

## MICROSTRUCTURAL EVALUATION IN HEAT-AFFECTED ZONE OF 9Cr-3W-3Co-Nd-B HEAT-RESISTANT STEEL

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### ABSTRACT

A newly developed ferritic heat-resistant steel; 9Cr-3W-3Co-Nd-B steel has higher creep rupture strength both in the base metal and welded joints than the conventional high-Cr ferritic heat-resistant steels. The creep rupture strengths of 9Cr-3W-3Co-Nd-B steel welded joints were below the lower limit of the base metal in long-term creep stage more than 20,000 hours. The creep rupture position was heat-affected zone (HAZ) from 1.0 to 1.5 mm apart from the fusion line on the welded joint specimen ruptured at 34,966 hours. The equi-axed subgrains and coarsened precipitates were observed in HAZ of the ruptured specimen. In order to clarify the creep fracture mechanism of the welded joints, the microstructures of HAZ were simulated by heat cycle of weld, then observed by EBSD analysis. Fine austenite grains formed along the prior austenite grain boundaries in the material heated just above  $A_{C3}$  transformation temperature, however there were no fine grains such as conventional steel welded joints. The prior austenite grain boundaries were unclear in the material heated at 1050 °C. The creep rupture life of the material heated at just above  $A_{C3}$  transformation temperature exceeded the lower limit of base metal and there was no remarkable degradation, although it was shorter than the other simulated materials. It is, therefore, concluded that the creep fracture of 9Cr-3W-3Co-Nd-B steel welded joint in long-term stage occurred at HAZ heated at from just above  $A_{C3}$  transformation temperature to 1050 °C. It is speculated that the fine austenite grains formed along the prior austenite grain boundaries and inhomogeneous microstructures cause the coarsening precipitates and recovery of lath structure during long-term creep deformation.

### INTRODUCTION

High Cr ferritic steels such as ASME Gr. 91, Gr. 92, and Gr. 122 have been widely used for large-diameter and thick-wall pipes for fossil-fired power plant boilers. However, it is well-known that the degradations of creep rupture strength are found in the base metal of these steels in long-term stage, and the abrupt rupture is occurred in fine grained heat-affected zone (FGHAZ) in these conventional steel welded joints, called type IV failure. Liu, et al. reported that the main cause of Type IV failure was the insufficient precipitates at various

boundaries<sup>[1]</sup>. To suppress this failure, the addition of boron and minimization of nitrogen were considered effective, because boron segregated on the grain boundaries and reduced grain boundary energy resulting in the suppression of  $\alpha$  to  $\gamma$  diffusive transformation<sup>[2],[3]</sup>.

Developed 9Cr-3W-3Co-Nd-B steel has good creep rupture strength in both base metal and welded joint owing to optimizing the contents of elements while considering not only creep properties but also weldability, ductility, and manufacturability<sup>[4]</sup>. The chemical composition ranges of 9Cr-3W-3Co-Nd-B steel are shown in Table 1. It has been already registered as ASME Code Case 2839.

In this study, in order to clarify the creep fracture mechanism of welded joints of developed steel, we evaluated the long-term creep rupture properties of the welded joints and observed the microstructures after creep deformation. Then, we fundamentally investigated the microstructures and creep rupture properties of HAZ.

*Table 1 Chemical composition ranges of 9Cr-3W-3Co-Nd-B steel (mass %)*

ASME Code Case	C	Si	Mn	P	S	Cr	Ni	W	Co	V	Nb+Ta	Nd	B	sol. Al	N	O
2839	0.05 /0.10	0.05 /0.50	0.20 /0.70	≤0.020	≤0.008	8.50 /9.50	≤0.20	2.5 /3.5	2.5 /3.5	0.15 /0.30	0.05 /0.12	0.010 /0.060	0.007 /0.015	≤0.030	0.005 /0.015	≤0.0050

