Journal of Nuclear Materials 386–388 (2009) 495–498

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Effect of thermal ageing on tensile and creep properties of JLF-1 and CLAM steels

Y.F. Li [a,b,*], T. Nagasaka [c], T. Muroga [c], Q.Y. Huang [b], Y.C. Wu [b]

[a] *The Graduate University for Advanced Studies, 322-6 Oroshi, Toki, Gifu 509-5292, Japan*
[b] *Institute of Plasma Physics, Chinese Academy Sciences, Hefei, Anhui 230031, China*
[c] *National Institute for Fusion Science, 322-6 Oroshi, Toki, Gifu 509-5292, Japan*

ABSTRACT

The thermal ageing treatments of JLF-1 and CLAM steels were performed at 823 K up to 2000 h to simulate in-service condition and at 973 K for 100 h to provide heavy overageing. Hardness, tensile and creep rupture tests were conducted in the aged and un-aged materials. The results showed increase in hardness at room temperature and improvement in creep properties after ageing at 823 K/2000 h for the both steels, implying the strengthening. However, softening and degradation of creep properties occurred after ageing at 973 K/100 h. Lower normalization and tempering temperature of CLAM steel were suggested to be responsible for higher hardness and tensile strength, lower minimum creep rate and longer rupture time, and also for higher susceptibility to the thermal ageing of relatively short term than those of JLF-1.

1. Introduction

Several types of reduced activation ferritic/martensitic steels (RAFMs) have been considered as promising candidates for blanket structural material in ITER-TBM and DEMO fusion reactors [1]. JLF-1 [2] and CLAM (Chinese Low Activation Martensitic) [3,4] were developed by Japanese university and China in recent years, respectively. The qualification of these materials for practical application requires an exhaustive understanding of their microstructure and mechanical properties. Of special relevance is ageing resistance behavior under long-term loading at the high temperatures of fusion reactor operation [5].

Although comparative evaluation of basic mechanical properties and microstructure for JLF-1 and CLAM steels was carried out [6], the knowledge about the effect of ageing is still limited for JLF-1 and not yet available for CLAM steel. In this work, the thermal ageing treatments were performed on these steels at 823 K up to 2000 h to simulate in-service condition and at 973 K to provide heavy overageing. The mechanical properties were investigated with focused attention on their tensile and creep properties.

2. Experimental

The materials used were JLF-1 (JOYO-II-HEAT) and CLAM (HEAT 0603) steels. Their chemical compositions are listed in Table 1. The JLF-1 steel has been supplied as plates of 25 mm thick in the normalized and tempered conditions. The CLAM steel was melted in a vacuum induction furnace into an ingot of 300 kg, followed by hot-forging and rolling into a 15-mm-thick plate in 2006. The detailed heat treatment conditions for both steels are given in Table 2.

Thermal ageing treatments were carried out at 823 K up to 2000 h in a muffle furnace, in which the samples were vacuum sealed in quartz capsules in order to avoid oxidation. Ageing at 973 K for 100 h was conducted in an infrared image furnace in a vacuum of $\sim 1 \times 10^{-4}$ Pa.

The Vickers hardness was measured at room temperature with a load of 300 g and loading time of 30 s, and the average values were calculated out of 5–10 data points for every result.

The SSJ specimens with a gauge size of $5 \times 1.2 \times 0.25$ mm^3 were machined parallel to the rolling direction. The tensile tests were conducted at an initial strain rate of 6.67×10^{-4} s^{-1} and the test temperatures ranged from room temperature (RT) to 873 K. The test at RT was conducted in air, while the tests at the elevated temperatures in a vacuum of 10^{-4} Pa. The 0.2% proof strength was measured as yield strength.

The uniaxial creep tests up to rupture were performed at two conditions, 823 K with the applied stress of 250 MPa and 923 K with 150 MPa, in a vacuum of $<1 \times 10^{-4}$ Pa using the specimen of the same shape as for the tensile tests.

3. Result and analysis

3.1. Hardness measurements

The hardness results are shown in Table 3.

