Continuous Observation of Cavity Growth and Coalescence by Creep-Fatigue Tests in SEM*

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Structural components operating at high temperatures in power plants are subjected to interaction of thermal fatigue and creep which results in creep-fatigue damage. In evaluating the life of those components, it is important to understand microscopic damage evolution under creep-fatigue conditions. In this study, static creep and creep-fatigue tests with tensile hold-time were conducted on a SUS 304 stainless steel by using a high-temperature fatigue testing machine combined with a scanning electron microscope (SEM), and cavity growth and coalescence behaviors on surface grain boundaries were observed continuously by the SEM. Quantitative analysis of creep cavity growth based on the observations was made for comparison with theoretical growth models. As a result, it was found that cavities nucleate at random and grow preferentially on grain boundaries in a direction almost normal to the stress axis. Under creep condition, the cavities grew monotonously on grain boundaries while remaining an elliptical shape. On the other hand, under creep-fatigue conditions, the cavities grew due to the effect of the local strain distribution around the grain boundary due to cyclic loading and microcracks of one grain boundary length were formed by coalescence of the cavities. Also, cavity nucleation and growth rates under the creep fatigue condition were more rapid than those under the static creep condition and the constrained cavity growth model coincided well with the experimental data for creep.

Key Words: Scanning Electron Microscope, Creep, Creep Fatigue, Cavity Growth Model, Damage Mechanisms

1. Introduction

In the main structural components of thermal and nuclear power plants, damage caused by the interaction of thermal fatigue and creep accumulates irreversibly. Extensive studies on the development of creep-fatigue damage evaluation methods⁽¹⁾ have been executed in order to establish life assessment techniques for preventing crack initiation. Existing creep-fatigue damage evaluation methods have been proposed based on creep-fatigue life properties and the failure appearance identified in the tests. Although simple evaluation methods such as the linear life fraction rule should be used in designing and assessing the remaining life of components, quantitative damage accumulating in local portions of the components can not be identified by such methods.

Thus in order to establish a precise method for quantitatively evaluating creep-fatigue damage in the local portion, it is necessary to clarify the microscopic damage mechanisms under the creep fatigue loading imposed in actual plants⁽²⁾.

In order to comprehend how the damage progresses throughout a creep-fatigue test, we examined cross sections of interrupted specimens of austenitic stainless steel (SUS 304) and continuously observed the microcrack initiation and propagation behaviors in predamaged miniature specimens under creep-fatigue conditions by using a high temperature creep-fatigue testing machine combined with a scanning electron microscope (SEM)⁽³⁾. As a result, we found that the period until cavity coalescence which results in microcracks is about 40% of the specimen failure. Thus in order to clarify the detailed microscopic damage mechanisms, it is important to examine the cavity nucleation and growth processes.

Extensive studies⁽⁴⁾ on the theoretical treatment of cavity growth behavior under creep conditions have been conducted. Hull and Rimmer⁽⁵⁾, who published a classic paper on a cavity growth model, formulated

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the growth rate equation by assuming that spherical cavities, when placed on an idealised square grid on the grain boundary plane in a direction normal to the stress axis, grow by the grain boundary diffusion mechanism. Chen and Argon⁽⁶⁾ and Needleman and Rice⁽⁷⁾ proposed a more realistic cavity growth model which considered the coupling effect of grain boundary diffusion and creep deformation in grains. Also, Dyson⁽⁸⁾ pointed out that constraints are imposed on the normal motion of grains, particularly when few grain boundary facets are cavitated and the stress is very low. However, the above-mentioned models have not been discussed based on observations of the cavity growth process.

In this study, static creep and creep-fatigue tests with tensile hold-time were conducted on a SUS 304 stainless steel by using a high-temperature fatigue testing machine combined with the SEM, and cavity growth and coalescence behaviors on surface grain boundaries were observed continuously by the SEM. Also, the creep cavity growth based on the observation results was quantitatively analyzed by comparing with theoretical growth models.

2. Experimental Procedure

The material tested in this study was austenitic stainless steel (SUS 304) with an average grain diameter of 40 μm . The chemical composition is shown in

Table 1 Chemical composition of SUS 304

SUS304									wt (%)
C	Si	Mn	P	S	Ni	Cr	Co	V	
0.05	0.47	0.83	0.03	0.009	9.11	18.63	0.21	0.06

Fig. 2 Appearance of high-temperature fatigue machine combined with SEM

Table 1. The heat treatment condition was 1 050°C for 12 min with water quenching. The dimensions of the specimen used in the creep and creep-fatigue tests are shown in Fig. 1. The specimen surface was polished in the longitudinal direction after machining and lightly etched by a mixture of hydrochloric acid (30 cc) + nitric acid (10 cc). In order to determine the region observed by the SEM, the mark shown in Fig. 1 was set by a Vickers hardness machine (applied load 200 g).

