



# High-cycle fatigue tests of modified 316 stainless steels under 20 MeV proton irradiation and thermal pulses

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## Abstract

In situ radiation damage and thermal pulses considerably modify the high cycle fatigue property of solution treated specimens of the modified 316L stainless steels, where the observed effects show a characteristic relationship with the chemical composition of the specimens. For the specimens which show a low work-hardening exponent, the fatigue life elongates under irradiation. In contrast, the fatigue life of the specimens which show a high work-hardening exponent is shortened under irradiation. A probable dislocation–defect interaction responsible for these effects is discussed. © 1998 Elsevier Science B.V. All rights reserved.

## 1. Introduction

The first wall of a fusion reactor is planned to be subjected to thermal stresses and 14 MeV neutron irradiation simultaneously. On the low cycle fatigue property, an effect of in situ radiation damage is negligible because the expected damage rate is too low to modify a violent dynamic defect process under the low cycle fatigue strains or stresses. On the other hand, the high cycle fatigue property is expected to be modified by in situ radiation damage because a dynamic defect process under the high cycle fatigue strains or stresses can be comparable with the damage rate (e.g. [1–8]). Since most of the structural reactor materials are expected to be subjected to the high cycle fatigue strains or stresses during the reactor operation, understanding of the effects of in situ radiation damage and in situ thermal pulse on the high fatigue property is important.

The preliminary high cycle fatigue tests on the Ti-modified AISI 316L stainless steel (316(ST–1) [4], seen in Table 1 and Fig. 4(a)) indicate that the effect of radiation damage on the fatigue life  $N_f$  in the  $\varepsilon_t$ -controlled tests at 333 K is much larger under irradiation than after irradiation, where  $\varepsilon_t$  is the total strain amplitude. The

further high cycle fatigue tests of various modified-316L stainless steels (316F and 316SI [9–11], seen in Table 1 and Fig. 4(b)) indicate that for the solution treated specimens the chemical composition mainly governs the fatigue hardening process and in situ radiation damage modifies the fatigue hardening process, resulting in the characteristic relationship between the effects of in situ radiation damage on the fatigue life  $N_f$  and the chemical composition of the specimens. In the present paper, these results will be reported together with further fatigue tests on 316SI specimens.

## 2. Experimental

Specimens were cut from the rolled sheets of 316SI with the chemical composition shown in Table 1. The chemical composition of 316(ST–1) reported in Ref. [4] and that of 316F reported in Ref. [9] are also compiled in Table 1, and the present results will be discussed together with the previous results on 316(ST–1) and 316F specimens. As will be shown in Fig. 4(a) and (b), the work hardening exponent  $n$  [12] is the highest in 316SI, the lowest in 316(ST–1) and intermediate in 316F. After mechanical polishing, 316SI specimens were annealed at 1370 K for 3.6 ks in a vacuum of  $2 \times 10^{-4}$  Pa and then quenched into water. As quenched specimens were electrolytically polished in order to remove

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

Material	C	Si	Mn	P	S	Cr	Ni	Mo	N	Ti
316SI <sup>a</sup>	0.005	1.38	1.35	0.014	0.009	17.32	13.59	2.34	0.001	–
316F <sup>a</sup>	0.038	0.04	0.23	0.003	0.002	16.77	13.95	2.32	0.011	–
316(ST-1) <sup>a</sup>	0.077	1.00	1.81	0.029	< 0.003	16.20	13.60	2.46	–	0.089

<sup>a</sup> Modified AISI 316L stainless steels, where the codes indicated are used only to refer to the specimens with the given chemical composition.

