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Mechanical properties of 8Cr–2WVTa steel aged for 30 000 h

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Abstract

A mill production plate of a reduced activation ferritic steel was thermally aged for up to 30 000 h at 400–650°C. Charpy impact tests, creep rupture tests and hardness tests were conducted. Both Vickers hardness number and creep strength decrease with aging at 650°C. The ductile–brittle transition temperature (DBTT) increases with both aging time and aging temperature. However, the DBTT does not exceed +20°C even after aging at 650°C for 30 000 h. Extracted residues and extraction replicas were analyzed metallurgically. The increase in DBTT is related mainly to the precipitation of Laves phase on the prior austenite grain boundaries. The rather low DBTT after aging is caused by the fine prior austenitic grain size. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Reduced activation ferritic steel has been a candidate alloy for the first wall of a Tokamak reactor [1]. The reason is that besides the relatively good resistance of the steel to void swelling and good thermal conductivity and thermal expansion, there is no essential engineering issue in constructing and fabricating large scale components [1–5]. On the other hand, both the mechanical properties of the weldments and the degradation of properties after long term service are still critical issues for judging the acceptability of the reduced activation ferritic steels [1,6]. These problems strongly depend on chemical composition in addition to service conditions and welding methods; therefore, round robin tests have been performed using the mill product of 8Cr–2WVTa steel as an IEA activity [7] and much effort has been focused on the shift in ductile–brittle transition temperature (DBTT) under irradiation [8]. However, a report on the DBTT shift after thermal aging is not yet available.

The first mill product of 8Cr–2WVTa steel (pre-IEA heat, hereafter) was also tested systematically and the results have been reported elsewhere [4]. The phase stability has also been discussed briefly on specimens aged for up to 10 000 h. The purpose of the present paper is to evaluate the precipitation behavior and the related mechanical properties of the pre-IEA heat aged for longer time, i.e., 30 000 h.

2. Experimental procedure

25 mm thick plates of the pre-IEA heat were normalized at 1040°C for 30 min, tempered at 740°C for 2 h and followed by aging at 400–650°C for up to 30 000 h in air. The chemical composition is as follows; 0.096% C–0.10%Si–0.15%Mn–7.71%Cr–0.18%V–2.1%W–0.04% Ta–0.0043%N with the balance Fe. Charpy impact tests were conducted using full size V notched specimens (JIS Z 2202, No. 4) and specimens for rupture tests were machined to 6 mm diameter and 30 mm gauge length. All of the specimens were machined from 1/4 thickness position. X-ray diffraction and chemical composition analysis were performed on residues extracted from the aged specimens. The details of the experimental procedure are described in previous work [9].

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3. Results

3.1. Mechanical properties

The absorbed energy at 0°C of the materials aged for 10 000 and 30 000 h is plotted along with short time aging data [4] in Fig. 1. The figure shows that the higher the aging temperature is, the earlier the absorbed energy decreases when the aging temperature is 500–650°C. The absorbed energy seems to approach about 50 J when the aging temperature is higher than 600°C. Fig. 2 shows that although the upper shelf energy decreases and DBTT increases after aging, the values still stay at acceptable levels, i.e., roughly 200 J and +10°C to +20°C, respectively, even after aging at 600°C or 650°C over 10 000 h. However, as shown in the top of Fig. 2, the decrease in Vickers hardness number at 650°C is larger than that at 600°C.

Fig. 3 shows the stress vs time to rupture diagram at 600°C on the aged specimens as compared with that in the normalized and tempered condition. Although the time to rupture is usually plotted against the logarithm of stress, in Fig. 3 the stress is plotted on a linear scale according to a new creep theory [10]. After aging at 600°C for 30 000 h, the time to rupture is shorter at higher stresses. However, at lower stresses the difference between the aged and the unaged material becomes smaller. On the other hand, aging at 650°C for 30 000 h causes a significant decrease in the time to rupture.

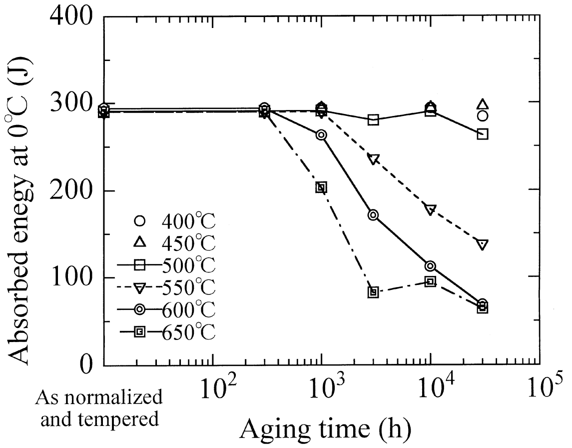

Fig. 1. Change in absorbed energy due to aging.

