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Life extension by cavity annihilation heat treatment in AISI 316 stainless steel under creep-fatigue interaction conditions

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The creep-fatigue life extension of AISI 316 austenitic stainless steel by heat treatment for cavity annihilation was investigated. Different heat treatments were conducted to find better conditions which could enhance the effect of treatments for the life extension. Cavities formed during creep-fatigue tests were found to be annihilated during solution heat treatment, and the cavity annihilation led the materials to have longer creep-fatigue lives. Having different heat treatments, it can also be suggested that the extension of the creep-fatigue life can be maximized by changing the ageing condition, after solution treatment, to have the lower density of grain boundary carbides that serve as nucleation sites for the cavities. The specimen aged at a higher temperature which has the lower value of cavity nucleation factor, P', has a longer extended creep-fatigue life. In this specimen, cavity re-generation during the loading after annihilation of previous cavities is retarded owing to the lower density of the grain boundary carbide.

1. Introduction

Many components operating in power-generation and aeronautics are subjected to complex stress-loading cycles at high temperature. A high temperature low cycle fatigue with tensile hold time at the peak tensile strain (creep-fatigue interaction), is one of the methods for understanding the damaging process of these components. Studies on this damage mechanism and the life prediction of these structures under creep-fatigue interaction conditions are very important for their future effective use. However, it would be more efficient if the life of these components could be extended by a proper treatment. Considering the creep-fatigue lives limited by the damages, it can be imagined that the creep-fatigue life could be extended if the formation of damage were retarded or the damage formed in the materials were removed.

It is generally known that grain boundary cavitation is the main damage mechanism in austenitic stainless steels under tensile hold creep-fatigue interaction condition [1–5]. The number of cavities nucleated during cyclic loading per unit area of grain boundary, n, which is proportional to the amount of plastic deformation was suggested to be

$$n = P\Delta\varepsilon_{\mathrm{p}}N \tag{1}$$

where P, $\Delta\varepsilon_{\mathrm{p}}$, and N were the cavity nucleation factor, the plastic strain range, and the number of cycles, respectively [2].

Generated cavities are thought to be grown during the hold time period at tensile peak strain by grain boundary diffusion of vacancies. To make a growth model of the cavities, the constant stress term of the Hull-Rimmer [6] model for diffusional growth of cavities was modified as a function of hold time, because of the load relaxation taking place during tensile hold time in the creep-fatigue test [2–4]. With assuming that $\sigma \gg 2\gamma/r$;

$$\frac{\mathrm{d}A}{\mathrm{d}t} = \frac{2\pi\delta D_{\mathrm{g}}\Omega\sigma(t)}{kTl} \tag{2}$$

where A is the cavitated area of a given cavity, l is the cavity spacing, δ is the effective width of the boundary,

D_g is the grain boundary diffusivity, Ω is the atomic volume, $\sigma(t)$ is the tensile peak stress relaxation term during hold time, and k, T give their usual meanings.

Considering cavity nucleation and growth at carbides on the grain boundary, the model for creep-fatigue life prediction was suggested by Hong *et al.* [2];

$$N_{\mathrm{cr}} = C(P\Delta\varepsilon_{\mathrm{p}})^{-3/5}\left\{\frac{\exp(-Q_{\mathrm{g}}/RT)}{T}\int_0^t \sigma(t)\,\mathrm{d}t\right\}^{-2/5} \tag{3}$$

where,

$$C = \left(\frac{4\pi\Omega\delta D_0}{5kA_{\mathrm{t}}}\right)^{-2/5} \tag{4}$$

where Q_g is the activation energy of grain boundary diffusion, C is a constant including the critical cavitated area (A_t) and P was considered to be a constant. P was, however, found to be a function of the plastic strain range,

$$P = P'\Delta\varepsilon_{\mathrm{p}}^{m'} \tag{5}$$

Considering the strain dependency of the cavity nucleation factor, the model was modified [4];

$$N_{\mathrm{cr}} = C\left(P'\Delta\varepsilon_{\mathrm{p}}^{m}\right)^{-3/5}\left\{\frac{\exp(-Q_{\mathrm{g}}/RT)}{T}\int_0^t \sigma(t)\,\mathrm{d}t\right\}^{-2/5} \tag{6}$$

where P', the new cavity nucleation factor, is known to be a materials constant related with the grain boundary carbide density and $m = m' + 1$. This model is known to be good for the accurate life prediction under creep-fatigue condition in which the failure is controlled by the grain boundary cavitational damage [3–4, 7].

However, the cavity growth during hold time will actually only take place when the applied stress perpendicular to the grain boundary, $\sigma(t)$, is greater than the surface tension forces which tend to collapse the cavity [6], even though this term is disregarded in Equation 2 because of its small value as compared to the applied stress. Considering the surface tension force, the condition for cavity growth is

$$\sigma(t) > \frac{2\gamma}{r} \tag{7}$$

where γ is the surface free energy and r is the radius of the cavity. If the applied stress is removed when the material is at high temperature, the cavity would be expected to collapse by losing vacancies to the grain boundary [8–12]. This annihilation of cavity means the recovery of the materials from the damage formed during the creep-fatigue interaction. And the recovery of the materials result in the extension of their lives.

