

Influence of Pre-Aging on Creep Rupture Strength of Tungsten Alloyed 9%Cr Ferritic Steel and Creep Damage Evaluation by Electrochemical Method

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The influence of pre-aging on creep rupture strength has been investigated to understand the creep damage mechanism of tungsten alloyed 9%Cr ferritic steel KA-STBA29/KA-STPA29. Additionally, we have also examined changes in strengthening factors during creep and thermal aging by a nano-indentation test. Experimental results revealed that the creep rupture strength of thermally aged materials decreased with prolonged pre-aging and that the decrease in solid-solution strengthening due to Laves phase precipitation caused the reduction of rupture strength. Based on the results, an electrochemical nondestructive procedure, which quantitatively detected the amount of Laves phase, has been proposed to evaluate creep damage accumulation in KA-STBA29/KA-STPA29. By this electrochemical method and a newly proposed parameter based on kinetics of Laves phase precipitation, a testing temperature or applied stress of creep-damaged materials can be nondestructively estimated with high accuracy.

1. INTRODUCTION

The efficiency improvement of thermal power plants can be achieved by elevating the temperature and pressure of steam, which would require advanced heat resistant materials with superior performances in such steam conditions. Recently 9-12%Cr ferritic steels have received particular attention as candidate steels for boiler headers and heat-exchanger tubes of an ultra super critical (USC) boiler. These martensitic steels have improved high temperature strength by precipitation strengthening attributed to Laves phase in addition to solid-solution strengthening by elements, such as tungsten and molybdenum and dispersion strengthening by MX type carbonitride particles, such as Nb(C,N) and VN^{(1),(2)}. However, it is not easy to quantitatively understand their strengthening factors and its change because of their complicated microstructure, and their material degradation mechanism have not as yet been thoroughly clarified.

In the present study, the influence of pre-aging on creep rupture strength was investigated to understand the creep damage mechanism of tungsten alloyed 9%Cr ferritic steel KA-STBA29/KA-STPA29. The change in strengthening factors during creep and thermal aging was also examined by a nano-indentation test. Additionally, based on the results, the change in electrochemical property of the steel caused by creep and thermal aging were investigated to establish an electrochemical nondestructive procedure for creep damage evaluation.

2. EXPERIMENTAL PROCEDURE

2.1 Materials and Creep Tests

Tungsten alloyed 9%Cr ferritic steels KA-STBA29/KA-STPA29 (T92/P92) were used in this study. The chemical compositions (weight percent) of the steels are as follows: 0.10 C; 0.04 Si; 0.46 Mn; 0.008 P; 0.001 S; 8.96 Cr; 0.47 Mo; 1.84 W; 0.2 V; 0.068 Nb; 0.001 B; 0.051 N; balance Fe (steel A) and 0.11 C; 0.10 Si; 0.45 Mn; 0.012 P; 0.003 S; 8.82 Cr; 0.47 Mo; 1.87 W; 0.19 V; 0.06 Nb; 0.002 B; 0.17 Ni; 0.047 N; 0.001Al; balance Fe (steel B). The heat treatments of the steel A and B consisted of normalizing at 1065°C for 1 h, tempering at 770°C for 1 h and post weld heat treatment at 740°C for 4 h, and normalizing at 1070°C for 2 h, tempering at 780°C for 2 h and post weld heat treatment at 745°C for 2 h, respectively. The steel B was subjected to the creep rupture test at temperature of 600°C at stress level of 170MPa after thermal aging at 650°C for duration ranging from 300 to 10000 hours to investigate the influence of

pre-aging on the creep rupture strength. Furthermore, the creep rupture and interrupted creep tests were conducted at temperatures 600–650°C at stress levels of 95–190 MPa using steel A and B, respectively, then these creep specimens were subjected to the below-mentioned electrochemical measurements.

