The Effect of Strain Amplitude, Temperature and *in situ* Irradiation by 20 MeV Protons upon the Fatigue Properties of 316 Stainless Steel

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The effect of various testing parameters upon the fatigue hardening associated with the fatigue induced precipitation in solution heat treated type 316 s.s. has been investigated in high cycle strain-controlled fatigue tests employing a large amplitude flexural resonant vibration of about 200 Hz. Miniaturized specimens were used at total strain amplitude (ϵ_t) up to about 6×10^{-4} at 323 K and 573 K. An investigation of the ϵ_t dependence of the apparent modulus suggests the fatigue induced precipitates to be very fine. It was further found that the synergistic effect of the existence of the fatigue induced damage and/or precipitates and rising temperature played an important role in revealing the subsequent enhancement of fatigue induced precipitation. The effect of ϵ_t and of ϵ_p (the plastic strain component of ϵ_t) upon the precipitation phenomenon revealed that the number density of the fatigue induced precipitates tended to increase with increasing ϵ_p or to decrease with increasing ϵ_t . A preliminary investigation concerning the effect of *in situ* irradiation by 20 MeV protons at 573 K upon the precipitation phenomenon reveals that the fatigue induced precipitates are possibly dispersed by the irradiation.

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I. Introduction

The first wall of a fusion reactor is expected to be subjected, under intensive neutron irradiation, to repeated thermal stresses during normal operation and when accidentally induced runaway impacts occur⁽¹⁾. However our knowledge of the effects of *in situ* irradiation upon the fatigue properties of the fusion reactor wall material remains limited⁽²⁾⁽³⁾.

Our work concerning this problem was started with room temperature (RT) 20 MeV proton irradiation of 316 s.s. under strain controlled high cycle fatigue tests during *in situ* irradiation and in the post irradiated stage⁽⁴⁾. In this earlier study the following results were found. The apparent modulus determined at about the mid-point of the fatigue test showed

a moderate softening for unirradiated specimens but a moderate hardening for the irradiated specimens. The latter effect caused the fatigue life of the irradiated specimens to be extended beyond that for unirradiated specimens. The effect of irradiation upon the fatigue life of the s.s. was more pronounced in the fatigue tests conducted under *in situ* irradiation than in those fatigue tests run after irradiation. However, an investigation of the effect of increase in the temperature upon the fatigue properties of the unirradiated specimens showed the fatigue induced hardening occurred above about 473 K, and this was considered to be associated with fatigue induced precipitation at that temperature or above⁽⁵⁾. These results suggest that *in situ* irradiation accelerated the fatigue induced precipitation to such an extent that fatigue hardening became apparent even at RT.

In the present work the effect of strain amplitude upon the fatigue induced precipitation has been studied at 323 K and 573 K on unirradiated specimens and a preliminary fatigue test at 573 K under *in situ* irradiation conditions has also been carried out.

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II. Experimental Procedure

The specimens used were cut from a rolled sheet of type 316 s.s. and their chemical composition and prior heat treatment were the same as those described in previous studies⁽⁴⁾⁽⁵⁾. In review, after mechanical polishing of the specimens, they were annealed at about 1370 K for 3.6 ks in a vacuum of 2×10^{-4} Pa and subsequently quenched into water. The specimens were then electrolytically polished, in order to remove a surface layer of about 50 μm and simultaneously to smooth out the specimen surface. The sample were then annealed again this time at about 573 K for 36 ks in a vacuum of 2×10^{-4} Pa to degass any hydrogen possibly picked up during the electrolytic polishing. The shape of the specimens was that of a rectangular cross-section U coil with thick ends as shown in Fig. 1(b) of Ref. (5). The thick ends were used for clamping the specimen and the thinner arms, about 0.12 mm in thickness, about 0.5 mm in width and about 16 mm in gage length, were used as vibrating reeds.

The grain size of the specimens was typically about 40 μm , which was about twice as large as that for the specimens used in our earlier studies. The origin of this difference in grain size is probably attributable to a difference in the rolling conditions of the sheets from which the specimens were made. The present specimens were placed in a small open-ended capsule, annealed in a group in the furnace and quenched directly from the furnace into water. On the other hand, the specimens employed in the earlier studies were annealed and then withdrawn from the furnace (unprotected) and subsequently quenched into water. Therefore, the quenching rate was faster for the present specimens compared with the previously studied samples. Some precipitation is considered to have occurred in the previously studied specimens, i.e. these specimens were somewhat hardened before the start of the fatigue tests as compared with the specimens employed in the current study. Due to these differences in the quenching rate and the grain size⁽⁶⁾, the total strain amplitude, ϵ_t , at the fatigue limit, is a little lower for the present

