1. Introduction

In recent years, several new ferritic steels having high creep strength have been developed for boiler pressure parts, with 11Cr–0.4Mo–2W–V–Nb–Cu steel (P122) having excellent corrosion resistance and approximately 1.3 times higher creep strength than Mod.9Cr–1Mo steel (P91)1). This steel has already been used for main steam pipes, high temperature reheat pipes and headers, as well as SH/RH tubes in modern large capacity fossil power plants.2) This steel consists of a tempered martensitic structure that is the same as for P91. The microstructure of the heat-affected zone (HAZ) in weldments is quite complicated because of the transformation and recovery caused by repeated heat cycles through welding processes. It is well known that weldments are weaker in creep strength than base metal in this test condition. The coarse and fine grained microstructures were observed, and the average grain sizes were measured. The HAZ adjacent to the base metal was characterized by a fine grained microstructure consisting of subgrains with low dislocation density. Hardness of the intercritical area between HAZ and the base metal was the lowest after PWHT and during creep. Creep cavities tended to form at the grain boundaries in the fine grained HAZ due to creep. Small cracks gathered with cavities were observed in the fine grained HAZ after creep, and these corresponded to the fracture portion. M23C6, M7C3 and MX type carbides had already precipitated in the HAZ before the creep test. A Laves phase arose at the grain boundary of the coarse and fine grained zones of the HAZ during the test. It is presumed that Laves phase precipitation in the coarse grained HAZ is slower than in the fine grained HAZ and base metal during creep.

2. Experimental Procedure

Table 1 shows the chemical composition of the tested steel plate, with a thickness of 34 mm. This plate was normalized at 1050°C for 100 min and tempered at 770°C for 360 min. Weldments were fabricated by GTAW process welding using matching filler metal concurrently developed. Post-weld heat treatment (PWHT) was applied to the weldment at 750°C for 2 h. Two types of uniaxial creep specimens, namely a large type specimen having a 30 mm square cross section and a 175 mm gauge length, and small type specimens having a 9 mm square cross section and a 25 mm gauge length, were taken from the weldment with the center of each specimen placed at the fusion boundary. Creep testing was conducted using two types of specimens at 650°C and 675°C, and ruptured in the fine grained HAZ, known as type IV failure. Weldments were known to be weaker in creep strength than base metal in this test condition. The coarse and fine grained microstructures were observed, and the average grain sizes were measured. The HAZ adjacent to the base metal was characterized by a fine grained microstructure consisting of subgrains with low dislocation density. Hardness of the intercritical area between HAZ and the base metal was the lowest after PWHT and during creep. Creep cavities tended to form at the grain boundaries in the fine grained HAZ due to creep. Small cracks gathered with cavities were observed in the fine grained HAZ after creep, and these corresponded to the fracture portion. M23C6, M7C3 and MX type carbides had already precipitated in the HAZ before the creep test. A Laves phase arose at the grain boundary of the coarse and fine grained zones of the HAZ during the test. It is presumed that Laves phase precipitation in the coarse grained HAZ is slower than in the fine grained HAZ and base metal during creep.

KEY WORDS: 12 %Cr steel; creep rupture strength; creep cavity; Laves phase; type IV failure.

| C   | Si  | Mn  | Cr  | Ni  | P   | S   | Mo  | W   | Cu  | V   | Nb  | B   | N   |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.12| 0.28| 0.61| 10.50| 0.34| 0.018| 0.001| 0.36| 2.05| 0.97| 0.21| 0.06| 0.0029| 0.069|


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**Microstructural Degradation of the HAZ in 11Cr–0.4Mo–2W–V–Nb–Cu Steel (P122) during Creep**

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the microstructure by optical microscope and SEM. Hardness distributions around the HAZ were measured each time that creep tests were interrupted. Creep cavities were observed by replica using SEM, and the densities were measured by counting the number of creep cavities in a particular area. Transmission electron microscope (TEM) observations were carried out by using thin foil specimens from the HAZs of the creep interrupted small specimens. The precipitates were identified using carbon extraction replicas observed by transmission electron microscope with EDS (TEM-EDS).