---

* Corresponding author. Address: The Graduate University for Advanced Studies, 322-6 Oroshi, Toki, Gifu 509-5292, Japan.
E-mail address: li.yanfen@nifs.ac.jp (Y.F. Li).

0022-3115/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.12.149

Table 1
Chemical compositions of JLF-1 and CLAM steels in wt%.

	Cr	W	C	Mn	V	Ta	O	N	S	Fe
JLF-1	9.00	1.98	0.09	0.49	0.20	0.083	0.0019	0.0150	0.0005	Bal.
CLAM	8.94	1.45	0.13	0.44	0.19	0.15	–	–	0.004	Bal.

Table 2
Heat treatment conditions for JLF-1 and CLAM steels.

Type of steel	Normalization	Tempering
JLF-1	1323 K/60 min/air cool	1053 K/60 min/air cool
CLAM	1253 K/30 min/air cool	1033 K/90 min/air cool

Table 3
Hardness data of JLF-1 and CLAM steels.

Type of steel	Heat treatments		
	No ageing	823 K/2000 h ageing	973 K/100 h ageing
JLF-1	214 ± 6	220 ± 4	205 ± 4
CLAM	234 ± 6	236 ± 6	217 ± 5

Table 3 shows that the hardness values of CLAM steel were higher than those of JLF-1 at all conditions. After ageing at 823 K for 2000 h the hardness increased slightly for both steels. However, ageing at 973 K for 100 h caused a decrease of hardness, but the softening of JLF-1 steel was smaller than that of CLAM.

3.2. Tensile properties

The tensile tests were conducted from RT to 873 K for all samples of JLF-1 and CLAM and the results are presented in Fig. 1(a)–(c).

The ultimate tensile strength (UTS) and yield strength (YS) of CLAM steel were higher and total elongation (TE) were smaller than those of JLF-1 steel at all conditions, in agreement with the hardness data. The UTS and YS at RT before ageing were 678 and 576 MPa for CLAM steel, and 625 and 510 MPa for JLF-1 steel, respectively. Both steels exhibit adequate strength and ductility level, comparable to other RAFM steels such as Eurofer'97 [7] and F82H [8].

As shown in the figure, no significant degradation of tensile properties (UTS, YS and TE) for both steels was detected by ageing at 823 K for 2000 h. Both steels display very stable tensile properties and small ageing effects. Similar behaviors after thermal ageing treatments were also observed for F82H [9] and Eurofer'97 steels [10]. However, ageing at 973 K for 100 h resulted in a decrease in UTS and YS, accompanied by an increase in TE. The differences between un-aged and ageing at 973 K/100 h became smaller with the increase of test temperature. The present results show that the influence of thermal ageing at 973 K is strong for these RAFM steels at the test temperature below the typical maximum blanket operation temperature of 823 K.

3.3. Creep properties

The uniaxial constant load creep tests were conducted at 823 K with the applied stress of 250 MPa and 923 K with 150 MPa. Figs. 2 and 3 show the effects of thermal ageing on minimum creep rate and rupture time, respectively. The minimum creep rate is plotted