## EXPERIMENTAL PROCEDURES

The welded joint tubes were prepared by automatic gas tungsten arc welding with two types of commercial filler wires; AWS A5.28 ER90S-G and A5.14 ERNiCr-3, then applying post weld heat treatment (PWHT) at 740 °C for 30 min. The tube size was 38.1 mm in outer diameter and 8.8 mm in wall thickness. The creep rupture tests of welded joints were conducted at 600 and 650 °C with applying stress of 78.5 to 215.7 MPa. The microstructures of crept specimen which ruptured at 34,966 hours tested at 650 °C with 78.5 MPa were observed by optical microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM). TEM observations were carried out by using thin foils which were thinned by focused ion beam (FIB) technique. In order to evaluate the microstructures of HAZ, normalized and tempered laboratory melted plate were prepared and applied welding heat cycles. Figure 1 shows simulated heat cycles with a heating rate of 67 °C/s, holding for 1 second at 750 °C, 910 °C, 1050 °C, 1150 °C and 1350 °C, and cooling by blowing helium gas. The microstructures after each cycle were observed by electron backscatter diffraction (EBSD) method. Then, their austenite grains were distributed by the reconstructed method<sup>[5]</sup>. Creep rupture specimens were cut out from bar test pieces which were applied simulated PWHT at 740 °C for 30 min after tempering heat treatment and heat cycle simulations of weld. It is noted that the holding temperature just above  $A_{C3}$  transformation temperature changed from 910 °C to 920 °C, because different material of 9Cr-3W-3Co-Nd-B steel was used for the creep tests. The creep tests were conducted at 650 °C with applying stress of 120 MPa.

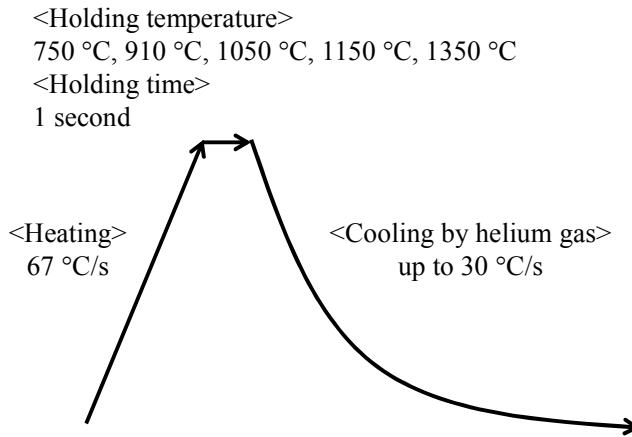


Figure 1: Heat cycle simulation of weld

## RESULTS AND DISCUSSION

### Creep Rupture Strength of Welded Joints

Figure 2 shows the creep rupture strength of 9Cr-3W-3Co-Nd-B steel welded joints tested at 600 °C and 650 °C with the average and lower limit curves of base metal. WM, BM, FL, and HAZ in the figure indicate that specimens ruptured at "Weld Metal", "Base Metal", "Fusion Line", and "Heat-Affected Zone", respectively. All specimens ruptured less than 20,000 hours exceeded the minimum strength curve of base metal. There was no remarkable difference in the rupture life between two types of welded joints. The ruptured positions, in contrast, were different depending on the weld metal. The welded joint with commercial filler ER90S-G ruptured at WM in short-term stage, whereas that with Ni-based filler ERNiCr-3 ruptured at base metal or fusion line. On the other hand, a slight degradation was confirmed in the long-term stage in both welded joints against lower limit curve of base metal. They ruptured at HAZ about 1.5 mm away from fusion line. However, the severe degradation of creep rupture strength against base metal exhibits over 5,000 hours at 600 °C in Gr. 91 and Gr. 92 steel welded joints [6],[7]. It is, therefore, suggested that the degradation is mitigated in 9Cr-3W-3Co-Nd-B steel welded joints compared with conventional steel welded joints, although more creep rupture tests are needed with lower stress.