2.2 Hardness Measurements

Vickers hardness test was carried out at room temperature under the load of 98 N to investigate the change in hardness due to creep and thermal aging. Additionally, in order to examine the variation of matrix hardness during creep and thermal aging, the gauge and grip sections of the interrupted creep specimens were subjected to a nano-indentation test, namely, ultra-microhardness test. The nano-indentation test was performed at room temperature under the load of 52.5 μ N using an atomic force microscope (AFM) and a three-sided pyramid diamond indenter. The measurement surface was electropolished in an electrolyte consisting of 95 pct acetic acid and 5 pct perchloric acid to remove work hardening layer. Next, the indentation was carried out on a relatively smooth region where precipitation was not observed.

2.3 Electrochemical Polarization Measurements

The gauge and grip sections of the creep test specimens were longitudinally cut into two halves. These samples were connected to a lead wire and then covered with epoxy resin, except for the cross sectional surfaces for electrochemical polarization measurements. The measurement surfaces were mechanically polished to a 1- μ m diamond finish. The surface area exposed to the test solution was 0.3–0.5 cm² and the remainder was electrically insulated using paraffin. The electrochemical polarization measurements were performed in a 1N-KOH at 30 $\pm$ 1°C. The potential of the specimens was scanned from the rest potential to +400 mV (vs SCE) at a sweep rate of 0.5 mV/sec to obtain anodic polarization curves.

3. RESULTS AND DISCUSSION

3.1 Influence of Pre-Aging on Creep Rupture Strength

The creep rupture tests were carried out at temperature of 600°C at stress level of 170MPa using the thermally aged materials. These test results showed that the time to rupture decreased as the duration of pre-aging increased and there was a good correlation between them. It is known that a tempered martensitic lath structure of Mod. 9%Cr steel shows no significant change by thermal aging at a temperature less than tempering

temperature⁽³⁾. Additionally, it has been previously reported by the authors⁽⁴⁾ that Laves phase was not present in the as-tempered material and its precipitation occurred markedly from the beginning of the thermal aging at 650°C. In this investigation, it was also revealed that, in contrast to Laves phase, the amount of $M_{23}C_6$ type carbide in thermally aged materials showed almost no variation throughout aging and was equivalent to that in the as-tempered material. Correlation between the above-mentioned rupture time and the amount of Laves phase, which had been measured using the electrolytic extraction method after the thermal aging, is shown in Fig. 1. As can be seen clearly in this figure, the rupture time decreases monotonously with increasing amount of Laves phase. This result suggests that the increase in amount of Laves phase is closely associated with the reduction of creep rupture strength due to pre-aging. In the next section, we focus on Laves phase and investigate the influence of Laves phase precipitation on creep rupture strength in detail using a nano-indentation test.

3.2 Material Degradation Due to Laves Phase Precipitation

In general, it is well known that Laves phase $(Fe,Cr)_2(W,Mo)$ contributes to precipitation strengthening during creep, but on the other hand it reduces tungsten and molybdenum in solid solution and decreases solid-solution strengthening. Hence, it was considered that the reduction of creep rupture strength due to pre-aging was caused by the decrease in solid-solution strengthening. In order to directly evaluate the decrease in solid-solution strengthening due to Laves phase precipitation, change in hardness of the matrix itself was examined using a nano-indentation test.

The indentations observed by AFM after the indentation tests had the shape of equilateral triangle and the length of one side were as small as $0.20\text{--}0.25\text{ }\mu\text{m}$. These length are smaller than the lath width (about $0.5\text{ }\mu\text{m}$ ⁽⁵⁾) after tempering. This result means that the indentation size reflects the hardness of the matrix itself (lath interior) where lath boundaries have no influence. That is, this hardness is equivalent to the hardness of iron (ferrite) itself strengthened with dislocation and solid-solution elements.

The indentation sizes measured on the interrupted creep specimens were larger than that measured on the as-tempered material. The projection area of indentation, which was regarded as being inversely proportional to hardness, was measured by using AFM and an image processor. The results measured on the interrupted creep specimens (650°C, 105 MPa, $t_r = 3066\text{ h}$) are given in Fig. 2, along with the results of Vickers hardness measurements, where each hardness is normalized by the hardness of the as-tempered material and plotted as a function of the creep life fraction. Hereafter, the normalized hardness of matrix measured by the nano-indentation test is defined as "nano-hardness".

