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In situ phase characterization in tempering and aging of Fe–Cr–W steels

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Abstract

The in situ measurements of hardness and X-ray diffraction have been performed at high temperature for Fe–Cr–W steels. The effect of tungsten concentration was investigated by comparing Fe–9Cr–2W–V–Ta steel and Fe–9Cr–3W–V–Ta steel. The tungsten addition increases hardness below 873 K. An aging test for 3600 s at 873 and 1073 K showed no clear change of hardness. A structural change from bcc to fcc and tungsten carbide coarsening are observed by X-ray diffractometry.

1. Introduction

Several types of low activation materials have been developed as candidates for fusion reactor structural components. Japanese Fe–9Cr–2W–V–Ta steel (JLF-1) is a reference alloy which has been tested under a research program conducted by Japanese universities [1–4]. In addition, weld joint and heat affected zone properties have also been characterized [3,5–8].

The structural material for fusion reactors will operate at high temperatures. Increasing the tungsten concentration would enhance the strength of JLF-1 at high temperature. For this purpose, a new alloy with increased tungsten content (Fe–9Cr–3W–V–Ta, JLS-2) was fabricated. In this study, in situ measurements at high temperature, such as hardness and phase characteristics by X-ray diffractometry, were performed for these Fe–Cr–W steels. The effect of tungsten concentration was investigated by comparing JLF-1 and JLS-2.

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2. Experimental

2.1. Sample preparation

Samples were JLF-1 ferritic steel and JLS-2 steel. The chemical composition of JLF-1 and JLS-2 steel is given in Table 1. These samples were normalized at 1323 K for 1 h and air-cooled. In addition, the samples were tempered at 1033 K for 1 h, and then air-cooled. Scanning electron microscope (SEM) images of JLF-1 and JLS-2 samples are shown in Fig. 1. It is evident that the microstructure of JLS-2 steel is almost the same as that of JLF-1.

2.2. Measurements

In situ Vickers hardness measurements at temperatures ranging from room temperature (300 K) to 1473 K were carried out by a high-temperature micro-hardness tester (QM-2, Nikon Co., Tokyo). The temperature was automatically controlled. The sample to be measured was heated in a vacuum (1.02×10^{-3} Pa). The testing load was 100 gf.

Sample crystal structures were determined by means of high-temperature X-ray diffractometry (RINT2200, Rigaku Co., Tokyo). The temperature was controlled

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Table 1
Chemical compositions of JLF-1 and JLS-2 series and heat treatments, normalized at 1323 K × 3600 s followed by air-cooling, tempered at 1033 K × 3600 s followed by air-cooling

	C	Cr	W	V	Ta	Mn	Ti	N	Si	Ni	Mo	Al	P	S
JLF-1	0.097	9.04	1.97	0.19	0.700	0.46	0.001	–	<0.1	<0.1	<0.1	–	–	–
JLF-2	0.096	8.99	2.97	0.24	0.810	1.00	–	0.0259	0.048	–	–	0.003	0.0020	0.0004

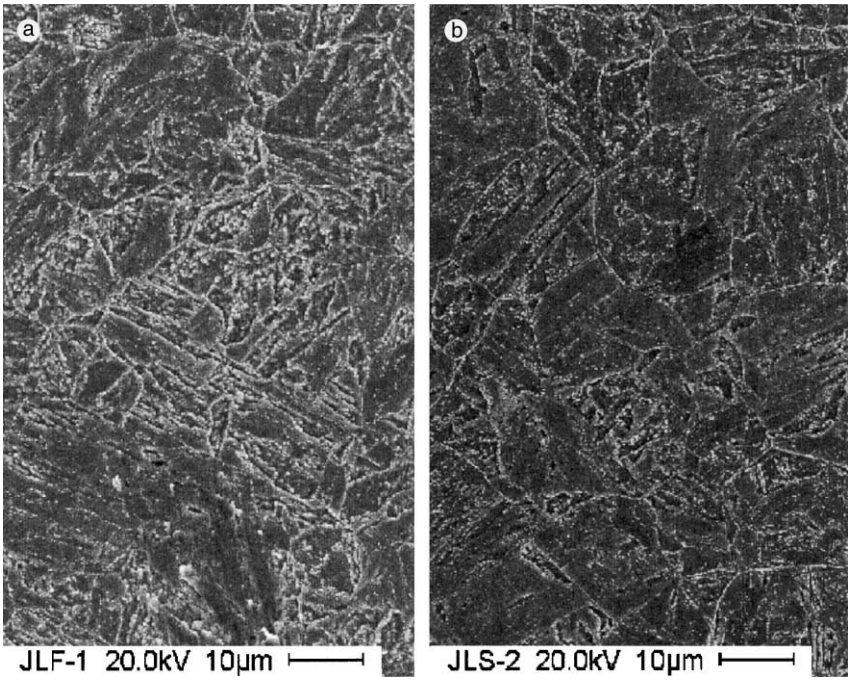

Fig. 1. SEM images of (a) JLF-1 and (b) JLS-2.