specimens when compared to the value for the earlier specimens. For example, at RT , ϵ_t was about $4\text{--}5 \times 10^{-4}$ at the fatigue limit for the present specimens and it was about 6×10^{-4} for the previous specimens studied, respectively. This difference in the specimen grain size and quenching rate gave us the opportunity to investigate the effect of ϵ_t on the fatigue induced precipitation phenomena in more detail, and this will be described later in the text. The fatigue testing method was almost the same as that described previously⁽⁴⁾⁽⁵⁾: In review, ϵ_t controlled tests were conducted employing large amplitude resonant flexural vibrations of the specimens at about 200 Hz in an atmosphere of about 10^2 PaHe. During the fatigue tests concurrent changes in the resonant period, P , and the drive force, DF , were measured almost continuously. The total strain amplitude was maintained constant by automatically adjusting the DF . To investigate the strain amplitude dependence of P , (SADP), ϵ_t was increased in steps from about 0.4×10^{-4} to a given larger value and then decreased in one step to about 0.4×10^{-4} . This procedure was employed repeatedly with the maximum total strain amplitude attained becoming successively larger.

The SADP observed in as prepared specimens showed that P remained almost constant (P_0) for all ϵ_t values well below the fatigue limit. As ϵ_t increased above these low values, P started to increase noticeably up to ϵ_t values slightly below the fatigue limit. On the other hand, for the fatigue tests with ϵ_t above the fatigue limit, P exhibited initially a steep increase (i.e. softening) which was followed by fatigue hardening at the intermediate stage of the fatigue test, i.e. P exhibited a maximum, P_s , where the specimen was the softest (see Fig. 1(a)). Since the strong increase in P was considered to be associated mainly with an increase in the plastic strain component ϵ_p of ϵ_t , the ratio P_s/P_0 has been used to monitor the relative strength of the plastic strain in the present fatigue tests. It will be shown (see eq. (5) of this paper) that ϵ_t may be considered to signify the apparent stress amplitude σ^* .

During the fatigue test the temperature (T) of the testing bed was controlled within ± 0.1 K.

However, the specimen temperature was higher than the bed temperature, and this temperature enhancement of the specimen increased as P/P_0 increased mainly due to the Joule heating associated with the higher driving current passing through the specimen. In the atmosphere of about 10^2 PaHe, the increase in temperature of the specimen was about 20 K, 50 K and 100 K for P/P_0 values of 1.1, 1.2, and 1.3, respectively. In the present study P_s/P_0 values fell in the range mainly between 1.1 to 1.2, but the maximum P_s/P_0 found was 1.3. It has been reported that the fatigue properties of 316 s.s. are not very sensitive to relatively small changes in temperature (i.e. < 100 K)⁽⁵⁾⁽⁷⁾. Therefore, the test temperature will be represented by that of the testing bed throughout this report. Also, it was observed that the increase in P due to a temperature increase was about $2.7 \times 10^{-2} P_0/100$ K which was negligibly small compared with the P changes associated with increasing values of ϵ_t .

In situ irradiation tests were performed in the same manner as described in Ref. (4) using 20 MeV protons at 573 K not at 323 K. The specimen was thin enough for the complete penetration of 20 MeV protons, so that the increase in temperature of the specimen due to the irradiation was only several degrees for the beam current density employed.

III. Experimental Results and Discussion

1. The general features of *in situ* changes in P and the DF observed during the fatigue tests

Figure 1(a) shows an example of the *in situ* changes in P and the DF observed during a fatigue test at 323 K for a current specimen, where P_0 was measured initially at $\epsilon_t = 0.4 \times 10^{-4}$ and then ϵ_t was rapidly increased to 5.5×10^{-4} prior to the start of fatigue testing. We observed in this case that $P_s/P_0 = 1.3$ ($P_0 = 5520 \mu\text{s}$). As can be seen in Fig. 1(a), the sudden increases in both P and DF at the initial stage of the test are followed by abrupt decreases at the early middle stage of test. The decreasing values of P and the DF are attributed to fatigue hardening which tends

Fig. 1 (a) An example of observed *in situ* changes in P and the DF during a fatigue test at 323 K with $P_s/P_0 \approx 1.3$ ($\epsilon_t = 5.5 \times 10^{-4}$). (b) A typical schematic drawing depicting the *in situ* changes in P shown in (a) with ϵ_t included.

towards saturation values as the fatigue test continues towards failure. In this case P_h/P_0 attained a value of about 1.1 at saturation (see Fig. 1(b)). At the very final stage of the test both P and DF increase again and the specimen fails with a life-time (N_f) of about 6.5×10^5 cycles in this case. For the current specimens these general features were always observed at 323 K and also at 573 K. This

characteristic change in P/P_0 during the fatigue test is schematically shown in Fig. 1(b) as well as the change in ε_t .

