

# Long-term thermal stability of reduced activation ferritic/martensitic steels as structure materials of fusion blanket

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In this work, the effect of thermal ageing on mechanical properties of JLF-1 (JOYO-II HEAT) and CLAM (HEAT 0603) steels have been studied at temperatures in the range of 823 - 973 K. The results showed that the hardness increased slightly and the creep properties improved after ageing at 823 K for 2000 h for the both steels. On the other hand, the hardness decreased after ageing above 823 K, especially at 973 K for 100 h, and the creep property degraded at 973 K for 100 h. The Larson-Miller parameter was shown to be appropriate for predicting the long-term creep properties from the short-term experiments at higher temperature with higher stress. By extrapolation to the typical design limit for the blanket, 823 K for 100 000h, the rupture stress was estimated to be about 135 MPa for the both steels. The present thermal aging treatments influenced the rupture stress by ~15 MPa.

Keywords: fusion blanket, reduced activation ferritic / martensitic steel, thermal stability, thermal ageing, creep properties, Larson-Millar parameter

## 1. Introduction

Blanket is one of the important components of fusion reactors, which provides the primary heat transfer and tritium breeding systems. Currently, reduced activation ferritic / martensitic (RAFM) steels are considered as the primary candidates for blanket structural materials because of their most matured industrial infrastructure and relatively good radiation resistance [1].

In fusion applications, they need to withstand high temperature under long-term loading. When the absolute service temperature was higher than about 698 K, the creep deformation of these steels will occur [2]. Since the maximum operating temperature of blanket structural materials will be determined by the thermal creep deformation, evaluation of the thermal creep performance in the blanket condition is the key necessity [3]. In addition, the thermal ageing during the operation may affect the creep properties [4]. However, the research to understanding the thermal ageing effects on the creep deformation is quite limited.

Since testing materials for the actual operating time is extremely costly and time-consuming, prediction of creep rupture performance based on the results of short-term creep experiments at higher temperature with higher stresses has been explored using stress-time parameters. Many efforts to estimate the long-term creep properties have been done for the steels which are being used in fission power plant [5,6], but limited data are available for RAFM steels [7]. No effort has been made to include the thermal aging effect on the prediction of the long-term

creep performance of RAFM steels.

In this work, thermal ageing at temperatures in the range of 823 - 973 K on JLF-1 and CLAM steels was carried out and the creep properties were tested. As the aging temperature, 823 K was chosen to test at the upper temperature limit in fusion blanket, and 973 K to accelerate the aging effects. The Larson-Miller parameter and Monkman-Grant equation was proposed to describe the long-term behavior necessary for the blanket design.

## 2. Experimental Procedure

The materials used are JLF-1 (JOYO-II-HEAT) and CLAM (HEAT 0603) steels. The chemical compositions of these two steels (in weight) are 9.00% Cr, 1.98% W, 0.49% Mn, 0.20% V, 0.083% Ta, 0.09% C, and balance Fe for JLF-1 and 8.94% Cr, 1.45% W, 0.44% Mn, 0.19% V, 0.15% Ta, 0.13% C, and balance Fe for CLAM. The heat treatments included quench and tempering. The quench treatments were carried out by heating at 1323 K for 60 minutes for JLF-1 and 1253 K for 30 minutes for CLAM and then cooled by air. The tempering treatments were carried out by heating at 1053 K for 60 minutes for JLF-1 and 1033 K for 90 minutes for CLAM and then cooled by air.

The SSJ specimens with a gauge size of 5 x 1.2 x 0.25 mm<sup>3</sup> were machined along the roll direction. Then, these specimens underwent ageing experiments in the temperature range of 823 to 973 K under high vacuum in order to avoid high oxidation of the material.

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The Vickers hardness was measured by using a Vickers Hardness Testing Machine under a load of 300 g with loading time of 30 s at room temperature.

