^\circ$ and $\langle 011 \rangle 60^\circ$ were found to be more susceptible to creep deformation, whereas $\langle 111 \rangle 60^\circ$ was relatively less sensitive. This disappearance of crystallographic relationship was more pronounced in Steel 1 than in Steel 2, implying that the difference in creep strength between the two steels could be attributed to the ease of migration and merging of block/packet boundaries.

KEY WORDS: creep strength enhanced ferritic steels; Type 2 specification; creep void; segregation; block/packet boundaries.

1. Introduction

To ensure the safe design and maintenance of equipment, it is essential to understand the latest trends in material standards and allowable tensile stresses. Gr. 91 type steel (9Cr-1Mo-V-Nb), which is widely used in thermal power plant boilers, exhibits heat-to-heat variation in long-term creep strength.¹⁻⁴⁾ Accordingly, its material specifications and allowable tensile stress have undergone multiple revisions.⁵⁻¹¹⁾ In recent years, ASME has newly standardized the specification of Gr. 91: Type 2,⁵⁾ which aims to ensure adequate creep rupture ductility and to minimize heat-to-heat variation in creep strength. The allowable tensile stress for Type 2 is set slightly higher than that for Type 1 (the specification equivalent to the previous standard before Type 2 was established).⁸⁾ Table 1 shows the chemical composition

requirements for ASME SA387/SA387M Gr. 91 Type 1 and Type 2.⁵⁾ It should be noted that Type 2 introduces upper limits for impurity elements such as Sb, As, and Sn.

As reported by Parker and Siefert,¹²⁾ Gr. 91 with relatively high impurity (Sb, As, or Sn) concentration tends to be poor creep ductility. Previous studies have demonstrated that the rupture time and reduction of area decreased by Sb and Sn¹³⁻¹⁵⁾ and that these elements promoted creep void formation.^{14,15)} Galliopoulou *et al.* reported that prior austenite grain boundary is one of the preferential creep void formation sites in Gr. 91.¹⁶⁾ Hou *et al.* successfully predicted the rupture time of Gr. 91 using constitutive equations combining creep void growth model.¹⁷⁾ Thus, creep void in creep strength enhanced ferritic (CSEF) steels, including Gr. 91, is one of the research fields of interest.¹⁶⁻¹⁹⁾ On the other hand, under uniaxial creep deformation, it is well known that Gr. 91 exhibits not intergranular fracture caused by the linkage of creep voids at grain boundaries, but transgranular

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Table 1. Chemical requirements for product analysis by SA387/SA387M Gr.91 in ASME BPV Section II-A (2019)⁵⁾ and chemical composition of test materials (mass%).

Standard/Test material	C	Si	Mn	P	S	Ni	Cr	Mo	V	Nb	Sol-Al
SA387/SA387M	0.06–	0.18–	0.25–	≤0.025	≤0.012	≤0.43	7.90–	0.80–	0.16–	0.05–0.11	≤0.02
Gr.91 Type 1	0.15	0.56	0.66				9.60	1.10	0.27		
SA387/SA387M	0.06–	0.20–	0.30–	≤0.020	≤0.005	≤0.20	8.0–	0.80–	0.16–	0.05–0.11	≤0.020
Gr.91 Type 2	0.15	0.40	0.50				9.50	1.05	0.27		
Steel 1	0.082	0.30	0.41	0.012	0.0006	0.11	8.45	0.94	0.20	0.08	0.008
Steel 2	0.087	0.29	0.41	0.011	0.0005	0.09	8.49	0.93	0.20	0.074	0.007
Standard/Test material	N	Sb	As	Sn	W	Ti	Cu	Zr	B	O	N/Al
SA387/SA387M	0.025–	–	–	–	–	≤0.01	–	≤0.01	–	–	–
Gr.91 Type 1	0.080										
SA387/SA387M	0.035–	≤0.003	≤0.010	≤0.010	≤0.05	≤0.01	≤0.10	≤0.01	≤0.001	–	4.0≤
Gr.91 Type 2	0.070										
Steel 1	0.052	0.008	0.019	0.018	0.03	0.001	<0.01	<0.001	<0.0001	0.003	6.5
Steel 2	0.052	<0.001	<0.001	<0.001	<0.01	0.001	<0.01	<0.001	<0.0001	0.001	7.4

fracture.^{20,21)}

Evaluating creep rupture ductility, elongation and creep intolerance parameter (λ) are also used.^{21–23)} λ is one of the parameters representing rupture ductility and is given by Eq. (1).^{24,25)}

$$\lambda = \varepsilon_f / \dot{\varepsilon}_{\min} t_r \dots\dots\dots (1)$$

Here, ε_f is elongation, $\dot{\varepsilon}_{\min}$ is minimum creep rate, and t_r is time to rupture. For Gr. 91, λ values of 5 or less are considered to indicate low ductility (creep intolerant material).^{21,26)} Although elongation and λ are also crucial properties, previous studies^{13–15)} have not addressed the impurities impact on them.

The first issue is that, despite their recent incorporation into the specification, our knowledge of the impurities impact remains limited. An even more important point is that no recent research findings (within the past five years) regarding the effects of impurities have been reported. In CSEF steels, the creep deformation behavior could differ between high-stress and low-stress regimes.^{27–29)} Moreover, as the rupture time increases, the reduction of area in Gr. 91 generally decreases, but may increase in long-term and low-stress regimes.²¹⁾ Creep tests conducted in previous studies^{13–15)} have been relatively short-term (less than 10 000 h); therefore, it is essential to clarify the impurity impacts on long-term creep properties.

Previous studies^{13–15)} have reported the degradation of creep strength by impurities, whereas microstructural analysis has been limited. The second issue is that the main cause of reduced creep strength in Gr. 91 steel with high impurity contents has not been sufficiently clarified, that is, whether it is due to promotion of creep void formation or to microstructural aspects. Our previous work³⁰⁾ revealed that impurities (Sb, As, and Sn) influenced the microstructure development in the heat-affected zone (HAZ) and the creep rupture strength of welded joints in Gr. 91 steel. These impurities suppressed the dissolution of $M_{23}C_6$ during welding. Therefore, the amount of $M_{23}C_6$ re-precipitated at the grain boundaries during post weld heat treatment was decreased, and pinning force exerted by $M_{23}C_6$ was reduced. Accord-

ingly, accelerated microstructural recovery and subsequent creep void formation in HAZ resulted in the degradation of creep rupture strength in the welded joint.³⁰⁾ Thus, to clarify the effects of impurities, it is indispensable to discuss focusing on the microstructures. The recovery of subgrain (or lath) structure with high dislocation density plays a key role in the creep deformation of CSEF steels.^{31–33)} In recent years, subgrain size and (geometrically necessary) dislocation density in CSEF steel can be quantitatively estimated using electron backscatter diffraction (EBSD).^{34,35)} It has also been reported that block and packet boundaries migrate and gradually disappear during creep,^{36,37)} however, it has not yet been clarified which type of boundary is more susceptible to creep. To discuss how impurities could affect creep strength, it is desirable to comprehensively analyze subgrain size, block/packet boundaries, creep voids, and other relevant characteristics.