Recovery of mechanical properties by heat treatment [13–17] and cavity annihilation under different heat treatments and stress conditions [8–11, 14] have been investigated. Most studies for recovery of properties or life extensions have been conducted from the view point of the effectiveness of reducing the damage. However, there are little reports considering the prevention of cavity re-generation during loading after the annihilation of previous cavities, which can be another effective method to extend the lives of high temperature materials.

TABLE I Chemical composition and heat treatment procedure of 316 stainless steel

C	Si	Mn	P	S	Cr	Ni	Mo	V	Fe
0.044	0.47	1.40	0.04	0.017	16.8	10.82	2.20	<0.04	bal.

Heat treatment before creep-fatigue tests
Solution treatment : 1050°C, 1 hr → water quenching
Ageing : 760°C, 50 hr → water quenching

In this work, creep-fatigue life extension of austenitic stainless steel by cavity annihilation is investigated. Different heat treatments are conducted to find better conditions which could enhance the effects of the treatments on the life extension by controlling not only the conditions of annihilation but also the re-generation of cavities. Microstructural studies are also conducted to observe the annihilation behavior of the cavities on the grain boundary.

2. Experimental procedure

Creep-fatigue tests were carried out with AISI 316 stainless steel whose chemical composition and heat treatment conditions before testing are shown in Table I. Ageing treatment is conducted to make the stable carbides precipitate on the grain boundary and to prevent further growth during the test. Total strain controlled low cycle fatigue tests with 10 minute tensile hold time at the maximum tensile strain were conducted at 600°C. The tested total strain ranges ($\Delta\varepsilon_t$) were ±1.0, ±1.5 and $\pm2.0\%$. The tests were interrupted at the pre-determined cycles (N_{stop}), i.e., $1/3N_{\mathrm{cr}}$ or $2/3N_{\mathrm{cr}}$, where N_{cr} is the critical number of cycles (i.e., fatigue life) at a given test condition [2, 5]. The interrupted specimens were then solution treated at 1050°C for 30 minutes to remove the cavities and to resolve the carbides on the grain boundary. After solution treatment the specimens were aged again with two different conditions, at 840°C for 40 hours and at 760°C for 50 hours, to investigate the effects of the ageing heat treatment on the life extension by controlling the size and distribution of grain boundary carbides which are known to be very important factors for cavity nucleation [3]. The test procedure is schematically shown in Fig. 1.

For the observation of grain boundary carbides and cavities, specimens were chilled in liquid nitrogen to be broken by impact and examined with scanning electron microscope (SEM). The second technique used in examining was a metallographic observation with SEM of the longitudinally sectioned specimen whose surface was polished and etched in a solution of 10 ml $HCl + 15$ ml $CH_3COOH + 10$ ml HNO_3.

3. Results and discussion

3.1. Life extension by cavity annihilation

It has been verified from the previous reports [2–4] that grain boundary cavities are continuously formed and grown with fatigue cycles under creep-fatigue interaction condition. It is also generally believed that cavities are nucleated at geometrical irregularities on the grain boundaries where high tensile stress concentration can be developed. In austenitic stainless steels the potential sites of stress concentration are the carbides precipitated on the grain boundary during an ageing treatment [18].

Fig. 2 shows the cavities and carbides on the grain boundary of the 316 stainless steel which were fractured by impact at liquid nitrogen temperature (LNT) after a creep-fatigue test at 600°C with $\Delta\varepsilon_t$ of ±2.0% and 30 minute tensile hold. Fig. 2a and c are the opposite sides of Fig. 2b and d, respectively. The cavities in Fig. 2a or c are observed to be formed at the equivalent position of carbides in Fig. 2b or d and vice versa. From this observation, it is clear that the grain boundary cavities are formed and grown at the carbides, as a dominant nucleation site.