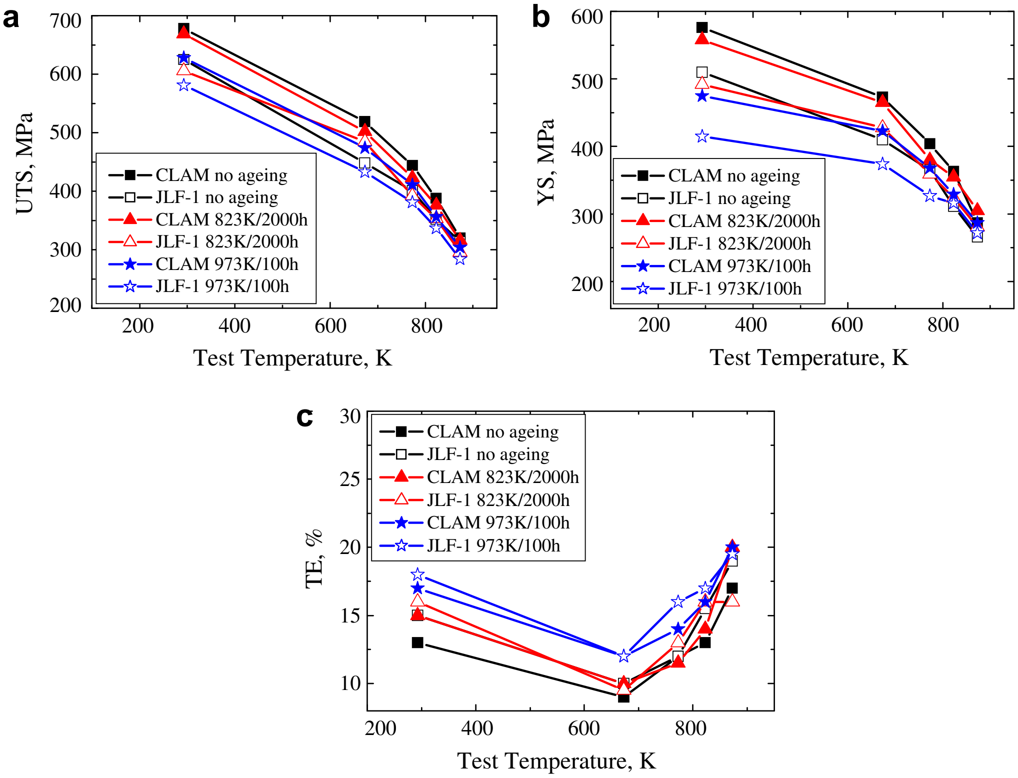

Fig. 1. Effect of thermal ageing on tensile properties of JLF-1 and CLAM steels: (a) UTS; (b) YS; (c) TE.

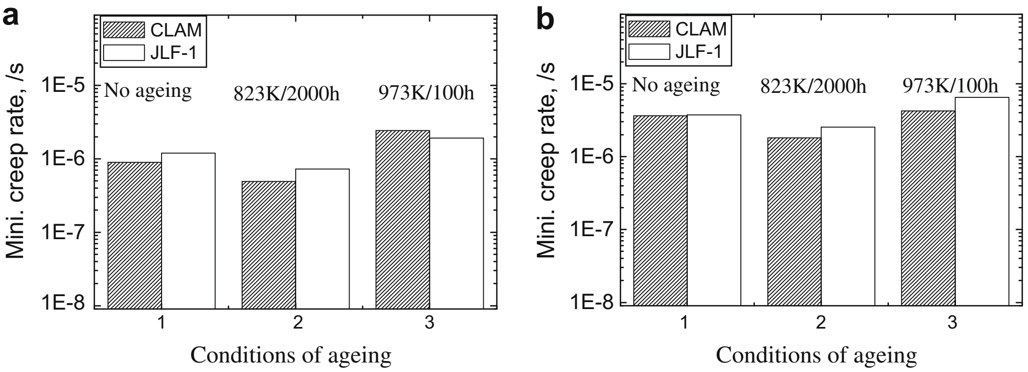

Fig. 2. Effect of thermal ageing on minimum creep rate for JLF-1 and CLAM steels tested at: (a) 823 K/250 MPa; (b) 923 K/150 MPa.