The aim of the present study is to investigate the effects of impurities (Sb, As, Sn) on the long-term creep properties of Gr. 91 steel and relations between creep strength and microstructure or creep voids. Hereafter, “impurities” refers to the three elements: Sb, As, and Sn. Creep tests exceeding 10 000 h were conducted using two test materials with varied impurity contents, and the microstructure before and after creep was examined. This study focuses on the crystallographic features of martensite and discusses the relationship between creep strength and microstructure.

2. Experimental Procedure

2.1. Test Material

The test materials used in the present study were the two types of Gr. 91 steel, which were the same as those employed in our previous work.³⁰⁾ The chemical compositions of the two steels are shown in Table 1. Hereafter, the steel with higher and lower impurity content is referred to as Steel 1 and Steel 2, respectively. Steel 1 meets the requirements of ASME SA387/SA387M Type 1, while Steel 2 meets those of Type 2. Details of manufacturing process of the steels are provided in the previous article.³⁰⁾ The steels

were normalized at 1 050°C for 1.5 h (fan cooling) and tempered at 760°C for 1 h (air cooling). The prior austenite grain sizes of both steels were approximately 20–30 μm .³⁰⁾

2.2. Creep Tests

To ensure that the rolling direction was parallel to the loading direction, creep test specimens were cut from the center of the plate thickness. The diameter and length of the gauge portion of the specimens were 6 and 30 mm, respectively. The creep strain tests were performed at 650°C under stress ranging from 60 to 90 MPa in air atmosphere. 650°C is higher than the service temperature of Gr. 91 steels in thermal power plant boilers, but it is commonly used for creep testing^{13–15,21)} especially to investigate the creep deformation behavior in the low-stress regime. Since the yield stresses ($\sigma_{0.2}$) of Steel 1 and Steel 2 at 650°C were 197 and 201 MPa, respectively,³⁰⁾ all creep test stress in this study met less than half of $\sigma_{0.2}$. In addition to creep tests carried out until failure, creep tests at 650°C and 60 MPa were discontinued for some specimens after approximately 5 000 h (Steel 1: 5 000 h; Steel 2: 5 011 h) and approximately 10 500 h (Steel 1: 11 029 h; Steel 2: 10 006 h).

2.3. Microstructural Characterization

Microstructural observations were performed by optical microscopy (OM) and scanning electron microscopy (SEM). OM samples were prepared using Vilella's reagent. Creep voids were quantitatively evaluated using OM images with a field of view of 900 $\mu\text{m} \times 680 \mu\text{m}$. Average number density, diameter, and area fraction of creep voids were determined using five OM images. SEM was carried out using two types of equipment: Zeiss Ultra55 and JSM-7100F (JEOL). The former equipment was operated at an accelerating voltage of 1 kV and used mainly to observe the precipitate (primarily M_{23}C_6). Under these conditions, the precipitate was observed as dark contrast. Five SEM images were obtained for each sample, with a field of view of 24 $\mu\text{m} \times 18 \mu\text{m}$, and the average particle diameter was estimated. The latter equipment was operated at an accelerating voltage of 15 or 25 kV, and back-scattered electron (BSE) images were acquired under conditions that emphasize channeling contrast (corresponding to electron channeling contrast imaging, ECCI). This condition was utilized to observe the subgrain (or lath) structure.

For analyzing martensite structure, EBSD was conducted using EDAX OIM system (Digiview camera) where the accelerating voltage was operated at 15 kV and step size was set as 0.5 μm . Each measurement covered a field of view of 300 $\mu\text{m} \times 300 \mu\text{m}$, which is sufficiently larger than the prior austenite grain size of both steels. The ROPA software (TSL Solutions) was used to estimate prior austenite grain boundaries (PAGB). ROPA enables estimation of PAGB from EBSD data based on the Kurdjumov-Sachs (K-S) relationship. In addition, using EBSD data, the common rotation axes and rotation angles (misorientation angles above 15°) of grain boundaries in martensite structure were analyzed. The subgrain size w was evaluated using the following equation.³⁵⁾

$$w = 3sN_t / 4N_\Phi \dots\dots\dots (2)$$

Here, s is step size, N_t is the total number of measurement

points, and N_Φ is the number of points with misorientation of 1–5° between neighboring points. Eq. (2) is suitable for evaluating the average size of subgrain in the whole field of view.

The hardness of the grip portion of crept specimens was measured by micro-Vickers hardness test using HMV-G-FA-D (SHIMADZU), applying a force of 9.8 N (1 kgf). The average value of five measurements was taken as the representative value. To investigate the total amount of precipitates, extraction residue analysis was performed. After the extraction residue was obtained using a 10% acetylacetone-based electrolyte, the composition of the residue was analyzed by inductively coupled plasma (ICP) emission spectrometry using ICPV-8000 (SHIMADZU).

3. Results

3.1. Creep Properties

Figure 1 shows the plot of stress vs. time to rupture at 650°C. For comparison, Fig. 1 also displays the average creep rupture strength of Gr. 91 (regression curve of all products).¹¹⁾ In all creep tests, time to rupture of the two steels exceeded the average strength of Gr. 91. Incidentally, the data in this study fall within 99% confidence interval of Gr. 91 steel,¹¹⁾ suggesting that the difference in creep strength between Steel 1 and Steel 2 is within the general variation observed for this type of steel. When comparing under the same stress conditions, Steel 1 tended to rupture slightly earlier than Steel 2.

Figure 2 shows the creep curves of creep strain vs. time, creep rate vs. time, and creep rate vs. creep strain under two conditions: 80 and 60 MPa. In Figs. 2(b), 2(e), no differences were observed between Steel 1 and Steel 2 in the deformation behavior prior to reaching the minimum creep rate (transient creep), but Steel 1 entered acceleration creep earlier than Steel 2. Accordingly, the minimum creep rate of Steel 1 was slightly higher than that of Steel 2. Focusing on the creep curve of creep strain vs. creep rate, a slight difference was found in the behavior after 95% of the lifetime ratio (the ratio of elapsed test time to rupture time) between Steel 1 and Steel 2. In Steel 1, the increase in creep strain associated with the rise in creep rate just before rupture was larger than that in Steel 2. Conversely, for Steel 2, the

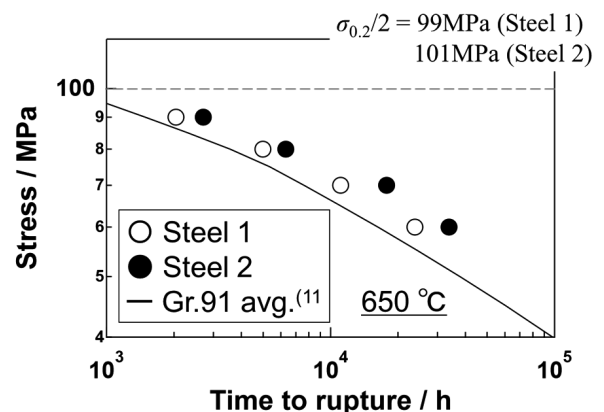


Fig. 1. Relationship between stress and time to rupture at 650°C. The solid line demonstrates the regression curve of the average creep rupture strength of Gr. 91 steel (all products).¹¹⁾