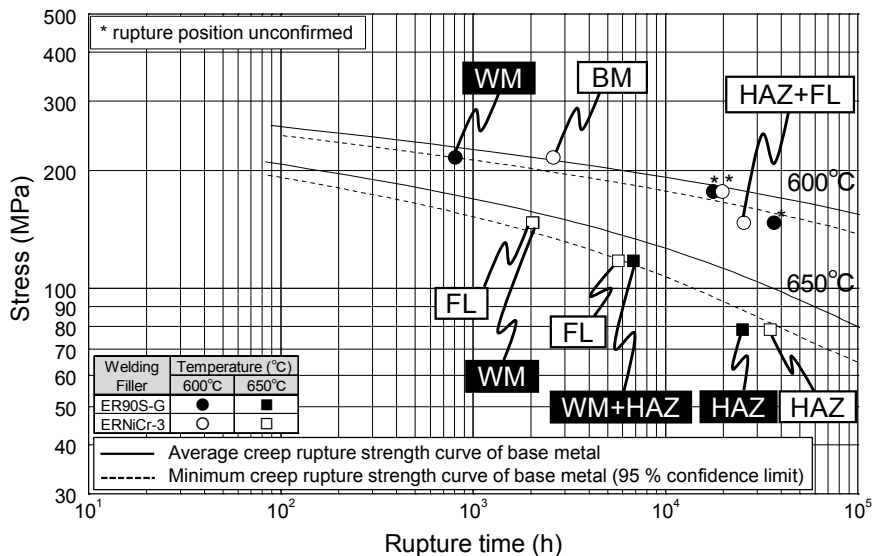


Figure 2: Creep rupture strength of 9Cr-3W-3Co-Nd-B steel welded joints

### Microstructures of Welded Joint after Creep Rupture

Figure 3 shows the optical micrograph of ruptured specimen of welded joint with ERNiCr-3 filler which ruptured at 34,966 hours tested at 650 °C with 78.5 MPa. The fracture was occurred at HAZ from 1.0 to 1.5 mm apart from the fusion line. The coarsened grains were observed in the middle in thickness of ruptured specimen. It is considered that this microstructure was influenced by the heat cycle of weld, then it changed from martensite structures to the coarsened grains during creep deformation. Figure 4 shows SEM micrographs of base metal (10 mm apart from FL) and HAZ about 0.5 mm apart from FL. It is noted that observed HAZ was different area from the coarsened grains as mentioned above. It is seen that there were about 1 μm of precipitates on the grain boundaries and fine precipitates inside grains in the microstructure of base metal. On the other hand, coarsened precipitates distributed mainly on the grain boundaries in the microstructure of HAZ. In addition, about 1 μm of creep voids were observed on the grain boundaries in HAZ. Thin foil for TEM observation was taken from the HAZ about 1.0 mm apart from FL by FIB technique. Figure 5 shows observed TEM micrograph. It is seen that there were remarkable equi-axed subgrains, and fine  $M_{23}C_6$  carbides and coarse Laves phase were found inside grains and on the grain boundaries, respectively. It is, therefore, considered that the creep fracture of 9Cr-3W-3Co-Nd-B steel welded joints in long-term stage occurred at HAZ with coarsened precipitates and a formation of equi-axed subgrains.

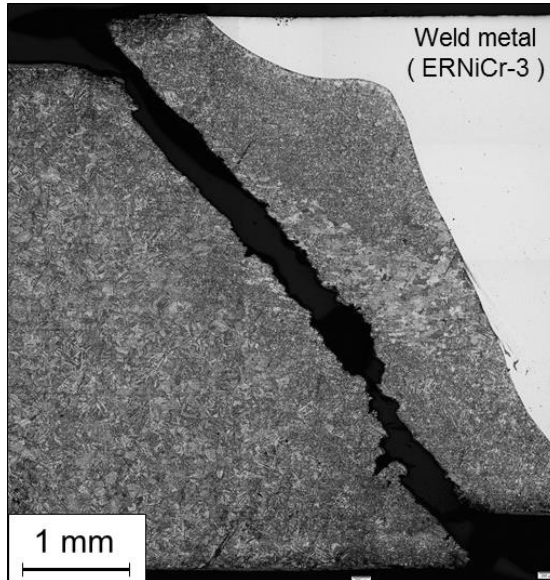


Figure 3: Optical micrograph of 9Cr-3W-3Co-Nd-B steel welded joint ruptured at 34,966 hours tested at 650 °C with 78.5 MPa

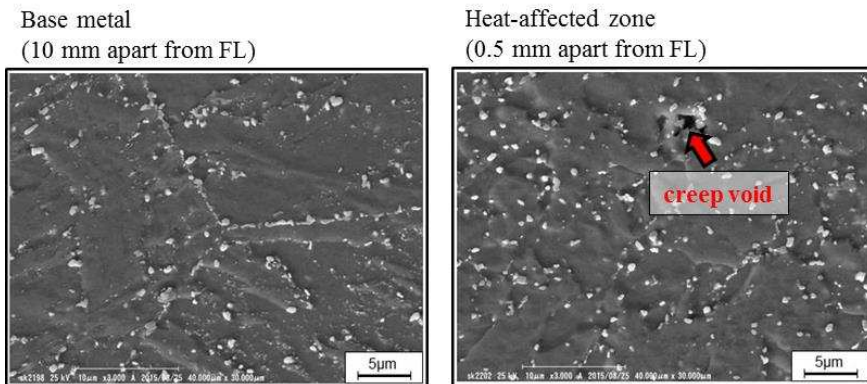


Figure 4: SEM micrographs of 9Cr-3W-3Co-Nd-B steel welded joint ruptured at 34,966 hours tested at 650 °C with 78.5 MPa

