Microstructure and Mechanical Properties of JLF-1 and CLAM Steels Exposed to Thermal Aging with Stress*)

Yanfen LI[1,2], Takuya NAGASAKA[1] and Takeo MUROGA[1]

[1] *National Institute for Fusion Science, 322-6 Oroshi-cho, Toki 509-5292, Japan*
[2] *Institute of Plasma Physics, Chinese Academy Sciences, Hefei 230031, China*

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In this work, effects of low levels of stress during aging at 823 and 973 K for 100 h on mechanical properties and microstructures were investigated for JLF-1 and CLAM steels. The results showed that hardness decreased and tensile strength increased after aging at 823 K for 100 h with stresses from 9 to 100 MPa for the both steels. The improvement of tensile properties was due to increase in number density of precipitates, especially with small size. On the contrary, hardness, tensile and creep strength decreased after aging at 973 K for 100 h, suggesting softening. The degradation of these properties was accelerated by applying stresses of 30 and 50 MPa during the aging. Decrease in number density of precipitates, partial recovery of martensitic structures and coarsening of lath width, were responsible for the degradation of mechanical properties at 973 K for 100 h and under applied stress.

Keywords: fusion blanket, reduced activation ferritic/martensitic steel, thermal aging, low levels of stress, mechanical property, microstructural evolution

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1. Introduction

Reduced activation ferritic/martensitic steels (RAFMs) are considered as the primary candidates for structural materials of fusion reactor blankets, because of superior irradiation resistances, low coefficient of thermal expansion, and their advanced technological base [1]. In the past two decades, several advanced RAFMs, including ORNL 9CrWVTa, F82 H, JLF-1, EUROFER 97, and the recent CLAM, have being developed [2, 3].

In the fusion application, these RAFM steels are exposed to high temperatures during long-term service, which result in recovery of microstructure and changes in mechanical property. This is called thermal aging [4, 5]. Studies on microstructure and mechanical property changes for JLF-1 and CLAM steels during long-term aging were performed in previous work [6-8]. The low levels of stress during aging, ≤ 100 MPa, which is the typical stress limit for structural materials in blanket design [1], may accelerate the recovery of microstructures. Although such information is important for evaluating thermal aging effects during the operation, data are quite limited. In this work, thermal aging tests with applied stresses were carried out followed by mechanical property tests and microstructural observations. The microstructure and mechanical properties are correlated.

author's e-mail: li.yanfen@nifs.ac.jp

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2. Experimental Procedure

The materials used were JLF-1 (JOYO-II HEAT) and CLAM (0603 HEAT). The chemical compositions and heat treatments are listed in Tables 1 and 2, respectively.

The SSJ specimens with gauge dimensions of 5 × 1.2 × 0.25 mm^3 were used for tests. The ground and mechanically polished specimen was mounted in a creep machine with a loading stress and held at 823 and 973 K for 100 h in a vacuum < 10^{-4} Pa. A non-contacting Zr foil surrounded the specimen to getter environmental impurities. A minimal tensile load (∼ 9 MPa) is necessary to keep the spec-

Table 1 Chemical compositions of JLF-1 (JOYO-II HEAT) and CLAM (0603 HEAT) (in weight %).

	JLF-1	CLAM
Cr	9.00	8.94
W	1.98	1.45
C	0.09	0.13
Mn	0.49	0.44
V	0.20	0.19
Ta	0.083	0.15
O	0.0019	0.0017
Fe	Bal.	Bal.

Table 2 Heat treatments for JLF-1 (JOYO-II HEAT) and CLAM (0603 HEAT) (AC: air cooling).

	Normalizing	Tempering
JLF-1	1323 K/60 min/AC	1053 K/60 min/AC
CLAM	1253 K/30 min/AC	1033 K/90 min/AC

imen position stable in the creep machine. Thus, aging with stress of 9 MPa is called an 'aged specimen' in this study. Those with larger stresses are called 'stress-aged specimen'. Aging temperature of 823 K was used as the typical upper operation temperature limit. According to Larson-Miller parameter, $P = T(\log t + 18)$ [9], the parameter value of aging at 973 K for 100 h is equal to that at 823 K for 440,000 h (~ 50 years). Thus aging at 973 K for 100 h was used to accelerate significantly thermal activated processes and can cover the most severe aging condition.