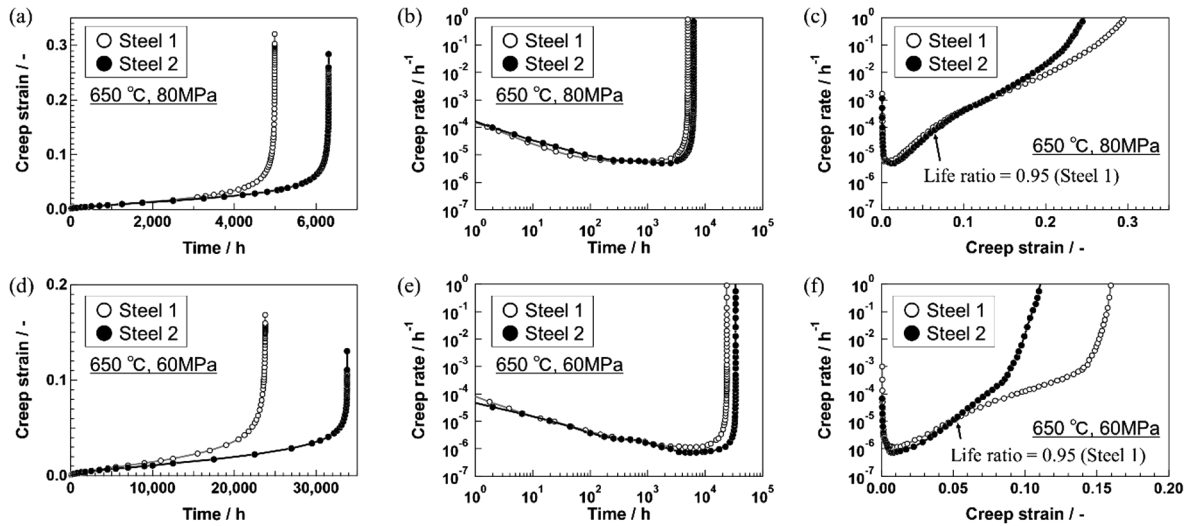


Fig. 2. Creep curves under (a)–(c) 80 MPa and (d)–(f) 60 MPa at 650 °C. (a), (d) Creep strain vs. time, (b), (e) creep rate vs. time and (c), (f) creep rate vs. creep strain.

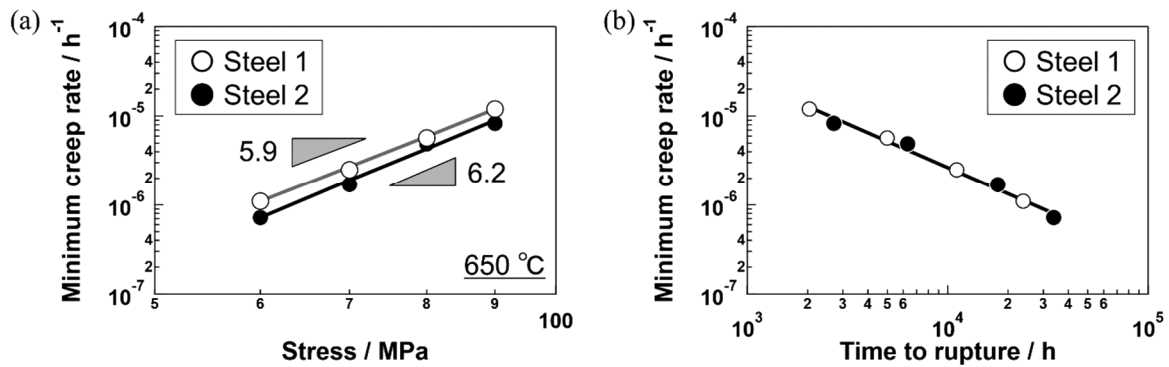


Fig. 3. Relationships between (a) minimum creep rate and stress, and (b) minimum creep rate and time to rupture.

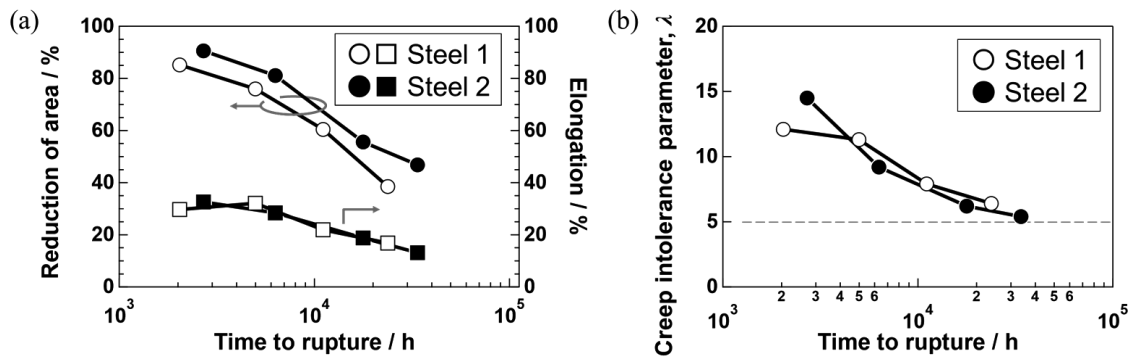


Fig. 4. Changes of (a) elongation, reduction of area, and (b) creep intolerance parameter (λ) against time to rupture.

degree of increase in creep rate was greater at a smaller strain compared to Steel 1. The relationship between stress and minimum creep rate is shown in Fig. 3(a). In the double logarithmic plot of Fig. 3(a), the experimental results for Steel 1 and Steel 2 were each arranged along straight lines. The slopes of these lines are equivalent to the stress exponent in the power law creep. The stress exponents for both Steel 1 and Steel 2 were approximately 6, which is consistent with values reported in previous studies.^{38,39)} The stress exponent of Gr. 91 may vary depending on temperature and stress.^{38–41)} However, the relationship between stress and minimum creep rate showed no inflection point as all creep tests in this study were conducted at stress levels below

$\sigma_{0.2}/2$. Figure 3(b) shows the relationship between minimum creep rate and rupture time (Monkman–Grant plot). All test results for Steel 1 and Steel 2 were on a single straight line. This indicates that minimum creep rate of Steel 1 was higher than that of Steel 2, resulting in slightly earlier time to rupture for Steel 1.

The reduction of area and elongation were plotted against time to rupture in Fig. 4(a). In both Steel 1 and Steel 2, the reduction of area decreased with increasing rupture time. The reduction of area for Steel 1 tended to be slightly lower than that for Steel 2, suggesting that impurities could contribute to decrease reduction of area. In contrast, the elongation for Steel 1 and Steel 2 followed a common curve. The

elongation remained almost constant up to approximately 4 000 h (90 MPa) but decreased with increasing rupture time beyond approximately 4 000 h (60–80 MPa). Figure 4(b) shows the relationship between creep intolerance parameter (λ) and time to rupture. As in the case of elongation, λ for Steel 1 and Steel 2 followed a common curve. The values of λ for all creep tests in this study were higher than 5.