On the other hand for the specimens used in the previous studies⁽⁴⁾⁽⁵⁾, which were already hardened by pretreatment precipitation, the fatigue tests were carried out at higher ε_t values. In these previous tests, the pronounced fatigue hardening as seen in Fig. 1(a) was observed only at temperatures in excess of 473 K. At 333 K, the *in situ* changes in P observed for the previous specimens were as follows: For a fatigue test with $\varepsilon_t = 6.9 \times 10^{-4}$ the fatigue lifetime (N_f) of 7.7×10^5 cycles is comparable to that shown in Fig. 1(a). For these previous specimens the initial softening of $P_s/P_0 \approx 1.06$ was followed by some very small fatigue hardening behavior, similar to that of run 2 shown in Fig. 5(b). For higher ε_t with smaller values of N_f , the *in situ* change in P during the middle fatigue stage exhibited only a moderate increase (see for example Fig. 5 with

$\varepsilon_t = 12.1 \times 10^{-4}$ of Ref. (4).

From these observations one can suggest that fatigue hardening is somewhat suppressed by increasing values of ε_t . This point will be pursued later in this paper.

2. The initial softening and the fatigue hardening as depicted by SADP

Figure 2(a) shows an example of the strain amplitude dependence of P , SADP, observed at 573 K under the following conditions: The SADP was measured initially by increasing ε_t stepwise from 0.4×10^{-4} to 3.3×10^{-4} for run 1, and then ε_t was decreased to 0.4×10^{-4} in one step. The similar procedure was repeated with the maximum total strain amplitude attained becoming successively larger for runs 2 through 6. After run 5, however, the specimen was subjected to a fatigue test with $P_s/P_0 \approx 1.25$ ($\varepsilon_t = 4.3 \times 10^{-4}$) for about 4.5×10^5 cycles. This induced some appreciable fatigue hardening as indicated by the arrow AA' in

Fig. 2 (a) An example of the SADP (strain amplitude dependence of P) observed at 573 K. For an as-prepared specimen, the SADP was measured repeatedly for runs 1 to 5 and then run 6 was determined following a fatigue test conducted after run 5. (b) Similar to (a) except that run 3 was observed following an aging treatment at 573 K for 3.6 ks after run 2 was completed (see text).

Fig. 2(a). During the measurements at each ϵ_t for the determination of the SADP, P remained constant for the lower ϵ_t values i.e. $<2 \times 10^{-4}$, but at higher ϵ_t values P exhibited first an initial increase which was followed by a tendency to decrease. Therefore, the measurements for the larger ϵ_t values were made when P had reached its maximum value, and subsequently ϵ_t was increased stepwise for further measurements. The P values shown in Fig. 2(a) correspond to the maximum value obtained at each ϵ_t . The abrupt increases in P observed above $\epsilon_t \doteq 3 \times 10^{-4}$ for runs 1 to 5 in Fig. 2(a) correspond with the sharp softening observed during the very initial stage of the fatigue test (see Fig. 1(a) and (b) of this text and also see Fig. 6 in Ref. (5)).

The following features of Fig. 2(a) should be noted. For run 1, P/P_0 remained almost constant at 1.00 ($P_0 = 5158 \mu\text{s}$) up to $\epsilon_t \doteq 2 \times 10^{-4}$ and then it started to increase for higher ϵ_t values. The SADP in run 2 was very similar to that in run 1, but a small softening and small hardening effect compared with that in run 1 is observed below and above $\epsilon_t \doteq 3 \times 10^{-4}$, respectively. Runs 3 to 5 show that this softening and hardening effect becomes more and more pronounced until finally a hump in the SADP is discernible at $\epsilon_t \doteq 3.5 \times 10^{-4}$ for run 5. Particularly in run 6, as ϵ_t increased, P gradually increased up to $\epsilon_t \doteq 4.5 \times 10^{-4}$ and then steeply rose with increasing ϵ_t value beyond the prior fatigue hardened point designated by A'. Runs 1 and 2 in Fig. 2(b) show results for another specimen similar to those of runs 5 and 6 of Fig. 2(a), where after run 1 the specimen was subjected to a fatigue test with $P_s/P_0 \doteq 1.2$ ($\epsilon_t = 4.2 \times 10^{-4}$) for 3.8×10^5 cycles. In Fig. 2(b), after run 2 the specimen was aged at 573 K for 3.6 ks in unstressed condition and then runs 3 and 4 were performed in sequence. Run 3 shows that aging caused rather pronounced hardening in the intermediate range of ϵ_t values, i.e. $1.5 \times 10^{-4} < \epsilon_t < 4.3 \times 10^{-4}$. Run 4 shows softening in the same intermediate ϵ_t range as just defined for run 3 and a very small amount of hardening at higher ϵ_t values greater than 4.3×10^{-4} as compared with the data for run 3.

From these results, it appears that once ϵ_t is

increased to a large value, softening occurs at low and intermediate values of ϵ_t in the subsequent run and the softening reverses to hardening at the larger values of ϵ_t in this subsequent run. This behavior is further enhanced by a fatigue test between runs (see run 6 in Fig. 2(a)). The latter hardening at ϵ_t values $> 3.5 \times 10^{-4}$ is considered to originate from fatigue induced precipitation in which the precipitates must be too small to be observable by electron microscope⁽⁵⁾. The softening at ϵ_t values $< 3.5 \times 10^{-4}$ is thought to be due to the disappearance of small precipitates or clusters by the dislocation cutting mechanisms.