After the aging, some specimens were removed for hardness measurements, tensile tests, and microstructural analyses. Some specimens remained in the creep machine for subsequent creep rupture tests. The detailed test conditions were already explained in previous work [6-8].

Aged disk-shape specimens and stress-aged specimens punched from gauge center of SSJ were used for microstructural examination by transmission electron microscope (TEM) using JEM-2000FX in Kyushu University. In addition, extraction residue tests were carried out for the aged specimens with a size of 10 mm × 20 mm × 1 mm. The description in detail is in Ref. [10].

3. Results and Discussion

3.1 Hardness measurement

The Vickers hardness was measured at room temperature (RT). Figures 1 and 2 show the dependences of hardness on stress during aging at 823 and 973 K for 100 h, respectively.

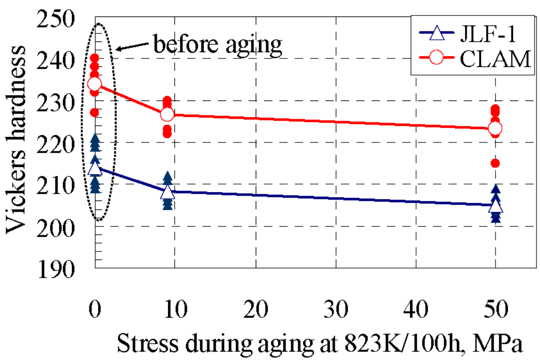

Fig. 1 Effect of stress during aging at 823 K for 100 h on hardness for JLF-1 and CLAM steels (RT).

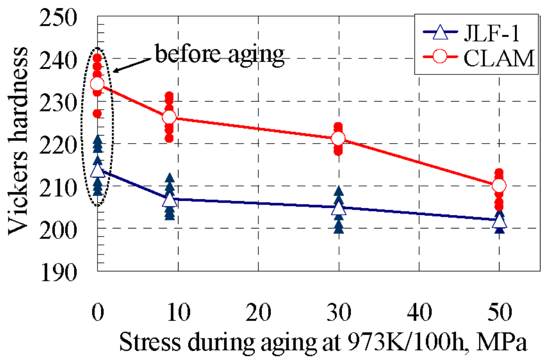

Fig. 2 Effect of stress during aging at 973 K for 100 h on hardness for JLF-1 and CLAM steels (RT).

Hardness decreased rapidly for JLF-1 and CLAM after aging at 823 and 973 K for 100 h, suggesting aging softening.

Hardness decreased further with increasing stress from 9 to 50 MPa, suggesting stress-assisted softening. The decrease was slightly larger after aging at 973 K than at 823 K. In addition, the stress-assisted decrease in hardness was larger for CLAM than for JLF-1.

3.2 Tensile properties

Figures 3 and 4 show the tensile properties after aging at 823 and 973 K for 100 h with stresses, respectively.

The results showed that, ultimate tensile strength (UTS) and yield stress (YS) increased without decrease in total elongation (TE) after aging at 823 K for 100 h, as shown in Fig. 3, suggesting hardening. This is not in agreement with the hardness data. The tensile strength did not increase further by the applied stress during the aging.

However, after aging at 973 K for 100 h, UTS and YS decreased, as shown in Fig. 4 (a), indicating softening. With increasing stress from 9 to 50 MPa, UTS and YS decreased further. On the contrary, TE increased after aging and stress-aging, as shown in Fig. 4 (b).