3. Results and Discussion

3.1. Microstructure of the HAZ

Figure 2 shows the optical microstructure of the weldment after PWHT. Small amounts of $\delta$-ferrite were seen in the HAZ near the fusion boundary. These were formed by heat-up and reverse-transformation due to heat input when welding. The microstructures at 0.5 and 1.0 mm away from the fusion boundary basically consisted of tempered martensite, but the diameters of the grains were smaller than that of base metal. The degradation of the creep strength of Ni based alloy and austenitic stainless steel is known to accompany decreasing grain size, and it is possible that HAZ microstructure having small grains affects the creep strength of the weldment. Figure 3 shows SEM micrographs of the weldment. Measurements of average grain diameter in the HAZ were conducted by means of the intercept method using SEM micrographs. The measured average grain size of prior austenite grains and subgrains in the base metal and the HAZ are shown in Fig. 4. Although the average grain size in the base metal was approximately 28 $\mu$m, those of fine grained area in the HAZ were from 0.8 to 1.0 $\mu$m. There was no change in average grain size at a distance of 2 mm or more from the fusion boundary. It is well known that the HAZs of welds predominantly consist of coarse grained martensite close to the fusion boundary, which becomes progressively finer as the distance from the fusion boundary increases, terminating in a fine intercritical structure. Even though a coarse grained microstructure was seen in a very limited area near the fusion boundary in this HAZ, a fine grained microstructure was clearly observed.

Precipitates were observed on the prior austenite grain boundaries and lath boundaries in the base metal as shown in Fig. 3(d). However, precipitates in line were observed to traverse grain boundaries at 1.4 mm away from the fusion boundary as shown in Fig. 3(c). Moreover the amount of precipitates at the grain boundaries was comparatively small. It appears that those grains in the area shown in Fig. 3(b) were rearranged and that the prior grain boundaries disappeared due to reverse transformation from heat input.

3.2. Creep Testing

The large and small specimens ruptured at 689.0 h and 340.8 h, respectively, after several creep interruptions. The
creep rupture times of these specimens were shorter than that of the base metal for these creep conditions. The macrostructure of the weldment after creep rupture is shown in Fig. 5. Both weldments ruptured in the fine grained zone at a location approximately 1.2 mm away from the fusion boundary in the HAZ, and the creep fracture mode were categorized as type IV failure. It was posited that the reason why the weldment ruptured in a shorter time than the base metal was that the creep strength of fine grained zone was weaker than other parts.

3.3. Hardness

Figure 6 shows changes in the hardness distribution in a small specimen weldment during creep. The hardness near the fusion boundary in the HAZ was the highest, and that of the intercritical area between the HAZ and the base metal was the lowest both before and during creep. The microstructure having the lowest hardness was located from 1.2 mm to 1.5 mm away from the fusion boundary, corresponding to the fine grained microstructure as shown in Fig. 3(c). The hardness of the weld metal, HAZ and base metal tended to be lowered due to creep, except for the area having the lowest hardness before creep. Hardness distribu-
tion changes in the large specimen were similar to those of the small specimens.

3.4. Changes in Microstructure due to Creep

3.4.1. Creep Cavities

Figure 7 shows the optical microstructure of the HAZ in the small specimen interrupted at 650°C×130 MPa for 231.4 h (t/tr 0.68). Creep cavities were generated and observed at the grain boundary. In order to investigate creep cavity formation in the HAZ, the distribution of creep cavity density was measured in direction to the stress axis using a small creep interrupted specimen. Figure 8 shows the creep cavity number density in a small specimen that was creep interrupted at 650°C×130 MPa (t/tr 0.68). The creep cavity density was the highest in the HAZ, which was located 1.3 mm away from the fusion boundary, and which corresponded to the part having the lowest hardness during creep as shown in Fig. 6. There was no creep cavity formation in the weld metal immediately before creep rupture.

The relationship between the maximum cavity number density in the HAZ of the large specimen and the creep interrupted time ratio was measured. Figure 9 shows that the creep cavity started to form in the HAZ at approximately t/tr = 0.5. There were only a few creep cavities observed in the coarse grained HAZ and base metal until creep rupture. The cavity number density in the HAZ increased with creep time. The behavior of increasing cavity number densities measured in locations A and B were different, and this must originate in the small differences of stress distribution and microstructure in the fine grained HAZ. Figure 10 shows a replica taken from the middle of the large specimen on the opposite side of the fracture. Small cracks by gathering cavities were observed at a distance of 1 mm away from the fusion boundary, although they were not observed on the outer surface of the specimen. The location of the small cracks corresponded to the fractured portion.

3.4.2. Precipitation

Precipitates in the HAZ were identified using a carbon extracted replica subjected to TEM-EDS. M₂₃C₆, M₇C₃ and MX type carbides had already precipitated in the HAZ after PWHT, although the amounts of precipitates were different due to the observation area in the HAZ. Figure 11 shows the areal fraction and average diameter of precipitates after PWHT measured from SEM micrographs of the HAZ. It
can be seen that the amount of precipitates in the fine grained zone is small compared with the base metal. The average diameter of precipitates near the fusion boundary was smaller than in other locations. Hasegawa et al.\(^5\) reported the precipitation behavior after the HAZ heat cycle and after PWHT in 0.08C–9Cr–0.5Mo–1.8W–0.2V–0.05Nb–0.05N steel; they showed that M\(_{23}C_6\) type carbides are easy to resolve by HAZ heat cycle, and that the amount of precipitation recovers after subsequent PWHT. It was considered that the peak temperature during welding near the fusion boundary in the HAZ was high enough to resolve the precipitates and to facilitate re-precipitation during PWHT. On the other hand, it appeared that only the fine precipitates were resolved, and that comparatively large precipitates ripened due to heat input. These results support the findings reported by Hasegawa et al.

