



## MICROSTRUCTURE AND CREEP STRENGTH OF GRADE 91 STEEL USED IN USC PLANTS

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### ABSTRACT

ASME Grade 91 steel seam-welded elbow pipe, which has been used in a USC plant (A-Plant) for about  $6 \times 10^4$  h, was investigated to clarify the microstructure and remaining creep life of the material at long-term region. SEM and TEM observations were conducted on specimens cut from the welded portions of the intrados and extrados of the elbow, and the number density of creep voids in fine-grained HAZ was measured in the wall-thickness direction. Then, creep rupture tests were performed to examine the remaining life of each portion of the base metal and welded joint. On the basis of the results, it was suggested that the microstructural changes were small and that the cumulative creep damage was also small for the elbow pipe during its use at the USC plant for about  $6 \times 10^4$  h. The present result was compared with the result of an investigation on Grade 91 steel elbow used in another USC plant (B-Plant) for about  $5 \times 10^4$  h. The A-Plant material had a creep life about ten times longer than that of the B-Plant material for not only the base metals but also the welded joint. Through the comparison of the investigation results, it was suggested that the difference in the creep deformation property between the base metals of the elbows was the main reason for the difference in their creep lives.

### INTRODUCTION

ASME Grade 91 steel has a high creep strength and has been used as the material of piping in 600 °C class thermal power plants in the world. However, the creep strength of the steel for a long-term region has been found to be lower than first expected. In 2005, the creep strength of the material was reevaluated in Japan using the latest data at the time <sup>[1],[2]</sup>. Then, reevaluation of the creep strength of the material was once again conducted on the basis of the new data set in Japan in 2010 <sup>[3],[4]</sup>. One of the reason of the reevaluations was that the amount of data on creep rupture of the steel for the long-term region is insufficient for evaluation. The materials used in actual power plants are useful in estimating the changes of material properties caused by long-term aging and damage at low stress conditions; however, there are only a limited number of reports on the kinds of Grade 91 steel. Thus, the observation of the microstructure and examination of the creep strength on long-term used Grade 91 steel are strongly demanded because operation times of power plants, which have used the steel as high temperature pipes, exceeds the longest rupture time of experimental data by far.

With the above background, CRIEPI and NIMS carried out a joint study on the observation of the microstructure and the assessment of the creep strength of elbow pipes of ASME Grade 91

steel used for a long time in 600 °C class thermal power plants. This paper summarizes results obtained in the study.

## MATERIALS

In this study, longitudinally welded elbow removed after their use as high-temperature reheat steam pipes for a long time in a thermal power plant were used as materials. The base metal is ASME Grade 91 steel plate (KA-SCMV28) with nominal dimensions of 711 mm diameter, 39 mm thickness, and 710 mm bending radius. Table 1 shows a summary of the chemical composition of the material.

Table 1: Chemical compositions of material

C	Si	Mn	P	S	Ni	Cr	Mo	V	Nb	N	wt%
0.11	0.33	0.45	0.007	0.002	0.07	8.55	0.97	0.19	0.08	0.051	
O	Al	Ti	Zr	Cu	As	Sn	Sb	B	Pb		
0.003	0.004	0.001	<0.001	0.01	0.002	<0.001	<0.001	0.0002	<0.001		

The operating conditions of the plant were a steam temperature of 593.6 °C, steam pressure of 4.7 MPa, and cumulative operating time of ~61,000 h. Here, the cumulative operating time includes the duration of operation at reduced temperatures.

The data for the observation of microstructure and those used in the creep rupture test were collected at the axial center (45°) of the intrados side of the elbow pipes (where the circumferential stress is maximum with respect to longitudinal welded joints during the application of internal pressure) and the extrados side of the elbow pipes, respectively.

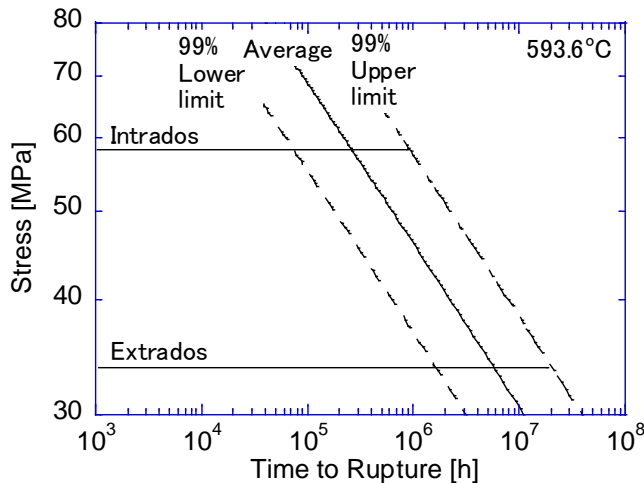


Figure 1: Creep strength property<sup>[4]</sup> of Gr.91 steel welded joint and hoop stresses applied to welded joints of the elbow