3.3 Creep properties

Uni-axial creep rupture tests were carried out at 823 K with 250 MPa for the aged and stress-aged specimens at 973 K for 100 h. The yield stresses at 823 K were 363 MPa for CLAM and 311 MPa for JLF-1, respectively [11],

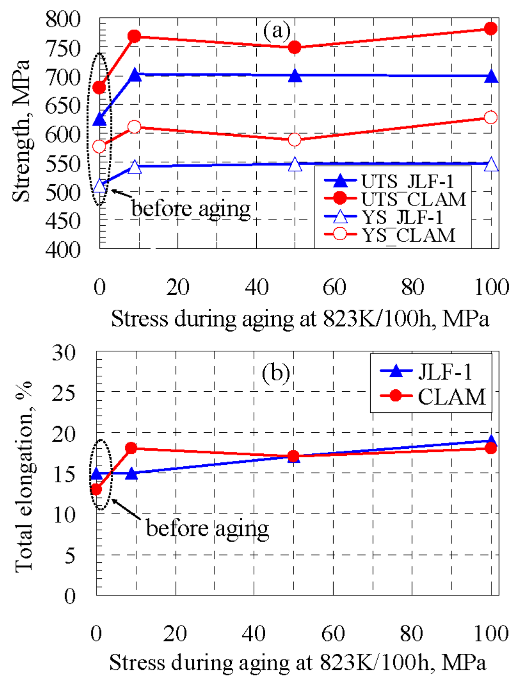

Fig. 3 Effect of stress during aging at 823 K for 100 h on tensile properties (RT): (a) UTS and YS, (b) TE.

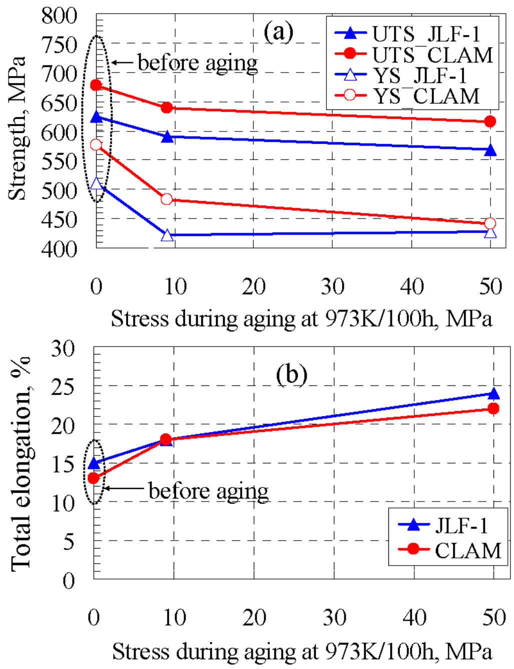

Fig. 4 Effect of stress during aging at 973 K for 100 h on tensile properties (RT): (a) UTS and YS, (b) TE.

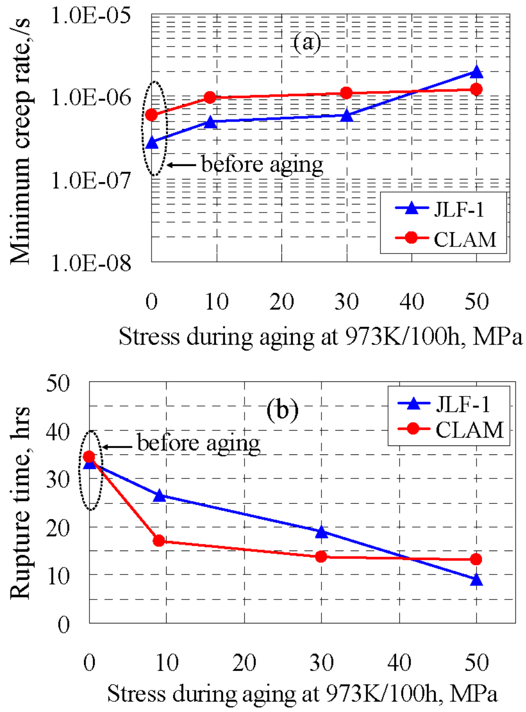

Fig. 5 Effect of stress during aging at 973 K for 100 h on creep properties (tested at 823 K/250 MPa): (a) minimum creep rate, and (b) rupture time.

which are higher than the creep stress of 250 MPa. Figure 5 shows the creep properties. Prior to the creep tests, deformation during the aging with highest applying stress of 100 MPa was < ~ 0.3%. The previous work on data reproducibility showed that scatter in the rupture time and minimum creep rate was < ~ 20% [11].