The reason for conclusion that the hardening is associated with precipitation but not with the dynamic strain aging will be discussed below. It is reported that the dynamic strain aging phenomenon is observed in austenitic stainless steels when cycled above temperatures of approximately 750 K and arises from dragging of such interstitial solutes as carbon and nitrogen⁽⁸⁾. The strain rate with which the considerable dynamic strain aging phenomenon can be revealed is considered to decrease strongly with decreasing temperature⁽⁹⁾⁽¹⁰⁾ and for 316 s.s., it is reported that the dynamic strain aging phenomenon at 723 K becomes to be observable for the strain rates below about $7 \times 10^{-4} \text{ s}^{-1}$ ⁽¹¹⁾. Therefore, the dynamic strain aging phenomenon can not be expected in the present experiments carried out at 573 K with the strain rates of the order of 10^{-1} s^{-1} . Furthermore, the behaviors of SADP shown in Fig. 2 are difficult to be explained by the dynamic strain aging. For example, if the hardening from A to A' in Fig. 2(a) and (b) had been due to the dynamic strain aging, the softening at low ϵ_t (below 3×10^{-4}) in the following runs would not have been observed.

Changes in ϵ_t with stress σ corresponding to changes in ϵ_t with P/P_0 values are schematically shown in the insert figure of Fig. 2(a). The σ versus ϵ_t curves shown in Fig. 2(a) can be explained in the following way. The as prepared specimens contain many precipitates or clusters of solute atoms with a distribution in size which we will characterize by their obstacle strength or resistance to the passage of dislocations. The simplification we propose here is

that there are only two kinds of obstacles to dislocation motion, weak ones and strong ones. We further assume the density of strong obstacles is much less than that of the weak ones. For the case where the density of strong obstacles is nearly zero and the dislocations can overcome the weak obstacles at $\sigma = \sigma_{y1}$, the change in σ with ϵ_t is depicted by curve 1' in Fig. 2(a). When the density of weak obstacles decreases and that of strong ones becomes finite but low in value, the yield stress for the weak obstacles decreases, but at higher values of ϵ_t another yield for the strong obstacles appears, so that the σ changes with ϵ_t as shown by curve 2' in Fig. 2(a). In addition, since the steep increase in P for ϵ_t in excess of 3×10^{-4} seen for runs 1 to 5 in Fig. 2(a) corresponds to the initial softening revealed in the fatigue test shown in Fig. 1, the above results suggest that fatigue induced precipitation may have commenced already during the initial softening stage.

Figure 3(a) shows similar results to those shown in Fig. 2, except the tests were conducted at 323 K. After runs 1, 2 and 3 in sequence were made, the specimen was heated to 573 K with a heating rate of about 0.03 K/s and aged at 573 K for about 3 ks. The specimen was then cooled to 323 K with a cooling rate of about 0.02 K/s. During the heating and cooling cycles, P was measured with $\epsilon_t \doteq 0.4 \times 10^{-4}$. The difference between the P 's evaluated during the warming up (P_w) and cooling down (P_c) at each temperature (shown in Fig. 3(b)) are plotted as $(P_w - P_c)/P_0$ versus temperature in Fig. 3(c). After the aging treatment at 573 K, runs 4 and 5 were obtained at 323 K. The results of runs 1, 2 and 3 in Fig. 3(a) are very similar to those obtained for runs 1 and 2 shown in Fig. 2(b). The results of runs 4 and 5 in Fig. 3(a) are also very similar to those of runs 3 and 4 in Fig. 2(b), but the hardening due to the aging treatment is more pronounced in Fig. 3(a) than in Fig. 2(b). Since the specimens used here were already annealed at about 573 K for 36 ks prior to the fatigue tests, the pronounced age hardening mentioned above is considered to be associated with fatigue induced damage and/or precipitates. This is probably also the origin for an observed enhanced

Fig. 3 (a) An example of the SADP (strain amplitude dependence of P) observed at 323 K. The SADP runs were made in the sequence 1 through 5 but after run 3 the specimen was aged at 573 K for 3 ks. (b) The change in P measured at $\epsilon_t = 4 \times 10^{-5}$ during the warm-up to 573 K after run 3 (P_w : the solid curve), that during aging at 573 K for 3 ks (shown by the arrow) and that during the subsequent cooling-down to 323 K (P_c : the broken curve). (c) The difference between P_w and P_c at each temperature seen in (b) is depicted as a function of T .

fatigue hardening at 573 K which will be described later. The changes in P shown in Fig. 3(b) or (c) suggest that dislocation pinning has occurred at about 330 K, 470 K, 500 K and 560 K and some dislocation depinning and/or rearrangement of pins at about 370 K and 573 K. The atoms or clusters responsible for these pinning and depinning effects are not known, however the results do suggest that possibly a variety of solute atoms or defect clusters may be involved in inducing the enhancement of the fatigue hardening at 573 K. On the other hand, it is reported that fatigue induced precipitates in austenitic stainless steel become observable in TEM only after an aging treatment for several hours or more at 800 K⁽¹²⁾. Therefore, the fatigue induced precipitates responsible for the fatigue hardening observed here are considered to be too fine to be resolvable by TEM.