The circumferential stress applied to the curved pipes is calculated using the equation of mean diameter. The stress applied to the intrados and extrados sides of the elbow pipes is 58.2 and 33.7 MPa, respectively. Figure 1 shows the relationship between the creep strength of the RHC

committee and the stress of the welded joints of Grade 91 steel at the operating temperature, where RHC committee stands for “Committee on Review on Reliability of High Temperature Strength Enhanced Ferritic Steels for Fitness-for-service of Thermal Power Component” and the values of the creep strength were based on the reevaluation conducted in 2010<sup>[3], [4]</sup>. From the stress and the RHC regression curve, the creep rupture time of the material was calculated, and then the creep damage was calculated from the ratio of the cumulative operating time to the creep rupture time. The creep damage at the intrados side of the welded region was 23% on the basis of the mean properties and 83% on the basis of the 99% lower limit. The creep damage at the extrados side of the welded region was 1% on the basis of the mean properties and 4% on the basis of the 99% lower limit. Here, the above values were calculated assuming that the steam temperature is constant at 593.6 °C over the cumulative operating time without considering the effect of the duration of operation at reduced temperatures.

## MICROSTRUCTURE OBSERVATION

### Measurement of Hardness

Figure 2 shows a typical distribution of hardness in the circumferential direction (intrados side of welded region) measured using a micro-Vickers-hardness testing machine. The load was 1.961 N and the indentation time was 10 s. The center of the abscissa corresponds to the center of the weld metal. The hardnesses of the base metal in the intrados and extrados sides were in the range of 210–220 HV. The hardnesses of the softest and hardest regions were 190–200 and ~260 HV, respectively, showing no distinct difference between the intrados and extrados sides. The hardness of the weld metal in the intrados side of the welded joint tends to be slightly lower than that in the extrados side. The degree of softening is unclear because the data of initial hardness are not known.

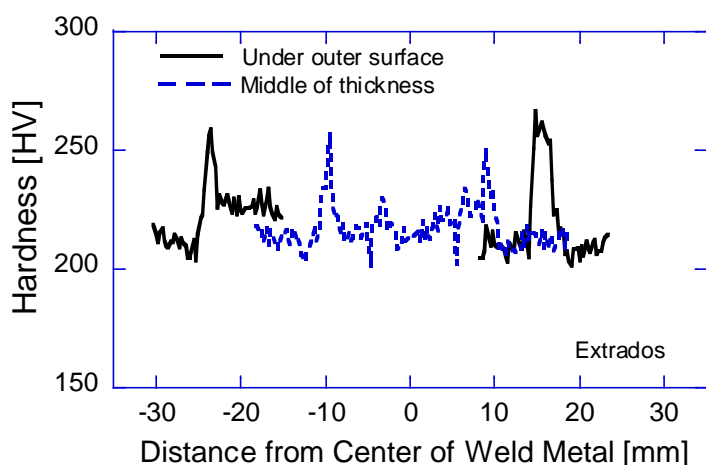


Figure 2: Hardness distribution in welded joint

### Measurement of Density of Voids

The number density of voids in the intrados side of the longitudinally welded region, where the circumferential stress is considered to be the highest during the steady operation, was measured.

In the present study, the measurement of the voids densities was conducted at six positions along fine-grain heat-affected zone (HAZ); that is, (1) on the outer surface, (2) in the vicinity of the outer surface, (3) in the welded shape discontinuous region, (4) at the center of the thick region, (5) at the cusp and (6) in the vicinity of the inner surface. Figure 3 reveals a measurement result for left and right HAZ. The number of voids in the outer surface tends to be greater than that in the inner surface. The number density of voids is  $\leq 100/\text{mm}^2$  at maximum, which is smaller than the generally reported value in ruptured welded joints of Grade 91 steel.