As previously reported,^{13–15)} Steel 1 with higher impurity concentrations tended to exhibit a slightly lower reduction of area. On the other hand, elongation and λ for Steel 1 were not necessarily lower compared to Steel 2. To discuss the creep rupture ductility, attention is focused on the specimens crept under 80 MPa, which exhibited relatively thin oxidation scale. The appearances of the specimens for Steel 1 and Steel 2 are shown in Figs. 5(a), 5(b). Based on these photographs, Fig. 5(c) shows the diameter of the specimens measured at 0.2 mm intervals within 15 mm on both sides of the fracture surface. It should be noted that this measurement method differs from the one used to obtain the reduction of area. Therefore, especially in the minimum diameter measured by the former method may slightly differ from those by the latter one; however, it was confirmed that there were no significant differences in the diameter sufficiently far from the fracture surface measured by both methods. As shown in Fig. 5(c), the slope of the plots near the fracture surface was gentler for Steel 1, suggesting that the decrease in diameter due to necking occurred over a wider range in Steel 1 than in Steel 2. This is consistent with the result that the increase in creep strain just before rupture was larger in Steel 1 (Figs. 2(c), 2(f)). As discussed in Section 4, although Steel 1 tended to exhibit a slightly lower reduction of area,

the elongation did not decrease due to sufficient deformation in the gauge portion, including the fracture surface.

3.2. Creep Voids Distribution and Microstructures

In the following sections, attention is focused on the conditions (60–80 MPa) where elongation decreased with increasing time to rupture. Cross-sectional OM images of the ruptured specimens under 80 and 60 MPa are shown in Fig. 6. The observation positions were at least 10 mm away from the fracture. Creep voids were present in all specimens, whereas linkage of several creep voids or cracks

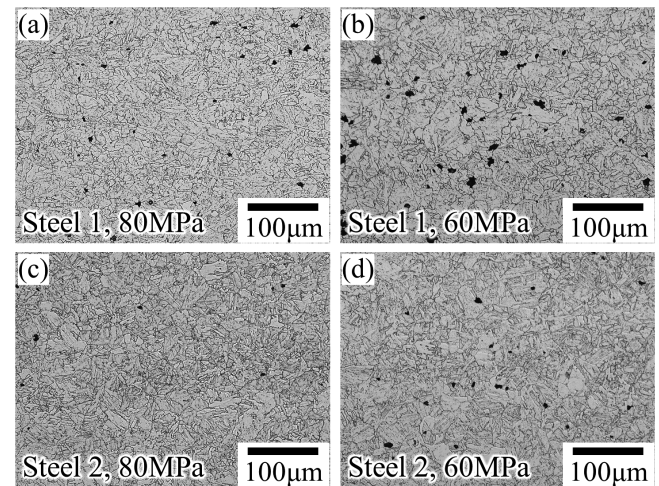


Fig. 6. OM images of ruptured specimens observed at least 10 mm away from the fracture surface in (a), (b) Steel 1 and (c), (d) Steel 2 crept under (a), (c) 80 MPa and (b), (d) 60 MPa.

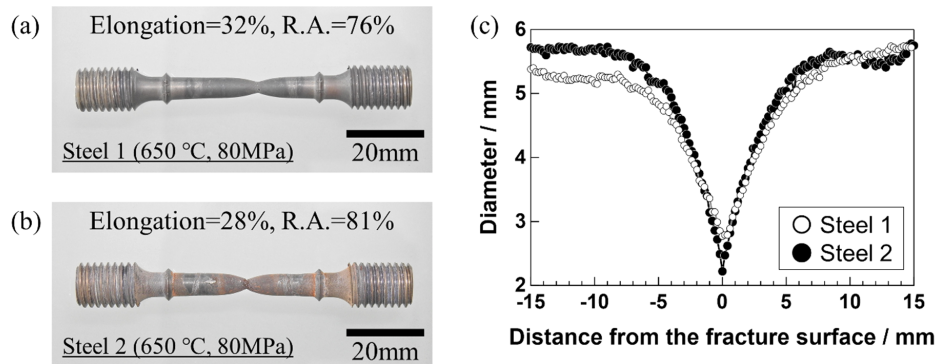


Fig. 5. Appearance of the creep ruptured specimens of (a) Steel 1 and (b) Steel 2 under 80 MPa. (c) Diameter change of ruptured specimens with respect to the distance from the fracture surfaces. (Online version in color.)

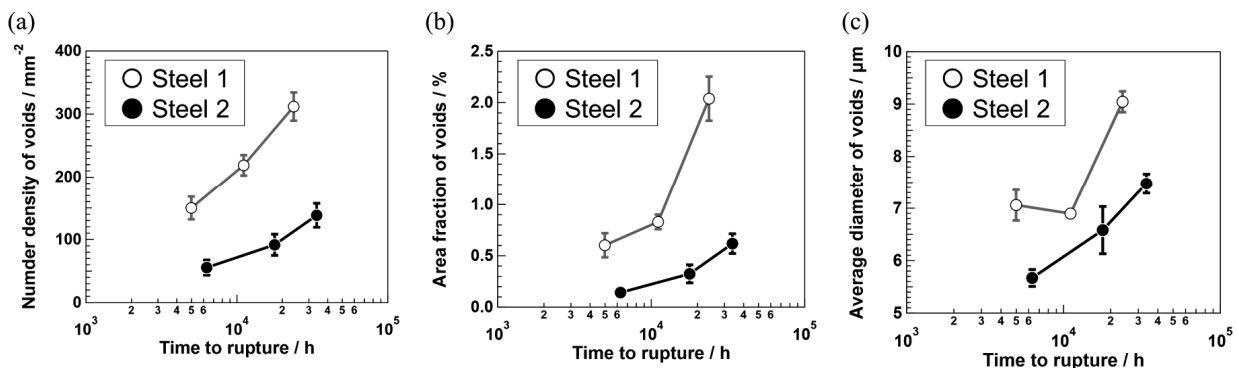


Fig. 7. (a) Number density, (b) area fraction, and (c) average diameter of creep voids with respect to time to rupture.

were hardly observed. The relationships between number density, average size, and area fraction of creep voids as a function of time to rupture are shown in Fig. 7. The values of these characteristics increased with increasing rupture time. Compared to Steel 2, more than twice as many creep voids were observed in Steel 1. Figure 8 shows OM images near the fracture surface. The observation results suggested the ductile fracture under both stress conditions. In the specimens ruptured under 80 MPa, the grains tended to be more elongated parallel to the direction of applied stress than those under 60 MPa. No significant differences were observed in the microstructure near the fracture surface between Steel 1 and Steel 2.

Figure 9 shows cross-sectional SEM images before and after the creep tests. Under these observation conditions, precipitate particles (mainly $M_{23}C_6$) are observed as dark contrast. The precipitate distributions did not significantly differ between Steel 1 and Steel 2. Figure 10 demonstrates the relationship between average particle diameter and time to rupture. With increasing creep time, coarsening of precip-

itate particles progressed. Error bars in Fig. 10 represent the scatter in the evaluated values between five fields of view. No significant differences were found between Steel 1 and Steel 2 in the average particle diameter, but the error bars in Steel 1 tended to be larger than Steel 2. The amount of precipitate was compared using extraction residue analysis.