The uniaxial creep tests up to rupture were performed at 823 to 923 K with the applied stress between 150 and 300 MPa in a vacuum of  $< 1 \times 10^{-4}$  Pa. The loading was carried out by a simple suspension, which had a high stability. Creep strain was measured by double linear variable differential transformers (LVDTs) with increased precision.

### 3. Results

#### 3.1 Hardness Measurement

The hardness results are plotted in Fig. 1. It 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 the both steels. On the contrary, softening occurred above 823 K. The softening of JLF-1 steel was smaller than that of CLAM.

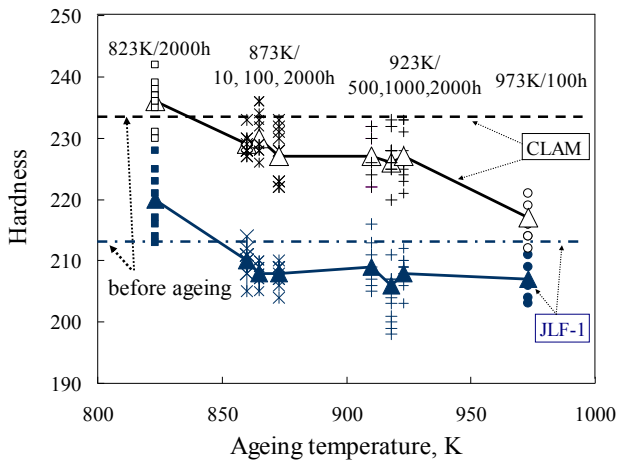


Fig. 1 Hardness change of JLF-1 and CLAM steels by thermal ageing.

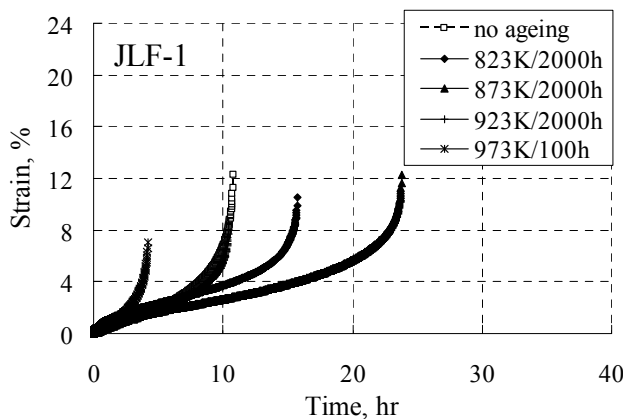


Fig. 2 Creep curves of JLF-1 steel at different ageing conditions (tested at 823 K with 250 MPa).

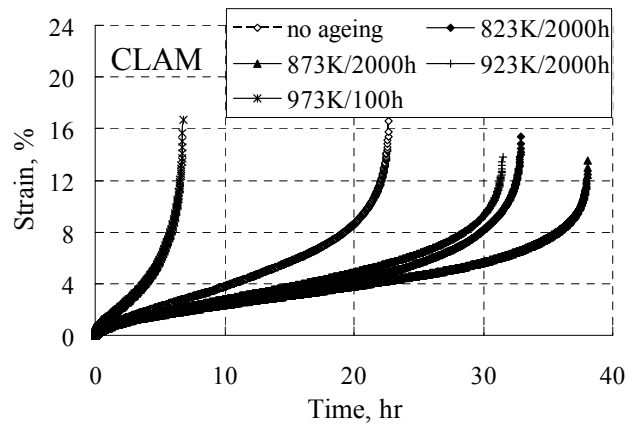


Fig. 3 Creep curves of CLAM steel at different ageing conditions (tested at 823 K with 250 MPa).

#### 3.2 Creep Properties

The uniaxial constant load creep tests were conducted at 823 to 923 K with the applied stress between 150 and 300 MPa. Figs. 2 and 3 show the strain dependence of the creep curve for different ageing conditions. The typical creep curves of the present steels, similar to that observed in other RAFMs, were composed of the primary or transient region, where the creep rate decreases with time, steady state region which is a linear process, and the tertiary or accelerated creep region characterized by an increased creep rate after reaching a minimum creep rate until the material rupture.