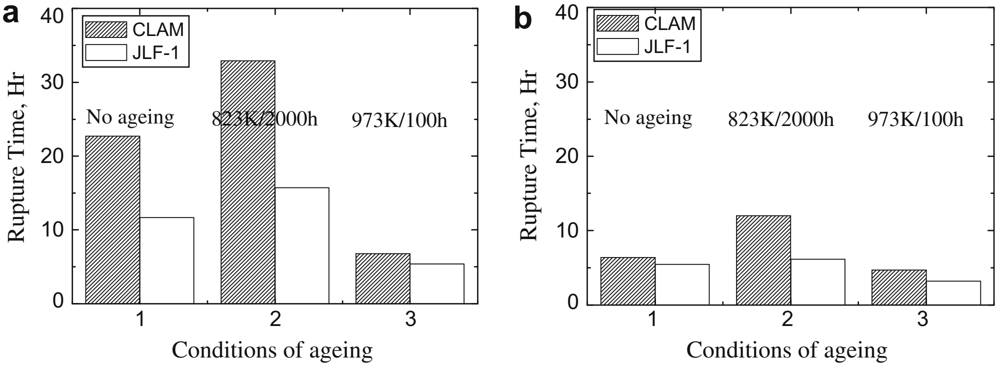

Fig. 3. Effect of thermal ageing on rupture time for JLF-1 and CLAM steels tested at: (a) 823 K/250 MPa; (b) 923 K/150 MPa.

on a logarithm scale. The data reproducibility was examined by repeating three tests for JLF-1 at 923 K with 150 MPa, which showed the maximum errors of 20% for the minimum creep rate and 29% for the rupture time.

After ageing at 823 K for 2000 h, the minimum creep rate decreased and the rupture time increased for both steels, relative to the un-aged case. It is worthwhile to mention that the thermal ageing at 823 K for 2000 h improved the creep properties. However, ageing at 973 K for 100 h caused a significant increase in the minimum creep rate and decrease in rupture time at test temperature of 823 K for the both steels.

The minimum creep rate of CLAM steel was smaller and rupture time was longer than those of JLF-1 in no-ageing condition. On the other hand, the differences between the aged and un-aged samples of CLAM were larger than those of JLF-1 especially for the rupture time, suggesting larger thermal ageing effect on CLAM than on JLF-1.

4. Discussion

Ageing resistance (change of structure and/or mechanical properties during long-term load at higher temperature) of RAFM steels is a very important criterion for application in fusion reactors.

In the present study, the thermal ageing at 823 K for 2000 h did not degrade the tensile properties and even improved the creep properties for both steels. This is different from the results for Eurofer'97 steel [11], which showed that creep rupture tests was not influenced by ageing at 823 K for 20000 h. The creep resistance of RAFM steels is considered to be affected by microstructural changes and microstructure stability. From the viewpoint of precipitation strengthening, suitable dispersion of the fine particles within the grains of the matrix is required, which can act as barriers to the movement of dislocations and the migrations of lath boundaries. Two kinds of precipitates with different morphologies, namely $M_{23}C_6$ and MX (Ta and V rich), have been detected on the normalized and tempered condition as well as in the aged conditions in most RAFM steels [12]. Ennis [13] reported that in P92 steel, during the first 3000 h of exposure at 873–923 K, an increase in size of $M_{23}C_6$ precipitates occurred. Hattestrand and Andren [14] also studied an isothermally aged P92 and found that coarsening of $M_{23}C_6$ took place, and MX (mainly VN) precipitates appeared to be relatively stable. While Sklenicka et al. [15] suggested that change in the dislocation substructure was much more important than indirect effects caused by particle evolution for modified 9–12% Cr steels (grades P91 and P92). In the cases of JLF-1 and CLAM steels before ageing, the microstructure contained $M_{23}C_6$ and fine MX particles as reported in a previous paper [6]. Therefore a possible strengthening mechanism could be the formation of fine MX precipitates during the ageing. However, the detailed examination of microstructure after ageing is necessary to identify the actual mechanism responsible for the increased creep resistance.

The recovery of martensite lath structures with high dislocation density can be accelerated by overageing [16]. As shown in the hardness, tensile and creep data, a significant softening occurred after ageing at 973 K for 100 h. It seems that the recovery of martensite lath and coarsening of precipitates are the reason for deterioration of mechanical properties. The investigations for microstructural evolution are also needed.