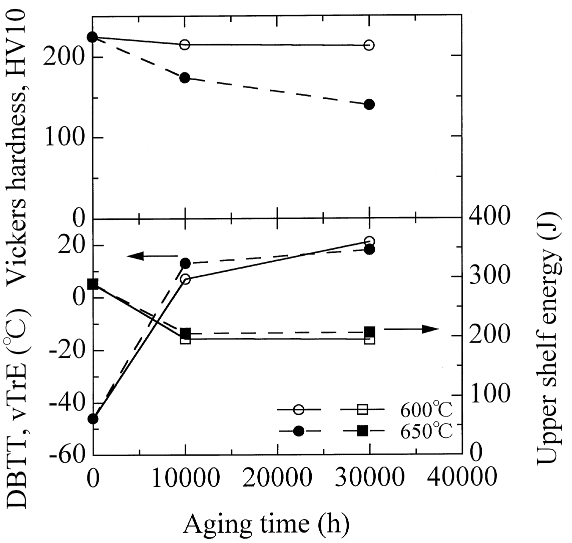

Fig. 2. Impact properties and hardness as a function of aging time.

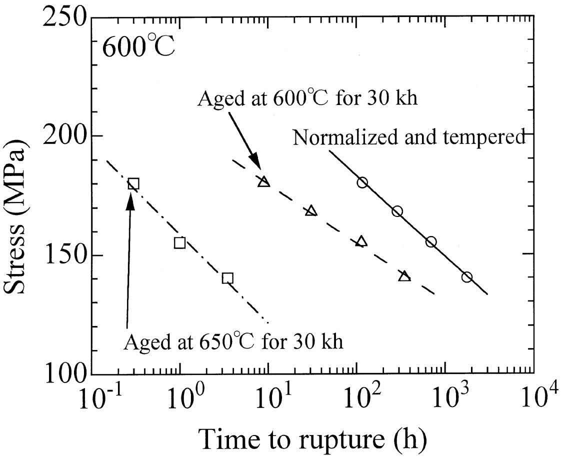

Fig. 3. Stress vs time to rupture diagram for the aged material.

3.2. Microstructure

Even after aging at 600°C for 30 000 h, the tempered martensite structure was maintained. However, the martensitic structure was almost gone and only ferritic grains were observed after aging at 650°C for 30 000 h. The etched surfaces and the extraction replicas of the aged specimens were observed under a scanning electron microscope (SEM) and transmission electron microscope (TEM) (Figs. 4 and 5, respectively). Table 1 shows the summary of the X-ray diffraction analysis of the extracted residues and the TEM and SEM observations.

Figs. 4 and 5 show that particles of 10–1000 nm precipitate both separately and like a net-work on the prior austenite grain, packet and lath boundaries in the normalized and tempered state. They are composed of mainly Cr and Fe and are identified as $Cr_{23}C_6$ (Table 1). The precipitates within the grains grow gradually and the number decreases with aging, but they are rather stable and the maximum size after aging is about 200 nm. On the other hand, the $Cr_{23}C_6$ carbide on the prior

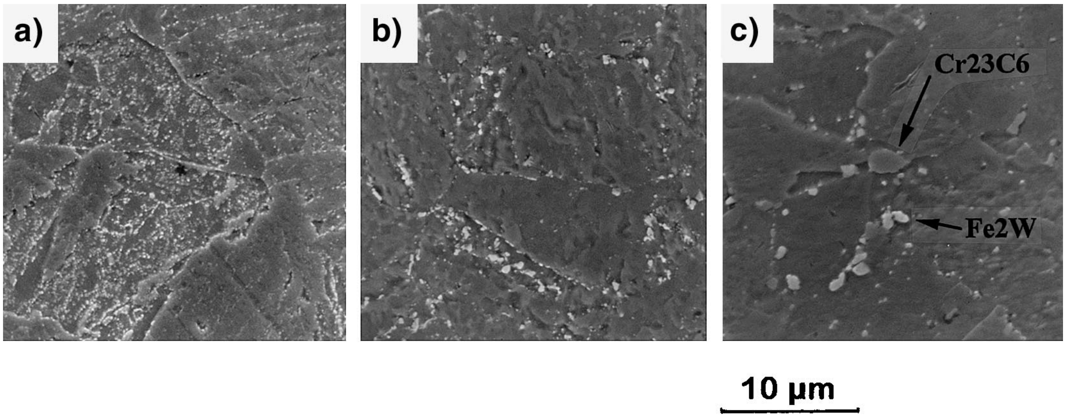

Fig. 4. Secondary electron images: (a) normalized and tempered; (b) aged at 600°C for 30 000 h; (c) aged at 650°C for 30 000 h.