For CLAM steel, after ageing at 823 to 873 K up to 2000 h, the minimum creep rate decreased and the rupture time increased. Similar to CLAM, the creep rupture time increased by ageing at 823 and 873 K for 2000 h for JLF-1. But further ageing at 923 K for 2000 h returned the properties to almost the level of no aging. On the other hand, ageing at 973 K for 100 h caused a significant degradation in creep properties, which is consistent with the results of hardness measurements. Although the minimum creep rate is smaller and rupture time is longer for CLAM than those of JLF-1, CLAM is more susceptible to thermal ageing than that of JLF-1.

### 4. Discussion

#### 4.1 Larson-Miller Parameter

Prediction of long-term creep rupture performance based on the results of short-term creep experiments at higher temperature with higher stresses has been carried out based on stress-time parameters. Larson-Miller parameter is one of the popular methods, which is based on the model of the rate processes [8]:

$$r_c = A \times \exp(-Q/RT) \tag{1}$$

where

$r_c$  = minimum creep rate

- A = constant
- exp = natural logarithm base
- Q = activation energy for process
- R = gas constant
- T = absolute temperature

Assuming the times of primary and tertiary creep are much shorter than that of the secondary creep and the tertiary creep begins when total strain reached a critical value ( $\epsilon_r$ ), the creep curve is simplified as schematically presented in Fig. 4. In this case, Equation (1) can be written as

$$1/t_r = B \times \exp(-Q/RT) \quad (2)$$

where  $t_r$  is the time to rupture and B is the constant.

Taking the logarithm for Equation (2), the Larson-Millar equation (LMP: Larson-Miller Parameter) can be derived:

$$T(C + \log t_r) = Q/2.3R = LMP \times 1000 \quad (3)$$

where  $C = \log B$

The Larson-Miller equation assumes that the activation energy Q, hence LMP, is independent from T and  $t_r$  but only the function of the applied stress  $\sigma$ . Assuming  $LMP = P_0 - \alpha\sigma$ , applied stress  $\sigma$  and  $LMP (= T(C + \log t_r) \times 0.001)$  should show linear relation (Larson-Miller diagram).

Larson-Millar parameter assumes that temperature and time can be interchanged, provided no important microstructural changes occurred during the test. When the creep mechanism changes, the use of this parameter for predicting long time performance is not accurate. It was also suggested to use the Monkman-Grant equation [9] together with the Norton law [10] for predicting creep properties of 9%Cr steels:

$$\log r_c = -1/n \times \log t_r + D \quad (4)$$

where n and D are the constants.

Figs. 5 and 6 present the minimum creep rate ( $r_c$ ) as a function of rupture time ( $t_r$ ) for JLF-1 and CLAM steels. From the figures, it can be seen that the constant n in eq. (4) is almost equal to 1. This means that the simplification shown in Fig. 4 and thus the Larson-Miller parameter are appropriate for the present prediction.

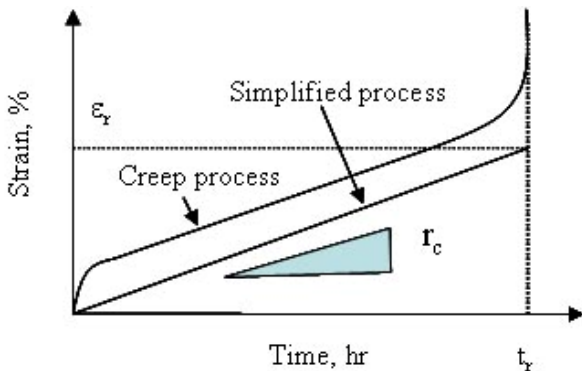


Fig. 4 Schematic illustration the creep curve and simplified process.