A specimen was fatigued up to the number of cycles which are high enough to have some cavities in the specimen, say, the 2/3 of the expected critical life at 600°C with total strain range ($\Delta\varepsilon_t$) of ±2.0% and 10 minute tensile hold. The specimen was then taken out of the tester and impact fractured at LNT to observe the damage formed during creep-fatigue test. Fig. 3a shows the fractured grain boundary with cavities formed during creep-fatigue loading at the carbides. Another two specimens were also creep-fatigued up to the $2/3N_{\mathrm{cr}}$

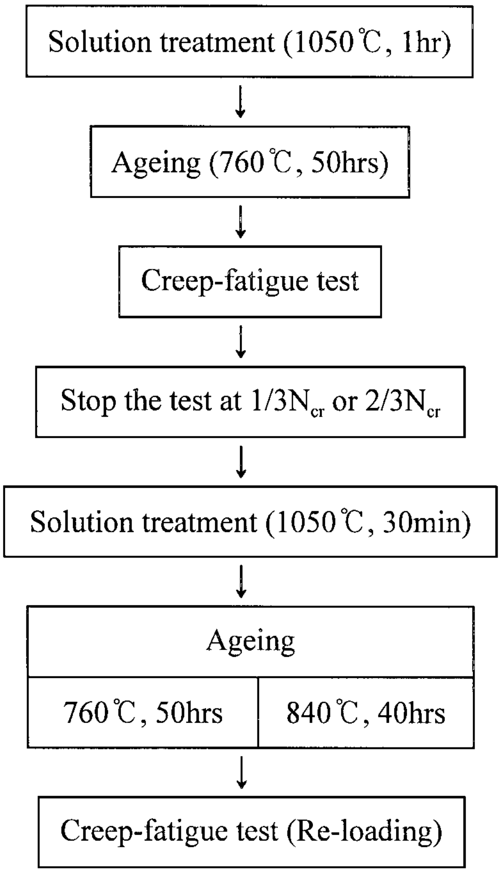

Figure 1 Schematic diagram showing the test procedures.

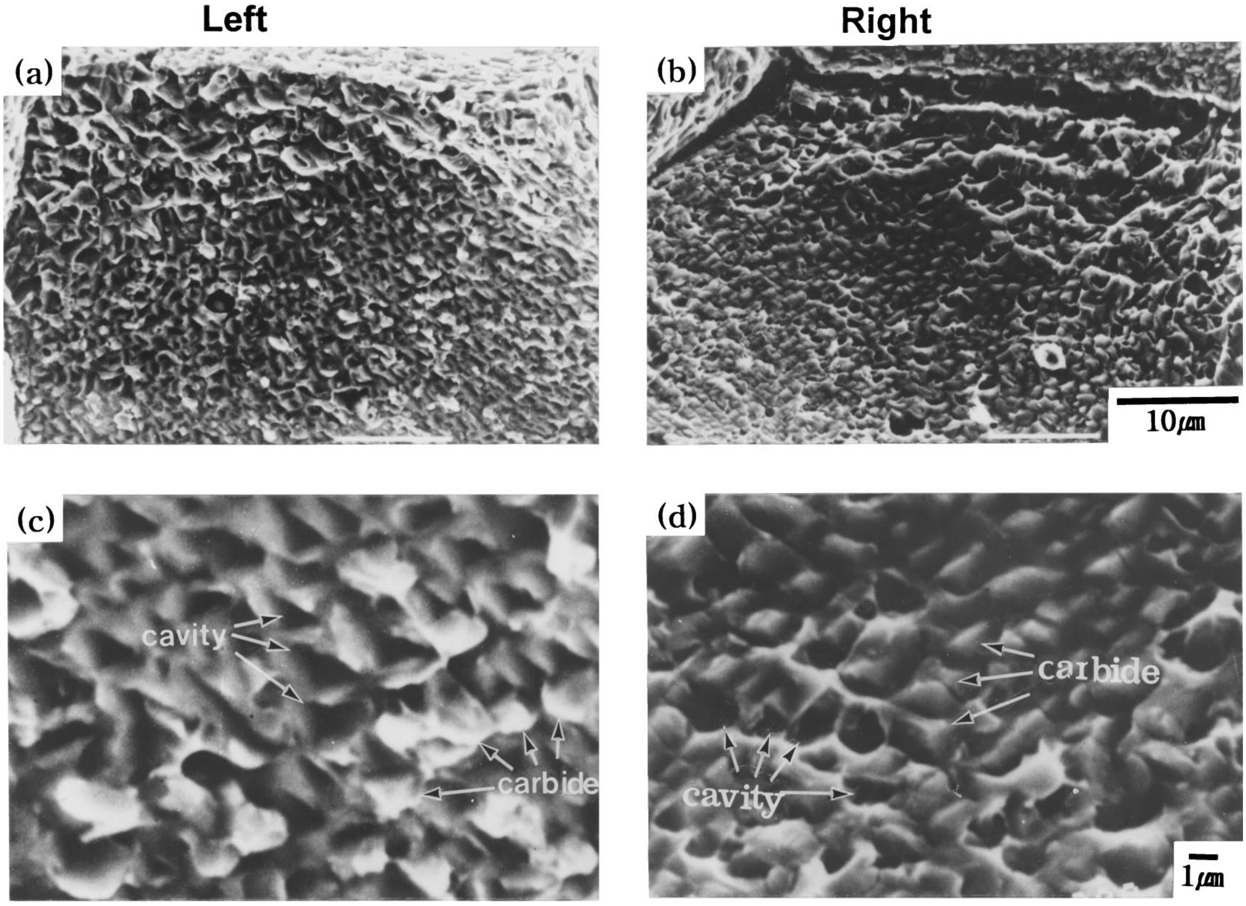

Figure 2 SEM micrographs showing the distribution of cavities and carbides on the matched grain boundary after creep-fatigue test (316S.S., 600°C, $\Delta\varepsilon_t = \pm 2.0\%$, $t_h = 30$ min) (a) grain boundary with cavities and carbides, (b) the opposite side of (a), (c) high magnification of (a), (d) high magnification of (b).