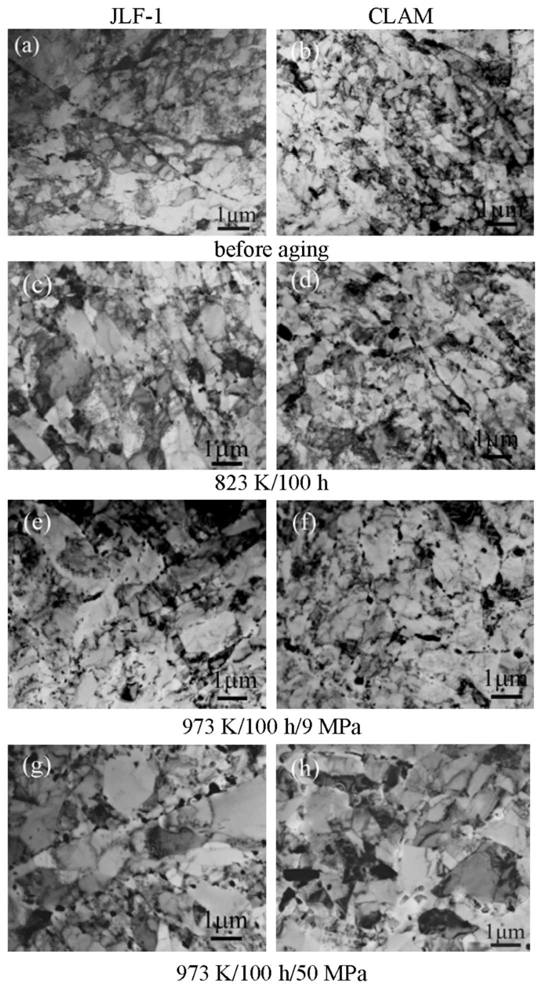

Fig. 6 TEM micrographs for JLF-1 and CLAM steels.

Aging at 973 K for 100 h degraded creep properties by increase in minimum creep rate and decrease in rupture time. In addition, with increasing stress from 9 to 50 MPa, the creep properties degraded further for JLF-1 and CLAM, suggesting stress-assisted softening.

3.4 Microstructural evolution

The microstructures were examined for JLF-1 and CLAM steels. Figure 6 shows microstructural evolutions before and after aging by TEM. Figure 7 shows density and size distribution of precipitates, which were derived from TEM images in high magnification.

The microstructure of these two steels consisted of a mixture of lath- and tempered-martensitic phases decorated with precipitates and dislocations before and after aging. Previous study by EDX showed that there were two types of precipitates before and after aging at 823-923 K for 2000 h, fine TaC with a size $\leq$ 40 nm and larger $M_{23}C_6$ as the most common precipitates [7]. Thus the type of precipitates after aging at 823 K for 100 h and with the stresses, carried out in this study, is expected to be similar.

After aging at 823 K for 100 h, the microstructure is similar to that before aging, as shown in Figs. 6 (c) and (d).

