Creep Stress Effect on the Precipitation Behavior of Laves Phase in Fe–10%Cr–6%W Alloys

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Effect of creep tensile stress on the precipitation behavior of Laves phase in Fe–10%Cr–W alloys has been investigated. Fe–10%Cr–W alloys are basic alloys of ferritic heat resisting steels, which have been being developed for the ultra-super critical pressure power generating plants. It has been clarified that the creep tensile stress enhances the Laves phase precipitation comparing with that in the same alloys aged under no stress.

KEY WORDS: ferritic heat resisting steel; Laves phase; tungsten; cobalt; embrittlement; creep; stress effect; aging treatment; precipitation; morphology.

1. Introduction

Recently, very much effort has been made on the development of the ferritic heat resisting steels used for the ultra-super critical pressure power generating plants.^{1,2)} Fe–10%Cr–W alloys are basic alloys of such ferritic resisting steels.³⁾ It is well known that Laves phase forms in these ferritic steels and this phase is considered to contribute to the precipitation strengthening at high temperature but to induce the embrittlement at low temperature.^{4,5)} However, general features of the Laves phase precipitation behavior is not clarified. Particularly, creep stress effect on Laves phase precipitation behavior has not been clear. In the present research, stress effect on the precipitation behavior of the Laves phase at the early stage of aging and creep in the Fe–10%Cr–6%W–(Co) alloys has been investigated.

2. Experimental Procedures

The materials were melted in a vacuum induction furnace. The chemical composition of the materials is shown in Table 1. 3% Co is added in the alloy B (6W–3Co) for the clarification of its effect, because the recently developed ferritic heat resisting steels contain Co,⁶⁾ while the details of the effect of Co has not been clarified except for the enhancement effect on the austenization at high temperature and the martensitic transformation on rapid cooling. 6% W is also added in the alloys to investigate the Laves phase (Fe₂W) precipitation behavior in the enhanced state, although the limit of W addition is suggested to be about 3% in the advanced ferritic heat resisting steels.³⁾

After hot rolling, specimens were solution treated at 1323 K for 3.6 ks and water quenched. Aging treatment was carried out at 873 and 923 K for 1.0 to 3.6×10³ ks.

Tensile stress of 80 MPa was also loaded on the creep test specimens at 873 and 923 K for 7.2×10² ks for the investigation of stress effect on the Laves phase precipitation behavior.

Vickers hardness (Load: 98 N) measurement was performed after the aging treatment. Transmission electron microscopy (TEM) observation was also made after the aging treatment or the creep stress loading.

3. Experimental Results and Discussion

The Vickers hardness change during the aging treatment at 873 and 923 K is shown in Fig. 1. Small hardness peaks are observed after about 10 and 1.0 ks aging at 873 and 923 K, respectively, in both alloys A and B. After these small peaks, both alloys indicate large hardness increase. Miyahara *et al.* have already recognized a similar hardness change in the Fe–10%Cr–Mo alloys and shown that this phenomenon is related to the characteristics of the Laves phase precipitation behavior.^{7–9)} That is, they have clarified that the small hardness peak is caused by the very fine disk-like precipitates formed on the {100} planes of the matrix before the bulky Laves phase formation and that the large hardness increase after the small hardness peak is due to the large volume of the bulky Laves phase precipitates.^{7–9)}

Table 1. Chemical composition of the materials used. (mass%)

Alloy	C	Si	Mn	S	Cr	W	Co
Alloy A (6W)	0.0014	0.03	0.003	0.0024	9.89	5.86	0.09
Alloy B (6W–3Co)	0.0013	0.02	0.0019	0.0021	10.16	6.16	3.03

Fig. 1. Vickers hardness (load: 98 N) change during the aging treatment at 873 K and 923 K of alloys A (6W) and B (6W-3Co).

Fig. 2. Tiny precipitates in the alloy A (6W) aged for 10.8 ks at 873 K.

Fig. 3. TEM observation on the Laves phase precipitation in the alloy A (6W). (a-1) aged at 873 K for 7.2×10^2 ks, (a-2) selected area diffraction pattern from the subboundary in the photo. (a-1), (b) crept with the load of 80 MPa at 873 K for 7.2×10^2 ks.

