Effects of *In-Situ* Proton-Irradiation and Thermal-Pulse on the High-Cycle Fatigue Properties of Low Carbon 316 Stainless Steels

H. Mizubayashi, K. Tateishi, H. Tanimoto and K. Nakata*

Institute of Materials Science, University of Tsukuba, Tsukuba 305, Japan

*** Hitachi Research Laboratory, Hitachi Ltd., Hitachi 317, Japan*

Abstract. For the modified 316L stainless steel in which the dislocation interaction governs the fatigue hardening process, the high-cycle fatigue tests were carried out between 300 and 403 K. The *in situ* irradiation tests and the *in situ* thermal-pulse tests indicate that dislocation rearrangements due to thermal-pulse give rise to an increase in the areal density of persistent slip bands (PSBs), resulting in elongation of the fatigue life N_f and an introduction of obstacles to dislocation motions, irradiation induced lattice defects or the fatigue induced precipitates, brings about a decrease in the areal density of PSBs, giving shortening of N_f .

1. INTRODUCTION

The first wall of a fusion reactor is planned to be subjected to thermal stresses and 14 MeV neutron irradiation simultaneously. For the dynamic mechanical properties such as the fatigue property, understanding of effects of *in situ* radiation damage and *in situ* thermal pulse is important. However, knowledge on these effects on the fatigue properties is very limited (e.g., [1-3]). One of reasons is a limited radiation damage rate which is not enough to modify considerably the low cycle fatigue property. However, the high cycle fatigue property is expected to be modified under the available radiation damage rate of 10^{-6} - 10^{-7} dpa/s, where the effects of *in situ* radiation damage may more clearly be investigated. Understanding of the high cycle fatigue property itself may also be important, because the most of the structural reactor materials is expected to be subjected to the high cycle fatigue stresses during the reactor operation. The preliminary work on the Ti-modified AISI 316L stainless steel (see [4] and 316(ST-1) below) indicates that the effect of radiation damage on the fatigue life N_f in the ϵ_t -controlled tests at 333 K, here elongation in N_f is much larger under irradiation than after irradiation, where ϵ_t is the total strain amplitude. The further study on the various modified-316L stainless steels [5,6] shows that the fatigue hardening mainly governs N_f of the solution treated specimens and *in situ* radiation damage or thermal pulse modifies the fatigue hardening process. That is, the elongation in N_f observed for 316(ST-1) is mainly due to enhancement of the fatigue induced precipitation under irradiation. After these results, we started the ϵ_p -controlled tests on the 316L stainless steels with the decreased carbon contents, where ϵ_p is the constituent plastic strain amplitude in ϵ_t and the dislocation behavior is expected to be pursued more quantitatively in the ϵ_p -controlled tests than in the ϵ_t -controlled tests. The preliminary tests at 333 K on 316SI (see Table 1) [6] indicate that N_f shows elongation for the thermal-pulse (ΔT) tests (the TP-tests) with ΔT below 10 K (see Fig.1) and shortening under *in situ* radiation damage (see Fig.2), where the effect of *in situ* radiation damage is found after the subtraction of the results of the TP-tests from the results of the pulse 20 MeV-proton irradiation tests (the pulse-PI-tests). To pursue the further insight into the fatigue process in 316SI, we carried out the TP-tests at 300 K with the increased ΔT of about 100 K and the continuous 20 MeV-proton irradiation tests (the PI-tests) at 403 K, because the effects of *in situ* radiation damage or thermal pulse on the fatigue hardening at 403 K have been compiled in [6] and the specimen temperature attained during thermal-pulses is about 400 K. Further for the no-loading tests (the NL-tests) without thermal-pulse nor proton irradiation, the fatigue properties at 300 K are practically the same to those at 333 K which have also been compiled in [6].