In order to identify the precipitates after creep, a carbon extracted replica taken from the HAZ was observed and analyzed by TEM-EDS. A Laves phase precipitation was confirmed in addition to the precipitation identified before creep. It was considered that the peak temperature during welding near the fusion boundary in the HAZ was high enough to resolve the precipitates and to facilitate re-precipitation during PWHT.

On the other hand, it appeared that only the fine precipitates were resolved, and that comparatively large precipitates ripened due to heat input. These results support the findings reported by Hasegawa et al.

In order to identify the precipitates after creep, a carbon extracted replica taken from the HAZ was observed and analyzed by TEM-EDS. A Laves phase precipitation was confirmed in addition to the precipitation identified before creep. It was confirmed that the Laves phase tended to precipitate at the grain boundary. Figure 12 shows the TEM-EDS spectra of the Laves phase observed in the fine grained zone, consisting of small amounts of Mo and Cr in addition to Fe and W. The existence of Cu was also detected but this might be strongly influenced by the mesh stage frames made with Cu for the replicas.

Laves phase can be easily distinguished from other precipitates because of the different contrast of the SEM backscattered electron image. It was confirmed that the Laves phase tended to precipitate at the grain boundary. Figure 13 shows the areal fraction of Laves phase precipitates in crept and aged specimens. This shows that the amount of Laves phase precipitated in the base metal of creep specimen at 650°C for 231.4 h is nearly equal to that
of the specimen aged at 650°C for 2 000 h. However, the amount of Laves phase precipitates near the fusion boundary is smaller than that of the base metal in the crept weldment. The reason for this is still not clear, but if creep stress were lower and creep time reached 2 000 h, the distribution of the Laves phase in the crept specimen might be the same as in the aged specimen, i.e., precipitation might be slow near the fusion line in the HAZ because of the lack of Laves phase precipitation sites due to grain coarsening.

3.4.3. Dislocation

TEM observation of the HAZ microstructure was conducted using specimens after PWHT and creep. Figure 14 shows the TEM microstructure at the fine grained HAZ having the lowest hardness before and during creep. The fine grained HAZ consisted of small subgrains with low dislocation density, with the subgrain size having grown due to creep. Since the microstructure of the fine grained HAZ had already recovered and had low dislocation density after PWHT, hardness would not tend to be further reduced due to decreasing dislocation density during creep. On the other hand, martensitic laths were observed in the pre-creep and crept specimens in the coarse grained HAZ near the fusion boundary, and the density of dislocation within the laths decreased due to creep. Microstructural changes in the base metal of the specimen were similar to that of the coarse grained HAZ.

The lower hardness in the fine grained HAZ was derived from the microstructure having low density without laths, and it was considered that the microstructure was inferior in terms of creep strength. The degradation of creep strength in the fine grained HAZ resulted in fracture known as type IV failure.

4. Conclusion

Microstructural degradation of the HAZ in 11Cr–0.4Mo–2W–V–Nb–Cu Steel (P122) during creep was investigated in order to better understand the mechanism of type IV failure. The results obtained were as follows:

(1) A coarse grained HAZ was seen in a very limited area near the fusion boundary, and a fine grained HAZ was observed.

(2) The test specimens were crept at 650°C and 675°C, and ruptured in the fine grained HAZ, referred to as type IV failure. Weldments were weaker in creep strength than the base metal in these test conditions.

(3) The hardness of the weld metal, coarse grained HAZ and base metal decreased with increasing creep damage, while hardness changed only slightly in the fine grained HAZ adjacent to base metal.

(4) The fine grained HAZ having low hardness consisted of subgrains with low dislocation densities, and the diameter of the subgrains grew during creep.

(5) Creep cavities tended to form on the grain boundaries in the fine grained HAZ due to creep. The densities of the creep cavities increased with the progress of creep damage. Small cracks gathered with cavities were observed in the fine grained HAZ after creep, corresponding to the fracture portion.

(6) M23C6, M7C3 and MX type carbides had already precipitated in the HAZ before the creep test. A Laves phase arose at the grain boundary of the coarse and fine grained zones of the HAZ during the creep test. It was considered that Laves phase precipitation in a coarse grained HAZ is slower than in a fine grained HAZ or base metal during creep.

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