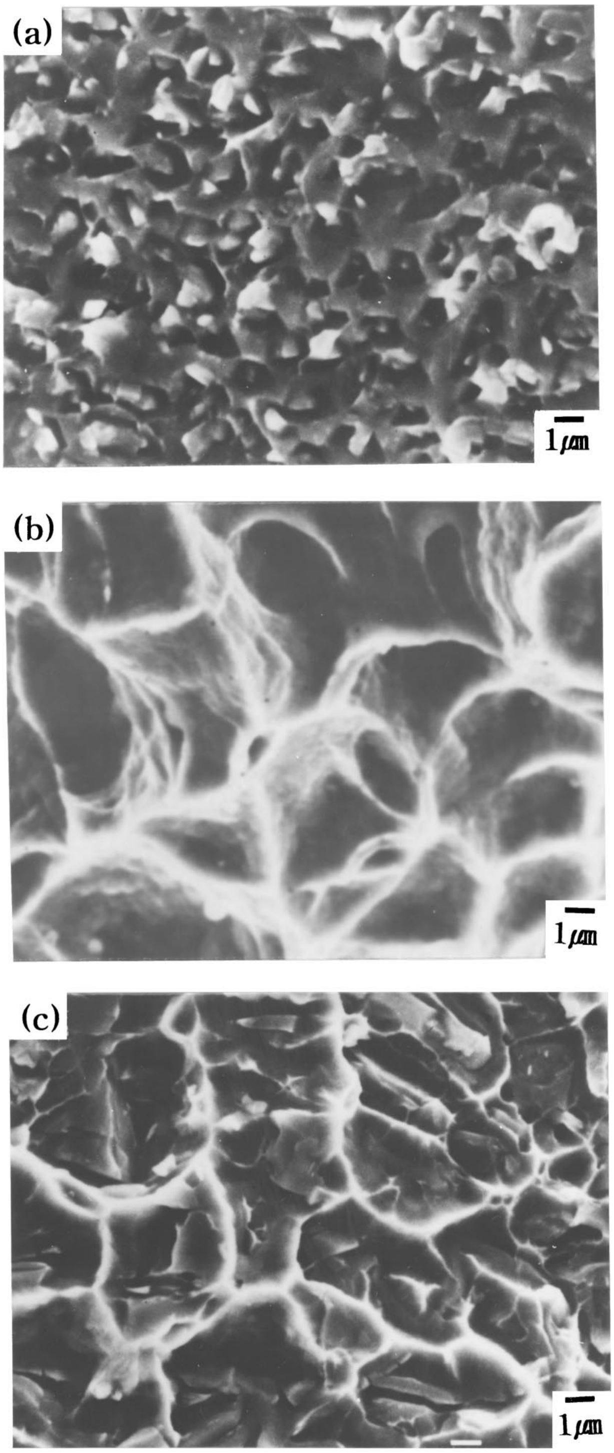

Figure 3 SEM micrographs showing surfaces fractured by impact at LNT after creep-fatigue test (316S.S., 600°C, $\Delta\varepsilon_t = \pm 2.0\%$, $t_h = 10$ min, $2/3N_{cr}$ stop) (a) without heat treatment, (b) after solution treatment (1050°C, 30 min), (c) after ageing (760°C, 50 hrs).

under the same condition. One of them was solution treated at 1050°C for 30 minutes and then impact fractured at LNT to see if there still are cavities and carbides on the grain boundary. Fig. 3b shows the fracture surface of this specimen to indicate no evidence of either cavities or carbides. The other was solution treated and then aged at 760°C for 50 hours to be also fractured at LNT. Fig. 3c is the fractured micrograph for this specimen aged for the re-precipitation of grain boundary carbides. In this figure no indication of grain boundary cavities is found either and this fractography is very similar to the fracture surfaces at LNT of the virgin specimen as shown in the previous study [7].

