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Effects of precipitation morphology on toughness of reduced activation ferritic/martensitic steels

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Abstract

Reduced activation ferritic/martensitic steels (RAFs) are leading candidates for structural materials of D-T fusion reactors. It is reported that 9Cr–2W–V, Ta steel (JLF-1), one of the RAFs, has superior phase stability, swelling resistance and mechanical properties against high-fluence neutron irradiation. Recently 9Cr–*x*W–V, Ta steels ($x = 2.5$, 3.0 and 3.5, JLS-series hereafter) were developed for use at higher temperatures. In this work, JLF-1 and JLS-series were thermal-aged at 823 and 923 K. Charpy impact tests were performed before and after thermal-aging. Microstructural features were observed using transmission electron microscope with energy dispersive X-ray spectrometer. From the results of Charpy impact tests, the ductile to brittle transition temperature was found to increase both by thermal-aging and by increasing tungsten content. This behavior was consistent with microstructural evolution of intergranular precipitates such as $M_{23}C_6$ and Laves phase coarsening.

1. Introduction

Cr–W–V, Ta steels are of interest as RAFs to simplify the special waste storage from fusion reactors after service [1]. It was reported that JLF-1 has been developed and proven to have good resistance against high-fluence neutron irradiation and good phase stability [2–4]. Recently, in order to achieve better energy conversion efficiency by using RAFs at higher temperatures in advanced blanket systems, the JLS-series has been prepared. These chemical compositions are based on JLF-1. In the series, improvement of high temperature mechanical properties was intended by increasing tungsten contents.

In a previous work [5], creep properties of JLF-1 and JLS-series were investigated to obtain fundamental understanding of high temperature mechanical properties. From the results, creep properties were found to improve with increasing tungsten content. From the view point of precipitation effects on mechanical properties, coarsened precipitates such as $M_{23}C_6$ and Laves phase (Fe_2W) at elevated temperatures degrade material toughness is well known.

In the present work, precipitation morphology was examined in detail and its effect on material toughness is studied.

2. Experimental procedure

Table 1 gives chemical compositions and heat treatments. The addition of tungsten instead of molybdenum improves creep rupture strength in high chromium

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Table 1
Chemical composition and heat treatments

	C	Mn	Si	Cr	W	V	Ta	Al	P	S	N
JLF-1	0.097	0.46	<0.1	9.04	1.97	0.19	0.070	0.003	0.0030	0.0020	0.0237
JLS-1	0.094	0.99	0.050	9.19	2.50	0.24	0.081	0.003	0.0020	0.0004	0.0267
JLS-2	0.096	1.00	0.048	8.99	2.97	0.24	0.081	0.003	0.0020	0.0004	0.0259
JLS-3	0.097	0.90	0.048	8.96	3.59	0.25	0.081	0.003	0.0020	0.0005	0.0270

Normalized at 1323 K × 3.6 Ks followed by air-cooling. Tempered at 1053 K × 3.6 Ks followed by air-cooling (JLF-1). Tempered at 1033 K × 3.6 Ks followed by air-cooling (JLS-series). PWHT at 1013 K × 10.8 Ks followed by furnace cooling.

steels, but excess tungsten addition leads to formation of δ-ferrite phase and Laves phase. These phases are known to degrade the toughness [6–9]. Therefore, the optimum amount of tungsten addition should be defined [10]. The reason why a post weld heat treatment (PWHT) is given is that PWHT will be performed during construction of fusion reactors for all actual structures contain weld joints of these materials.

Aging was carried out for 1000, 3000 h at 823, 923 K. After that, the toughness of JLF-1 and JLS-2 was investigated by Charpy impact testing, using 1.5 mm mini-size specimen of 1.5 by 1.5 by 20 mm containing a 0.3 mm 30° V-notch with a root radius of 0.08 mm.

For microstructural observations with transmission electron microscope (TEM), electrolytic thinning was performed by using a solution of methanol with 10 vol.% sulfuric acid. In order to examine precipitation morphology in detail, the carbon extraction replica method was used. Extraction replicas were examined by TEM with energy dispersive X-ray spectrometer (EDS). Additionally, extracted residues were prepared by using a methanol with 5 vol.% hydrochloric acid (for 12 h with current density of 40–50 mA/cm^2). They were analyzed by means of X-ray diffractometry (XRD) and weighted and investigated with EDS.

3. Result and discussion

3.1. 550 °C aged specimens

Fig. 1 shows the results of Charpy impact testing before and after aging at 550 °C for 1000 and 3000 h, for JLF-1 and JLS-2, respectively. XRD results are summarized in Table 2.

3.1.1. JLF-1 aged at 550 °C

From Fig. 1, the ductile to brittle transition temperature (DBTT) decreases with aging for JLF-1. However, changes in extracted residues were not observed.