As can be seen in Fig. 2, the Vickers hardness measured on the grip section shows no significant change and that on the gauge section decreases slowly during creep. In contrast to the changes in Vickers hardness, the nano-hardness measured on the grip section decreases gradually with the life fraction, while that measured on the gauge section decreases abruptly at an early stage of creep life. The decrease in nano-hardness measured on the gauge section is no longer pronounced when the life fraction exceeds about 0.3. It is also noted that the nano-hardness measured on the gauge section of the creep-ruptured specimen is only one half that of the as-tempered material.

It is known^{(3), (6)} that dislocation density in lath interior decreases dramatically until creep rate reaches minimum, and it decreases gradually with creep strain after that. From the relationship between the average creep rate obtained from the interrupted creep tests and the nano-hardness, it was revealed that the nano-hardness decreased significantly before the creep rate reached minimum. This change was consistent qualitatively with the above-mentioned change in dislocation density. Thus, the marked decrease in nano-hardness of the gauge section at an early stage of creep life seems to correspond mainly to the decrease in dislocation density in lath interior.

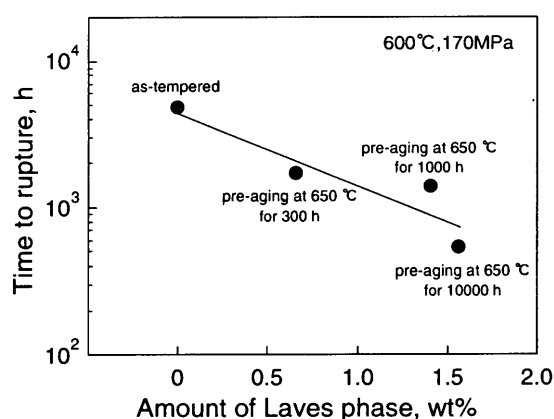


Fig. 1. Time to rupture plotted as a function of amount of Laves phase.

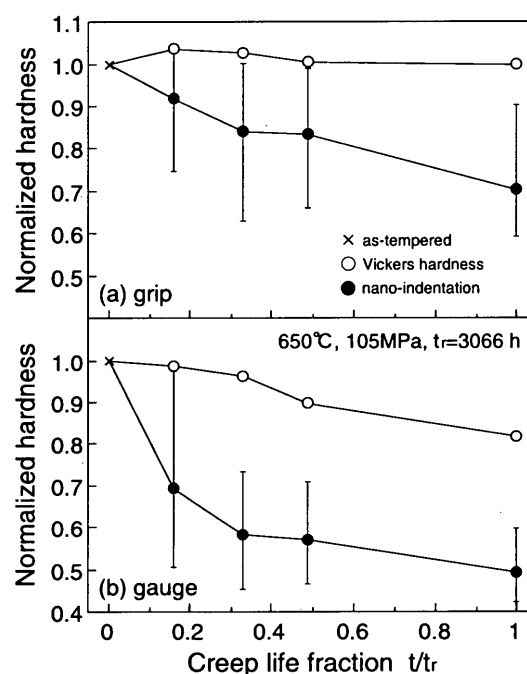


Fig. 2. Changes in normalized hardness as a function creep life fraction.

On the other hand, the decrease in nano-hardness of the grip section was considered to be caused by the decrease in solid-solution strengthening, because, as described above, dislocation substructure shows no significant change by thermal aging at a temperature less than tempering temperature. The amount of tungsten and molybdenum in solid solution was calculated by assuming that the chemical composition of Laves phase and $M_{23}C_6$ type carbide were $(Fe_{1.53}Cr_{0.45})(W_{0.72}Mo_{0.30})$ and $(Fe_{4.46}Cr_{16.12}W_{1.20}Mo_{1.22})C_6$ ⁽⁷⁾, respectively. A plot of the nano-hardness of grip section against the amount of tungsten and molybdenum in solid solution showed that the former decreased monotonously as the latter decreased. This result confirms that the decrease in nano-hardness due to thermal aging is caused by the decrease in solid-solution strengthening. Consequently, it is considered that the reduction of creep rupture strength due to pre-aging is caused primarily by the matrix softening, which is attributed to Laves phase precipitation.