2. EXPERIMENTAL

Specimens were cut from the rolled sheets of 316SI with the chemical composition shown in Table 1. After mechanical polishing, specimens were annealed at 1370 K for 3.6 ks in a vacuum of 2×10^{-4} Pa and then quenched into water. As quenched specimens were electrolytically polished, in order to remove the surface layer of about 0.05 mm and to smooth out the surfaces, and then annealed again at 573 K for 36 ks in a vacuum of 2×10^{-4} Pa to degas hydrogen picked up during the electrolytical polishing. The grain size was typically 0.05 mm. The fatigue tests were carried out using the flexural vibrations of a U-shaped reed specimen, where the middle of the U-shape forms a composite reed with thickness of 0.12 mm and a gage length of 16 mm and two open ends of the U-shape are thick for clamping. Excitation of the flexural vibrations was made electromagnetically and the fatigue tests were conducted in the atmosphere of 10^2 Pa He to suppress a heat-up of a specimen (see [4,6] for details). Period P of the resonant flexural vibrations of a specimen in the elastic range will be referred to as P_0 below, where $1/P_0$ here is about 200 Hz. The fatigue tests were started by an increase in ϵ_t beyond micro yielding. For the ϵ_t -controlled tests (see Fig. 1 in [5]), P shows a strong increase during several 10^3 cycles after the increase in ϵ_t , and then turns to decrease due to the fatigue hardening, where ϵ_p is proportional to $(P-P_0)/P_0$. With increasing fatigue cycles, the fatigue hardening tends towards saturation and at the fatigue cycles of about $0.9N_f$, P turns to increase reflecting a decrease in the cross-sectional area of the specimen due to the start of crack propagation. That is, the most of N_f is governed by the dislocation behaviors. In the present ϵ_p -controlled tests, P was held at a given value P_h by adjusting ϵ_t during the fatigue hardening and then, after the saturation of the fatigue hardening, ϵ_t was kept at an attained ϵ_t . For the TP-tests, a thermal pulse ΔT for 10 s was given every 30 s using halogen lamp light, where the specimen temperature rises to 90 % of the ultimate ΔT in a few seconds. For the PI-tests or the pulse-PI-tests, irradiation was made using 20 MeV protons of 0.02-0.03 A/m² from the tandem accelerator at University of Tsukuba, where the radiation damage rate estimated is about 1×10^{-7} dpa/s for 0.025 A/m².

Table 1. Chemical composition of the material used (mass%)

Material	C	Si	Mn	P	S	Cr	Ni	Mo	N
316SI*	0.005	1.38	1.35	0.014	0.009	17.32	13.59	2.34	0.001

* The modified AISI 316L stainless steel, where the code indicated is used only to refer the material with the given chemical composition.

3. RESULTS

Figure 1 shows the $(P_h - P_0)/P_0$ vs. N_f data observed for the NL-tests at 300 K here or at 333 K [6], the TP-tests at 333 K with ΔT below 10 K [6] and the TP-tests at 300 K with $\Delta T = 70 \sim 130$ K, where TP with ΔT below 10 K brings about elongation of N_f but TP with increased ΔT of $70 \sim 130$ K gives rise to shortening of N_f . Figure 2 is a redrawing of Fig. 8 in [6], where the $(P_h - P_0)/P_0$ vs. N_f data observed for the pulse-PI-tests at 333 K are compared with the results for the TP-tests at 333 K with ΔT below 10 K. As mentioned in the section 1, the *in situ* radiation damage at 333 K causes shortening of N_f , especially in the low $(P_h - P_0)/P_0$ range. Figure 3 shows the $(P_h - P_0)/P_0$ vs. N_f data observed for the NL-tests at 403 K and the PI-tests at 403 K, where a redrawing of the data of the NL-tests at 300 K or 333 K is also given. The $(P_h - P_0)/P_0$ vs. N_f data observed for the NL-tests at 403 K

Figure 1. $(P_h - P_0)/P_0$ vs. N_f observed for the fatigue tests at 300-330 K (see text). ●, 1=NL-tests; ○, 2=TP-tests with ΔT below 10 K; □, 3=TP-tests with $\Delta T = 70 \sim 130$ K. The lines 1 to 3 are tentatively fitted to the data.

show good agreement with those observed for the NL-tests at 300 K or 333 K. The outline of the effects of *in situ* radiation damage on N_f at 403 K is very similar to that of *in situ* radiation damage on N_f at 333 K. The results of the scanning electron microscope (SEM) observation of the specimen surfaces will be mentioned in the section 4.

4. DISCUSSION

A scatter in the present $(P_h - P_0)/P_0$ vs. N_f data is relatively large due to the limited thickness of the present specimens, in order to allow the complete penetration of 20 MeV protons. That is, the limited number of grains along the thickness, two or three here, are mainly responsible for the scatter in N_f , presumably reflecting the crystallographic dependence of the dislocation behavior. However, the outline of the $(P_h - P_0)/P_0$ vs. N_f data is expected to represent that of polycrystalline specimens. It is noted that the line 1 for the NL-tests shown in Fig.1 or Fig.3 falls on the extension from the ϵ_p vs. N_f data reported for the low cycle fatigue tests of the 316L stainless steel [7].