Figure 11(a) shows the results of the analysis for the total amount and composition of precipitates in the initial material and the grip portion of the crept specimens exposed for about 5 000 h at 650°C. Although Sb, As, and Sn were also analyzed, their values were all below 0.001%. The amounts of MX and $M_{23}C_6$ can be estimated from the sums of V and Nb, and Cr, Fe, and Mo, respectively. First, the amount of $M_{23}C_6$ before and after the exposure is compared. In both Steel 1 and Steel 2, much more $M_{23}C_6$ was precipitated in the grip portion exposed for about 5 000 h at 650°C than in the initial material. This is because the equilibrium volume fraction of $M_{23}C_6$ is higher at the creep test temperature (650°C) than at the tempering temperature (760°C).⁴²⁾ Figure 11(a) indicates that Cr and Mo contents increased, and Fe content decreased in $M_{23}C_6$ after exposure during the creep test. This result is consistent with previous studies.^{43–46)} Next, the amount of $M_{23}C_6$ in Steel 1 and Steel 2 is

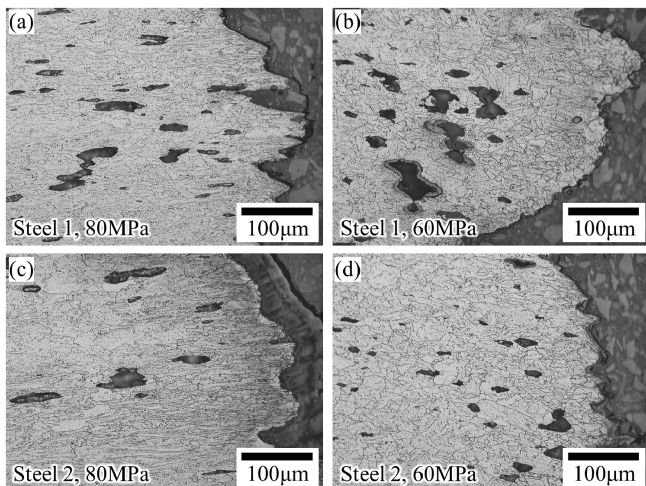


Fig. 8. OM images of ruptured specimens observed near the fracture surface in (a), (b) Steel 1 and (c), (d) Steel 2 crept under (a), (c) 80 MPa and (b), (d) 60 MPa.

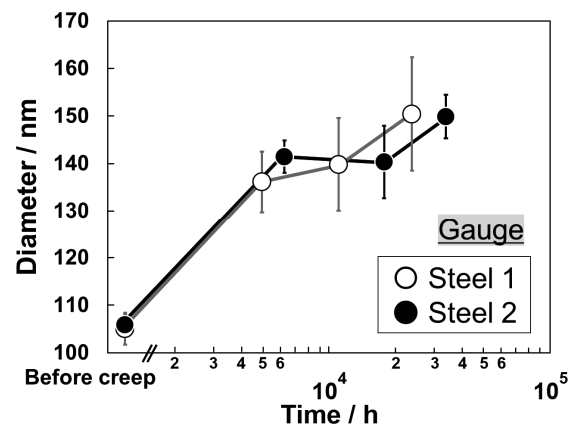


Fig. 10. Change of the average diameter of precipitates in the gauge portion of crept specimens with respect to time.

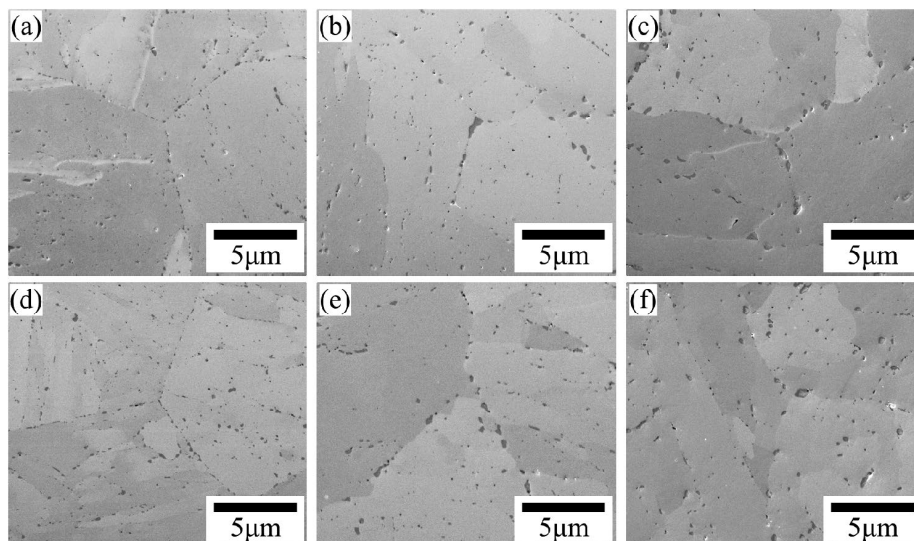


Fig. 9. SEM images of (a)–(c) Steel 1 and (d)–(f) Steel 2. (a), (d) Before creep, (b), (e) crept under 80 MPa, and (c), (f) 60 MPa.

compared. In both the initial material and the grip portion, Steel 1 had a slightly lower amount of $M_{23}C_6$ than Steel 2. It was also revealed that the amount of MX was comparable between Steel 1 and Steel 2.

For comparison, the equilibrium amounts of $M_{23}C_6$ and MX were estimated using Thermo-calc with SSOL4 database. The results are shown in Fig. 11(b). The calculated total amounts of precipitates were generally consistent with the experimental results. The total amount of precipitates (experimental values) for Steel 1 and Steel 2 corresponded to 97 and 95% of their calculated values. A comparison between Figs. 11(a), 11(b) shows that the compositional changes in $M_{23}C_6$ during exposure at creep test temperature (the increased Cr and Mo, and the decreased Fe) could be reasonably predicted by the calculations. The lower amount of calculated $M_{23}C_6$ in Steel 1 is attributed to low C concentration (see Table 1). According to the calculations, Sb, As, and Sn are all partitioned into the matrix phase. In addition, since the impurity content in Steel 1 was sufficiently low, it is thermodynamically suggested that impurities are unlikely

to have affected the amount of $M_{23}C_6$; however, experimental validation remains a future issue.

Figure 12 shows the hardness of the grip portions of the crept specimens. After approximately 15 000 h, Steel 1 tended to exhibit slightly lower hardness than Steel 2,

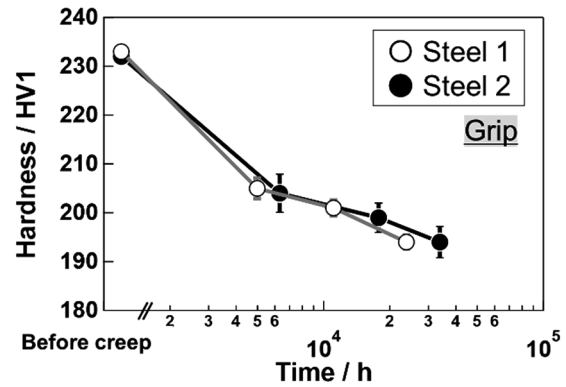


Fig. 12. Hardness of grip portion in the crept specimens.