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  $2 \times 10^{-4}$  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 Refs. [4,10] 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  $1/P_0$  is about 200 Hz. The fatigue tests were started by an increase in the total strain amplitude  $\varepsilon_t$  beyond microyielding. For the  $\varepsilon_t$ -controlled tests (see Ref. [4] and Fig. 4(a) for 316(ST-1)),  $P$  shows a strong increase during several thousands cycles after the increase in  $\varepsilon_t$ , and then starts to decrease due to the fatigue hardening, where the plastic strain amplitude  $\varepsilon_p$  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  $0.9N_f$ ,  $P$  starts to increase reflecting a decrease in the cross-sectional area of the specimen due to the start of crack propagation. That is, for smooth specimens, most of  $N_f$  is governed by the dislocation behavior. In the present plastic strain amplitude,  $\varepsilon_p$ , controlled tests,  $P$  was held at a given value  $P_h$  by adjusting the total strain amplitude,  $\varepsilon_t$ , during the fatigue hardening and then, after the saturation of the fatigue hardening,  $\varepsilon_t$  was kept at an attained  $\varepsilon_t$ . The fatigue tests without proton irradiation or thermal pulses will be referred to as the no-load tests (the NL-tests), hereafter. For the thermal pulse tests (the TP-tests), a thermal pulse  $\Delta T$  of 10 s was given every 30 s using a halogen lamp light, where the specimen temperature rises to 90% of the ultimate  $\Delta T$  in a few seconds. For the proton irradiation tests (the PI-tests) or the pulse-PI-tests, irradiation was made using 20 MeV protons of 0.02–0.03 A/m<sup>2</sup> from the tandem accelerator at the University of Tsukuba, where the radiation damage rate estimated is about  $1 \times 10^{-7}$  dpa/s for 0.025 A/m<sup>2</sup>. The present fatigue tests were conducted at 300 and 403 K.

### 3. Results

Fig. 1 shows the  $(P_h - P_0)/P_0$  vs.  $N_f$  data observed for 316SI specimens; the NL-tests at 300 K [11] and 333 K [10], the TP-tests at 333 K with  $\Delta T$  below 10 K [10] and the TP-tests at 300 K with  $\Delta T = 70$ –130 K [11]. The low cycle NL-tests on 316L stainless steel reported in Ref. [13] indicate that the dependence of  $N_f$  on  $\varepsilon_p$  is insensitive to the testing temperatures. In the present high cycle NL-tests on 316SI specimens, as will be shown in Fig. 3, the dependence of  $N_f$  on  $(P_h - P_0)/P_0$  is insensitive to the testing temperatures. Therefore, the  $(P_h - P_0)/P_0$  vs.  $N_f$  data for the NL-tests at 300 K and those at 333 K will be put into one group as the NL-tests at 300–333 K, hereafter. As seen in Fig. 1, the TP-tests with  $\Delta T$  below 10 K brings about elongation of  $N_f$  but the TP-tests with increased  $\Delta T$  of 70–130 K gives rise to shortening of  $N_f$ . The SEM observation of the specimen surfaces (not shown here) indicates that the areal density of slip bands observed after the TP-tests with  $\Delta T$  below 10 K is higher than, and that after the TP-tests with  $\Delta T$  of 70–130 K is lower than after the NL-tests, respectively.

Fig. 2 shows the  $(P_h - P_0)/P_0$  vs.  $N_f$  data observed for the pulse-PI-tests at 333 K on 316SI specimens [10], which are compared with the  $(P_h - P_0)/P_0$  vs.  $N_f$  data for the TP-tests with  $\Delta T$  below 10 K shown in Fig. 1 because the present pulse proton irradiation was accompanied by a thermal pulse with  $\Delta T$  below 10 K. It is seen in Fig. 2 that for 316SI specimens the in situ radiation damage at 333 K causes shortening of  $N_f$ , especially in the low  $(P_h - P_0)/P_0$  range around  $(P_h - P_0)/P_0 = 0.12$ . The SEM observation of the specimen surfaces (not shown here) indicates that the areal density of slip bands observed after the pulse-PI-tests is lesser than after the TP-tests with  $\Delta T$  below 10 K, especially for the low  $(P_h - P_0)/P_0$  range around  $(P_h - P_0)/P_0 = 0.12$ .

Fig. 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 data at 403 K on 316SI specimens, where a redrawing of the  $(P_h - P_0)/P_0$  vs.  $N_f$  data for the NL-tests at 300–333 K are also given. Although the areal density of slip bands observed after the NL-tests at 403 K was much higher than after the NL-tests at 300–333 K (not shown here), the dependence of  $N_f$  on  $(P_h - P_0)/P_0$  found for the NL-tests at 403 K was very similar to that for the NL-test at 300–333 K.