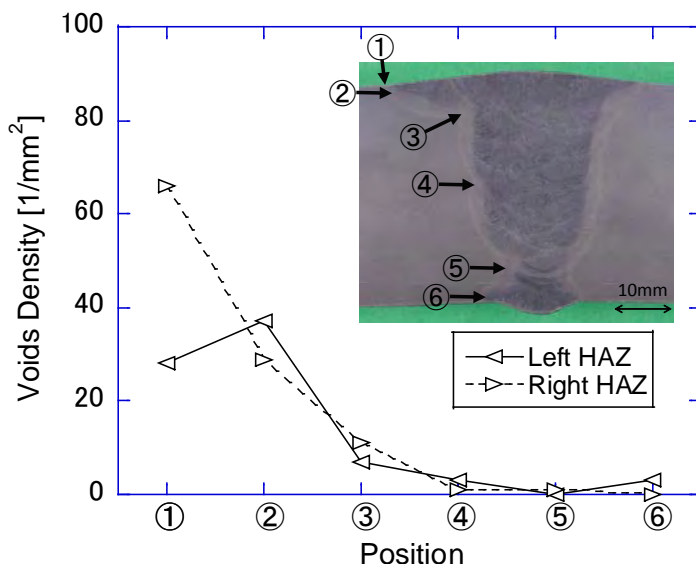
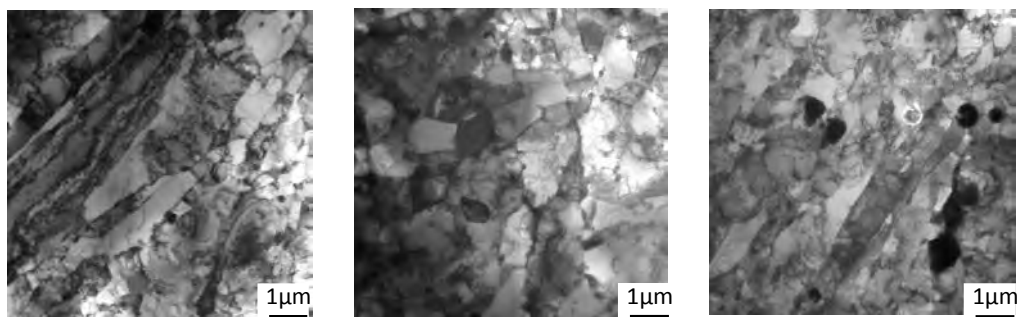


Figure 3: Voids density distribution in HAZ region along thickness direction

### Observation of Microstructure by Transmission Electron Microscopy (TEM)

The microstructures of the welded region in the intrados and extrados sides (base metal, fine-grain HAZ, coarse-grain HAZ, and weld metal) were observed by TEM. The position of the observation by TEM was the center of the thick region. Thin film specimens were collected from the material so that the observation surface was perpendicular to the direction of circumferential stress. The 250  $\mu\text{m}$ -thick film specimens collected from the material using a cutter were roughly polished to a thickness of 100–200  $\mu\text{m}$  and finished by electrolytic polishing.

Figure 4 shows typical TEM images (bright-field images) of the (1) base metal, (2) fine-grain HAZ and (3) weld metal in the intrados side. A typical martensitic structure is observed in the base metal. A subgrain structure with an equiaxial lath structure is observed in the fine-grain HAZ. It is generally known that an equiaxial subgrain structure exists in the HAZ of welded joints after Post Weld Heat Treatment (PWHT). Therefore, the equiaxial subgrain structure observed in this study is not considered to be formed during the service operation at the power station. The coarse-grain HAZ has a finer lath structure than that in the base metal and has a high dislocation density. In weld metal, inclusions in addition to the typical lath structure were observed. This tendency was similarly observed in the extrados side.