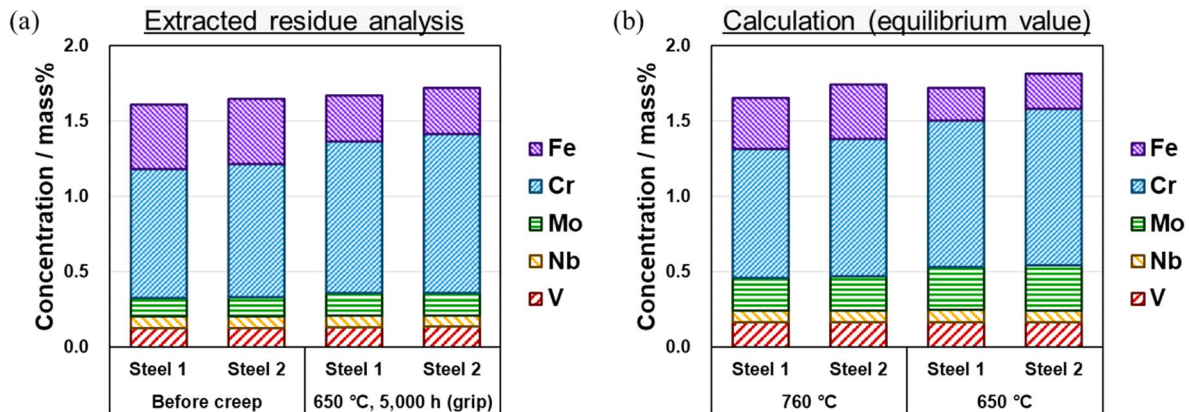


Fig. 11. (a) Extracted residue analysis results exhibiting the concentrations of precipitates in the initial materials (after tempered at 760°C) and the grip portion of creep specimens exposed for approximately 5 000 h at 650°C. (b) The equilibrium concentration at 760 and 650°C calculated by Thermo-Calc with SSOL4 database. (Online version in color.)

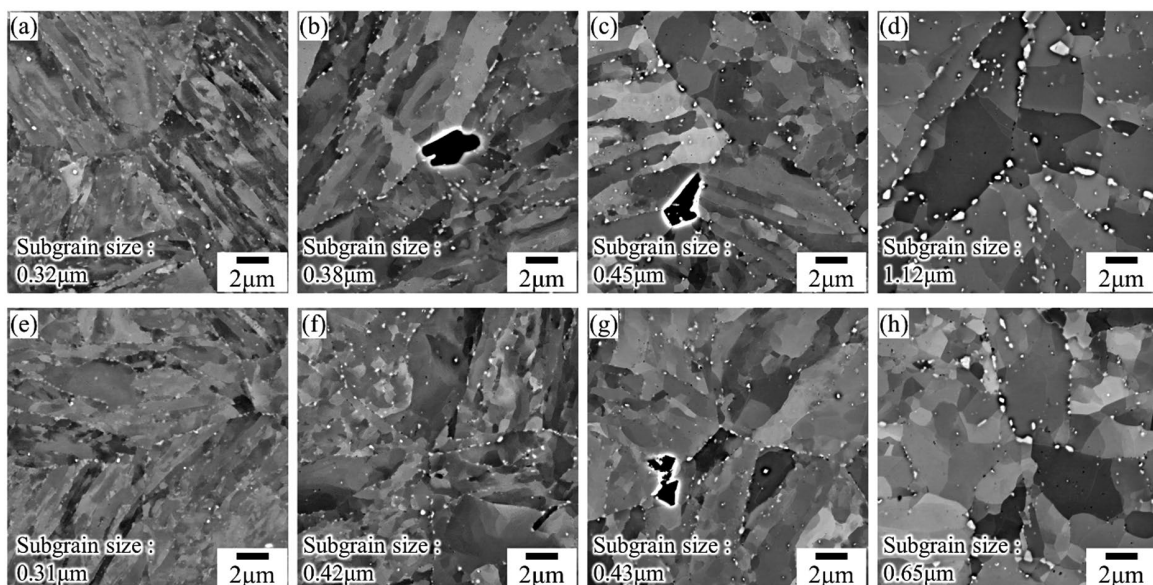


Fig. 13. BSE images of (a)–(d) Steel 1 and (e)–(h) Steel 2. (a), (e) Before creep, (b), (f) crept for approximately 5 000 h and (c), (g) approximately 10 500 h, and (d), (h) ruptured specimens under 60 MPa.

implying that microstructural recovery in Steel 1 may have progressed more rapidly than in Steel 2. To clarify the microstructural changes associated with creep deformation, the gauge portions of the crept specimens under 60 MPa were examined. As described in section 2, the creep times for interruption tests were set at two conditions: one before the transition to acceleration creep (at around minimum creep rate), and the other during acceleration creep for both Steel 1 and Steel 2 (see Fig. 2). Cross-sectional BSE images of the initial material and crept specimens are shown in Fig. 13. In Steel 1, creep voids were observed in the specimen crept for approximately 5 000 h, but their amount was very limited. On the other hand, in Steel 2, creep voids were hardly observed in the specimen crept for approximately 5 000 h. In the specimen of Steel 2 crept for approximately 10 500 h, a small number of creep voids were observed. In the BSE images in Fig. 13, subgrain structures were observed. With increasing creep time, microstructural recovery appeared to progress, and the subgrains seemed to coarsen. The subgrain size estimated by Eq. (2) is also shown in the figure. When comparing the initial materials, the subgrain sizes of Steel 1 and Steel 2 were comparable. Similarly, in the specimens crept for up to approximately 10 500 h, the estimated subgrain sizes of both steels were comparable; however, in the ruptured specimens, the subgrain size was larger in Steel 1.

4. Discussion

From Fig. 4(a), when comparing under the same stress conditions, the reduction of area for Steel 1 was lower than that for Steel 2, whereas elongation for Steel 1 was higher than that for Steel 2. In the first half of this section, the association between reduction of area and elongation is discussed using the following estimation results. Attention is focused on Fig. 5(c), which shows the diameter of the ruptured specimens under 80 MPa with a relatively thin oxide scale.

Here, the total deformation amount of the gauge portion (l) was estimated, where $l = l_{\text{hetero}} + l_{\text{homo}}$. l_{hetero} and l_{homo} are the contributions to l from the regions within and beyond 7.5 mm on both sides of the fracture surface, respectively. First, l_{hetero} was obtained by the following procedure. From the diameter at each measurement point (d_i) in Fig. 5(c), the cylindrical volume $V_i (= \pi d_i^2 p / 4)$ was calculated at the measurement intervals ($p = 0.2$ mm). Assuming that this volume is constant before and after creep deformation, the original length along the longitudinal direction (p_i') before

deformation was calculated from V_i and the initial diameter ($d_0 = 6$ mm). Here, $p_i' = 4V_i / \pi d_0^2 = d_i^2 p / d_0^2$. This calculation was performed over a range of 7.5 mm on both sides of the fracture surface (total length of 15 mm), and the sum ($\Sigma p_i'$) was obtained. The value obtained by subtracting $\Sigma p_i'$ from 15 mm was taken as l_{hetero} . Next, the total deformation amount of the gauge portion l was obtained by multiplying the gauge length by the elongation. l_{homo} was then estimated as $l - l_{\text{hetero}}$.

The estimation results are shown in Table 2. For Steel 1, both the contributions of l_{hetero} and l_{homo} to the elongation were greater than those for Steel 2. Greater contribution of l_{hetero} in Steel 1 was attributed to wider range of necking compared to Steel 2. Table 2 reveals sufficient deformation of Steel 1 in the gauge portion, including the fracture surface. Therefore, although Steel 1 showed a lower reduction of area, elongation did not accordingly decrease. For specimens ruptured under stresses below 80 MPa, it was difficult to perform similar estimations due to the significant influence of oxide scale. However, considering that the increase in creep strain just before rupture was also greater in Steel 1 than in Steel 2 under 60 MPa (see Fig. 2), it is expected that a similar trend to Table 2 would be obtained under 60 MPa as well.