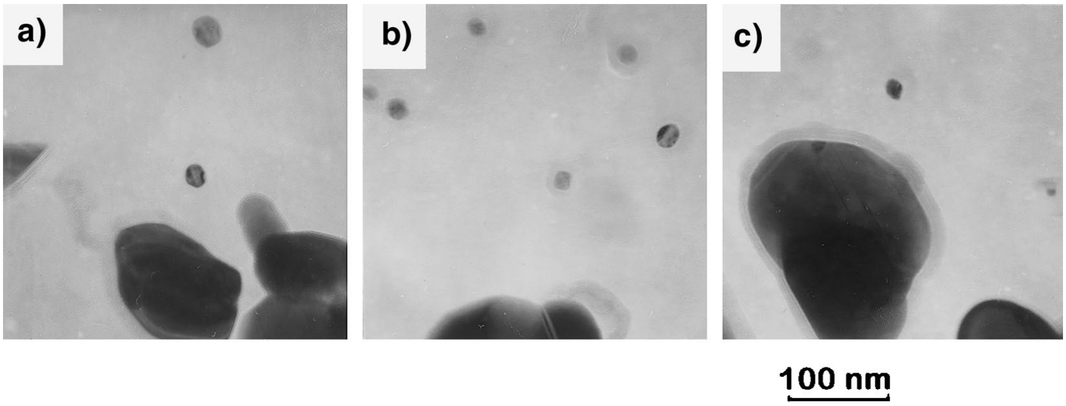

Fig. 5. Transmission electron microscope images of extracted replicas: (a) normalized and tempered; (b) aged at 600°C for 30 000 h; (c) aged at 650°C for 30 000 h. Fine particles are identified as TaC.

Table 1
Identified precipitates and their sizes

Heat treatment	Identified precipitate		Size of boundary precipitate (µm)[a]	
	XDM	TEM	Fe2W	$Cr_{23}C_6$
Normalized and tempered	$Cr_{23}C_6$	$Cr_{23}C_6$, (Fe2W),TaC (27 nm)[b]	–	1.00
600°C 10 kh	$Cr_{23}C_6$, Fe2W	–	1.83	3.17
600°C 30 kh	$Cr_{23}C_6$, Fe2W	$Cr_{23}C_6$, Fe2W, TaC (17 nm)	1.27	0.83
650°C 10 kh	$Cr_{23}C_6$, Fe2W	–	1.53	1.17
650°C 30 kh	$Cr_{23}C_6$, Fe2W	$Cr_{23}C_6$, Fe2W, TaC (23 nm)	1.80	2.60

[a] Maximum length along boundary is measured under SEM.
[b] Average diameter of TaC.

austenitic grain boundaries grow rapidly with aging, and large particles of about 3000 nm were found occasionally after aging at 650°C for 30 000 h (large gray precipitate in Fig. 4(c)).

Blocky and massive precipitates are found mainly on the prior austenitic grain boundaries after aging for longer than 10 000 h. They are mainly composed of Fe, W and Cr and are identified as Laves phase (Table 1 – white precipitates in Figs. 4(b) and (c) and are symbolized as Fe_2W). The maximum boundary precipitate for each SEM view at 3000x was measured and the average values are also listed in Table 1.

Besides these relatively large particles, fine particles of 5–50 nm are still observed in all of the specimens (Fig.

5). These are composed mainly of Ta with a small amount of V, Ti and Cr. The small particles found in TEM look to be extremely stable and grow very slowly during aging. They are judged to be TaC and not TaN, because the steel contains very little nitrogen.

Table 2 shows the chemical compositions of the extracted residues. While the total amount of precipitate increases slightly with increasing aging time at 600°C, at 650°C it decreases with aging time, and is smaller than the amount of precipitates in material aged at 600°C. Moreover, the contents of both W and Cr are also clearly decreasing with increasing aging time at 650°C.