(1) Base metal

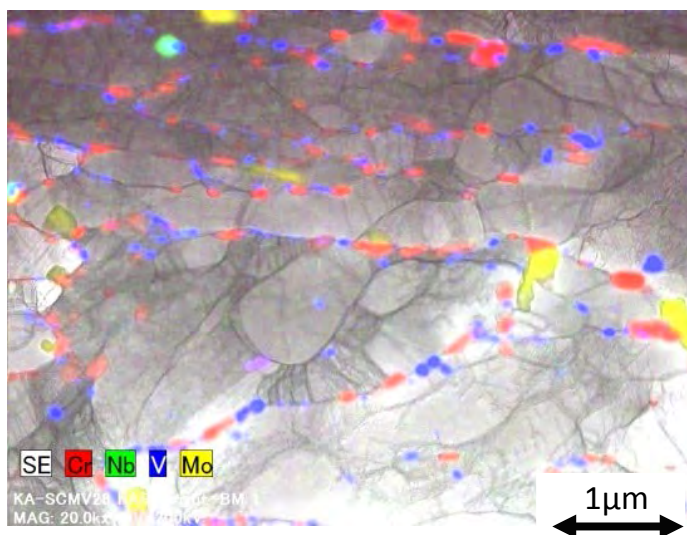
(2) Fine grain HAZ

(3) Weld metal

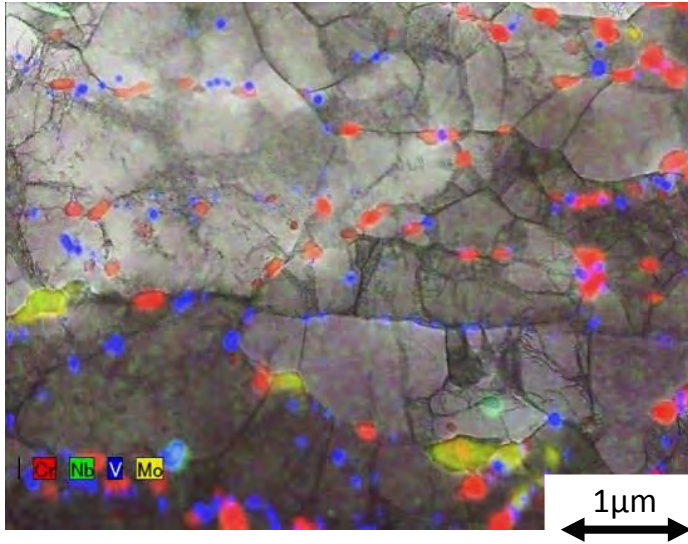
Figure 4: TEM Observation (BF Image)

The results of TEM observation indicated that the martensitic structure and subgrain structure in the base metal, fine-grain HAZ, coarse-grain HAZ, and weld metal were not markedly recovered, suggesting that the deterioration of the material caused by the change in dislocation structure during the service at the power station is considered to be insignificant.

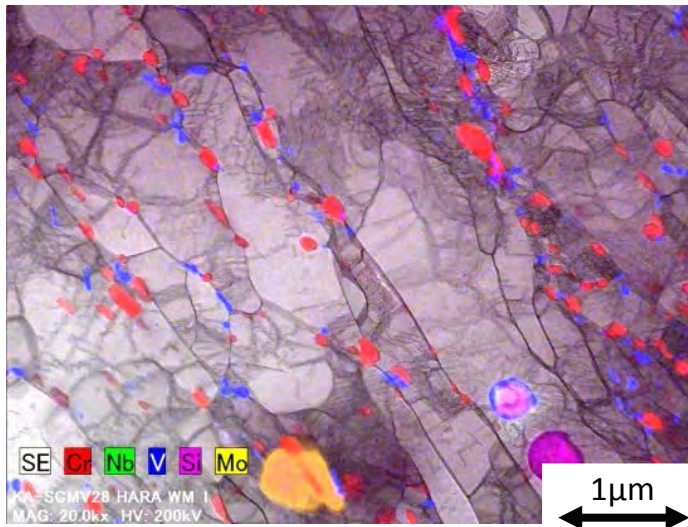
Figure 5 shows the TEM images (bright-field images) of the (1) base metal, (2) fine-grain HAZ, and (3) weld metal in the intrados side, over which element map images are superimposed.  $M_{23}C_6$  (red), MX (V-rich particles, blue; Nb-rich particles, green), and Laves phase (yellow) are found to precipitate. In the weld metal, Si-based inclusions (pink) are observed. In all regions,  $M_{23}C_6$  and MX are distributed on block boundaries and lath boundaries (or sub-boundaries). MX is also distributed inside the lath structure. This distribution tendency of the precipitations in the extrados side is similar to that in the intrados side.



(1) Base metal



(2) Fine grain HAZ



(3) Weld metal

Figure 5: Element mapping

It is reported that in the base metal of Grade 91 steel with a high Ni content (0.28 mass%), many Z-phase particles precipitate and many MX particles disappear after 50,000–80,000 h of the creep test at 600 °C, while few Z-phase particles appear and few MX particles disappear for the steel with a low Ni content (0.04 mass%). In Fig. 5, many MX particles are present and few Z-phase particles are found. Then, the base metal used in this study is considered to have a low Ni content. Actually, the Ni content of the present base metal is 0.07 mass%, which is revealed in Table 1. In summary, it was thought that the distribution of precipitations in the material is normal and the degree of degradation of the precipitations during the service operation at the high temperature is negligible.