It is reasonable to consider that the decreased reduction of area for Steel 1 is due to the higher density of creep voids.⁴⁷⁾ In our previous study,³⁰⁾ segregation of Sn near PAGB in Steel 1 was observed by three-dimensional atom probe tomography. Therefore, creep voids formed at PAGB were quantitatively estimated using the specimen crept for approximately 10 500 h under 60 MPa. Figure 14 shows IPF and PAGB maps for Steel 1 and Steel 2. Creep voids appeared black in IPF map and white in PAGB map, respectively. It is found that most of creep voids formed at PAGB. Similar observations were conducted for three fields in Steel 1 and five fields in Steel 2 (due to the low density of creep voids in Steel 2). The number density of creep voids formed at PAGB [number/mm²] was evaluated as 207 ± 28 for Steel 1 and 89 ± 18 for Steel 2, revealing that the number

Table 2. Magnitude of the deformation l in the ruptured specimens under 80 MPa. l_{hetero} and l_{homo} demonstrate the contribution to the deformation in the regions within and beyond 7.5 mm from the fracture surface, respectively.

	l	l_{homo}	l_{hetero}
Steel 1	9.6 mm	2.6 mm	7.0 mm
Steel 2	8.5 mm	2.2 mm	6.3 mm

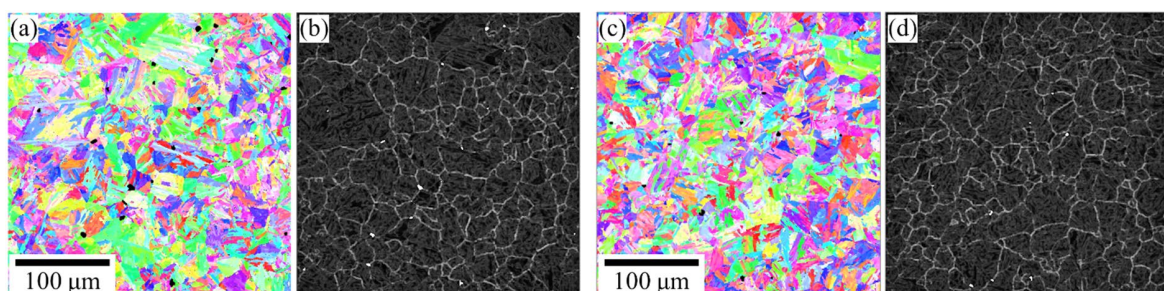


Fig. 14. EBSD maps of (a), (b) Steel 1 and (c), (d) Steel 2 crept for approximately 10 500 h under 60 MPa. (a), (c) IPF and (b), (d) PAGB maps. (Online version in color.)

of creep voids formed in Steel 1 was more than twice that in Steel 2. When impurities segregate near grain boundaries, the surface energy of voids generated at grain boundaries is considered to decrease; therefore, the critical nucleus size of creep voids decreases.^{48,49} Accordingly, impurity segregation promotes the formation of creep voids, resulting in a slight decrease in reduction of area for Steel 1. On the other hand, no clear correlation was observed between elongation or the creep intolerance parameter λ and impurity concentration. When evaluating the creep ductility of Gr. 91, it is desirable to comprehensively take these indicators into consideration.

Compared to Steel 2, many creep voids formed in Steel 1, which could raise interest in their potential impact on creep strength. For example, in Gr. 92 steel, it has been reported that the creep void formation adjacent to coarse Laves phase particles can be a factor in the decrease of long-term creep rupture strength.^{36,50} In the present study, however, even though fewer creep voids were observed under higher stress conditions (Figs. 7(a), 7(b)), differences in the minimum creep rate and time to rupture between Steel 1 and Steel 2 were found under all stress conditions. Sekido *et al.* reported no significant difference in creep rupture strength between two heats of P91 with different rupture ductility and amounts/distributions of creep voids.⁴⁷ Furthermore, according to the creep fracture mechanism map for Gr. 91 steel proposed by Shrestha *et al.*,⁵¹ all creep test results in the present study fall within the microstructural degradation

domain (ductile fracture region). Considering these experimental facts, it is suggested that the factors responsible for higher minimum creep rate and earlier rupture in Steel 1 are not necessarily related to creep voids. Although there was also a difference in W concentration between the two steels (see Table 1), slightly higher concentration of W, a strengthening element, in Steel 1 was inconsistent with the creep test results in this study. The differences in creep strength and rupture ductility between Steel 1 and Steel 2 could not be explained by W concentration.

From Fig. 10 and Figs. 13(a), 13(e), there were no significant differences in the initial microstructures between Steel 1 and Steel 2. However, hardness changes in the grip portion of the crept specimens (Fig. 12) imply that microstructural recovery in Steel 1 progressed more rapidly than in Steel 2. In other words, Steel 1 may have exhibited the microstructure with lower creep resistance compared to Steel 2. Based on Figs. 2(c), 2(f) and 5(c), it is suggested that localized deformation near the fracture surface accompanied by rupture (necking) in Steel 1 was more extensive than in Steel 2. These creep test results also support the possibility that the microstructure of Steel 1 exhibited lower creep resistance.

Incidentally, packet and block boundaries migrate and merge during creep deformation. Therefore, focusing on the common rotation axes and rotation angles (misorientation angles) of grain boundaries, martensite structure was analyzed.^{52,53} In this analysis, the threshold angle (allowable error) for the rotation axis was set to 5°. Histograms of mis-