In the subsequent section, we take note of Laves phase, which is a critical factor of the reduction of creep strength, and investigate changes in electrochemical property due to thermal aging and creep to develop a methodology for creep damage evaluation.

3.3 Change in Electrochemical Property Due to Creep

Figure 3 shows examples of anodic polarization curves obtained from the as-tempered material and the gauge and grip sections of creep-ruptured specimen (650°C, 95 MPa, $t_r = 12115$ h). As can be seen in this figure, the peak current density appears at around +200 mV and increases due to creep and thermal aging. Hereafter, this peak current density is defined as " I_p ".

The I_p values measured on the creep-ruptured and interrupted creep test specimens, which had undergone various degree of creep damage under a wide variety of temperature and stress conditions, are plotted as a function of the Larson-Miller type aging parameter (LMP) in Fig. 4. The I_p measured on the thermally aged material (grip section of creep-ruptured specimen) shows the tendency to increase gradually with increasing LMP, while that on the gauge section of creep-ruptured specimen rises suddenly in the region of high LMP. When the results of the creep-ruptured specimens and the thermally aged materials are compared, it is found that the former are larger than the latter even though they have the same thermal history and the I_p value reflects not only the thermal effect but also the stress or strain effect on creep damage. Similarly, the I_p values measured on the interrupted creep specimen reflects the stress effect and lie approximately between the I_p values of the creep-ruptured specimens and the thermally aged materials.

3.4 Electrochemical Detection Mechanism

To clarify the electrochemical detection mechanism, namely, the cause of the difference in electrochemical property between the creep-damaged and the as-tempered materials, potentiostatic differential etching was performed on the creep-ruptured specimen. The specimen was potentiostatically polarized at the peak potential (+200 mV) in 1N-KOH solution. The experimental result showed that Laves phase and $M_{23}C_6$ type carbide were selectively dissolved at the peak potential. This result indicates that the I_p value corresponds mainly to the selective dissolution volume of Laves phase and $M_{23}C_6$ type carbide. It also means that the increase in I_p due to thermal aging and creep reflects the increase in amount of Laves phase and/or $M_{23}C_6$ type carbide or their activated dissolution.

As described before, it has been determined that the amount of Laves phase increased significantly with prolonged thermal aging from the beginning of aging, whereas that of $M_{23}C_6$ type carbide showed almost no variation throughout aging. Furthermore, it was also revealed that the I_p values measured on the gauge and grip sections differed and the former had a tendency to become larger than the latter even if the amount of precipitates was the same. These results suggest that the increase in I_p due to creep reflect not only the increase in amount of Laves phase but also the change in electrochemical property (dissolution characteristics in 1N-KOH) of Laves phase. That is, there is a possibility that the chemical composition of Laves phase precipitated in the creep-damaged materials differs from that in the thermally aged materials.

In order to confirm the above-mentioned possibility, Laves phase was extracted from the gauge and grip sections of the creep-ruptured specimens (650°C, 95 MPa, $t_r = 12115$ h and 650°C, 115 MPa, $t_r = 3738$ h) using replica films and the chemical composition of Laves phase was determined by SEM-EDX. The obtained results are given in Fig. 5, along with the result of thermally aged material reported by Hald⁽⁷⁾. As can be seen in this figure, the chemical compositions measured on the grip sections are in relatively good agreement with that reported by Hald. Chromium concentration in Laves phase of the gauge section is approximately 25 at% and is larger than that of the grip section, which is as small as about 15 at%. According to the increase of chromium concentration, iron concentration in Laves phase of the gauge section becomes smaller compared with that of the grip section.