Heat-Affected-Zone (1.0 mm apart from FL)

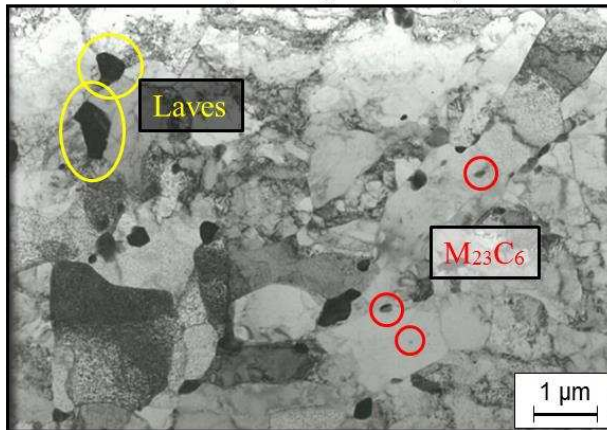


Figure 5: TEM micrograph of 9Cr-3W-3Co-Nd-B steel welded joint ruptured at 34,966 hours tested at 650 °C with 78.5 MPa

### Microstructures of Heat-Affected-Zone

Figure 6 shows the orientation maps of 9Cr-3W-3Co-Nd-B steel after welding heat cycle simulations and reconstruction of prior austenite grains. The grain boundaries where the misorientation is larger than 15 degrees are described by a black line in the figure. Martensite lath structure was observed in the material heated at 750 °C which was below  $A_{C1}$  transformation temperature. The reconstructed austenite grains are suggested the microstructure formed during normalizing heat treatment. Fine austenite grains formed along the prior austenite grain boundaries in the material heated at 910 °C, however there were no fine grains such as conventional steel welded joints [8]-[10]. It is assumed that no fine grains form in this temperature due to the suppression of diffusive transformation with the segregation of boron at the prior austenite grain boundaries [2]. The prior austenite grain boundaries were unclear in the material heated at 1050 °C. This microstructure was considered to be formed with the coarsened fine grains along the prior austenite grain boundaries observed in the material heated at 910 °C and shear type inverse transformation and recrystallization. Homogeneous grains were confirmed in the material heated at 1150 °C. There were prior austenite grains of mixed sizes in the material heated at 1350 °C. Austenite and delta-ferrite phases are stable at 1350 °C thermodynamically, so that the microstructure is estimated to be formed with the austenitic inverse transformation and the formation of delta-ferrite.

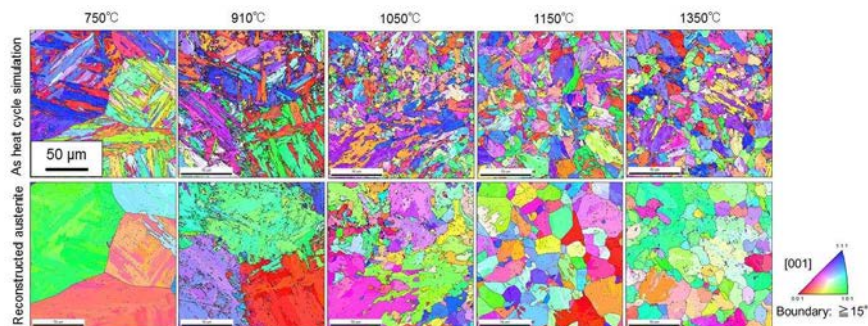


Figure 6: Orientation maps of microstructures after heat cycle simulation of weld and reconstructed austenite microstructures

### Creep Rupture Strength of Heat-Affected-Zone

Figure 7 shows the creep rupture strength of tempered and simulated HAZ specimens with heat treatment at 740 °C for 30 min. It is noted that the specimen heated at 1050 °C is before testing. It can be seen that creep rupture life of the material heated at 920 °C was shorter than the other materials, however it exceeded the lower limit of base metal and there was no remarkable degradation of creep strength in the material heated just above  $A_{C3}$  transformation temperature such as reported Gr. 92 steel<sup>[11]</sup>. It is reported that the main cause of Type IV failure was the insufficient precipitates at various boundaries<sup>[1]</sup>. The microstructure of the material heated at 920 °C had fine grains along grain boundaries shown in figure 6, so that the precipitates on the grain boundaries might be insufficient resulting in the shorter life than the other materials. On the other hand, the material heated at 1350 °C had enough creep rupture strength. It is assumed that the particles precipitated during tempering dissolved in heat cycle of weld and generated on newly grain boundaries during PWHT resulting in good creep rupture strength.