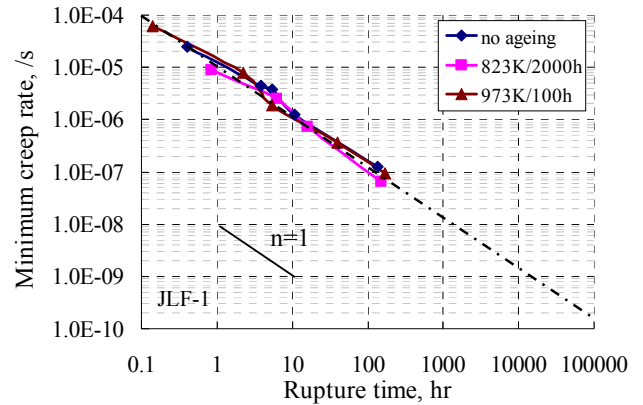


Fig. 5 Minimum creep rate as a function of rupture time (Monkman – Grant equation) for JLF-1 steel.

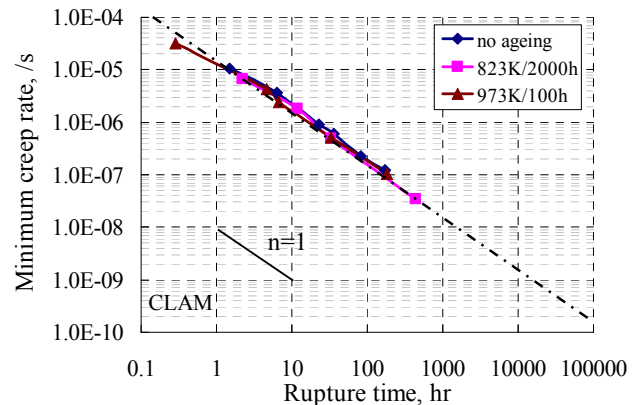


Fig. 6 Minimum creep rate as a function of rupture time (Monkman – Grant equation) for CLAM steel.

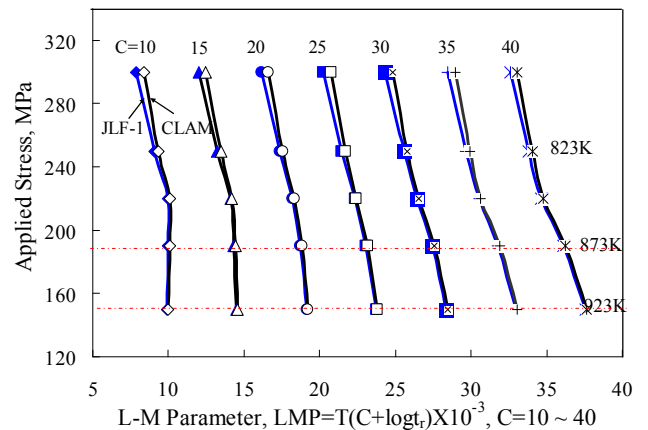


Fig. 7 Fitting of the Larson-Millar parameter with applied stress for various values of C for JLF-1 and CLAM steels before ageing

The C in Larson-Miller equation is a material constant. By the fitting of the present experimental data, the C = 30 is considered to be suitable for JLF-1 and CLAM steels, as shown in Fig. 7.

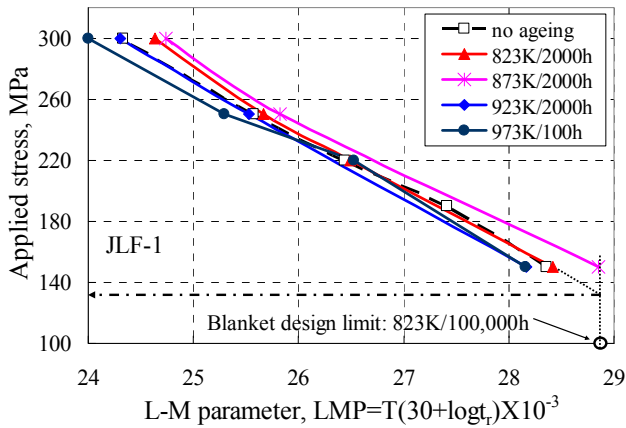


Fig. 8 Applied stress as a function of Larson-Miller Parameter for JLF-1 steel.

