

Effect of tempering temperature on Z-phase formation and creep strength in 9Cr–1Mo–V–Nb–N steel

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Abstract

The effect of the tempering temperature on Z-phase formation and creep strength in 9Cr–1Mo–V–Nb–N steel was examined with particular attention to the precipitation sequence of MX, M₂X, and Z-phase during creep exposure. Tempering at a lower temperature provided a high dislocation density and a fine lath structure. Tempering at 953, 1003, and 1038 K provided [M₂₃C₆, M₂X, NbX], [M₂₃C₆, M₂X, NbX, VX], and [M₂₃C₆, NbX, VX] phases, respectively. The creep strength of steel tempered at 953 K was the highest among the steels studied, even in the long term. No large decrease in creep strength was observed in steel tempered at 953 K. The Z-phase was observed after long-term creep in steel tempered at 1003 or 1038 K. In steel tempered at 953 K, a VX rather than a Z-phase formed during creep, and this was accompanied by consumption of M₂X. Retardation of the Z-phase formation can retard the creep strength degradation in steel tempered at 953 K.

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1. Introduction

Because they have good resistance to creep and to oxidation, steels containing 9–12% Cr play an important role in improving the thermal efficiency of power plants; however, the decrease in creep strength at a long term is a problem for such steels [1], leading to accidents with components in power plants. Type IV cracking causes degradation of the creep strength of welded joints [2], whereas intergranular fracture [3], preferential recovery around the prior austenite grain boundary [1], and Z-phase formation [4] contribute to a loss of creep strength in the base metals. The formation of the Z-phase [Cr(V,Nb)N] during creep exposure causes a decrease in creep strength because the formation of the Z-phase consumes MX [(V,Nb)(C,N)] carbonitride [5], a crucial strengthener in 9–12% Cr steels [6]. It has been reported that an increase in the Cr content promotes Z-phase formation [7,8]. To retard Z-phase formation, the Cr and N contents of steels should be as low as possible [7–9]. In addition to the alloying elements, tempering conditions can also affect

Z-phase formation. In 9–12% Cr steels, tempering at a higher temperature, such as 1053 K, results in the formation of M₂₃C₆ and MX. In this case, the presence of MX is closely related to Z-phase formation [8], because the composition of MX carbonitride is almost the same as that of the Z-phase. On the other hand, Agamennone et al. have reported that the formation of M₂X as a result of tempering at 843 or 973 K promotes Z-phase formation in 12Cr–2W–5Co steel, in contrast to tempering at 1033 K [10]. Their results indicate that tempering at higher temperatures can retard Z-phase formation during creep. However, Ishii et al. [11] have reported that M₂X formed by tempering at 843–923 K dissolves with subsequent formation of MX during creep at 873 K in 10Cr steels. In short, the precipitation sequence of MX, M₂X, and Z-phase during creep is not clear.

In actual power plants, steels such as ASME Gr.91 and Gr.92 are already in use in boilers. The ASME Boiler and Pressure Vessel Code [12] stipulates a minimum tempering temperature for these steels (Gr.91, Gr.92: 1003 K). Suzuki reported that tempering at 1003 and 1053 K provides (M₂₃C₆, MX, M₂X) and (M₂₃C₆, MX), respectively, in 9Cr–1Mo–V–Nb–N, a steel that is similar to Gr.91 [13]. The difference in the initial microstructure can affect Z-phase formation as mentioned above. In short, the tendency of creep-strength degradation can vary among Gr.91

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Table 1
Chemical compositions (mass%) of the steels examined

	C	Si	Mn	P	S	Ni	Cr	Mo	V	Nb	Al	N
ASME-T91	0.0900	0.29	0.35	0.009	0.002	0.28	8.70	0.90	0.22	0.072	0.001	0.044
Steel A-1, A-2	0.0870	0.30	0.45	0.002	0.001	0.03	8.89	1.06	0.22	0.08	0.001	0.049

Table 2
Heat treatment conditions of the steels examined

	Normalizing	Tempering
ASME-T91	1323 K, 10 min A.C.	1038 K, 30 min A.C.
Steel A-1	1323 K, 30 min A.C.	953 K, 30 min A.C.
Steel A-2	1323 K, 30 min A.C.	1003 K, 30 min A.C.

steels, even those of the same standard, depending on whether or not Z-phase formation occurs.