The testing equipment which is shown in Fig. 2 was an electric-hydraulic servo high-temperature fatigue testing machine combined with a SEM which allows us to continuously observe how the damage extends on the specimen surface during the tests. The maximum load capacity is 10 kN and the maximum operating temperature is 1 000°C. Axial strain was measured by using a high-temperature strain gage type extensometer.

Creep tests were performed under a constant load

Fig. 1 Miniature test specimen for testing in SEM

Fig. 3 Stress waveform

of 147 MPa at 600°C. Creep fatigue tests were performed under stress-control of 147 MPa with hold times of 10 and 60 min at the maximum tensile stress at 600°C. The stress waveform is shown in Fig. 3. The surface was observed by the SEM with magnifications of 400 and 1 000 at intervals of about 20 hours through the duration of the tests.

In order to quantify cavity growth behavior obtained from the observations, the cavity length $2c$ was defined as the distance between points at which a cavity edge and a grain boundary intersected. The cavity growth rate is given by

$$\left[\frac{dc}{dt} \right]_i = \frac{c_{i+1} - c_i}{t_{i+1} - t_i} \quad (1)$$

where c_i is the cavity half length and t_i is the testing time. The growth rate at coalescence is not included in the equation.

3. Results

3.1 Overall results of SEM observation

Observation of the specimen surface during the creep-fatigue test with a hold time of 60 min showed that many cavities had formed on grain boundaries after 23 hours. After 191 hours, the number of cavities had increased continuously and these might have nucleated at random on grain boundaries. At 425 hours, microcracks of one grain boundary length nucleated on grain boundaries in a direction almost normal to the stress axis. As mentioned in the following section, these microcracks were caused by cavity coalescence. In the creep and creep fatigue tests with a hold time of 10 min, the progress of damage was almost the same.

In order to examine the state of internal damage, cross sections of specimens interrupted after 637 cycles of the 60 min hold time test and after 2 331 cycles of the 10 min test were observed by the SEM. It was found that microcracks on surfaces grew on the inside to about half of the grain boundary length, and the number of cavities on the inside was fewer than those on the surface.

These observations suggest that microscopic damage evolves under creep fatigue conditions by cavities nucleating on grain boundaries at random due to the influence of the crystal direction and micro-

Fig. 4 Typical continuous SEM observation results under creep condition

structure. Furthermore, cavities, which exist on grain boundaries in a direction almost normal to the stress axis, grew by local stress acting on a grain boundaries and coalesced into microcracks due to the effect of cyclic loading. Microcracks on the surface propagate along grain boundaries and the damage then gradually progresses into the inside of the specimen through the nucleation and growth of cavities.

3.2 Continuous observation of cavity growth behavior

Typical continuous observation results of cavity growth behavior by the SEM under creep condition are shown in Fig. 4. At 45 hours, a number of cavities nucleated on the grain boundary indicated by arrow

(a) in Fig. 4. These cavities grew monotonously on the grain boundaries while remaining an elliptical shape, and then coalesced into microcracks of one grain boundary length at 618 hours. On the other hand, shallow pits were observed on the grain boundary indicated by arrow (b) at 92 hours. These pits may be the nuclei of cavities. These grew toward the inside of the specimen with increasing time and became large enough to be observed easily at a magnification of 1 000. As previous considerations for cavity nucleation mechanisms, Raj and Ashby⁽⁹⁾ considered it to be the stress concentration at grain boundary particles caused by grain boundary sliding. Our observations, however, indicate that the nuclea-

tion mechanism involves a cohesion of atom vacancies due to thermal activity on grain boundaries.

Typical continuous observation results of cavity growth behavior under creep fatigue condition are shown in Fig. 5. At 23 hours, cavities in diameter of about $2\text{ }\mu\text{m}$ were observed on the grain boundaries. The cavity indicated by arrow (c) in Fig. 5 grew while remaining a lens like shape. It then deformed from "lens like" to "V shaped" at 75 hours. This mechanism is thought to be due to inhomogeneous local strain distribution within the grains around the cavity. The cavity then recovered to a lens like shape and a microcrack of one grain boundary length was formed when coalesced unstably with adjoining cavities due to the effect of cyclic loading at 286 hours.

These observations show that, under the creep condition, the cavities grow monotonously on grain boundaries while remaining an elliptical shape. On the other hand, under the creep-fatigue condition, the cavities grow irregularly due to the effect of the accumulated local strain distribution within grains around the cavity due to cyclic loading and microcracks of one grain boundary length are formed by unstable coalescence of cavities.