from room temperature to 1473 K under a nitrogen gas atmosphere.

3. Results and discussion

3.1. Hardness changes with temperature

Fig. 2 shows changes in Vickers hardness (H_v) against temperature for JLF-1 and JLS-2 steels. The hardness of JLS-2 is 25% higher than that JLF-1 at 373 K. The difference in hardness becomes small at 673 K. The hardness of JLS-2 is greater than JLF-1 up to 1073 K. The maximum rate of hardness change was found at 973 K. The hardness decreases slowly with increasing temperature above 1073 K.

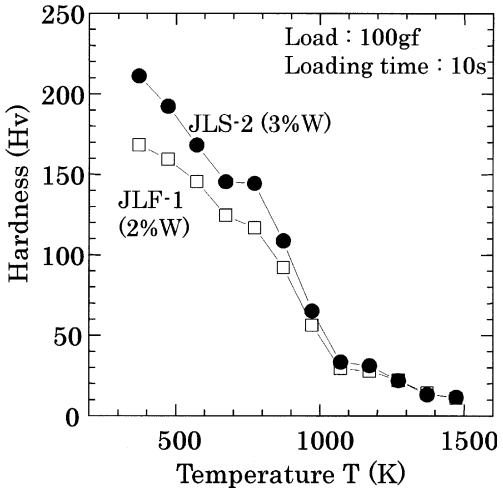

Fig. 2. Change in hardness H_v against temperature for JLF-1 and JLS-2.

3.2. Aging

Fig. 3(a) shows the change in hardness against an aging time for JLF-1 and JLS-2 at an aging temperature

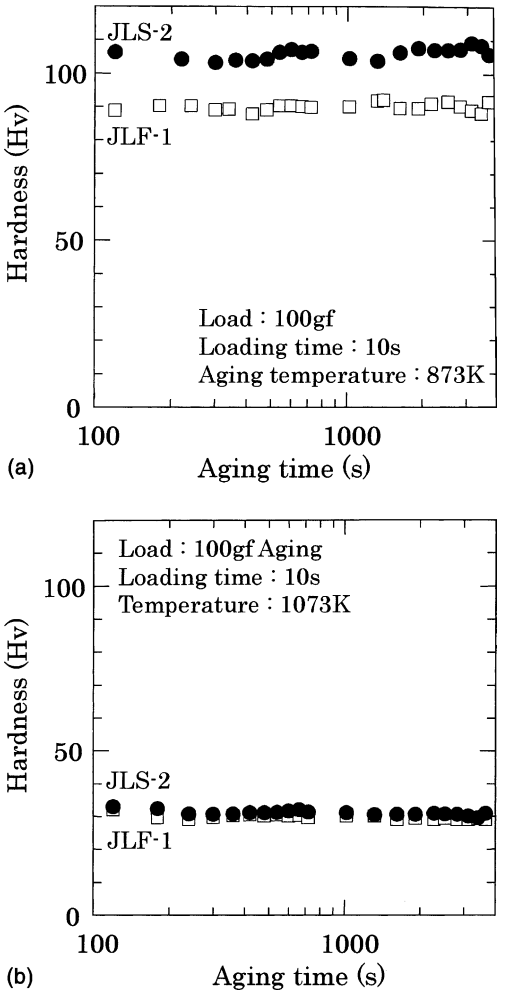

Fig. 3. Change in hardness against aging time at (a) 873 K and (b) 1073 K.