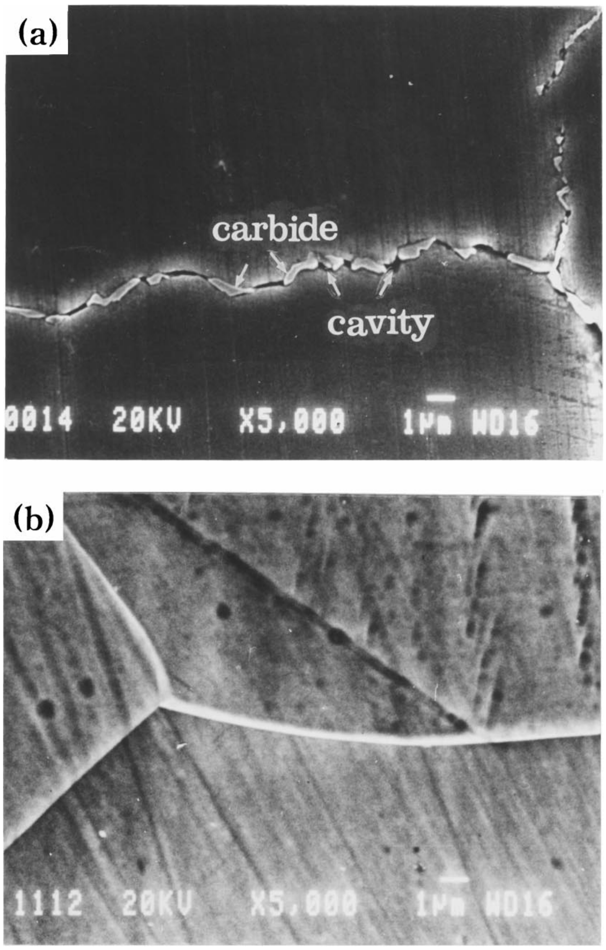

Figure 4 SEM micrographs showing the grain boundary carbide and cavity distribution (316S.S., 600°C, $\Delta\varepsilon_t = \pm 1.0\%$, $t_h = 10$ min, $2/3N_{cr}$ stop) (a) before heat treatment, (b) after solution treatment (1050°C, 30 min).

Other observations of carbides and annihilation of cavities during the solution heat treatment were conducted by the metallographic sectioning technique. The results are shown in Fig. 4 which indicates the same results as in Fig. 3. A creep-fatigue test at 600°C with $\Delta\varepsilon_t$ of ±1.0% and 10 minute tensile hold was interrupted at $2/3N_{cr}$ and the specimen was cut along the loading axis and then one part of the specimen was observed by SEM. The other part of the specimen was solution treated at 1050°C for 30 minutes and the cut surface was then observed by SEM. As previously mentioned, cavities have been formed at the carbides existing along the grain boundary after $2/3N_{cr}$ under creep-fatigue interaction as shown in Fig. 4a. After solution treatment, however, not only the carbides but also the cavities on most grain boundaries were annihilated as shown in Fig. 4b.

These two observations of cavity annihilation with different techniques indicate that cavities formed during creep-fatigue interaction can be annihilated by heat treatment, especially by the solution treatment. If this defect free specimen is tested again for creep-fatigue deformation, it has to have new cavity nucleation and growth again and that leads to the extension of the creep-fatigue life of the specimen.

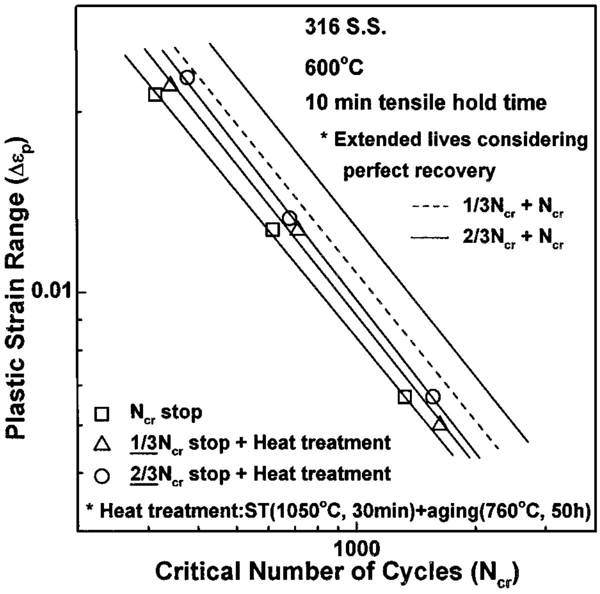

Figure 5 Relationship between plastic strain range and the critical number of cycles for different N_{stop}.