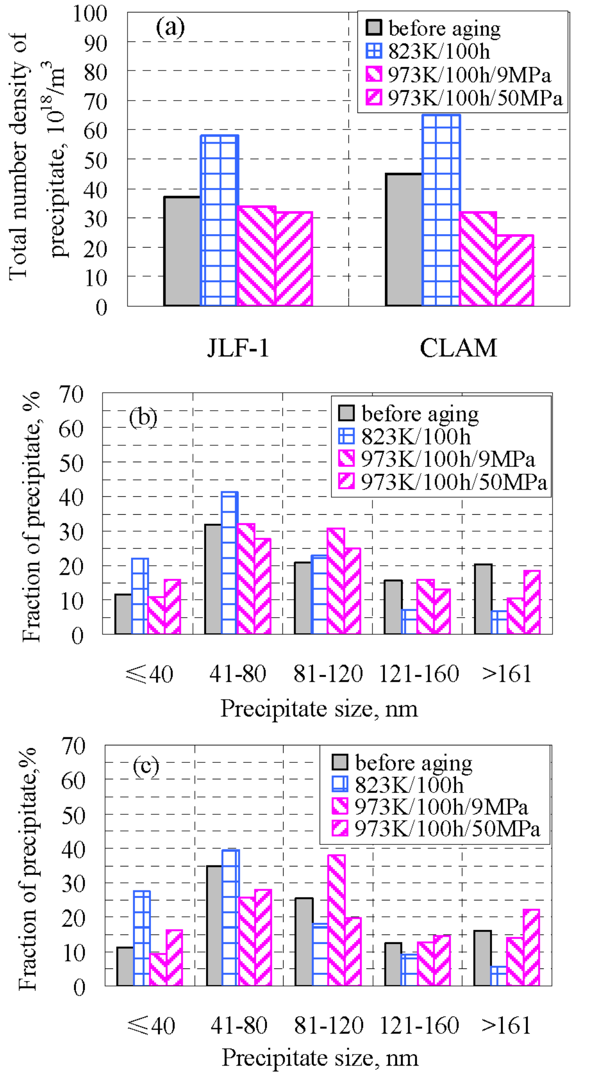

Fig. 7 Total number density (a) and size distribution of precipitates for JLF-1 (b) and CLAM (c).

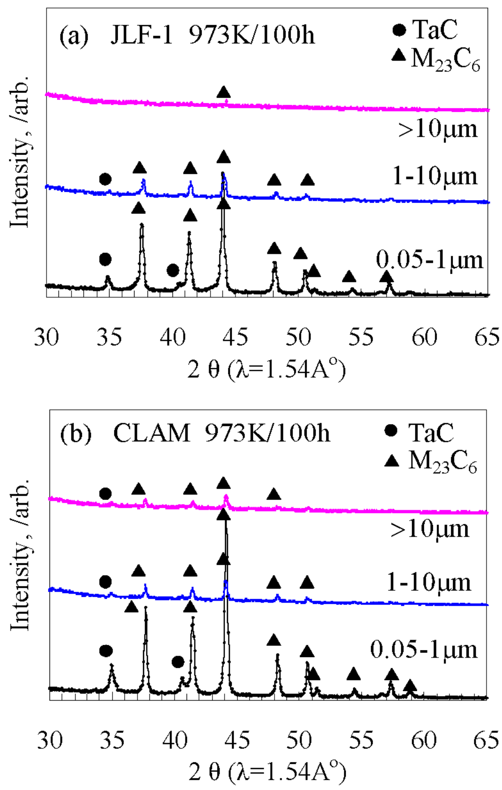

Fig. 8 XRD spectra for the extraction residues after aging at 973 K for 100 h: (a) JLF-1, and (b) CLAM.

However, total number density of precipitates increased significantly for the both steels, as shown in Fig. 7 (a). The increase in precipitates density was mostly because of the increase in density of precipitates with size < 80 nm (Figs. 7 (b) and (c)). The fine particles (mostly TaC) are difficult to grow and can slow the recovery of dislocation and lath structures, which is a benefit for long-term aging or creep resistance [4]. Thus, the increase in fine precipitates is responsible for the improvement of tensile properties after aging at 823 K for 100 h and with stress.

After aging at 973 K for 100 h, partial recovery of martensitic laths and dislocation structures occurred, as shown in Figs. 6 (e) and (f). The recovery was enhanced by applying stress of 50 MPa (Figs. 6 (g) and (h)). In addition, total density of precipitates decreased slightly for JLF-1, and decreased significantly for CLAM after aging and with stress, as shown in Fig. 7 (a). The decrease in precipitates density leads to the decrease in hardness, tensile and creep properties.