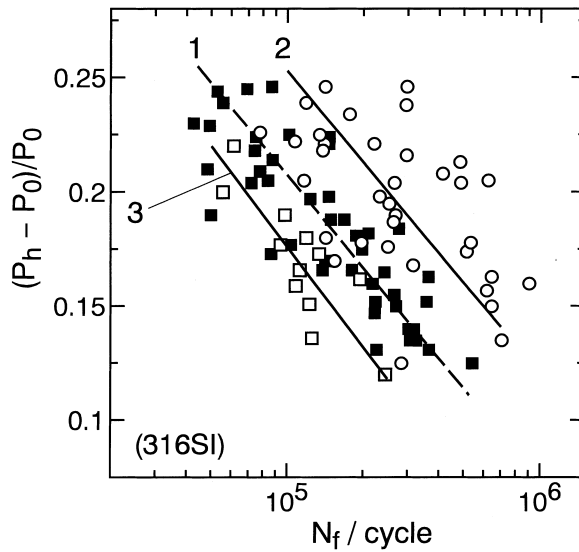


Fig. 1.  $(P_h - P_0)/P_0$  vs.  $N_f$  observed for the high-cycle fatigue tests at 300–333 K for 316SI specimens (see text): ■,1 = NL-tests; ○,2 = TP-tests with  $\Delta T$  below 10 K; □,3 = TP-tests with  $\Delta T = 70$ –130 K. The lines 1–3 are tentatively fitted to the data.

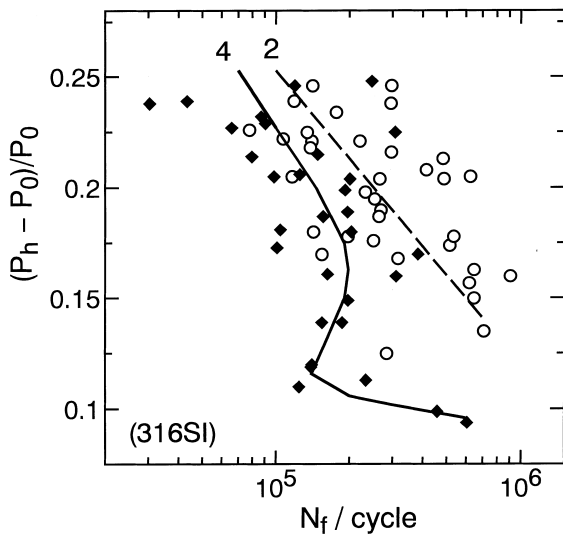


Fig. 2.  $(P_h - P_0)/P_0$  vs.  $N_f$  for the pulse-PI-tests at 333 K of 316SI specimens (◆,4) is compared with that for the TP-tests with  $\Delta T$  below 10 K shown in Fig. 1 (○,2). The lines 2 and 4 are tentatively fitted to the data, see [10].

The effect of the in situ radiation damage on the dependence of  $N_f$  on  $(P_h - P_0)/P_0$  at 403 K is very similar to that found at 333 K, i.e. the in situ radiation damage causes shortening of  $N_f$ , especially in the low  $(P_h - P_0)/P_0$  range around  $(P_h - P_0)/P_0 = 0.14$ . The results for the SEM observation of the specimen surfaces after the PI-tests at 403 K (not shown here) are also very similar to those mentioned for the pulse-PI tests, i.e. the areal density of slip bands observed after the PI-tests is lower