To verify this idea, some more creep-fatigue tests were conducted under three different strain ranges with 10 minute tensile hold time. The tests were stopped at $1/3N_{\mathrm{cr}}$ or $2/3N_{\mathrm{cr}}$ and solution treated and aged under the above mentioned conditions and then re-tested. The results of the creep-fatigue tests are plotted in Fig. 5 in terms of the plastic strain range with the critical number of cycles (N_{cr}). In the heat treatment cases, because of the slight change of mechanical properties during the heat treatment, the plastic strain range under the same total strain range test was also changed. Therefore, the average values of plastic strain range ($\Delta\varepsilon_{\mathrm{p}}$) obtained from the test before heat treatment ($\Delta\varepsilon_{\mathrm{po}}$) and that after heat treatment ($\Delta\varepsilon_{\mathrm{ph}}$) were taken for analysis and then calculated as follows;

$$\Delta\varepsilon_{\mathrm{p}} = \frac{\Delta\varepsilon_{\mathrm{po}} \times N_{\mathrm{stop}}}{N_{\mathrm{stop}} + N_{\mathrm{crh}}} + \frac{\Delta\varepsilon_{\mathrm{ph}} \times N_{\mathrm{crh}}}{N_{\mathrm{stop}} + N_{\mathrm{crh}}} \qquad (8)$$

The creep-fatigue lives of the specimens tested under various conditions were found to be extended with the heat treatment as shown in Fig. 5. Considering the results of SEM observations in Fig. 3 and 4, it can be concluded that the extension of creep-fatigue lives is owing to the annihilation of cavities by solution treatment. It can be also understood that the creep-fatigue lives of the specimens interrupted at $2/3N_{\mathrm{cr}}$ is extended longer compared with those interrupted at $1/3N_{\mathrm{cr}}$. The specimen tested up to at $2/3N_{\mathrm{cr}}$ is expected to be more damaged than that of $1/3N_{\mathrm{cr}}$ because the former one is subjected to creep-fatigue loading for more cycles than the latter one before stopping the test. Therefore, for the specimen interrupted at $2/3N_{\mathrm{cr}}$, more damage (cavities) is recovered by the heat treatment and this caused further life extension.

As previously mentioned, when the austenitic stainless steel was tested under creep- fatigue interaction condition, the material would fail due to the accumulation of a critical amount of the grain boundary cavitation damage [2–4]. Fig. 6, which is the fractograph observed by SEM after impact fractured at LNT, shows the grain boundary cavities formed during creep-fatigue tests. Fig. 6a is the result of the specimen which was subjected to creep-fatigue interaction test at 600°C with $\Delta\varepsilon_t$ of ±1.0% and 10 minute tensile hold up to the critical number of cycles ($N_{\mathrm{cr}} = 1330$ cycles). Fig. 6b is the fractograph of the specimen tested under the same condition up to the new critical number of cycles ($N_{\mathrm{crh}} = 1180$ cycles) after stopping the test at $1/3N_{\mathrm{cr}}$ of previous loading (450 cycles) followed by heat treatment for cavity annihilation. It can be found in Fig. 5 that the interrupted and heat treated specimen has smaller cavitated area than the specimen loaded up to N_{cr} without heat treatment even though the former one was tested for a longer time. This result may also indicate that cavities were annihilated during heat treatment and then formed again during the creep-fatigue loading.

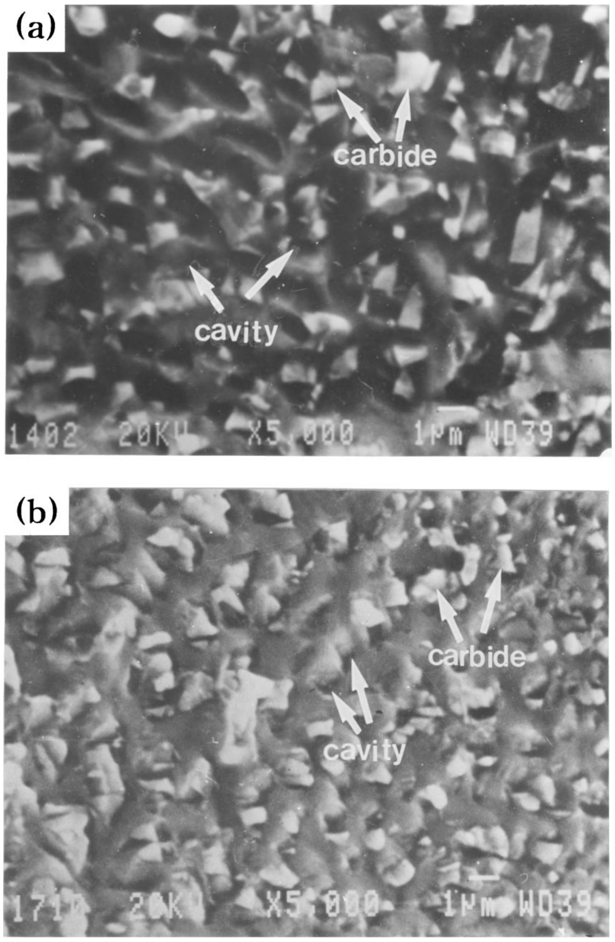

Figure 6 SEM micrographs showing cavities on the grain boundary fractured by impact at LNT(316S.S., 600°C, $\Delta\varepsilon_t = \pm 1.0\%$, $t_{\mathrm{h}} = 10$ min) (a) N_{cr} stop, (b) $1/3N_{\mathrm{cr}}$ stop + Solution treatment + ageing + N_{crh} stop).