The size distributions of precipitates after aging at 973 K for 100 h and with stress are also shown in Figs. 7 (b) and (c). For the aged specimens, 973 K for 100 h with 9 MPa, the fraction of precipitates with size of 81-120 was increased for the both steels. While the stress-aged specimens, 973 K for 100 h with 50 MPa, showed a slight increase in fraction of precipitates with size ≤ 40 nm for the both steels. On the other hand, the precipitates with sizes of 41-80 nm decreased and those of 81-120 nm increased for JLF-1. While precipitates with sizes of 41-120 nm decreased and those > 160 nm increased for CLAM. This suggests the possible growth of $M_{23}C_6$. The $M_{23}C_6$ can grow up easily relative to TaC during long-term aging and creep, deteriorating the mechanical properties [12]. Coarsening of precipitates was enhanced by stress in this study. However, further characterization is necessary to define the effects of stress on these precipitates evolution.

After aging at 973 K for 100 h, other type of precipitates such as Laves and M_6C phases seem not to appear according to time-temperature-precipitation (TTP) diagram of F82H steel [13]. Additional aging of the specimens was carried out with larger size without stress for the residue tests because SSJ specimens did not satisfy the requirement of the residue test. As shown by the XRD spectra from the extraction residue in Fig. 8, no brittle Laves phase but TaC and $M_{23}C_6$ precipitates were detected after aging at 973 K for 100 h.

The martensitic lath width was measured and the results are shown in Table 3. After aging at 823 K for 100 h

Table 3 Average width of martensitic laths for JLF-1 and CLAM steels (unit: nm)

steels	conditions of specimens		
	Before aging	973K/100 h /9MPa	973K/100 h /50MPa
JLF-1	500 [11]	650	700
CLAM	440 [11]	550	590

with stress, the lath width showed almost no change. However, the lath width was significantly wider after aging at 973 K for 100 h. Moreover, the applied stress of 50 MPa accelerated the coarsening of martensitic laths.

Thus, the decrease in precipitates density and increase in martensitic lath width, as well as the recovery of dislocation structures, contributed to the degradation of mechanical properties at 973 K for 100 h and with stress.

CLAM shows a higher hardness and tensile strength than JLF-1. This is because of finer prior austenitic grain size for CLAM (5-10 mm) than for JLF-1 (8-15 mm), and smaller lath width for CLAM (440 nm) than for JLF-1 (500 nm) before aging [11]. However, softening of CLAM at 973 K was also larger, suggesting a greater effect of aging and stress-aging than in JLF-1. This result was similar to that observed in the previous work, which showed that CLAM was more susceptible to thermal aging than JLF-1 [6]. The higher susceptibility of CLAM is probably due to lower normalizing and tempering temperature of CLAM than that of JLF-1 [14].

4. Conclusion

Aging experiments at 823-973 K for 100 h under stresses of 9 to 100 MPa were conducted for JLF-1 and CLAM steels followed by mechanical property tests and microstructural observations. The main results are summarized as follows.

(1) Hardness decreased after aging at 823 and 973 K for 100 h and decreased further with increasing stress from 9 to 50 MPa.
(2) Tensile properties were improved after aging at 823 K for 100 h, suggesting hardening by aging and stress-aging. While tensile strength decreased significantly after aging at 973 K for 100 h, suggesting softening. The effect of applying stress during aging on tensile strength was not significant.
(3) Creep properties were degraded after aging at 973 K for 100 h and degraded further by applying stress.
(4) The microstructure was similar to that before aging, while total number density of precipitates, especially with small size, increased significantly after aging at 823 K for 100 h. This is responsible for the improvement of tensile properties after aging.
(5) Total density of precipitates decreased slightly for JLF-1, but decreased significantly for CLAM after aging at 973 K for 100 h. The martensitic laths were coarsened, accompanied by partial recovery of lath and dislocation structures. All these changes were accelerated by applying stress during the aging. These are the reasons for the degradation of mechanical properties.

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