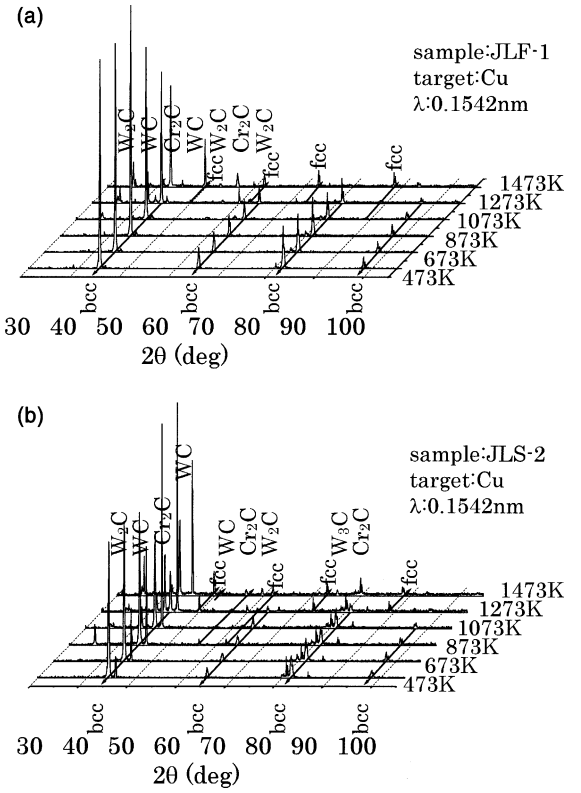

Fig. 4. Change in X-ray diffraction pattern with aging temperature for (a) JLF-1 and (b) JLS-2.

of 873 K. The hardness dose not change significantly with aging time. The H_v of JLS-2 is about 18% larger than that of JLF-1.

Fig. 3(b) shows the effect of aging at 1073K. The hardnesses of JLF-1 and JLS-2 are almost the same. The hardness dose not change significantly with aging time up to 3600 s.

3.3. X-ray diffractometry

Fig. 4(a) and (b) shows X-ray diffraction patterns of JLF-1 and JLS-2 samples aged at various temperatures. Samples annealed at 673 K, a representative service temperature for these steels, show the body centered cubic (bcc) crystal structure.

A crystal structure change from bcc to face centered cubic (fcc) as well as tungsten carbide coarsening were observed by X-ray diffractometry. The crystal structure of the sample heated to 1273 K is bcc with chromium carbide. The sample heated to 1473 K is fcc with tungsten carbide. Based on X-ray diffraction analysis results, the bcc phase was not found at 1473 K, whereas the fcc phase was not detected below 1073 K. At 1273 K both phases are detected. However, a higher X-ray intensity for the fcc phase was detected for JLS-2 than for JLF-1 at 1273 K. Thus the transformation from bcc to fcc seems to be enhanced by an increase in the tungsten content.

4. Conclusion

In situ measurements at high temperature, such as hardness and phase characteristics by X-ray diffraction, have been performed for JLF-1 (Fe–9Cr–2W) steel and JLS-2 (Fe–9Cr–3W) steel. JLS-2 shows higher hardness than JLF-1 up to 1073 K. Aging for 3600 s shows no clear change of hardness for temperatures greater than 1073 K. The structural change from bcc to fcc and tungsten carbide coarsening were observed by X-ray diffraction. The transformation from bcc to fcc appears to be enhanced by an increase in the tungsten content.

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References

[1] A. Kohyama, Y. Kohno, K. Asakura, H. Kayano, J. Nucl. Mater. 212–215 (1994) 684.
[2] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Diets, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.
[3] T. Hasegawa, Y. Tomita, A. Kohyama, J. Nucl. Mater. 258–263 (1998) 1153.
[4] A. Kohyama, Y. Kohno, M. Kuroda, A. Kimura, F. Wan, J. Nucl. Mater. 258–263 (1998) 1319.
[5] N. Inoue, T. Muroga, A. Nishimura, O. Motojima, J. Nucl. Mater. 258–263 (1998) 1248.
[6] A. Nishimura, N. Inoue, T. Muroga, J. Nucl. Mater. 258–263 (1998) 1242.
[7] N. Inoue, T. Muroga, A. Nishimura, O. Motojima, K. Oguri, Y. Nishi, Y. Katoh, A. Kohyama, Proceedings of International Symposium on Environment-Conscious Innovative Materials Processing with Advanced Energy Sources, 24–27 November 1998, Kyoto, Japan.
[8] N. Inoue, T. Muroga, A. Nishimura, T. Nagasaka, O. Motojima, S. Uchida, H. Yabe, K. Oguri, Y. Nishi, Y. Katoh, A. Kohyama, J. Nucl. Mater. 283–287 (2000) 1187.