Even though the cavities formed during the creep-fatigue test are removed by the solution heat treatment, the damages in the materials seem not to be fully removed. If the test was interrupted at N_{stop} and the damage of specimen was completely recovered with the heat treatment, the total creep-fatigue lives after re-testing had to be extended with the value of N_{stop} because the creep-fatigue life after the interruption should be the value of N_{cr} which is the life without interruption. These expected fatigue lives of the tests interrupted at $1/3N_{\mathrm{cr}}$

and $2/3N_{cr}$ are shown in Fig. 5 as a dashed line and a solid line, respectively. Comparing these tested and expected lives, one may see that the tested creep-fatigue lives in this study were extended much less than the expected values considering the complete recovery as shown in Fig. 5. Therefore, this result indicates that the tested materials were not recovered completely with the heat treatment condition conducted in this study.

Fig. 6 is another proof of the imperfect recovery. The cavitated area of the heat treated specimen (see Fig. 6b) is smaller than that of the specimen tested up to N_{cr} (see Fig. 6a). This indicates that the interrupted and heat treated specimen was broken before it became enough damage at grain boundary caused by cavitation. It means that not only cavities but also other damages which could not be removed during the heat treatment were also introduced to the specimen during the creep-fatigue test. (This extra damage may be surface cracks and their effect will be discussed in further publications.)

3.2. Enhancement of life extension

As previously mentioned, cavities in austenitic stainless steels are nucleated at carbides on the grain boundaries where high tensile stress concentration can be developed during creep-fatigue tests. According to the results of Choi *et al.* [3], the cavity nucleation factor, P', is proven to be the materials constant indicating the number of cavities formed on grain boundaries during creep-fatigue test. It is known to be closely related with the density of grain boundary carbides which act as dominant sites for cavity nucleation. They reported that the creep-fatigue life is increased with decreasing P' value which is caused by lowering the density of the grain boundary carbides. In this study, the carbides were resolved with annihilation of cavities and then reformed on the grain boundary again during ageing after the solution treatment. Considering the re-precipitation of carbides during the ageing treatment, it can be considered that the creep-fatigue life after the heat treatment can be increased by controlling the density of grain boundary carbides using a special ageing condition. In other words, if the density of grain boundary carbides becomes a lower value, the extension of creep-fatigue life can be achieved.

To obtain the different carbide distributions, two following different ageing conditions were conducted after solution treatment at 1050°C for 30 minutes with the specimens which were creep-fatigue tested up to the 2/3 of their creep-fatigue lives: 760°C for 50 hours and 840°C for 40 hours. Fig. 7a and Fig. 7b show the carbide distribution on the grain boundary after ageing at 760°C for 50 hours and at 840°C for 40 hours, respectively. The linear densities of carbides were measured with an image analyzer. They are of 9.344×10^5/m in case of the specimen aged at 760°C (see Fig. 7a) and of 4.253×10^5/m for that aged at 840°C (see Fig. 7b). It is found that the higher is the ageing temperature, the lower is the carbide density.

Using Equation 5, the cavity nucleation factors of the specimens with different ageing treatments were obtained from the relationship between the plastic strain range and the value of P. This was calculated from the results of creep-fatigue tests and Equation 3 as shown in Fig. 8. Values of P' for the specimen aged at