3. Some detailed observations concerning fatigue hardening

Figure 4(a) and (b) show the effect of temperature and/or P_s/P_0 on the fatigue hardening process. Figure 4(a) shows *in situ* changes in P during a fatigue test at 323 K for $P_s/P_0 \doteq 1.2$ ($\epsilon_t = 5.4 \times 10^{-4}$) and $P_s/P_0 \doteq 1.3$ ($\epsilon_t = 5.5 \times 10^{-4}$). Figure 4(b) shows the same parameters as Fig. 4(a), except that the tests were made at 573 K for $P_s/P_0 \doteq 1.2$ ($\epsilon_t = 4.2 \times 10^{-4}$) and $P_s/P_0 \doteq 1.3$ ($\epsilon_t = 4.6 \times 10^{-4}$). As mentioned previously, the fatigue hardening was very steep at the early stage of fatigue and then it tends towards saturation, i.e. P reaches its minimum value P_h near the end of the intermediate fatigue stage (see also Fig. 1(b)). One may evaluate the relative magnitude of the fatigue hardening effect using the ratio $(P_s - P_h)/(P_s - P_0)$. This fatigue hardening ratio is about 60% and 64% for $P_s/P_0 \doteq 1.2$ and 1.3 at 323 K, respectively (Fig. 4(a)). This ratio is about 87% and 89% for $P_s/P_0 \doteq 1.2$ and 1.3 at 573 K, respectively (Fig. 4(b)). In general, this fatigue hardening ratio seems to increase mildly with increasing P_s/P_0 , i.e. with increasing ϵ_p . This ratio is enhanced markedly as the test temperature increases.

As mentioned in the section III-1, the fatigue hardening observed in the previous studies was much smaller in magnitude than

Fig. 4 (a) An example of *in situ* changes in P observed during fatigue tests with $P_s/P_0 \doteq 1.2$ and $\doteq 1.3$ at 323 K at various values of ϵ_t as noted. (b) An example of *in situ* changes in P observed during fatigue test with $P_s/P_0 \doteq 1.2$ and $\doteq 1.3$ at 573 K at various values of ϵ_t as noted.

that observed here, due possibly to the higher ϵ_t values used in the earlier studies. Also, the results shown in run 4 of Fig. 2(b) or in run 5 of Fig. 3(a) suggest that the fatigue hardening is somewhat suppressed by the large ϵ_t previously applied. This point is further clarified in Fig. 5(a)–(c). Figure 5(a) shows an example of the *in situ* changes in P/P_0 observed during an extended fatigue test to failure on one specimen at 323 K. The specimen was at first tested with $P_s/P_0 \doteq 1.12$ ($\epsilon_t = 5.1 \times 10^{-4}$) for about 7×10^5 cycles (run 1) and then ϵ_t was increased to 5.2×10^{-4} (run 2), so that P_s/P_0 was again about 1.12 (the value of P_0 used for run 2 was the same as that used in run 1). Similarly, the test was interrupted after runs 2 and 3 to raise ϵ_t so that P_s/P_0 remained approximately constant. The specimen failed during the softening stage in run 4. Comparison of runs 1 and 2 suggests that increase in ϵ_t has caused the fatigue hardening ratio to decrease. Figure 5(b) employed the procedure similar to that

Fig. 5 (a) An example of the *in situ* change in P/P_0 observed during an extended fatigue test on one specimen at 323 K. This test was interrupted after runs 1, 2 and 3 respectively (see figure) to raise ϵ_t so that P_s/P_0 remained approximately constant at 1.12. (b) Similar to (a) with the exception that only two ϵ_t values were employed and run 1 was made very long so that fatigue hardening was established at 323 K. (c) Similar to (a) except that only three ϵ_t values were employed and the test was conducted at 573 K.

depicted in Fig. 5(a), with the exception that only two ϵ_t values were used and run 1 was made very long so that fatigue hardening was

established or almost saturated at 323 K. Run 2 dose show a markedly reduced fatigue hardening ratio possibly associated with the higher value of ϵ_t employed. Figure 5(c) shows similar behaviors to those shown in Fig. 5(a) but the test was conducted at 573 K not at 323 K. The decreasing fatigue hardening ratio with increasing ϵ_t is markedly observed with this 573 K test.