3.1.2. JLS-2 aged at 550 °C

In Fig. 1, the DBTT decreased after a 1000 h age, but increased after a 3000 h age for JLS-2. In the aging conditions, M_6C developed with increasing aging time. In the 1000 h aged specimen, no change of extracted residue amount was observed. So, this response is expected to be as same as in the case of the JLF-1 1000 h aged specimen. In the 3000 h aged specimen, the amount of extracted residues increased and thermal embrittlement was observed. It is considered that the degradation in toughness was caused by coarsening of $M_{23}C_6$ and M_6C.

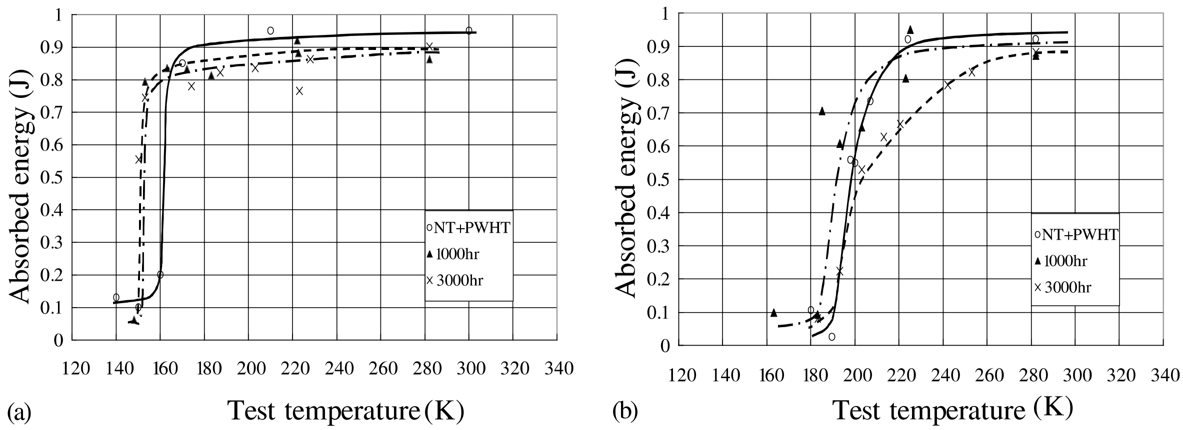

Fig. 1. Charpy impact test results of 550 °C aged specimens. (a) JLF-1; (b) JLS-2.

Table 2
XRD results of extracted residues

Temperature (°C)	Precipitate	JLF-1			JLS-2		
		NT + PWHT	1000 h	3000 h	NT + PWHT	1000 h	3000 h
550	$M_{23}C_6$	O	O	O	O	O	O
	M_6C	O	O	O	O	O	O
	Laves	×	×	×	×	×	×
650	$M_{23}C_6$	O	O	O	O	O	O
	M_6C	O	O	×	O	×	×
	Laves	×	×	O	×	O	O

3.2. 650 °C aged specimens

Fig. 2 shows the results of Charpy impact testing. before and after aging at 650 °C for 1000 and 3000 h. XRD results are summarized in Table 2. Comparing the 550 and 650 °C conditions, significant thermal embrittlement was observed in both specimens. In addition to that, a change in extracted residue amounts was observed. Microstructural evolutions under these conditions are given in Fig. 3. Schematic illustrations of the microstructural evolutions are given in Fig. 4.

3.2.1. JLF-1 aged at 650 °C

In Fig. 2, the DBTT increased and USE (Upper Shelf Energy) decreased due to thermal aging. From XRD analysis of extracted residues (Table 2), M_6C disappeared and Laves phase appeared. The amount of extracted residue increased up to 1000 h, but the change between 1000 and 3000 h was very small. Comparing the 1000 and 3000 h aged specimens, toughness was degraded due to thermal aging, but the change in extracted residue amounts was not significant. The difference between these specimen conditions is the precipitation of Laves phase. So, it is expected that toughness is independent of the amount of precipitate and that Laves phase causes a severe effect. Laves phase had precipitated and aggregated on prior austenite grain boundaries preferentially (Fig. 3).