Our aim was to clarify the effects of the initial microstructure, such as M_2X and MX phases, on Z-phase formation and creep strength in the long term in 9Cr–1Mo–V–Nb–N steel, which is similar to Gr.91.

2. Experimental procedure

The chemical compositions of the steels that were studied are listed in Table 1. ASME-T91 was used as a reference material. The composition of steel A is in the range specified in ASME-T91. The Ni content of steel A is relatively lower than that of T91. However, the Ni content of steel A is in the range specified in ASME-T91 (≤ 0.40 mass%). To change the initial microstructure, samples were tempered at several different temperatures: the heat treatment conditions are summarized in Table 2. Creep tests were conducted at 873 K under a constant load in air by using specimens with a gauge diameter of 6 mm and a gauge length of 30 mm. The microstructures were examined

by conventional transmission electron microscopy (TEM) and by scanning TEM in conjunction with energy-dispersive X-ray analysis (STEM-EDX) at 200 kV. For the TEM studies, thin foils and extracted carbon replicas were prepared from the samples after tempering and from the gauge portions of crept samples. Elemental mapping by STEM-EDX was performed to determine the distribution of precipitates for each phase, such as $M_{23}C_6$, M_2X , MX , Laves phase, and Z-phase. The presence of Z-phase was confirmed in a selected area of $255 \mu\text{m}^2$ for each sample. The Z-phase can be seen in both the Cr and V maps because the Z-phase contains V, Nb, Cr, and N [14]. On the other hand, MX carbonitrides are visible only in the V map because MX carbonitrides contain mainly V, Nb, N, and C. Therefore, the Z-phase was distinguished from MX carbonitride by comparing the V and Cr maps. We also confirmed that particles detected in both the Cr and V maps are Z-phase particles by electron-diffraction pattern analysis [14]. The details of the method used to identify the Z-phase have been reported in our previous paper [14].

3. Results and discussion

3.1. Effect of tempering temperature on initial microstructure

Fig. 1 shows TEM micrographs of the initial microstructure after tempering. The dislocation density is higher and the lath width is smaller after tempering at a lower temperature. The hardness of steels A-1, A-2, and T91 were HV302, HV258,

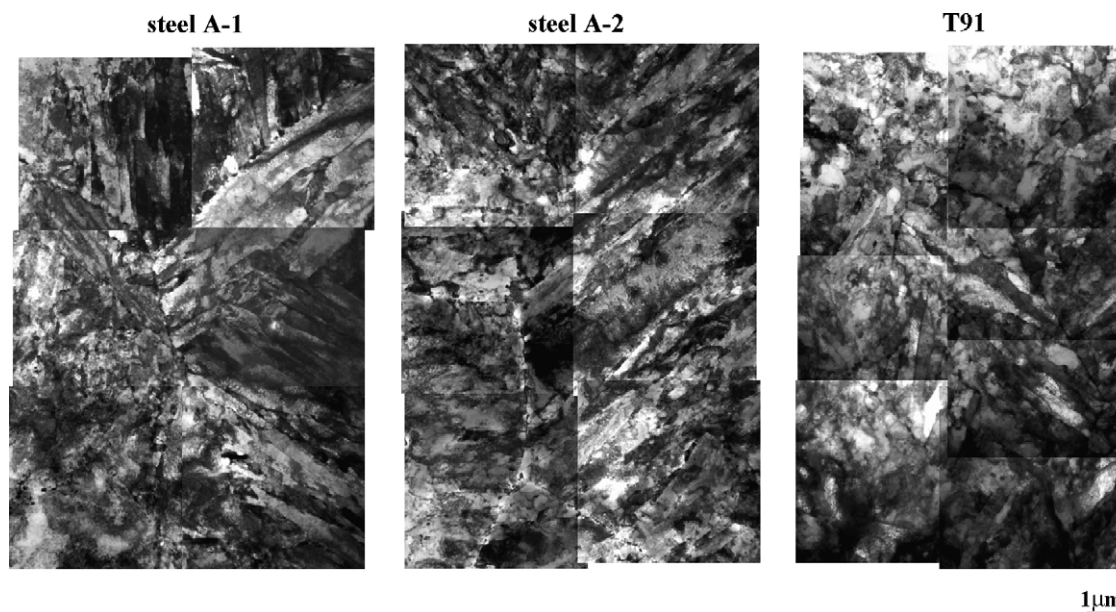


Fig. 1. TEM micrographs after tempering for all steels.

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