4. Discussion

4.1. Evaluation of microstructure

An equilibrium state of the microstructure can be simulated using THERMO-CALC [11] and the results of the calculation are also listed in Table 2. In the calculation Ta is ignored, because reliable thermodynamical data on Ta are not available. The table shows that the computer simulation for the equilibrium state seems to roughly explain the experimental results, i.e., the contents of the major elements of the extracted residues approach the calculated equilibrium values. Moreover, the total amount of precipitate decreases with increasing aging temperature. These calculations are confirmed to be reasonable by Morimoto et al. on a similar alloy, i.e., 9Cr1.8W 0.5Mo0.2VNbN steel; the prediction by THERMO-CALC roughly coincides with the chemical compositions of residues extracted from the specimens crept for about 10 000 h at 600°C [12].

The microstructure and the hardness number for the aged specimen at 650°C suggest that de-carburization might have occured during aging. Therefore, a chemical analysis was conducted on the aged specimens, and it was found that carbon content only slightly decreases during aging at 650°C, i.e., 0.082% for 30 000 h. The thermodynamical simulation for the de-carburized state was also conducted and the results are listed in Table 2. As shown in the table the de-carburization can explain the decrease in Cr contents during the aging at 650°C. However, the computer simulation on the de-carburized specimen predicts a slight increase in the amount of Fe_2W and no change in total W contents in the residues. Therefore, the observed distinct decrease in W contents with aging time have not been explained metallurgically. To clarify the phenomenon further work is needed.

4.2. Mechanical properties

It has been reported that the absorbed energy decreases when the aging temperature is higher than 550°C because of the rapid increase in precipitation [13]. A similar phenomenon was found in the mill production plate as shown in Figs. 1, 4 and 5. The detailed model of the growth of the precipitates has not yet been established. However, if the absorbed energy, ${}_vE_0$, is assumed to decrease from the initial level according to the following regression equation,

$$ {}_vE_0 = {}_vE_0(i) \cdot \left[1 - A\left\{t \exp\left(\frac{-Q}{RT}\right)\right\}^n\right], $$

we obtain $n = 0.525$ and $Q = 141.2$ kJ/mol, where, n, Q, R, T, A and ${}_vE_0(i)$ are the time exponent, the apparent activation energy, the gas constant, the absolute aging temperature, a constant and the initial value, respectively. The multiple regression correlation coefficient is 91%. The obtained time exponent of about 0.5 suggests that the rate of degradation is determined by a diffusion-controlled process in a semi-infinite body [14]. This interpretation leads to the hypothesis that the absorbed energy is lowered by the massive precipitates grown on the prior austenite grain boundaries. The activation energy is much lower than that for diffusion in alloy steels, which suggests that the defect-assisted diffusion may take place.

Table 2
Chemical analysis of extracted residues and the calculated values by THERMO-CALC (mass%)

Heat treatment	Fe	W	Cr	Si	V	Ta	C	Total	$Cr_{23}C_6$	Laves
Normalized and tempered	0.45	0.24	0.85	0.005	0.02	0.025	–	1.59	–	–
600°C 10 kh	0.80	1.10	1.05	0.02	0.02	0.031	–	3.02	–	–
600°C 30 kh	0.87	1.22	1.09	0.02	0.02	0.031	–	3.25	–	–
600°C calculated	0.90	1.43	1.33	0.00	0.01	–	0.096	3.76	1.92	1.82
650°C 10 kh	0.74	1.01	1.00	0.01	0.02	0.032	–	2.81	–	–
650°C 30 kh	0.70	0.81	0.93	0.01	0.02	0.030	–	2.50	–	–
650°C calculated	0.77	1.08	1.20	0.00	0.01	–	0.096	3.15	1.96	1.18
650°C calculated (0.082%C)	0.74	1.08	1.05	0.00	0.01	–	0.082	2.95	1.67	1.26

Table 3
Solute element in ferrite (mass%)

Heat treatment	W	V	Ta
Normalized and tempered	1.78	0.17	0.014
600°C 10 kh	0.90	0.17	0.004
600°C 30 kh	0.78	0.17	0.000
650°C 10 kh	1.15	0.17	0.005
650°C 30 kh	1.22	0.17	0.003

When the aging condition is longer than 10 000 h at 600°C and 650°C, the DBTT and the upper shelf energy each seem to approach a constant value as shown in Fig. 2. The impact properties generally depend on hardness, particle size, the amount of precipitate and the chemical composition of the matrix. Little hardness change at 600°C shown in Fig. 2 suggests that the dislocation structure changes little during aging. As shown in Table 2 and Fig. 5, precipitation of Fe_2W phase approaches the equilibrium state and most of the Fe_2W precipitates are on the prior austenite grain boundaries. Moreover Table 1 and Fig. 5 reveal that the size of the maximum boundary precipitates is larger than 1000 nm after aging for longer than 10 000 h. Therefore, the impact properties seem to become constant after aging longer than 10 000 h.