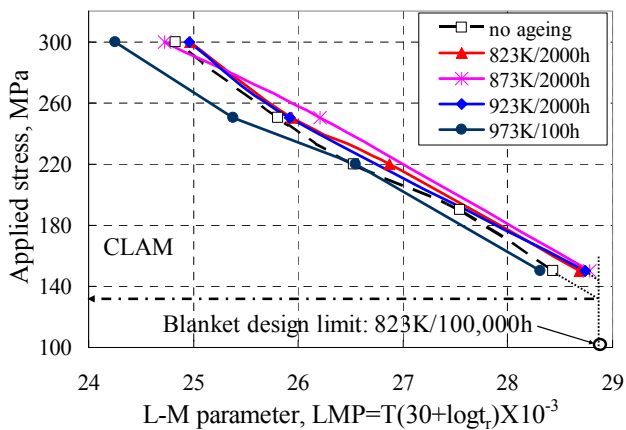


Fig. 9 Applied stress as a function of Larson-Miller Parameter for CLAM steel.

Figs. 8 and 9 present a Larson-Miller diagram with different ageing conditions. The diagrams show the increase of LMP by ageing at 823 and 873 K and the decrease by ageing at 973 K. In addition, the rupture stress was estimated to be about 135 MPa by predicting the typical design limit for blanket, 823 K for 100 000h.

Figs. 8 and 9 also show that the estimated rupture stress can be change by about 15 MPa by the prior thermal ageing. These uncertainty need to be considered as the possible thermal ageing effect during the thermal creep processes.

Based on ASTM VIII guideline, acceptable stress limit of  $2/3 \times 135 = 90 \text{ MPa}$  is derived for the both steels.

**4.2 Thermal Activation Analysis**

The present experiments and analysis showed that hardening and increase in the creep activation energy took place by aging at 823 K, and softening and the decrease in the creep activation energy occurred by aging at 973 K. In

this section correlation of the hardness data is attempted based on the thermal activation process.

In this analysis ageing is assumed to be induced by migration of the constituent species (most probably C) with activation energy of  $E_m$ . The total number of jumps of the species during the aging is given by:

$$\nu = \nu_0 \times \exp(-E_m / kT) \times t_a \tag{4}$$

where

- $\nu_0$  = jump frequency,  $10^{13}/\text{s}$
- $E_m$  = migration energy, eV
- $k$  = Boltzsmann energy,  $0.8625 \times 10^{-4} \text{ ev/K}$
- $T$  = absolute temperature, K
- $t_a$  = aging time

Figs. 10 and 11 show the hardness change against the total number of jumps assuming the migration energy of 1.6, 2.0 and 2.4 eV for JLF-1 and CLAM, respectively. As shown in the figures, the total number of jumps is not an appropriate correlation parameter in any case of the activation energy. This means that the hardness change by aging is not thermally activated processes with a particular activation energy.

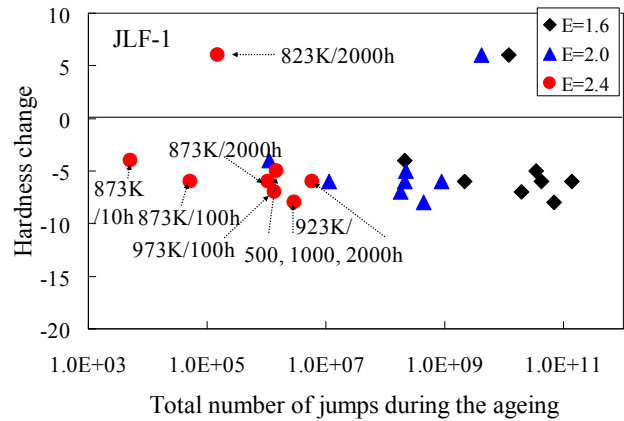


Fig. 10 Hardness change vs total number of jumps in different ageing conditions for JLF-1 steel.

