Change in Mechanical Properties by High-Cycle Loading up to Gigacycle for 316L stainless steel

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Received April 21, 2019

At the J-PARC pulsed spallation neutron source, a target vessel made of 316L stainless steel suffers radiation damage under high proton and neutron radiation environment. In addition to the radiation damage, the target vessel suffers cyclic impact stress caused by the proton beam-induced pressure waves. We have observed cyclic hardening/softening phenomena during investigating gigacycle (10^9 cycles) fatigue strengths of solution annealed and cold-worked 316L by an ultrasonic fatigue test. In this study, to elucidate the cyclic hardening/softening phenomena up to the gigacycle loading, dislocation densities of the cyclic-loaded specimens by the ultrasonic fatigue method were measured using neutron diffraction method. The results showed that the dislocation of the cold-worked 316L was rearrangement and re-accumulate by cyclic loading whereas the dislocation densities of the solution annealed 316L was increased with increasing in the number of loading cycles. For both cases, change in the dislocation densities by cyclic loading were well correlated with the change of mechanical properties; hardness, residual strength.

KEYWORDS: gigacycle fatigue, cyclic hardening, cyclic softening, dislocation density, neutron diffraction

1. Introduction

A pulsed spallation neutron source, which generates neutrons by injecting high-intensity 3 GeV pulsed proton beams into liquid mercury, is installed in the Japan Proton Accelerator Research Complex (J-PARC). A target vessel which contains liquid mercury and made of 316L stainless steels, suffers proton and neutron radiation environment. The target vessel suffers cyclic impact stress at a strain rate of 50 s^{-1} caused by proton beam-induced pressure waves in addition to exposure the radiation environment [1]. Since the designed lifetime of the vessel is 5000 hours at the rated beam power of J-PARC (1 MW at 25 Hz), the vessel endures approximately 10^9 cyclic loading during operation.

In the region of gigacycle (10^9 cycles) loading, it is known that fatigue failure tends to be occurred below the fatigue limit which is decided based on the conventional fatigue tests up to 10^7 cycles. Internal fracture originating from internal defects such as inclusions, so-called fish eye fracture, occurs in the gigacycle region for high-strength steels [2]. In contrast, for the austenitic stainless steels, it was reported that the fatigue failure originating from the surface to be dominant in the solution-annealed 316L SS [3, 4].

In the previous study, the authors investigated the gigacycle fatigue strength of 316L SS by an ultrasonic fatigue testing method. The result showed that the fatigue failure due to surface cracking occurred when the number of cycle higher than 10^7, and obviously fatigue limit was not recognized up to 10^9 cycles [4]. Furthermore, cyclic hardening represented by the change of hardness and the residual strength of a solution annealed 316L SS was observed. By contrast, in the cases of 10% and...
20% cold-worked 316L SS for simulating the accumulation of dislocation by the irradiation, cyclic softening seems to be caused by the dislocation annihilation before \(10^6\) cycles and cyclic hardening appeared above \(10^7\) cycles [5]. The cyclic hardening and softening phenomena of 316L SS under conventional fatigue conditions are reported. It was reported that the cyclic softening observed in the initial stage of fatigue and subsequent cyclic hardening are depending on the temperature [6]. Furthermore, the microstructure change by cyclic loading is well correlated with the fatigue life [7].

In this study, mechanical properties change by cyclic loading up to gigacycle under high-strain rate for the solution annealed (SA) 316L stainless steel and cold-worked (CW) 316L stainless steel were investigated by focusing on the change in the dislocation density. The dislocation density of the cyclic loaded specimens were measured using the neutron diffraction method, and correlation between the dislocation density and the mechanical properties were investigated.