4. Discussion of the apparent fatigue induced precipitation phenomena

The present results concerning the changes in P during fatigue tests suggest the following: The number density (n_f) of the fatigue induced precipitates responsible for fatigue hardening increases rapidly during the initial stage of the fatigue test and tends towards a saturation value occurring near the end of the intermediate stage of the fatigue test. Furthermore, n_f appears to increase with increasing ϵ_p and also with rising T but to decrease with increasing ϵ_t . As a first attempt to discuss these results quantitatively, we suggest that the time rate of change in n_f is given by the following kinetic equation,

$$dn_f/dt = \alpha(T) \cdot \epsilon_p - n_f \cdot \beta(\epsilon_t), \quad (1)$$

where $\alpha(T)$ and $\beta(\epsilon_t)$ are increasing functions of T and ϵ_t , respectively. The basic reasoning behind eq. (1) is as follows. With increasing ϵ_p , which means a higher plastic strain rate for a constant frequency fatigue test, the formation rate of vacancies and interstitials will be increased⁽¹³⁾ and also the mobility of drift of light atoms being dragged during the dislocation movements of the fatigue cycle⁽¹²⁾ will be enhanced. With increasing T , the diffusion flux of these defects and/or solute atoms is enhanced exponentially. The contribution of these effects to dn_f/dt is represented by the first term on the right hand side of eq. (1). As mentioned in the section III-2, the fatigue induced precipitates are considered to be extremely fine (i.e. not resolvable by TEM), so that the smaller ones are probably cut and dispersed by moving dislocation⁽¹⁴⁾. This latter effect should become more enhanced with increasing σ or with increasing ϵ_t and is represented by the second term on the right hand side of eq. (1). The assumption that T , ϵ_p and ϵ_t all remain cons-

tant during the fatigue test yields the following solution for eq. (1),

$$n_f = (\alpha(T) \cdot \varepsilon_p / \beta(\varepsilon_t)) \cdot (1 - \exp(-\beta(\varepsilon_t) \cdot t)). \quad (2)$$

Equation (2) shows that n_f increases fast initially with t or with the number of fatigue cycles and then tends towards a saturation value after long times or numbers of cycles. In the present fatigue tests, both T and ε_t were controlled and held constant but ε_p must have decreased due to fatigue hardening. This decrease in ε_p will modify the time dependence and saturation level of n_f , but the saturation behavior in general will not change.

The current theory concerning precipitation hardening upon dislocation motion⁽¹⁵⁾ predicts that the flow stress (σ) is roughly proportional to the square root of the precipitate density ($n^{1/2}$),

$$\sigma \propto n^{1/2} \quad (3)$$

and

$$\Delta\sigma = \sigma - \sigma_s \propto n^{1/2} - n_s^{1/2}, \quad (4)$$

where the quantities with the subscript s denote that they are taken as a standard. Under the present experimental conditions, a change in the apparent stress σ^* can be estimated from a change in P through the following relation,

$$\sigma^* \propto P^{-2} \cdot \varepsilon_t, \quad (5)$$

and if we normalize the relation with P_s as a standard, we obtain:

$$\Delta\sigma^* / \sigma_s^* = (\sigma^* - \sigma_s^*) / \sigma_s^* \propto (P_s / P)^2 - 1. \quad (6)$$

Corresponding to this normalization, we propose that for the fatigue hardening phenomena observed here, n in eq. (4) can be described by the following equation,

$$n = n_f + n_s, \quad (7)$$

where the clock for the elapsed time(t) in eq. (2) concerning n_f is started as P attains the value P_s . Therefore, n_s denotes the density of precipitates existing already at $t=0$ or as P attains the value P_s . The combination of eqs. (3) to (7) yields,

$$(P_s / P)^4 - 1 = n_f / n_s, \quad (8)$$

and from eqs. (2) and (8) one can expect that for the initial part of the intermediate fatigue stage $(P_s / P)^4 - 1$ increases according to eq. (2). These predictions have been verified in the hardening process as shown in Fig. 6(a), (b) and (c). The combination of eqs. (2) and (8) yields,

$$\ln((P_s / P)^4 - (P_s / P)^4) = -\beta(\varepsilon_t) \cdot t + \ln(\alpha(T) \cdot \varepsilon_p / \beta(\varepsilon_t) \cdot n_s), \quad (9)$$

where $(P_s / P)^4 = (\alpha(T) \cdot \varepsilon_p / \beta(\varepsilon_t) \cdot n_s) + 1$, i.e. a linear time dependence of the left side term of eq. (9) may be expected. In Fig. 6(a), the left side term of eq. (9) is plotted against the number of cycles, which elapsed after P had reached its maximum value P_s , for the results shown in Fig. 4(a) but only for the initial 1×10^5 cycles (shown by solid lines in the figure). In making these plots, however, a value of $(P_s / P)^4$ in eq. (9) was not known, so that it was determined as the parameter which gives the best fit of the observed $(P_s / P)^4$ values to eq. (9) as a whole. The value of $(P_s / P)^4$ thus obtained was denoted by $(P_s / P)^4_{\text{calc. } \infty}$ and used in Fig. 6(a). In Fig. 6(a), the broken lines are expected from eq. (9). Deviations of the observed results from eq. (9) are seen in both initial and later cycles which will be discussed later. From these broken lines, the parameters $(\alpha(T) \cdot \varepsilon_p / \beta(\varepsilon_t) \cdot n_s)$ and $\beta(\varepsilon_t)$ in eq. (9), which are assumed to remain constant over certain elapsed cycles concerned, can be determined as 0.28 and 2.33×10^{-5} /cycle for $P_s / P_0 \doteq 1.2$ and 0.64 and 2.52×10^{-5} /cycle for $P_s / P_0 \doteq 1.3$, respectively. In Fig. 6(b) the results shown in Fig. 6(a) are replotted as $((P_s / P)^4 - 1)$ versus the number of cycles. Except for the early cycles and the later cycles in excess of about 8×10^4 cycles, one can see an excellent agreement between the observed results (solid curves) and the results calculated through eq. (9) (broken curves). Figure 6(c) shows similar results but obtained from the data shown in Fig. 4(b) observed at 573 K. In Fig. 6(c), one can see again an excellent agreement between the observed and calculated results except for the very early cycles and for the later cycles in excess of about 8×10^4 cycles.