We now discuss the results of the various tests at 300 K or 333 K. As mentioned in Fig.3, the $(P_h - P_0)/P_0$ vs. N_f data for the NL-tests at 403 K show good agreement with those for the NL-tests at 300 K or 333 K, being in agreement with that N_f of the solution-treated 316L stainless steel is not sensitive to the test temperatures [7]. It is noted that the heart of the TP-tests is the abrupt change in the specimen temperatures, i.e., gradual changes in the specimen temperatures hardly modify N_f . For the TP-tests at 333 K with ΔT below 10 K shown in Fig.1, since the fatigue hardening process for the isothermal tests at 333+10 K is practically the same to that at 333 K, the elongation in N_f for the TP-tests should be due to dislocation rearrangements during TPs, suggesting an increase in the areal density of persistent slip bands (PSBs) because of a decrease in the plastic strain per PSB. In contrast, the results of the pulse-PI-tests at 333 K shown in Fig.2 suggest that an introduction of obstacles to dislocation motions, irradiation induced defects here, gives rise to a decrease of PSBs, resulting in the shortening in N_f . For the TP-tests at 300 K with $\Delta T=70\sim 130$ K, one can expect both dislocation rearrangements and an introduction of obstacles, the fatigue induced precipitates here (see Fig.6 in [6]), during TPs, and the results of N_f suggest that the effect of the latter is predominant.

The SEM observation indicates that the above view is the case: In the present test conditions, the specimens failed through the transgranular mode and striations are observed on the fracture surface. On the surfaces of the specimens subjected to NL-tests at 300 K or 333 K, PSBs are observed for about 20 % of constituent grains and no visible slip bands for the remaining grains. Referring to the specimens subjected to the NL-tests at 300 K or 333 K, the fraction of constituent grains which show PSBs is about twice for the specimens subjected to the TP-test at 333 K with ΔT below 10 K, about one half for the specimens subjected to the pulse-PI-tests at 333 K and much smaller for the TP-tests at 300 K with $\Delta T=70\sim 130$ K, respectively. That is, the close correlation between the PSBs and N_f evolutions is found.

For the tests at 403 K, both enhanced dislocation motions and the fatigue induced precipitation are expected for the NL-tests. The $(P_h - P_0)/P_0$ vs. N_f data for the NL-tests, however, indicate that the fatigue induced precipitation plays the minor role on the fatigue process during the NL-tests at 403 K, predicting that for the PI-tests, irradiation induced lattice defects can serve as obstacles to dislocation motions and the probable irradiation dispersion of the fatigue induced precipitates [6] plays the minor role. The results of N_f found for the PI-tests at 403 K support this view. The SEM observation for the specimens subjected to the PI-tests at 403 K will be reported elsewhere after attenuation of radio activity of the specimens.

Figure 2. $(P_h - P_0)/P_0$ vs. N_f observed for the tests at 333 K. ○, 2=TP-tests with ΔT below 10 K; ▲, 4=pulse-PI-tests. The lines 2 and 4 are tentatively fitted to the data. See text.

The present results suggests that the dislocation interaction plays the major role on the high cycle fatigue properties of 316SI. As a matter of fact, the work hardening exponent n [8] estimated from the line 1 shown in Fig. 1 is as high as $n=1$ (see [6] for details). For the effects of *in situ* radiation damage on N_f , 316SI reveals shortening of N_f at 333 K or 403 K. On the other hand, an opposite and an intermediate effects of *in situ* radiation damage on N_f are observed for various 316L stainless steels: Elongation of N_f at 333 K is found for 316(ST-1) in [4] in which the work hardening exponent n is as low as about 0.2 and fatigue induced precipitates can be observed as black dots in TEM observation [5]. Shortening of N_f for the higher $(P_h - P_0)/P_0$ range and elongation of N_f for the lower $(P_h - P_0)/P_0$ range are observed for 316F in [5] in which the work hardening exponent n is about 0.33 and the fatigue induced precipitation is intermediate between 316(ST-1) and 316SI. That is, the systematic relationship between the effect of *in situ* radiation damage on N_f and the fatigue hardening process is found. To clarify this issue, the further work is now in progress.

5. CONCLUSION

The high-cycle fatigue tests of 316SI were carried out between 300 and 403 K. The no loading tests indicate that the dislocation interaction governs the fatigue properties of 316SI. The *in situ* irradiation tests and the *in situ* thermal-pulse tests indicate that dislocation rearrangements due to thermal-pulse give rise to an increase in the areal density of PSBs, resulting in elongation of the fatigue life N_f and an introduction of obstacles to dislocation motions, irradiation induced lattice defects or the fatigue induced precipitates, brings about a decrease in the areal density of PSBs, giving shortening of N_f . In contrast, as reported in the previous work, for the 316L stainless steels in which the fatigue induced precipitation governs the high-cycle fatigue properties, an introduction of obstacles of dislocation motions gives rise to elongation of N_f .

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Figure 3. $(P_h - P_0)/P_0$ vs. N_f . ●, 1=NL-tests at 300-333 K (see text); ■=NL-tests at 403 K; □, 5=PI-tests at 403 K. The lines 1 and 5 are tentatively fitted to the data.