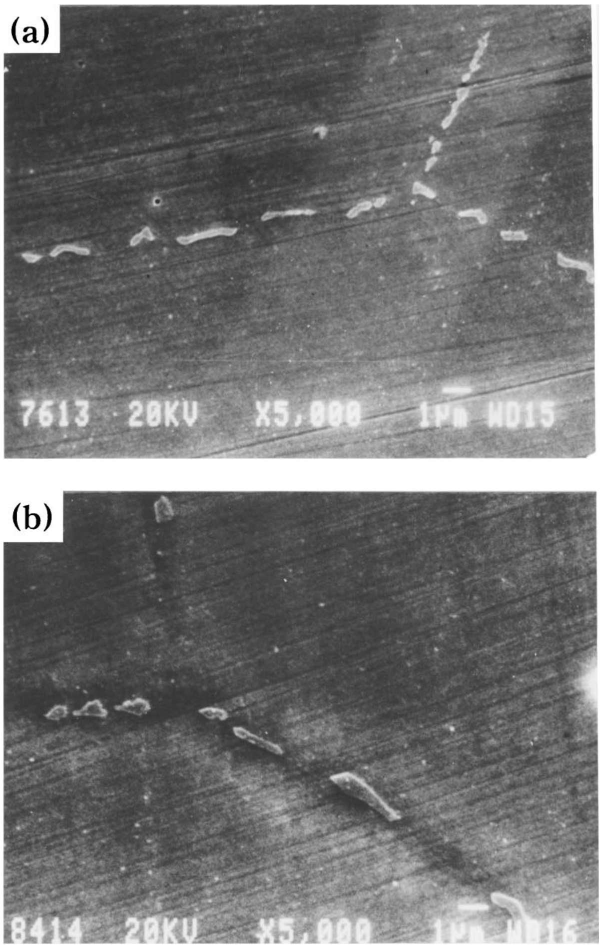

Figure 7 SEM micrographs showing the grain boundary carbide and cavity distribution after different heat treatments (316S.S., 600°C, $\Delta\varepsilon_t = \pm 1.0\%$, $t_h = 10$ min, $2/3N_{cr}$ stop, solution treatment at 1050°C, 50 h) (a) aged at 760°C for 50 h, (b) aged at 840°C for 40 h.

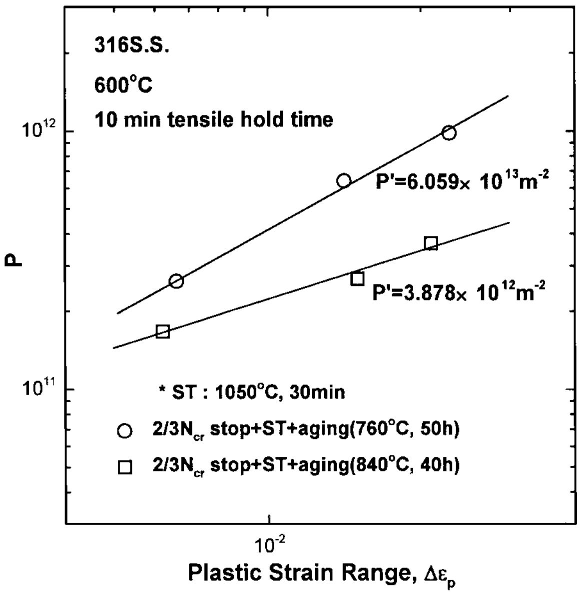

Figure 8 The value of the cavity nucleation factor, P', of the specimens with different ageing calculated from the relation between the value of P with the plastic strain range.

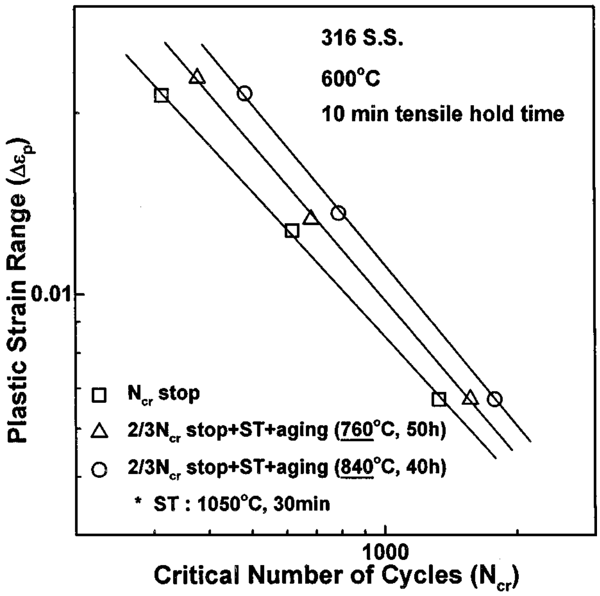

Figure 9 Relationship between plastic strain range and the critical number of cycles for different ageing conditions.

840°C and that aged at 760°C are 3.878×10^{12} m^{-2} and 6.059×10^{13}m^{-2}, respectively. The specimen which has lower density of grain boundary carbides shows the lower value of P'.

Fig. 9 shows the effect of the different ageing conditions on the extension of creep- fatigue live. As expected, considering the grain boundary carbide densities after the ageing treatment, specimens aged at 840°C for 40 hours having a lower value of P', show more extended creep-fatigue lives owing to lower density of grain boundary carbides than those aged at 760°C for 50 hours. Conclusively, with these results of different heat treatments, it can be suggested that the creep-fatigue life can be extended by cavity annihilation of austenitic stainless steels. And that this will be maximized as the heat treatment condition is controlled to have lower density of grain boundary carbides. It is also verified that the cavity nucleation factor P' is an important factor in controlling the creep-fatigue life.

4. Conclusion

1) Grain boundary cavities formed during the tensile hold time under a creep-fatigue test can be annihilated by solution heat treatment, and the cavity annihilation leads the materials to have longer creep-fatigue lives.

2) The life extension can be maximized with controlling the density of grain boundary carbides by changing the ageing condition followed by solution treatment. Higher ageing temperature gives longer creep-fatigue life after cavity annihilation because of related lower density of grain boundary carbides.

3) In the creep-fatigue life prediction model, the cavity nucleation factor, P', is verified again to be closely related to the density of grain boundary carbides. This result is very useful in controlling the life of austenitic stainless steel under creep-fatigue interaction condition.

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