The parameters $(\alpha(T) \cdot \varepsilon_p / \beta(\varepsilon_t) \cdot n_s)$ and $\beta(\varepsilon_t)$ found in Fig. 6(c) are 0.36 and 1.78×10^{-5} /cy-

Fig. 6 (a) The data shown in Fig. 4(a) just after P reached its maximum value P_s up to 1×10^5 cycles are replotted as $\ln((P_s/P)^4_{\text{calc.}\infty} - (P_s/P)^4)$ versus the number of fatigue cycles. (b) The data shown in Fig. 4(a) just after P reached its maximum value P_s up to 1×10^5 cycles are replotted as $((P_s/P)^4 - 1)$ versus the number of fatigue cycles. (c) Similar to (b) except that the data are from Fig. 4(b) obtained at 573 K. The dashed lines in Fig. 6(a) to (c) represent the best fit of eq. (9) to the data (see text).

cycle for $P_s/P_0 \approx 1.2$ and 0.68 and 1.97×10^{-5} /cycle for $P_s/P_0 \approx 1.3$, respectively. These results obtained through the analysis employing eq. (9) are shown in Fig. 7(a) to (c). Figure 7(a) shows a plot for $\beta(\epsilon_t)$ versus ϵ_t . Since $\beta(\epsilon_t)$ denotes the probability for fine precipitates to be cut and dispersed by moving dislocations,

this mechanical dispersion process is expected to be rather strongly total strain amplitude dependent (i.e. through ϵ_t) and mildly or not at all affected by temperature in the range employed here. These expectations are confirmed by the data shown in Fig. 7(a). In Fig. 7(b) and (c), since the ratio P_s/P_0 is considered to

Fig. 7 In (a), (b) and (c), values of the parameters $\beta(\epsilon_t)$, $(\alpha(T) \cdot \epsilon_p / \beta(\epsilon_t) \cdot n_s)$ and $\alpha(T) \cdot \epsilon_p / n_s$ in eq. (9) determined for the results shown in Fig. 6(a) or (b) and (c) are plotted against ϵ_t or P_s/P_0 , appropriately (see text).

monitor the relative strength of ϵ_p , the parameters $(\alpha(T) \cdot \epsilon_p / \beta(\epsilon_t) \cdot n_s)$ and $\alpha(T) \cdot \epsilon_p / n_s$ are plotted against P_s/P_0 , respectively. Since $(\alpha(T) \cdot \epsilon_p / \beta(\epsilon_t) \cdot n_s)$ is expected to represent a saturation value of n_t/n_s (see eqs. (8) and (9)), the results of Fig. 7(b) which shows this value to increase with increasing P_s/P_0 and also with increasing temperature are not unreasonable. The combination of the results obtained in Fig. 7(a) and (b) enables $\alpha(T) \cdot \epsilon_p / n_s$ to be determined, which corresponds with the formation rate of fatigue induced precipitates normalized by the n_s value (see eq. (1)). Values for this parameter are shown in Fig. 7(c). It is seen in Fig. 7(c) that $\alpha(T) \cdot \epsilon_p / n_s$ tends to increase with increasing P_s/P_0 , i.e. with increasing ϵ_p , for both cases obtained at 323 K and 573 K. This result is again reasonable. For the effect of temperature seen in Fig. 7(c), the values of $\alpha(T) \cdot \epsilon_p / n_s$ at around $P_s/P_0 = 1.2$ are larger at higher temperature, which seems reasonable,

however, those obtained at $P_s/P_0 = 1.3$ show a reversed temperature effect. In a previous study⁽⁵⁾, the fatigue hardening observed at constant ϵ_t (above the fatigue limit) showed a clear tendency to increase with increasing temperature (see Fig. 7 in Ref. (5)), so that $\alpha(T)$ in the present study is expected to increase with increasing temperature. The decrease of $\alpha(T) \cdot \epsilon_p / n_s$ with increasing temperature observed for $P_s/P_0 = 1.3$ in Fig. 7(c) is due probably to an accidentally somewhat higher value of n_s in the specimen used at 573 K compared with this value for the specimen tested at 323 K. On the other hand, the deformation induced martensitic transformation which plays an important role in controlling the low temperature mechanical properties of austenitic stainless steels tends to be enhanced with decreasing temperature⁽¹²⁾. The fatigue induced hardening observed here exhibiting an opposite temperature dependence can not be associated with the formation of martensitic particles.