2. Experiment

2.1 Preparation of specimens

Type 316L stainless steel, which is the structural material of the mercury target vessel, was used for the experiment. The steel was solution annealed (SA). Some of the SA steels were cold-rolled up to 10% (10% CW) and 20% (20% CW) reduction in thickness for simulating irradiation-hardened states by increasing the dislocation density. Figure 1 shows the schematic drawing of fatigue test specimen. The longitudinal direction of the specimen was set perpendicular to the rolling direction. The hour-glass shape of specimen is designed for obtaining the resonance frequency of 20 kHz to apply the cyclic loading. The ultimate tensile strengths of SA, 10% CW and 20% CW steels before cyclic loading were 652 MPa, 775 MPa and 882 MPa, respectively.

2.2 Cyclic loading up to gigacycle

Cyclic loading up to the gigacycle (\(10^9\) cycles) was applied using an ultrasonic fatigue testing system (Shimadzu, USF-2000). The specimen fixed on the end of an ultrasonic horn was loaded in tension and compression (Stress ratio \(R = -1\)) at 20 kHz [4]. The strain rate was approximately \(10^2\) s\(^{-1}\). The cyclic loading tests were interrupted at arbitrary numbers of loading cycles which were lower than the fatigue limits. Table I shows the conditions of the stress amplitudes and the interrupted numbers of loading cycles for three steels.

2.3 Mechanical properties measurements

The quasi-static tensile tests were performed to measure the ultimate tensile strength as the residual strength using a part of the cyclic stress-loaded specimens. The parallel part of the specimen was gripped and tensile stress was applied at the constant crosshead speed of 5 mm/min using a hydraulic testing machine (Shimadzu, 4890-servopulser).

| Material | Stress amplitude | Number of loading cycles |
|----------|------------------|--------------------------|
| SA 316L  | 190 MPa          | 0, 5\(\times\)10\(^6\), 5\(\times\)10\(^7\), 10\(^8\), 5\(\times\)10\(^9\), 10\(^9\), 2\(\times\)10\(^8\), 5\(\times\)10\(^9\), 10\(^9\) |
| 10% CW 316L | 320 MPa       | 0, 10\(^7\), 10\(^8\), 10\(^9\), 2\(\times\)10\(^7\), 2\(\times\)10\(^8\), 5\(\times\)10\(^8\), 10\(^9\) |
| 20% CW 316L | 390 MPa       | 0, 5\(\times\)10\(^7\), 5\(\times\)10\(^8\), 10\(^8\), 5\(\times\)10\(^9\), 10\(^9\), 2\(\times\)10\(^8\), 5\(\times\)10\(^9\), 10\(^9\) |
Some parts of cyclic loaded specimens were cut in a longitudinal direction and buff-polished for the hardness measurement. Load and depth curves were measured using the microhardness tester (Shimadzu, DUH-W201S) with the Berkovich tip to investigate the change in the microhardness by cyclic loading. The universal hardness, $H_u$, was evaluated using the following equation:

$$H_u = \frac{L_{max}}{26.43D_{max}^2}$$

(1)

where $L_{max}$ is the maximum load applied to the specimen and $D_{max}$ is the maximum depth of indent [8]. The maximum load and loading rate were 29.4 mN and 1.47 mN/s, respectively.

### 2.4 Dislocation density measurement

The dislocation densities in cyclic loaded specimens were measured by neutron diffraction experiments using the Engineering Materials Diffractometer TAKUMI at BL19 of the Materials and Life science experimental Facility (MLF) in J-PARC [11]. The measurement enables to investigate the characteristics and the behaviors of the microstructure inside bulk materials using the high-resolution and high-intensity pulsed neutron diffraction. Figure 2 shows the schematic drawing of the measurement setup. Longitudinal direction of specimen was aligned 45° to the incident neutron beam, and the diffracted neutrons were collected using a pair of 90° scattering detectors. Gauge volume of $5 \times 5 \times 5$ mm$^3$ was used, which is determined by the incident beam slit and the radial collimator [12]. A profile analysis to evaluate dislocation information was conducted using the Convolutional Multiple Whole Profile (CMWP) procedure [13, 14].