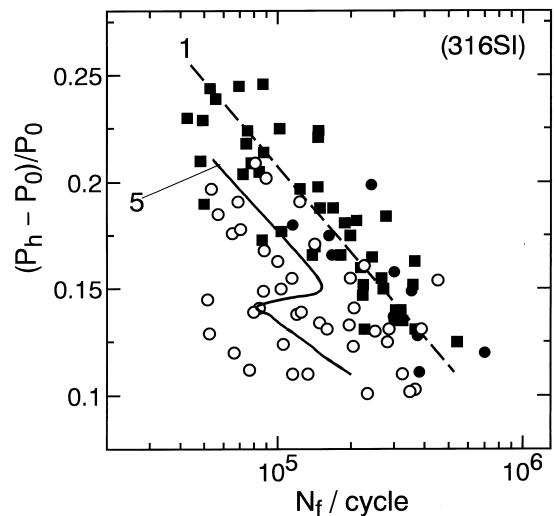


Fig. 3.  $(P_h - P_0)/P_0$  vs.  $N_f$  for the PI-tests at 403 K of 316SI specimens (○,5) is compared with that for the NL-tests at 403 K of 316SI specimens (●,1), where  $(P_h - P_0)/P_0$  vs.  $N_f$  for the NL-tests at 300–333 K (■,1) is also plotted. The lines 1 and 5 are tentatively fitted to the data.

than after the NL-tests, especially for the low  $(P_h - P_0)/P_0$  range around  $(P_h - P_0)/P_0 = 0.14$ .

#### 4. Discussion

Fig. 4(a) shows the  $\epsilon_t$  vs.  $N_f$  data for the NL-tests and the PI-tests at 333 K on 316(ST - 1) specimens reported in Ref. [4]. Fig. 4(b) shows the  $(P_h - P_0)/P_0$  vs.  $N_f$  data

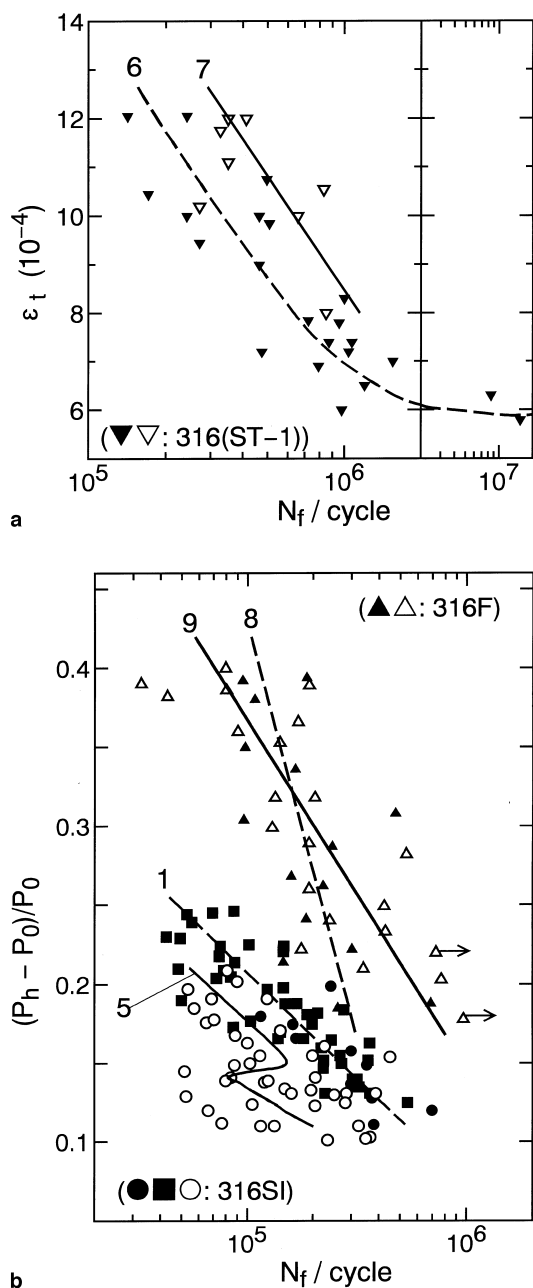


Fig. 4. (a) For 316(ST-1) specimens,  $\epsilon_t$  vs.  $N_f$  for the PI-tests at 333 K of ( $\nabla$ ,7) is compared with that for the NL-tests ( $\blacktriangledown$ ,6). The lines 6 and 7 are tentatively fitted to the data, see [4]. (b)  $(P_h - P_0)/P_0$  vs.  $N_f$  for the PI-tests ( $\triangle$ ,9) and that for the NL-tests ( $\blacktriangle$ ,8) at 333 K of 316F specimens [9] are compared with  $(P_h - P_0)/P_0$  vs.  $N_f$  for the PI-tests and the NL-tests at 403 K of 316SI specimens shown in Fig. 3.