The deviations of the observed results from eq. (9) in both the initial and later cycles of fatigue shown in Fig. 6(a) to (c) will be discussed. The initial deviation corresponds to hardening more rapid than that due to the obstacle particle density increase. As shown in Fig. 1(a), the steep softening at the initial stage of fatigue is caused by dislocation motion overcoming obstacles and the resultant increase in the dislocation density. This will soon turn to the hardening due to increased interaction between dislocations at around the maximum point of P , and then a stable dislocation structures will be established. This transient hardening due to dislocation density increase should overlap the precipitation hardening of eq. (9), and is considered to be the probable origin of the deviation in the initial cycles of fatigue. As for the deviation between our theory and experiment in the later fatigue cycles, i.e. in excess of about 8×10^4 cycles, this deviation is due probably to the fact that in the later cycles the fatigue induced precipitates grew up and became more stable against dislocation cutting, so that eq. (9) was no more valid.

Finally Fig. 8 shows the preliminary results obtained to ascertain the effect of *in situ* irradiation by 20 MeV protons upon the fatigue

Fig. 8 Preliminary results for the change in P observed during a fatigue test with *in situ* intermittent irradiation by 20 MeV protons at 573 K.

hardening at 573 K. In this case during the fatigue test with $P_s/P_0 \approx 1.2$ ($\epsilon_t = 4.5 \times 10^{-4}$) at 573 K, *in situ* irradiation by about 1.1×10^{12} p/cm²·s was made intermittently twice as shown in the figure. At this relatively low dose rate, the observed effect of the *in situ* irradiation upon fatigue hardening is rather small. One can discern a small trend of the fatigue hardening to be mildly suppressed as a result of the irradiation. In the earlier work at 333 K⁽⁴⁾, the *in situ* irradiation brought about a hardening effect in those particular specimens. Without the irradiation those fatigue specimens exhibited a moderate softening at the intermediate fatigue stage. This difference is considered to originate from the fact that the *in situ* irradiation can enhance fatigue induced precipitation, e.g. through the formation of vacancies and interstitials, and induce fatigue hardening. Simultaneously, however, the radiation damage could also disperse the fatigue induced precipitates and induce suppression of the fatigue hardening. It appears that the earlier specimens with larger precipitates underwent fatigue hardening whereas a mild suppression of the hardening occurred for the specimens used in the present study. Further investigations concerning the effect of *in situ* irradiation upon the fatigue process are now in progress and will be reported elsewhere.

IV. Summary and Conclusion

Stain controlled fatigue tests of solution

treated type 316 s.s. were performed on miniature specimens employing large amplitude flexural resonant vibrations of about 200 Hz at 323 K and 573 K. In the present work, the effect of the total strain amplitude (ϵ_t) upon the fatigue process was investigated in detail. In the current study, specimens of a rather large grain size and little "as prepared" precipitation enabled the fatigue properties to be evaluated at relatively low values of ϵ_t . In previous studies at higher ϵ_t values, typically 10×10^{-4} at 333 K and 7×10^{-4} at 573 K, the following observations were made: The *in situ* change in the apparent modulus during the intermediate stage of the fatigue tests exhibited some small softening at 333 K. Hardening due to fatigue induced precipitation (called fatigue hardening) became detectable only at temperatures in excess of 473 K. Furthermore, *in situ* irradiation at 333 K enhanced the subsequently observed high temperature fatigue hardening. In the present study however, the following observation have been made. At rather low values of ϵ_t of about 5×10^{-4} , fatigue hardening occurs even at 333 K, and this hardening decreases as ϵ_t increases at constant ϵ_p . Furthermore, the fatigue hardening appears to increase as the plastic strain component ϵ_p of ϵ_t increases. These fatigue hardening phenomena are shown to be semi-quantitatively understood using a kinetic model for the precipitate production rate given by eq. (1). From the strain amplitude dependence of the resonant period P , (SADP), it is inferred that fatigue induced precipitation commences even during the initial softening phase of the fatigue tests. Furthermore, it appears that these precipitates can be dispersed by the dislocation motion during fatigue and hence the precipitates must be very small. The effect of thermal aging at 573 K on SADP suggests that the fatigue induced damage appears to play an important role in promoting fatigue induced precipitation. Finally, preliminary experiments have been carried out to discern the effect of *in situ* irradiation by 20 MeV protons on the fatigue properties of this steel. The results suggest that the fatigue induced precipitates are dispersed mildly by the irradiation. Further investigations concerning the effects of *in situ*

proton irradiation upon these fatigue phenomena are now in progress and are forthcoming.

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