4. Discussion

4.1 Difference in cavity growth behavior under creep and creep-fatigue conditions

Cavity growth curves obtained under the creep condition are shown in Fig. 6. This figure corresponds to arrow (a) in Fig. 4, and the numbers 1 to 5 in the figure are indicated the cavities on the grain boundary in Fig. 4. Cavity length increases monotonously with time, and the cavity growth behavior on other grain boundaries was also almost the same. The time to cavity coalescence in correspondence for 4 and 5 is about 300 hours, and the coalescence time in correspondence with other numbers is about 1 500 hours. It

Fig. 5 Typical continuous SEM observation results under creep-fatigue condition

Fig. 6 Cavity growth behavior under creep condition

is considered here that the difference in coalescence time depends on the cavity spacing and cavity growth rate. Although the effect of cavity nucleation time was also considered, this could be negligible because the time difference to cavity nucleation was about 20 hours. The relationship between the cavity growth rate calculated by Eq. (1) and the cavity half-length is shown in Fig. 7. It was found that the cavity growth rate did not have scattered values over all cavity half-lengths and its value was between 10^{-13} and 10^{-12} m/s.

The cavity growth behavior under the creep-fatigue condition corresponding to Fig. 5 is shown in Fig. 8. The cavity length increased and decreased cyclically with increasing time in contrast to the monotonous cavity growth behavior under the creep condition. It is considered that the increasing and decreasing behavior was caused by cavity shape deformation, for example from "lens-like" to "V-shaped", due to the effects of cyclic loading. Furthermore, cavity coalescence was faster than that under the

Fig. 7 Cavity growth rate under creep condition

Fig. 8 Cavity growth behavior under creep fatigue condition

creep condition. This was caused by an instability coalescence, such as debonding of grain boundaries, with adjoining cavities. The relationship between the cavity growth rate and cavity half-length is shown in Fig. 9. Data for three grain boundaries (A, B and C) in a direction almost normal to a stress axis taken from the SEM photographs is shown. It was found that the cavity growth rate had a large scattered band over all cavity half-lengths, in contrast with that under the creep condition in Fig. 7. Particularly, the positive growth rate was about 10^2 times faster than that under the creep condition. It was not found in this study, however, whether the difference in cavity growth rates under the creep and creep-fatigue condition was caused by the rapid plastic strain rate at the moment of stress hold or by accumulated plastic strain due to cyclic loading within the grains around a cavity. As a future task, it is necessary to clarify the relationship between the macroscopic strain history in response to external loading and the local microscopic strain within grains and/or around grain boundaries.

4.2 Comparison of creep cavity growth models

In this section, two kinds of cavity growth models are examined based on the experimental data obtained in this study.

(A) Diffusion controlled cavity growth model (model 1)

This is the most basic model for cavity growth and was suggested by Hull and Rimmer⁽⁵⁾. They formulated the growth rate equation based on some assumptions in which spherical cavities grow only by a grain boundary diffusion controlled mechanism and are placed on an idealized square grid in the grain boundary plane in a direction normal to the stress axis. The cavity growth rate is given by

$$\frac{dc}{dt} = \frac{D}{\pi} \left\{ \frac{1}{c} - \frac{1}{c_0} \right\} \frac{\sigma^\infty - \sigma_0}{\frac{1}{2} \left(\frac{c^2}{c_0} + c_0 \right) - c}, \quad (2)$$

Fig. 9 Cavity growth rate under creep fatigue condition

The cavity growth rate is compared between experimental results under the creep condition and two kinds of model in Fig. 7. The cavity growth rate estimated by the grain boundary diffusion controlled model, i.e. model 1, was faster than that obtained in experiments. On the other hand, it was found that the cavity growth rate estimated by model 2, which takes into account the effect of the constraint from adjoining grains, was more appropriate. Here, as c approaches c_0 in Eqs. (2) and (6), the cavity growth rate in model 1 increases suddenly, which indicates "rupture". On the other hand, growth rate in model 2 does not increase significantly, which indicates that cavity coalescence forms microcracks of one grain-boundary length. These results show that model 2 is reasonable for modeling the cavity growth behavior.

In this study, although we do not examine the cavity growth rate under creep-fatigue conditions theoretically, the experimental data shown in Fig. 9 are important for comprehending the creep-fatigue damage mechanisms.

5. Conclusions

In this study, static creep and creep fatigue tests with tensile hold time were conducted on a SUS 304 stainless steel by using a high-temperature fatigue testing machine combined with a SEM, and cavity growth and coalescence behaviors on surface grain boundaries were observed continuously by the SEM. Continuous SEM observations showed that cavity nucleation occurred by shallow pits, then grew along grain boundaries while remaining an elliptical shape under the creep condition. On the other hand, under the creep fatigue condition, cavities nucleated by the same mechanism grew while subjected to the effects of cyclic loading.

Quantifying the cavity growth behavior, cavity length increased monotonously with time under creep condition and the growth rate was about 10^{-12} m/s. Also, cavity coalescence was caused by contact with adjoining cavities. On the other hand, under the creep-fatigue condition, the cavity length increased and decreased cyclically with time and the cavity coalescence was caused by unstable debonding of grain boundaries. Cavity growth models which have been

proposed previously were examined for the creep condition. By comparing calculated results with experimental data obtained in this study, it was found that the constrained grain boundary diffusion growth model was approximate.

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