3.2.2. JLS-2 aged at 650 °C

As found in Fig. 2, toughness was degraded with increasing aging time for JLS-2 aged at 650 °C. The thermal embrittlement for the 1000 h treatment was more significant than that between 1000 and 3000 h. In addition, the increase in extracted residue amount was similar. Laves phase was observed in both aged specimens. However, the increase in extracted residue between 1000 and 3000 h was smaller than that for the 1000 h treatment. So, it is believed that Laves phase precipitation saturated before 1000 h. In Fig. 3, Laves phase on lath boundaries disappeared on aging from 1000 to 3000 h, and instead, Laves phase precipitated and aggregated on prior austenite grain boundaries

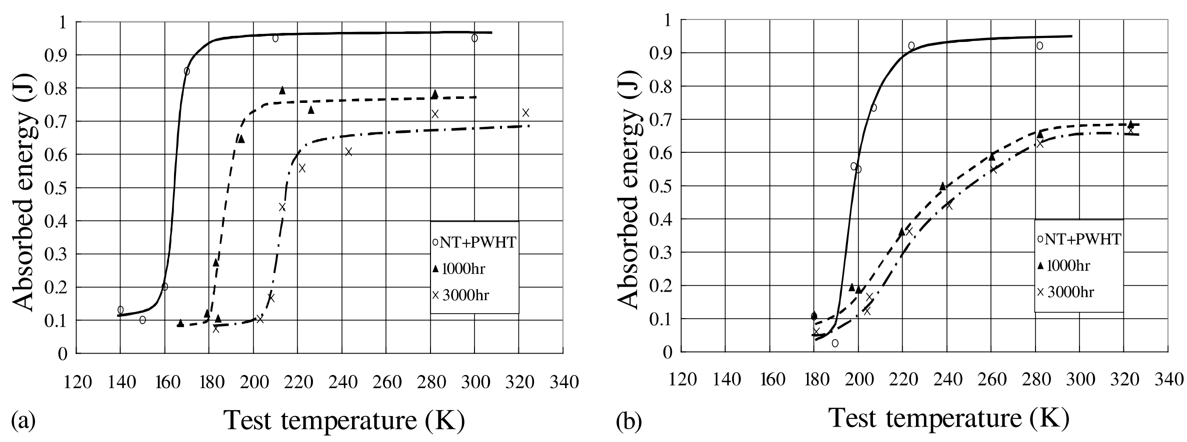

Fig. 2. Charpy impact test results of 650 °C aged specimens. (a) JLF-1; (b) JLS-2.

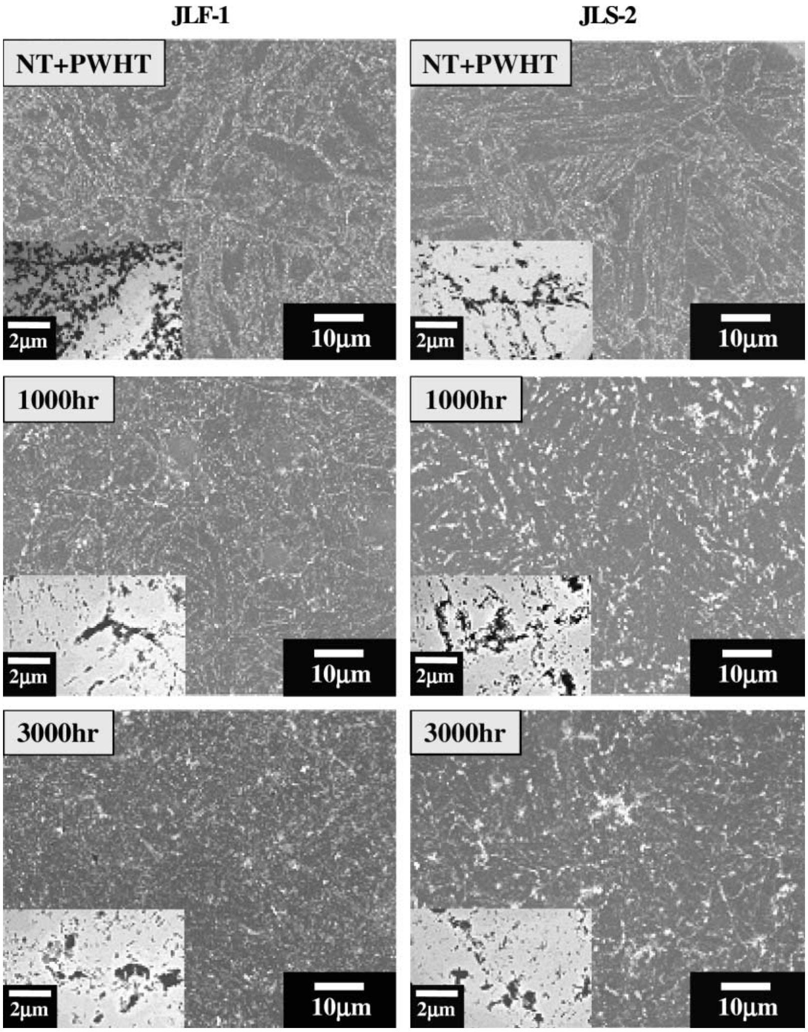

Fig. 3. Precipitation morphology of 650 °C aged specimen.

preferentially. Similar precipitation morphology was reported previously [11]. Laves phase is thought to be incoherent with the matrix and precipitates in distorted areas, such as on lath boundary, prior austenite grain boundaries and in the vicinity of other precipitates, such as $M_{23}C_6$. So the interface between the Laves phase and the matrix is considered to be a site of generation of a crack. As Laves phase precipitates, sites for crack generation increase and therefore the precipitation morphology degrades toughness.