# CREEP RUPTURE TEST

## Test Method

To estimate the remaining lifetime of the material used for a long time, specimens for the creep rupture test were cut out from the middle thick region of the elbow pipe. Two types of specimen, welded joint and base metal specimens, were prepared. The welded joint specimens were prepared to include the HAZ of both sides, and the base metal specimens were from the position adjacent to the region from which the welded joint specimens were obtained. The specimens are a solid bar type with dimensions of diameter 10 mm and length 50 mm at gauge portion. Three test conditions, i.e., constant temperature (650 °C) with stresses of 50, 60, and 70 MPa, were adopted for welded joint specimens, and another three test conditions, i.e., constant temperature (650 °C) with stresses of 60, 70, and 80 MPa, were adopted for base metal specimens.

## Test Result

Figure 6 shows the results of creep rupture tests of specimens collected from the material used for a long time, along with the mean curves of a base metal of Grade 91 plate steel and a welded joint of virgin material (RHC committee) [3],[4]. For the base metal, the rupture time of the extrados side is shorter than that of the intrados side, and for the welded joint, the rupture time of the intrados side is shorter than that of the extrados side. Note that the difference between the rupture times on the extrados and intrados sides is only ~20% of the total life time for both base metal and welded joint specimens. Compared with the mean curve of the virgin material, the rupture times of the base metal and welded joint specimens are longer. The creep ductility and deformation properties of these specimens are discussed later.

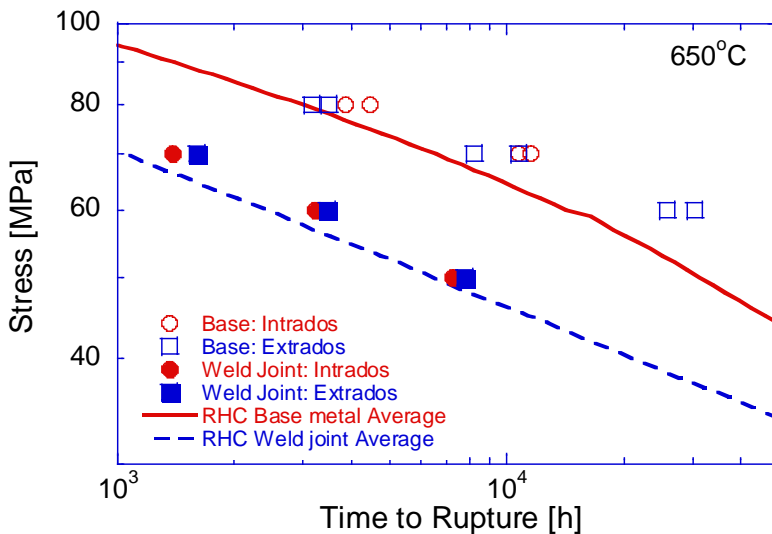


Figure 6: Creep test results of long-term used materials in comparison with RHC curves [3], [4]

For the welded joint specimens (both intrados and extrados sides), rupture occurred in the weld metal, as shown in Fig.7. We conducted scanning electron microscopy (SEM) observation with

the ruptured specimens and found that creep voids were present not only in the weld metal but also in the fine-grain HAZ in the base metal. The observation result suggested creep damage proceeded in the HAZ in the base metal, although rupture occurred in the weld metal.