Total amount of chromium precipitated as Laves phase was calculated and the correlation with the I_p was investigated. This examination showed that the I_p increased with increasing amount of chromium and there was linear relationship between them

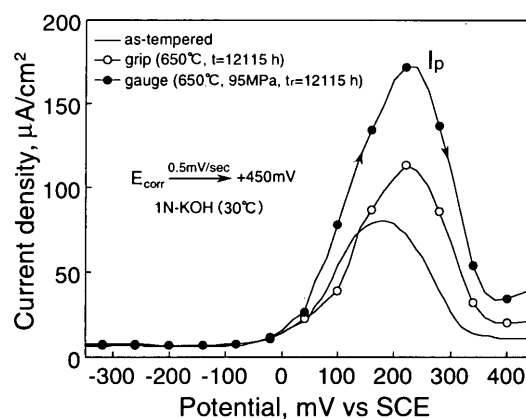


Fig. 3. Examples of anodic polarization curves.

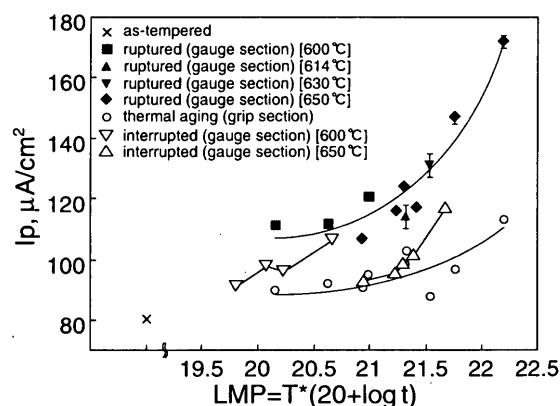


Fig. 4. Change in peak current density I_p as a function of LMP.

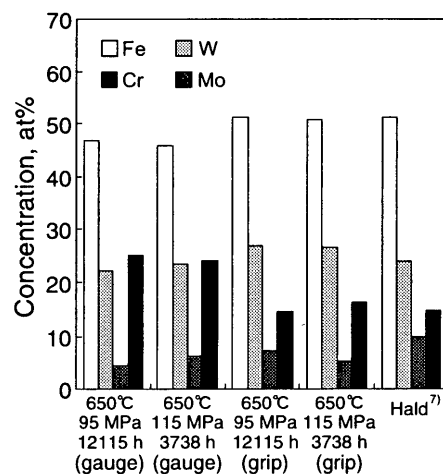


Fig. 5. Chemical compositions of Laves phase extracted from creep-ruptured specimens.

irrespective of the gauge or the grip sections. From this result, the increase in I_p due to thermal aging and creep seems to correspond the increase in amount of chromium precipitated as Laves phase rather than that of Laves phase. The dissolution of Laves phase is considered to be activated by the increase in chromium concentration and/or the decrease in iron concentration, because the peak potential (+180~+240 mV), which the I_p appears, lies in the domains of corrosion and passivation in E-pH equilibrium state diagrams of chromium and iron⁽⁸⁾, respectively. Shweinsberg et al.⁽⁹⁾ reported that the anodic current density corresponding to dissolution of carbide and sigma phases in 1N-NaOH correlated well with the [Cr]/[Fe] ratio in precipitate zones of aged AISI 347

grade stainless steel. The above results coincide with the authors' given assumption.

3.5 Damage Evaluation Based on Kinetics of Laves Phase Precipitation

As can be seen in Fig. 5, there is little difference in chemical composition of Laves phase between two gauge sections. From this result, it was not seemed that the chemical composition of Laves phase precipitated in creep-damaged materials strongly depended on the magnitude of stress and/or duration of rupture time. In this section, we rearrange the I_p measured on the creep-damaged materials using a newly proposed parameter, which is based on the kinetics of Laves phase precipitation, on the assumption that the constitutional element concentrations in Laves phase precipitated in the creep-damaged materials show almost no variation.