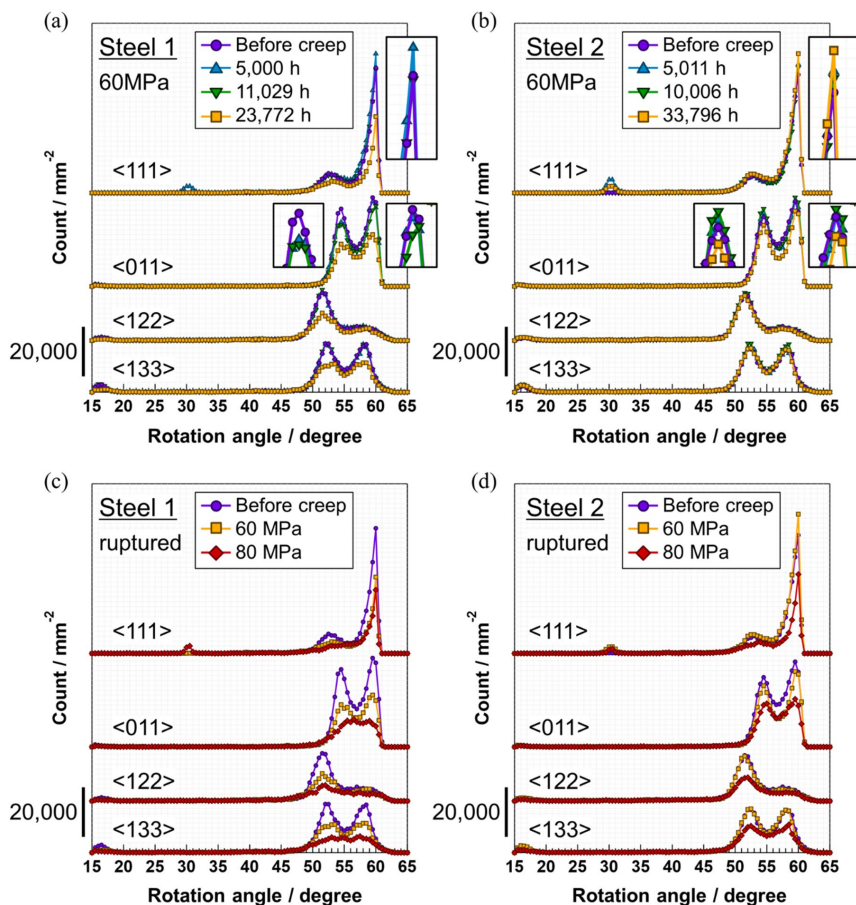


Fig. 15. Histograms for the rotation angles of grain boundaries classified by each type of common rotation axis in (a), (c) Steel 1 and (b), (d) Steel 2. Comparison between (a), (b) before and after creep under 60 MPa, and (c), (d) 80 and 60 MPa. (Online version in color.)

orientation angles for four common rotation axes ($\langle 011 \rangle$, $\langle 111 \rangle$, $\langle 122 \rangle$, $\langle 133 \rangle$) in the specimens crept at 650°C under 60 MPa are shown in Figs. 15(a), 15(b). Most of the peaks observed in $\langle 111 \rangle$ and $\langle 011 \rangle$ histograms are related to block boundaries, while peaks in the $\langle 122 \rangle$ and $\langle 133 \rangle$ histograms are relevant to packet boundaries.⁵⁴⁾

First, changes in the histograms with increasing creep time were examined. In Steel 1, $\langle 011 \rangle 53^\circ$ and $\langle 011 \rangle 60^\circ$ peaks slightly decreased at around 11 000 h. Up to approximately 11 000 h, the histograms of $\langle 111 \rangle$, $\langle 122 \rangle$, and $\langle 133 \rangle$ showed little change. In the ruptured specimen, peaks in all four histograms decreased. The decreased crystallographic relationships of block/packet boundaries imply that their migration and merging progressed with creep deformation. This migration and merging are more likely to occur at block boundaries than at packet boundaries.^{37,54)} At approximately 11 000 h, although $\langle 011 \rangle 53^\circ$ and $\langle 011 \rangle 60^\circ$ peaks decreased, the decrease in $\langle 111 \rangle 60^\circ$ peak was hardly observed. This suggests that block boundary with $\langle 111 \rangle 60^\circ$ is the least susceptible to creep deformation among the block boundaries. It has been reported that the grain boundary energy of $\langle 111 \rangle 60^\circ$ is less than half that of other block boundaries.⁵⁵⁾ Assuming that lower grain boundary energy correlates with sensitivity to creep, the changes in the histograms in Figs. 15(a), 15(b) are reasonable. In contrast, in Steel 2, all histograms showed little change at about 10 000 h, and only the $\langle 011 \rangle 53^\circ$ and $\langle 011 \rangle 60^\circ$ peaks slightly decreased in the ruptured specimen. Next, comparing the initial materials of Steel 1 and Steel 2, there were no significant differences in the shapes of the four histograms. However, the four histograms in Steel 1 crept for approximately 11 000 h were comparable to those in the ruptured specimen of Steel 2. This indicates that the crystallographic relationship of block boundaries in Steel 1 disappeared earlier. Comparing the ruptured specimens, the decrease in the histogram peaks was more pronounced in Steel 1 than in Steel 2.

Figures 15(c), 15(d) show histograms of misorientation angles for each common rotation axis in the specimens ruptured under 80 MPa. For comparison, histograms for the specimens ruptured under 60 MPa are also shown in Figs. 15(c), 15(d). First, comparing 60 MPa and 80 MPa, it is evident in both Steel 1 and Steel 2 that the peaks in histograms disappeared to a greater extent under higher stress. Next, focusing on the histograms of specimens ruptured under 80 MPa, a comparison between Steel 1 and Steel 2 reveals that the decrease in peaks was more pronounced in Steel 1, as in the case of 60 MPa.

According to Tamura *et al.*, the stabilization of grain boundaries (particularly block boundaries) has the effect of delaying the onset of acceleration creep and improving creep strength.⁵⁶⁾ In other words, based on the results in Fig. 15, this study clarified that the differences in creep strength and rupture ductility between Steel 1 and Steel 2 were attributable to the ease of migration and merging of block/packet boundaries. Relatively large scatter in the average diameter of $M_{23}C_6$ in Steel 1 (see Fig. 10) implies the possibility that coarsening kinetics of the particles were affected by impurities due to certain factors, such as changes in the interfacial energy. Moreover, the amount of $M_{23}C_6$ in Steel 1 was slightly lower than that in Steel 2 (see Fig. 11). Consider-

ing these experimental results, $M_{23}C_6$ may have affected the ease of migration and merging of block/packet boundaries. Based on the thermodynamic calculation, the difference in $M_{23}C_6$ amount between the two steels originated from the concentration of C (see Table 1). Although many researchers investigated the causes of heat-to-heat variation of creep strength in Gr. 91 steel,¹⁻⁴⁾ no reports have pointed out a significant impact of C in the range of commercial composition of this type of steel. Therefore, it is doubtful that the difference in the ease of migration and merging of block/packet boundaries between the two steels was solely attributable to $M_{23}C_6$. One possible scenario is changes in the grain boundary energy induced by segregation.⁵⁷⁾ The grain boundary energy may correlate with susceptibility to creep deformation, as discussed above. However, it is not easy to evaluate the effect of impurities on the grain boundary energy using any computational or experimental methodology due to the possibility of site competition or interactions between solute atoms in multicomponent alloys. Elucidating the essential effects of impurity elements on creep strength remains a subject for future investigation.

5. Conclusions

Creep tests were conducted using the two steels: one containing high impurities (Steel 1) and the other with low impurities (Steel 2). The key findings of this study are as follows.

(1) Steel 1 exhibited a higher minimum creep rate and ruptured in a slightly shorter time. Both Steel 1 and Steel 2 exceeded the average creep strength of Gr. 91 steel under all test conditions.

(2) Steel 1 tended to show a lower reduction of area due to higher density of creep voids. On the other hand, the deformation in the gauge portion, including the fracture surface, was greater in Steel 1 than in Steel 2. Consequently, no clear correlation was observed between impurity concentrations and either elongation or the creep intolerance parameter (λ).

(3) Fewer creep voids were observed under higher stress conditions, whereas the minimum creep rate and time to rupture were different between Steel 1 and Steel 2 under all stress conditions. This experimental result suggests that creep voids are unlikely to be a main factor affecting creep strength.

(4) The disappearance of crystallographic relationship at block/packet boundaries during creep was more pronounced in Steel 1 than in Steel 2, implying that creep strength of Gr. 91 steels could be related to the stability of block/packet boundaries. Although impurity elements and $M_{23}C_6$ may have been related to the disappearance of the above crystallographic relationship, fundamental elucidation of the mechanism remains an issue for future work.

Statement for Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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