4. Summary

DBTT decreased due to 550 °C thermal aging in some specimens. This phenomenon was caused by recovery in the matrix such as decrease of dislocation density and rearrangement of dislocations.

Thermal embrittlement was observed in all specimens aged at 650 °C. Toughness was degraded significantly with the precipitation of Laves phase. Laves phase is incoherent with the matrix and precipitated on distorted areas and precipitated on prior austenite grain boundaries with recovery of the martensite lath structure. The interface between Laves phase and the matrix is a site for crack nucleation, because Laves phase is incoherent with matrix. Sites for crack generation increase with increasing Laves phase and such precipitation morphologies degrade toughness. In JLS-2, it is expected that thermal embrittlement saturated before 1000 h. The increase in extracted residue between 1000 and 3000 h was smaller than that between NT + PWHT and 1000 h, but

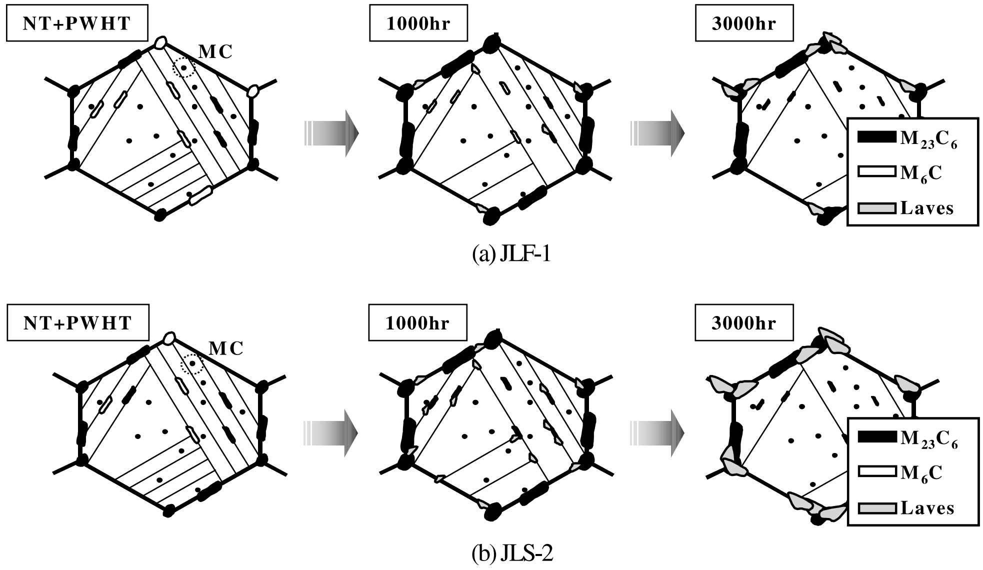

Fig. 4. Schematic illustrations of precipitation morphology of 650 °C aged specimens.

precipitation morphology was changed significantly. Thermal embrittlement was caused by precipitation of Laves phase rather than precipitate morphology changes.

References

[1] R.L. Klueh, P.J. Maziaz, J. Nucl. Mater. 155–157 (1988) 602.

[2] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1996) 138.

[3] Y. Kohno, A. Kohyama, M. Yoshino, K. Asakura, J. Nucl. Mater. 212–215 (1994) 707.

[4] K. Shiba, A. Hishinuma, J. Plasma Fus. Res. 74 (1998) 436.

[5] H. Sakasegawa, T. Hirose, A. Kohyama, Y. Katoh, T. Harada, T. Hasegawa, ASTM STP 1405, p. 546.

[6] F. Abe, H. Araki, T. Noda, M. Okada, J. Nucl. Mater. 155–157 (1988) 656.

[7] F. Abe, T. Noda, M. Okada, J. Nucl. Mater. 195 (1992) 51.

[8] Y. Hosoi, N. Wade, S. Kunimitsu, T. Urita, J. Nucl. Mater. 141–143 (1986) 461.

[9] K.W. Tupolme, D. Dulieu, G.J. Butterworth, J. Nucl. Mater. 179–181 (1991) 684.

[10] F. Abe, H. Araki, T. Noda, Metall. Trans. A 22 (1991) 2225.

[11] M. Tamura, H. Hayakawa, A. Yoshitake, A. Hisinuma, T. Kondo, J. Nucl. Mater. 155–157 (1988) 620.