In general, the precipitation rate of Laves phase is expressed as the following Johnson-Mehl-Avrami-type equations^{(7),(10)}.

$$w(t) = 1 - \exp\left[-(t/\tau)^{3/2}\right] \quad (1)$$

$$1/\tau = 2D \cdot (4\pi N/3)^{2/3} \left[(C_o - C_e^\alpha)/(C_e^\beta - C_e^\alpha) \right]^{1/3} \quad (2)$$

$$D = D_o \exp(-Q/RT) \quad (3)$$

Where $w(t)$ is relative amount of tungsten precipitated at time t , C_o is equilibrium tungsten concentration in ferrite after tempering at 770°C, C_e^α and C_e^β are equilibrium tungsten concentrations in ferrite and Laves phase at temperature T , respectively, N is nucleation site number, Q is activation energy (230 kJ/mol), D_o is frequency factor (0.29×10^{-4} m²/sec) and R is gas constant (8.314 J/mol K). Since an activation energy is a function of stress (σ), Eqs. (1)-(3) can be rewritten as follows:

$$w(t) = 1 - \exp\left\{-[c \cdot t \cdot F(T) \cdot \exp(-Q(\sigma)/RT)]^{3/2}\right\} \quad (4)$$

$$F(T) = (N)^{2/3} \left[(C_o - C_e^\alpha)/(C_e^\beta - C_e^\alpha) \right]^{1/3} \quad (5)$$

$$Q(\sigma) = Q_o - d\sigma \quad (6)$$

where Q_o is 230 kJ/mol and c and d are constants. Because C_e^α and C_e^β are function of temperature, $F(T)$ is also a function of temperature. From Eq. (4), it is presumed that the ΔI_p , which is the difference between the I_p values measured on the creep-damaged and the as-tempered materials and reflects the amount of Laves phase, can be well arranged by the following parameter:

$$P(t, T, \sigma) = t \cdot F(T) \cdot \exp(-Q(\sigma)/RT) \quad (7)$$

Hereafter, this parameter is defined as "P parameter". The changes in ΔI_p with P parameter were investigated using the interrupted creep specimens, when $F(T)$ and $Q(\sigma)$ were fixed, that is, both temperature and stress were constant. This investigation revealed the ΔI_p increased with prolonged duration of creep testing time t and there was linear correlation between them, when the temperature and stress were 650°C and 105 MPa, respectively. Similar relationship was observed in the interrupted creep specimens performed under the other test condition. Hence, it was postulated that the ΔI_p was correlated linearly with P parameter irrespective of testing conditions, then the relationship between them was represented as follows:

$$\Delta I_p = P(t, T, \sigma) + \Delta I_{p0} \quad (8)$$

where ΔI_{p0} was considered to be ΔI_p after relatively short term creep test and correspond to ΔI_p at the termination of Laves phase nucleation. In this study, the ΔI_{p0} was determined to be 6.9 $\mu\text{A}/\text{cm}^2$ by the correlation between the ΔI_p and P parameter obtained from the interrupted creep specimens (650°C, 105 MPa).

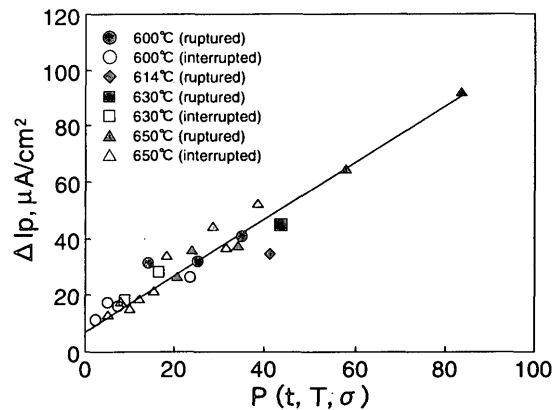


Fig. 6. Change in ΔI_p as a function of P parameter.