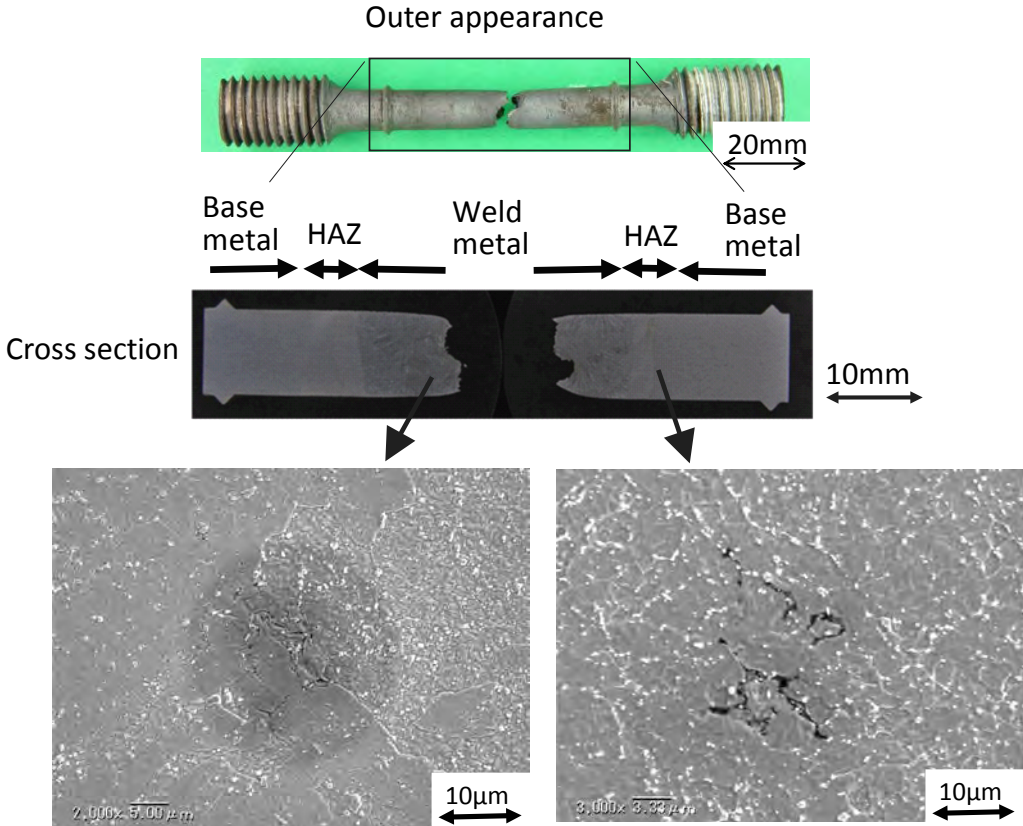


Figure 7: Ruptured specimen of welded joint (Intrados, 650°C, 50MPa)

## COMPARISON WITH ANOTHER MATERIAL USED FOR LONG-TERM

One of the authors has carried out the observation and creep rupture test of another Grade 91 steel used for a long-term [5]. In the following discussion, the material used for a long-term and examined in this study is named plant A material and another material used for a long-term and examined previously [5] is named plant B material. The results of the creep rupture test of plant A and B materials are compared. Plant B material was used in high-temperature-reheated and longitudinally welded elbow steam pipes. The steam temperature was 600 °C, the cumulative operating time was ~54,000 h (including the duration of operation at reduced temperatures), and the circumferential stress applied to the welded region in the intrados side was 48.8 MPa (calculated using the equation of the mean diameter for curved pipes). The creep damage evaluated in terms of the ratio of the cumulative operating time to the RHC regression curve [4] is 13% on the basis of mean properties and 46% on the basis of the 99% lower limit. For plant B material, specimens were collected from the middle thick region in the intrados and extrados sides at the center of the elbow axis direction (~45°).



Figure 8 shows a comparison of creep rupture tests of plant A material with those of B material [5]. The rupture time of the base metal specimens of plant A material is approximately ten times longer than that of plant B material and the rupture time of welded joint specimens of plant A material is approximately five times longer than that of plant B material. As mentioned above, the creep damage in plant A material was calculated to be approximately two times greater than that in plant B material on the basis of the loading conditions (temperature, circumferential stress, and operating time) and the RHC regression curve [4]. Therefore, the remaining lifetime of plant A material was estimated to be shorter than that of plant B material; however, the results of the creep rupture test indicated the opposite, where all rupture occurred in the HAZ in plant B material.