for the NL-tests and the PI-tests at 333 K on 316F specimens reported in Ref. [9], where a redrawing of the  $(P_h - P_0)/P_0$  vs.  $N_f$  data for 316SI specimens shown in

Fig. 3 is also given. The work hardening exponent  $n$  [12] estimated from the  $N_f$  data for the NL-tests is about 1 for 316SI, about 0.3 for 316F and about 0.2 for 316(ST-1), respectively. In 316(ST-1) specimens, the fatigue hardening observed in the high cycle fatigue tests is mainly governed by the fatigue enhanced precipitation [9], resulting in a low value of the work hardening exponent. Further the precipitates are too large to be dispersed by 20 MeV proton irradiation [9], suggesting that the elongation of  $N_f$  in the  $\epsilon_t$ -controlled PI-tests observed in 316(ST-1) specimens reflects enhancement of the fatigue hardening under proton irradiation which causes a decrease in the constituent plastic strain amplitude in the  $\epsilon_t$ -controlled tests.

In contrast, for 316SI specimens, the work hardening exponent is as high as 1 and on the specimen surface after the fatigue tests well-developed slip bands are observed with a high areal density. The morphology of slip bands in 316SI is similar to that of persistent slip bands (PSBs) in annealed copper specimens after the NL-tests, except that the width and height of a slip band in 316SI specimens are considerably less than those of a PSB in copper specimens. It is known that for the  $\epsilon_p$ -controlled tests, an increase (a decrease) in the areal density of PSBs brings about a decrease (an increase) in the local- $\epsilon_p$  in PSBs, resulting in elongation (shortening) of the fatigue life. The close relationship between the surface morphology and the  $(P_h - P_0)/P_0$  vs.  $N_f$  data observed in 316SI specimens suggests that an effect of proton irradiation or thermal-pulses on PSBs governs their effects on the fatigue life. For the elongation of  $N_f$  in the TP-tests with  $\Delta T$  of 10 K, we suppose that the local- $\epsilon_p$  in PSBs was not equal among PSBs and the PSBs with a lower local- $\epsilon_p$  could preferentially be activated under thermal pulses with  $\Delta T$  of 10 K through dislocation rearrangements. It is noted that the heart of the TP-tests is an abrupt change in specimen temperatures, i.e. gradual changes in the specimen temperatures hardly modify  $N_f$ . In contrast, for the TP-tests at 300 K with  $\Delta T = 70$ –130 K, one can expect both dislocation rearrangements and an introduction of the fatigue induced precipitates (see Fig. 6 in Ref. [10]) during thermal pulses. The decrease in  $N_f$  for the TP-tests with  $\Delta T = 70$ –130 K shown in Fig. 1 suggests that the effect of the fatigue induced precipitation is predominant for the TP-tests. Both the shortening of  $N_f$  in 316SI specimens due to in situ radiation damage at 333 K shown in Fig. 2 and that at 403 K shown in Fig. 3 are surmised to be related to irradiation induced hardening. The strong shortening of  $N_f$  due to in situ radiation damage around  $(P_h - P_0)/P_0 = 0.12$  at 333 K shown in Fig. 2 and that around  $(P_h - P_0)/P_0 = 0.14$  at 403 K shown in Fig. 3 possibly indicate that the irradiation induced precipitation takes place in 316SI, where the precipitates can be dispersed by dislocation motions when the plastic strain amplitude during the fatigue tests is increased.

The work hardening exponent and the effect of in situ radiation damage on  $N_f$  in 316F shown in Fig. 4(b) are intermediate between those in 316SI and in 316(ST – 1). These results indicate the close relationship between the effect of in situ radiation damage on the fatigue life and the chemical composition. To clarify this issue, further work is now in progress.

## 5. Conclusion

From the high cycle fatigue tests on solution treated smooth specimens of modified 316L stainless steels, the close relationship between the effect of in situ radiation damage on the fatigue life and the chemical composition of the specimens is found. The present work demonstrates that understanding of the effect of in situ radiation damage on the fatigue property is important for the first wall materials.

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