The hardness, tensile and creep strength of CLAM steel were higher than those of JLF-1 in un-aged case. However, the differences in these properties of CLAM between aged and un-aged conditions were also larger than those of JLF-1. The different heat treatments are considered to be the reason. As demonstrated in the previous work [6], the lower normalization and tempering temperature and higher content of Ta for CLAM steel led to finer prior austenite grains and smaller width of martensites laths than those of JLF-1, which were considered to be responsible for larger tensile strength. The higher strength of CLAM steel is thought in this paper to be the reason for the lower minimum creep rate and longer rupture time than those of JLF-1. However, the present study suggested that, because of lower heat treatment temperature, CLAM steel is also more susceptible to thermal ageing than JLF-1. Thermodynamic calculations for longer term ageing such as in-service condition need to be done to confirm the suggestion.

5. Conclusion

The effect of thermal ageing treatment on mechanical properties of JLF-1 and CLAM steels was investigated. The following conclusions were obtained.

By ageing at 823 K for 2000 h, hardness at RT increased, tensile properties did not change significantly, and creep properties improved for both steels relative to no-aged case. These results imply that the strengthening occurred by the ageing. By overageing at 973 K for 100 h, hardness at RT and tensile strength decreased and creep properties degraded.

CLAM steel had lower normalization and tempering temperature and higher Ta content, which was considered to result in larger tensile strength, lower minimum creep rate and longer rupture time than those of JLF-1. On the other hand, the present results, although only relatively short-term ageing effect was investigated, suggest that CLAM steel could be more susceptible to thermal ageing than JLF-1 because of its lower heat treatment temperature.

Acknowledgements

This work was carried out based on NIFS Budget Code NIFS07UCFF003 and NIFS07GCFF003. This work was also supported by JSPS-CAS Core University Program.

References

[1] T. Muroga, M. Gasparotto, S.J. Zinkle, Fus. Eng. Des. 61&62 (2002) 13.
[2] A. Kohyma, Y. Kohno, M. Kuroda, A. Kimura, F. Wan, J. Nucl. Mater. 258–263 (1998) 1319.
[3] Q. Huang, C. Li, Y. Li, M. Chen, M. Zhang, L. Peng, et al., J. Nucl. Mater. 367–370 (2007) 142.
[4] Y. Wu, J. Nucl. Mater. 367–370 (2007) 1410.
[5] Y. de Carlan, A. Alamo, M.H. Mathon, G. Geoffroy, A. Castaing, J. Nucl. Mater. 283–287 (2000) 672.
[6] Y. Li, Q. Huang, Y. Wu, T. Nagasaka, T. Muroga, J. Nucl. Mater. 367–370 (2007) 117.
[7] B. van der Schaaf, F. Tavassoli, C. Fazio, E. Rigal, E. Diegele, R. Lindau, et al., Fus. Eng. Des. 69 (2003) 197.
[8] S. Jitsukawa, M. Tamura, B. van der Schaaf, R.L. Klueh, A. Alamo, C. Petersen, et al., J. Nucl. Mater. 307–311 (2002) 179.
[9] J. Lapeña, M. Garcia-Mazario, P. Fernández, A.M. Lancha, J. Nucl. Mater. 283–287 (2000) 662.
[10] R. Lindau, A. Möslang, M. Schirra, Fus. Eng. Des. 61-62 (2002) 659.
[11] M. Rieth, M. Schirra, A. Falkenstein, P. Graf, S. Heger, H. Kempe, et al., Eurofer 97 – Tensile, Charpy, Creep and Structural Tests, FZKA 6911, 2003.
[12] P. Fernández, A.M. Lancha, J. Lapeña, R. Lindau, M. Rieth, M. Schirra, Fus. Eng. Des. 75–79 (2005) 1003.
[13] J.P. Ennis, in: W.T. Bakker, J.D. Parker (Eds.), Proceedings of the 3rd Conference on Advances in Materials Technology for Fossil Power Plants, The Institute of Materials, London (UK), 2001, p. 187.
[14] M. Hattestrand, H.-O. Andren, Micron 22 (2001) 789.
[15] V. Sklenicka, K. Kucharová, M. Svoboda, L. Kloc, J. Bursík, A. Kroupa, Materials Characterization 51 (2003) 35.
[16] H. Sakasegawa, T. Hirose, A. Kohyama, Y. Katoh, T. Harada, K. Asakura, Fus. Eng. Des. 61&62 (2002) 671.