TEM observation of the present research also suggests that the hardness change of the alloys A and B is due to the same mechanism of the Laves phase precipitation behaviors. A TEM photograph on the alloy A aged at 873 K for 10.8 ks, which indicates the first small hardness peak, is shown in **Fig. 2**. Coherent strain is observed in the matrix around the tiny precipitates and the small hardness peak is considered to be caused by these tiny precipitates.

Figures 3 and **4** show the Laves phase formation in the alloys A and B, which are aged at 873 K for 7.2×10^2 ks under the load of creep stress of 80 MPa or at unloaded state. 80 MPa is about one half of 0.2% proof strength of the alloys A and B at 873 K. The creep stress effect on the Laves phase precipitation is clearly observed in these figures. Figure 3(a-1) shows that, in the alloy A aged at the stress unloaded state, very tiny precipitates are formed on the dislocation network in a subboundary which is certified by a diffraction pattern (Fig. 3(a-2)) and that the precipitates within the grains are also formed only on the dislocations. On the other hand, as shown in Fig. 3(b), slightly larger precipitates are formed uniformly in the matrix under

the creep stress loading. Accordingly, it can be seen from Fig. 3 that the creep stress enhances the Laves phase precipitation at the early stage of aging. Figure 4 also shows that the creep stress enhances the Laves phase formation in the alloy B. Furthermore, comparing Fig. 4 with Fig. 3. It is seen that the addition of Co enhances the precipitation of the Laves phase.

Quantitative analysis for the number density of the Laves phase precipitates has been made on the alloys A and B aged or crept at 873 K, using TEM observation results, as shown in **Fig. 5**. Specimen thickness of TEM observed area was obtained from the extinction distance and contour. The number density per unit volume, N_v (m^{-3}), of the Laves phase precipitates can be obtained from the slope of the line shown in Fig. 5 and the N_v values are got as followings, the aged alloy A; $2.4 \times 10^{10} \text{ m}^{-3}$, the crept alloy A; $4.7 \times 10^{10} \text{ m}^{-3}$, the aged alloy B; $3.4 \times 10^{10} \text{ m}^{-3}$ and the crept alloy B; $4.9 \times 10^{10} \text{ m}^{-3}$. These values indicate that the creep stress increases the number density of the Laves phase precipitates. Average precipitates size in the crept specimen is also slightly larger than that in the aged specimens, as

shown in Figs. 3 and 4. Accordingly, it is certified quantitatively that the creep stress enhances the Laves phase precipitation in the alloys A and B aged or crept at 873 K. The above N_v analysis results also indicate that Co has small effect to increase the number density of Laves phase precipitates, but the comparison between Figs. 3 and 4 shows that Co has a effect to enhance the growth of the Laves phase precipitates. Accordingly, it can be concluded that Co has a effect to enhance the preipitation of the Laves phase in the stress loaded state, at lease, at the early stage of creep deformation at 873 K.

Figure 6 shows the Laves phase in the alloy B aged at 923 K for 7.2×10^2 ks under the creep stress of 80 MPa or at stress unloaded state. In Fig. 6, slightly darker image of the Laves phase particles, that is, thicker particles of the Laves phase are observed in the crept specimen than in the aged specimen. Accordingly, this figure also shows that creep stress enhances the Laves phase precipitation at 923 K. Although the mechanism of such creep stress effect on the

Fig. 5. Specimen thickness dependences of number density per unit area (N_a/m^{-2}) of the Laves phase precipitates in the alloys A (6W) and B (6W-3Co) aged or crept at 873 K for 7.2×10^2 ks.

Fig. 4. TEM observation on the Laves phase precipitation in the alloy B (6W-3Co). (a) aged at 873 K for 7.2×10^2 ks, (b) crept with the load of 80 MPa at 873 K for 7.2×10^2 ks.

Fig. 6. TEM observation on the Laves phase precipitation in the alloy B (6W-3Co). (a) aged at 923 K for 7.2×10^2 ks, (b) crept with the load of 80 MPa at 923 K for 7.2×10^2 ks.

Laves phase precipitation cannot be clearly understood at the present time, the dragging effect of dislocation for W atoms at the stress loading is considered to be one of important factors for the enhancement of precipitation and growth of the Laves phase.

4. Conclusions

The creep stress effect on the Laves phase formation behavior in Fe–10%Cr–6%W alloys has been investigated. The creep stress enhances the Laves phase formation during the aging at 873 K and 923 K. One of the mechanisms for such clear creep stress effect is considered to be due to the dragging effect of dislocations for W atoms, which enhances the movement of W atoms.

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