From Eqs. (7) and (8), the following equations can be derived.

$$\Delta I_p - \Delta I_{p0} = t \cdot F(T) \cdot \exp(-Q(\sigma)/RT) \quad (9)$$

$$\ln(\Delta I_p - \Delta I_{p0}) = \ln t + \ln F(T) - Q_o/RT + d\sigma/RT \quad (10)$$

When temperature does not vary, a plot of $\ln(\Delta I_p - \Delta I_{p0}) - \ln t$ against σ/RT gives a straight line with the slope d because Q_o/RT and $\ln F(T)$ are constant. The value of d resulted in 310 from the examination using the creep-ruptured specimens at 650°C, and $Q(\sigma)$ was represented as follows:

$$Q(\sigma) = 230000 - 310\sigma \quad (11)$$

Similarly, it is recognized that a plot of $\Delta I_p - \Delta I_{p0}$ against $t \cdot \exp(-Q(\sigma)/RT)$ gives a straight line with the slope $F(T)$ from Eq. (9). The investigation using the creep-ruptured and interrupted creep specimens at 600°C, 630°C and 650°C revealed that $F(T)$ had an Arrhenius-type temperature dependence and was expressed by the following equation.

$$F(T) = \exp[57.0 - 33000(1/T)] \quad (12)$$

Based on the obtained $Q(\sigma)$ and $F(T)$, the ΔI_p measured on all creep-ruptured and interrupted creep specimens were rearranged using P parameter. The obtained result is given in Fig. 6, where an excellent single correlation can be seen irrespective of test conditions though there are some scattering results. This result supports that the increase in I_p due to creep reflects the increase in amount of Laves phase, to be exact, chromium precipitated as Laves phase. The relationship between the ΔI_p and P parameter was calculated from Fig. 6 by the least-squares method and the following equation was obtained:

$$\Delta I_p = 0.997P(t, T, \sigma) + 6.9 \quad (13)$$

The test temperature of each creep specimen was estimated by Eq. (13), assuming the applied stress to be known, then the predicted test temperatures were compared with the actual ones. The major part of the test temperatures were estimated with accuracy of $\pm 5^\circ\text{C}$ though a few predicted temperatures of the creep specimens carried out at 600°C were slightly higher than the actual ones. Similarly, most of the applied stresses were estimated with accuracy of ± 10 MPa.

Thus, the amount of Laves phase, which is the critical factor of the reduction of creep strength, and the test temperature or applied stress of creep damaged materials can be nondestructively evaluated by measuring the I_p and P parameter. It is expected that the present electrochemical technique can be an appropriate tool to evaluate creep damage accumulation in tungsten alloyed 9%Cr

ferritic steels.

4. CONCLUSIONS

In the present study, the influence of pre-aging on creep rupture strength was investigated to understand the creep damage mechanism of tungsten alloyed 9%Cr ferritic steel KA-STBA29/KA-STPA29. The change in strengthening factors during creep and thermal aging was also examined by a nano-indentation test. Additionally, based on the results, the change in electrochemical property of the steel caused by creep and thermal aging were investigated to establish an electrochemical nondestructive procedure for creep damage evaluation. From the results of this study, the following conclusions can be drawn.

- (1) The creep rupture strength of the thermally aged materials decreases as the duration of pre-aging increases. Laves phase is considered to be closely associated with the reduction of rupture strength, because the rupture time decreases with increasing amount of Laves phase.
- (2) Nano-indentation tests revealed that the matrix softening during thermal aging was caused by the decrease in amount of tungsten and molybdenum in solid solution, which was attributed to Laves phase precipitation. This decrease in solid-solution strengthening is considered to cause the above-mentioned reduction of rupture strength.
- (3) The peak current density I_p , which appears at a specific potential during potentiodynamic polarization measurement in 1N-KOH, increases with thermal aging and creep. The I_p reflects not only the thermal effect but also the stress effect on creep damage.
- (4) The I_p value corresponds to the selective dissolution volume of $M_{23}C_6$ type carbide and Laves phase and the increase in I_p due to creep (ΔI_p) reflects the increase in amount of chromium precipitated as Laves phase.
- (5) The ΔI_p is uniquely correlated with a newly proposed P parameter based on the kinetics of Laves phase precipitation, irrespective of test conditions. The test temperature or applied stress of creep-damaged materials can be nondestructively

evaluated by measuring I_p and P parameter with high accuracy.

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