As a results of the microstructural observations and evaluation of creep rupture strength shown in figures 6 and 7, it is considered that the creep fracture of 9Cr-3W-3Co-Nd-B steel welded joint in long-term stage occurred at HAZ heated at from just above  $A_{C3}$  transformation temperature to 1050 °C. It is speculated that the fine austenite grains formed along the prior austenite grain boundaries and inhomogeneous microstructures cause the coarsening precipitates and recovery of lath structure during creep deformation.

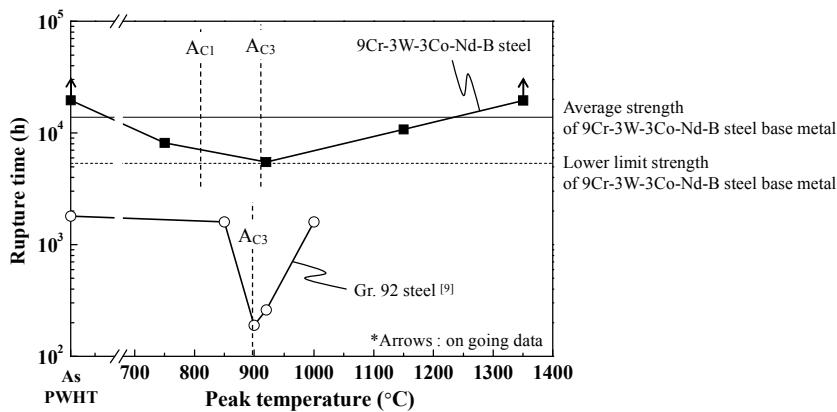


Figure 7: Creep rupture strength of simulated HAZ specimens tested at 650 °C with 120 MPa

## CONCLUSIONS

To understand the creep fracture mechanism of developed 9Cr-3W-3Co-Nd-B steel welded joints, we evaluated creep rupture strength of the welded joints, then observed the microstructures after creep deformation. Furthermore, we investigated the microstructures and creep rupture life of HAZ.

(1) 9Cr-3W-3Co-Nd-B steel welded joints had superior creep rupture strengths to conventional steel welded joints, and there were no severe degradation of creep rupture life even in long-term stage such as conventional high-Cr ferritic steel welded joints.

(2) The microstructural observations of HAZ after long-term creep deformation clarified the distribution of coarsened precipitates and the formation of equi-axed subgrains.

(3) The microstructural observations on the materials simulated HAZ revealed there were no fine grains such as conventional steel welded joints in the material heated at just above  $A_{C3}$  transformation temperature, although the fine austenite grains formed along the prior austenite grain boundaries. In addition, the prior austenite grain boundaries were unclear in the material heated at 1050 °C.

(4) The creep rupture life of the material heated at just above  $A_{C3}$  transformation temperature exceeded the lower limit of base metal and there was no remarkable degradation, although it was shorter than the other materials.

It is, therefore, concluded that the creep fracture of 9Cr-3W-3Co-Nd-B steel welded joint in long-term stage occurred at HAZ heated at from just above  $A_{C3}$  transformation temperature to 1050 °C. It is speculated that the fine austenite grains formed along the prior austenite grain boundaries and inhomogeneous microstructures cause the coarsening precipitates and recovery of lath structure during long-term creep deformation.



## REFERENCES

- [1] Liu, Y. *et al.*: Metallurgical and Materials Transaction A. 44A, 4626 (2013)
- [2] Abe, F. *et al.*: Materials at High Temperatures. 23 (3–4), (2006)
- [3] Liu, Y. *et al.*: Metallurgical and Materials Transaction A. 45A, 1306 (2014)
- [4] Hamaguchi, T. *et al.*: Nippon Steel & Sumitomo Metal Technical Report, No. 119, 32
- [5] Hata, K. *et al.*: Nippon Steel & Sumitomo Metal Technical Report, No. 114, 26
- [6] Yaguchi, M, *et al.*: PVP2012-78393 (2012)
- [7] Watabe, T., *et al.*: Tetsu-to-Hagane, Vol. 90(2004), No. 4, 206
- [8] Matsui, M. *et al.*: ISIJ International. 41, S126 (2001)
- [9] Hasegawa, Y. *et al.*: Tetsu-to-Hagané. 90 (10), 609 (2004)
- [10] Sakthivel, T. *et al.*: Materials Science and Engineering A. 591, 111 (2014)
- [11] Matsui, M. *et al.*: ISIJ International, Vol. 41 (2002), Supplement, S126-S130