The prior austenite grain size, i.e., JIS #8, is fine due to the presence of TaC and the fine grains are kept stable after long time aging. Therefore, the impact properties are considered to be maintained at moderate levels after long term aging.

4.3. Solute atoms in ferrite

Table 3 shows the solute concentrations in ferrite which are calculated as the differences between the total and the residues for each aged specimen. About 50% of W is in the matrix even after aging at 650°C for 30 000 h. About 85% of V is in the matrix after aging.

Regarding Ta, Jayaram and Klueh [15], using an atom probe field ion microscope on very similar alloys, found that most of Ta, i.e., about 0.07%, is dissolved in the ferritic matrix in the normalized and tempered condition. However, using inductively coupled plasma atomic emission it was found that in the present steel only about 35% of total Ta, i.e., 0.014%, is dissolved in the matrix in the normalized and tempered condition. Tantalum is precipitated as TaC during the subsequent aging, but about 30 ppm of Ta is still in the matrix after aging at 600–650°C.

5. Conclusion

The mill production plate of 8%Cr–2%W–0.2%V–0.04%Ta steel was thermally aged at 400–650°C for up to 30 000 h and mechanical properties have been studied metallurgically, which lead to the following conclusions.

1. Acceptable impact properties – a DBTT of 20°C and upper shelf energy of 200 J – are retained even after aging at 650°C for 30 000 h, if the prior austenite grain size is fine.
2. Degradation in DBTT is caused by massive precipitation of Laves phase, mainly on prior austenite grain boundaries. However, the DBTT approaches a limiting value when the precipitation approaches the equilibrium state.
3. Tungsten and Vanadium remain in solution in ferrite – about 50% and 85% of the total value, respectively. Most of Ta precipitates as TaC, but about 30 ppm of Ta is still in the ferrite after the aging at 600–650°C for 30 000 h.

References

[1] E.E. Bloom, J. Nucl. Mater. 258–263 (1998) 7.
[2] Fusion Reactor System Laboratory (Ed.), Concept Study of the Steady State Tokamak Reactor (SSTR), JAERI-M 91-081, June 1991.
[3] M. Tamura, H. Hayakawa, M. Tanmura, A. Hishnuma, T. Kondo, J. Nucl. Mater. 141–143 (1986) 1067.
[4] N. Yamanouchi, M. Tamura, H. Hayakawa, A. Hishinuma, T. Kondo, J. Nucl. Mater. 191–194 (1992) 822.
[5] M. Tamura, Shinsozai 4 (1993) 60.
[6] A. Nishimura, N. Inoue, T. Muroga, J. Nucl. Mater. 258–263 (1998) 1242.
[7] Proceedings of the IEA Workshop on Ferritic/Martensitic Steels, held in Tokyo, 26–28 October 1992, vols. I and II, March 1993.
[8] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 258–263 (1998) 1269.
[9] M. Tamura, K. Shinozuka, K. Masamura, K. Ishizawa, S. Sugimoto, J. Nucl. Mater. 258–263 (1998) 1158.
[10] M. Tamura, H. Esaka, K. Shinozuka, ISIJ Int. 39 (1999) 380.
[11] B. Sundman, B. Jansson, J.-O. Andersson, CALPHAD 9 (1985) 153.
[12] H. Morimoto, T. Tanaka, S. Ookita, M. Fuji, Quart. J. Jpn. Weld. Soc. 46 (1998) 512.
[13] M. Tamura, H. Hayakawa, A. Yoshitake, A. Hishinuma, T. Kondo, J. Nucl. Mater. 155–157 (1988) 620.
[14] R. Wagner, R. Kampmann, Mater. Sci. Technol. 5 (1991) 213.
[15] R. Jayaram, R.L. Klueh, Metall. Trans. A 29 (1998) 1551.