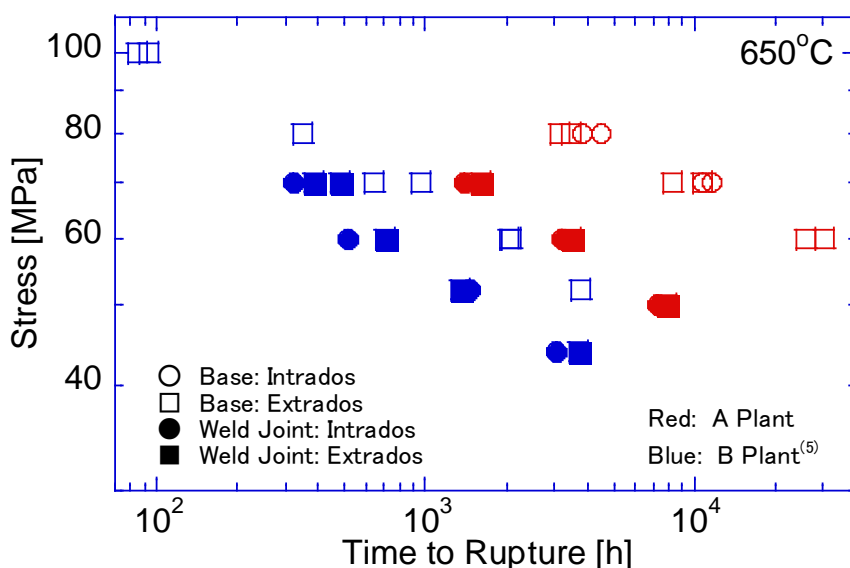


Figure 8: Comparison of creep test data of long-term used materials

Figure 9 shows the reduction in the areas of plant A and B materials obtained in the creep rupture test compared with that of the other virgin material of Grade 91 steel (base metal and welded joint)[6]. For the base metal specimens, there is no distinct difference in the reduction in area among plant A material, plant B material and virgin material. With increasing rupture time, the reduction in the area tends to slightly decrease. For the welded joint specimens, rupture occurred in the weld metal in plant A material, and in the HAZ in both plant B material and virgin material. The reduction in the area of plant B material is equivalent to or higher than that of virgin material; the reduction in creep rupture ductility caused by long-time use or creep damage is not observed. For the specimens collected from the intrados side, the reduction in the area in the specimen, in which rupture occurred in the HAZ (plant B material), tends to be smaller than the reduction in the area in the specimen, in which rupture occurred in weld metal (plant A material). However, for the specimens collected from the extradados side, there was no distinct difference in the reduction in the area relative to the position of rupture. Overall, with increasing rupture time, the reduction in the area of the welded joint specimens tends to decrease, similarly to the base metal specimens.

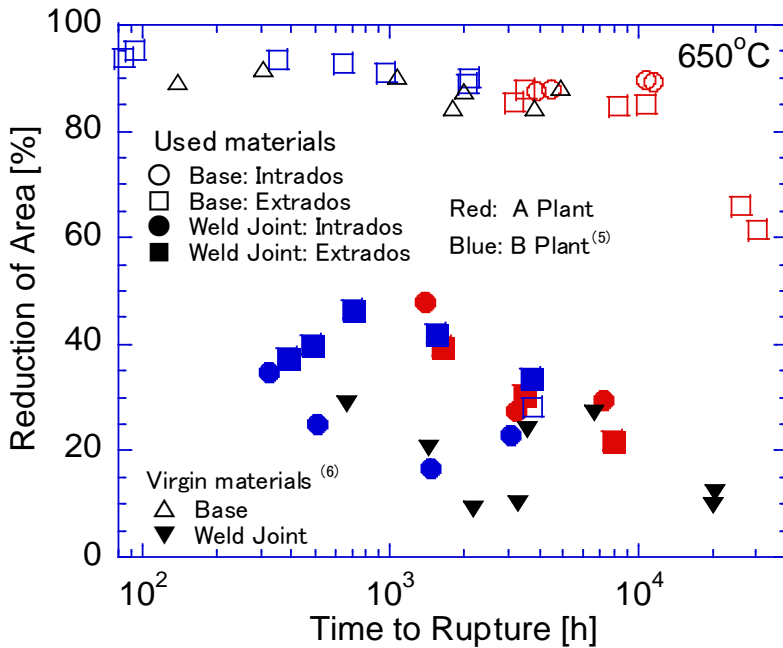


Figure 9: Comparison of reduction of area of long-term used and virgin materials

Figure 10 shows the minimum creep strain rate of base metal specimens of plant A material, plant B material and virgin material. The relationship between stress and the minimum creep strain rate of plant A material is similar to that of virgin material, while the minimum creep strain rate of plant B material is significantly higher (5–10 times).