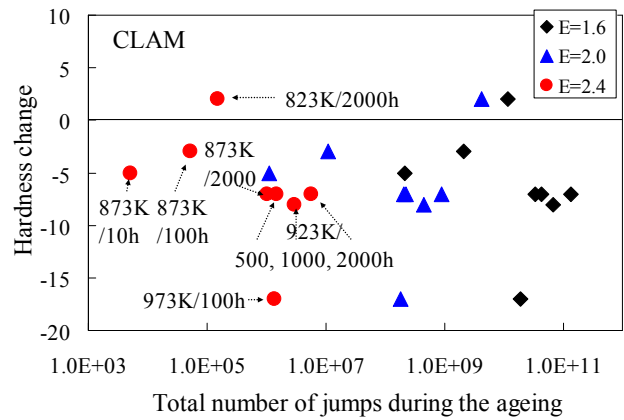


Fig. 11 Hardness change vs total number of jumps in different ageing conditions for CLAM steel.

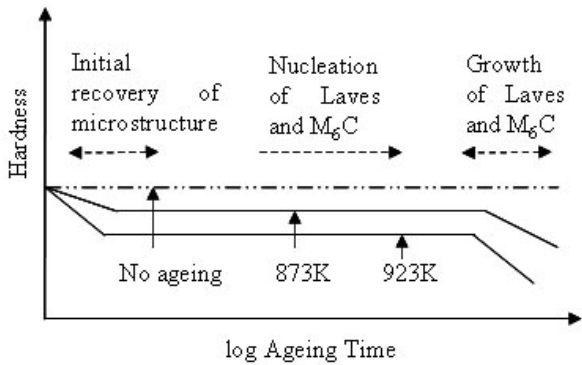


Fig. 12 The proposed dependence of hardness on ageing time at 873 to 923 K for JLF-1 and CLAM steels.

The authors' previous study showed that the hardening by aging at 823 K for 2000 h is due to the formation of fine TaC precipitates [11]. Therefore, the increase in the creep activation energy by ageing at 823 K for 2000 h was also considered to be originated from the precipitation of TaC.

However, by the ageing at 873 to 923 K, the hardness is almost independent from the aging time as shown in Figs. 10 and 11. It seems that the softening took place only in the initial ageing time as schematically shown in Fig. 12. In this case further softening needs formation of new phases such as Laves and  $M_6C$ . This is consistent with the observed precipitation in F82H by Tanigawa et al [12], which showed that the present aging conditions are before the formation of those phases.

## 5. Summary

The ageing experiments were carried out for JLF-1 and CLAM in the temperature range of 823 to 973 K followed by mechanical properties tests. The conclusions of the study are listed below:

(1) The hardness increased slightly after ageing at 823 K for 2000 h for the both steels. However, ageing at  $> 823$  K caused a decrease in hardness.

(2) The minimum creep rate decreased and the rupture time increased after ageing at 823 and 873 K for 2000 h for the both steels, which suggested that the activation energy for the creep process increased. However, the creep property degraded significantly after ageing at 973 K for 100 h, indicating the decrease in the energy.

(3) The Larson-Miller parameter was used to predict long-term creep performance from the short-term experiment at higher temperature. The rupture stress of 135 MPa was predicted for the typical design limit of blanket, 823 K for 100 000h. The present thermal aging treatments influenced the rupture stress by  $\sim 15$  MPa.

(4) From the activation analysis, it was suggested that the present ageing conditions are located after the

initial microstructural recovery and before the softening and by formation of Laves and  $M_6C$  phases.

## Acknowledgements

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