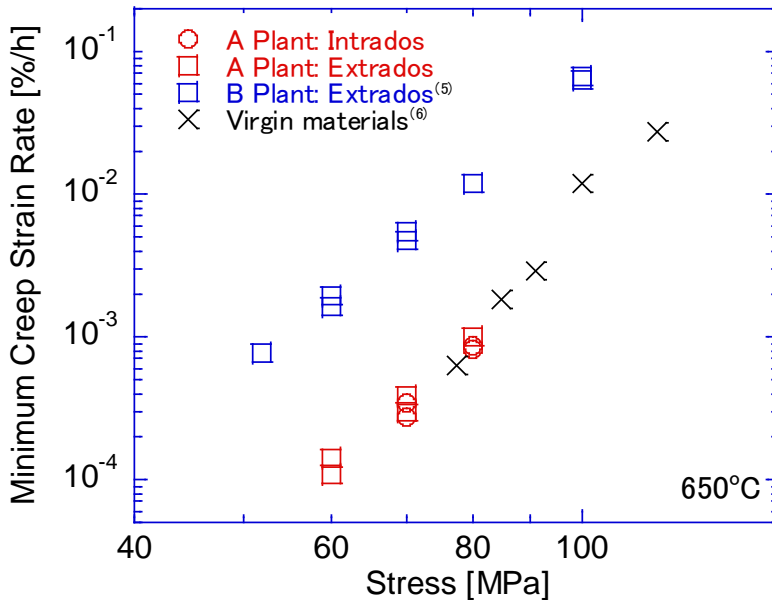


Figure 10: Relationship between minimum creep strain rate and stress of long-term used and virgin materials

From the above, the remaining lives of base metal specimens and welded joint specimens determined in the creep rupture test were found to be markedly different between plant A and B materials; however, there was no marked difference in the reduction in the area between plant A and B materials. In contrast, there was a significant difference in the minimum creep strain rate of base metal specimens between plant A and B materials. Therefore, it was suggested that one of the causes of the difference in the lifetimes of specimens of plant A and B materials may be the difference in the creep deformation property of the base metal, though the operating temperatures of plants A and B differ by 6 °C and this should also be taken into consideration in the interpretation and assessment of the results.

Figure 11 shows relationship between the minimum creep strain rate and rupture time for base metal. The relationship for welded joint was also plotted for reference in the Fig.11, where the creep strain rate actually means nominal creep strain rate of gauge portion in the case of welded. For base metal and welded joint, it seemed that there was unique relationship between the creep strain rate and the rupture time, i.e., Monkman-Grant relationship held true for each material.

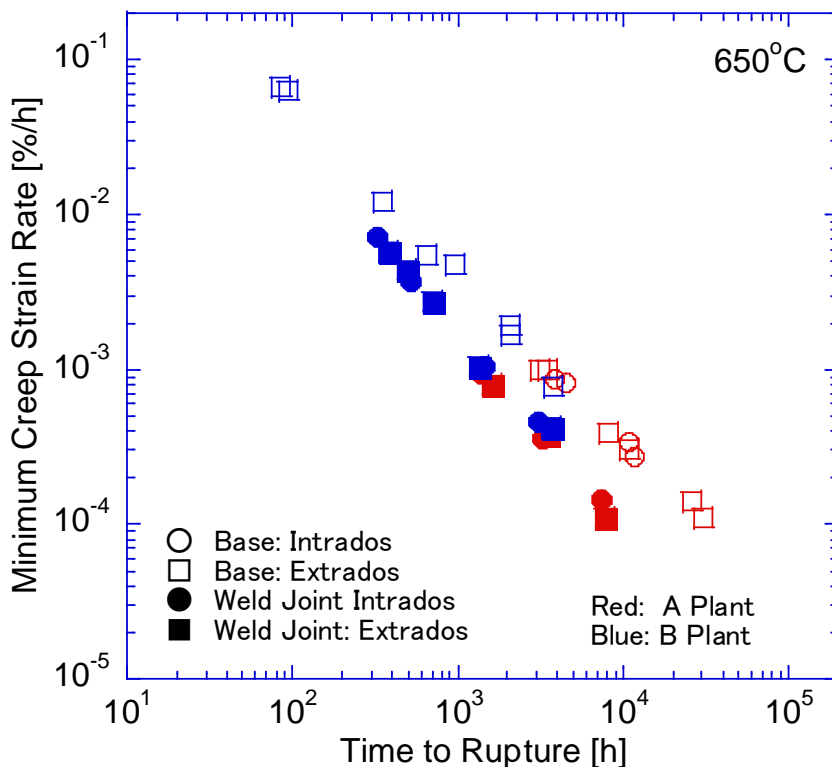


Figure 11: Relationship between minimum creep strain rate and time to rupture of long-term used materials

## CONCLUSIONS

In this study, the microstructure observation and the creep rupture test of ASME Grade 91 steel used for long-term at power plant were conducted. The results indicated that recovery of the microstructure of the material was not observed, and the creep strengths of base metal and welded joint specimens were equivalent to or higher than the mean values of the virgin material. Regarding the welded joint, a rupture occurred in the weld metal while creep voids were observed not only in the weld metal but also in the fine-grain HAZ in the base metal. Comparison with another long-term used Grade 91 steel suggested remaining lives of welded joints remarkably depended on creep deformation properties of the base metal of each welded pipe.

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