### 3. Results and discussions

#### 3.1 Mechanical properties change

Figure 3 shows the change in the residual strength as a function of the number of loading cycle for SA [5], 10% CW [5], and 20% CW steels. In the case of cyclic loading test of SA steel, up to $10^8$ cycles the residual strength is hardly changed, and beyond $10^8$ cycles the strength seems to increase. In contrast, for CW steels, the residual strengths obviously decreased by cyclic loading around $10^6$ cycles, but they gradually recovered after that, regardless of the ratio of cold-working.

Figure 4 shows the change in the universal hardness as a function of the number of loading cycles. It is noted that the universal hardness plotted in Fig. 4 is the averaged value of 5 data measurement in the same condition. It can be seen that the hardness gradually increases with the number of loading cycle for SA. In the cases of 10% and 20% CW steels, decreases in the hardness that suggest the occurrence of cyclic softening were observed at cycles below $10^6$. By contrast, above $10^6$ cycles, cyclic hardening is seen for both the 10% and 20% CW steels. The similar trends were observed also in the hardness and residual strength change as results from the previous cyclic loading with different stress amplitudes [5].

#### 3.2 Dislocation densities

The observed and CMWP-fitted neutron diffraction profiles before cyclic loading are typically shown in Fig. 5. The profiles are plotted as a function of $K$ for the horizontal axis. $K = 1/d$, where $d$
is the lattice spacing. It is noted that the numbers in the figure denotes the mirror index, and A and M denotes austenite and martensite, respectively. SA steel is the austenitic stainless steel with the face-centered cubic (FCC) crystal structure, however, it can be seen that the martensite structure with the body-centered cubic (BCC) which might be formed by machining during the specimen preparation is observed in the specimen before cyclic-loading. Figure 6 shows the intensity of CMWP-fitted neutron diffraction profiles for [200] plane. It is known that the width of the diffraction profile tends to be correlated with the dislocation density. The full width at half maximum (FWHM) of profiles for SA, 10%, and 20% steels are 0.017, 0.023, 0.030 nm$^{-1}$, respectively, showing that the values increase with the increasing in the degree of cold-working.

Figure 7 shows the change in the dislocation density as a function of the number of loading cycle. In the case of SA steel, although the values close to the detectable limit, the dislocation density increase with the number of loading cycle. By contrast, in the case of 10% CW steel, the dislocation density slightly decreases during the cyclic test up to $10^8$ cycles, then increases at $10^9$ cycles. In the case of 20% CW steel, the dislocation density decreases by applying cyclic load at the beginning and tends to increased again for the cycles higher than $10^6$. The difference in the inflection point of the dislocation density change by cyclic loading between 10% and 20% CW steels is caused by the difference of pre-existed dislocation density of each specimen. The trend of change in the dislocation...
density as a function of the number of loading cycle is relatively well correlated with the hardness change by cyclic loading.

Figure 8 shows the change in the dislocation arrangement parameter, $M^*$, as a function of the number of loading cycle. When the value of $M^*$ is much larger than 1.0, the material has the random arrangement of dislocations. By contrast, the material has a dipole arrangement or a strongly correlated arrangement of dislocations in the case of the value of $M^*$ is lower than 1.0 [13, 15]. In the case of SA steel, $M^*$ was kept much larger than 1.0 regardless of the number of loading cycle. Since the reliability of the quantitative values for SA steel are insufficient, the values are not shown in the figure. By contrast, for the 10% and 20% CW steels, $M^*$ values before cyclic loading are already lower than 1.0 for both steels, and the value is smaller in 20% CW steel than 10% CW steel due to the larger cold-working reduction ratio. These results suggest that the steel seems to have a cell structure by cold-rolling, and the crystallite size is decreased by applying the large strain. Furthermore, it can be seen from Fig. 8, $M^*$ of 10% CW steel slightly decreases with increasing the number of loading cycle. In the case of 20% CW steel $M^*$ increases by applying cyclic loading at the beginning, and then gradually decreased with increasing the loading cycle. These trends suggest that the rearrangement of the dislocation might be occurred and formed high-dense cell structure by cyclic loading.

In the case of the mercury target vessel for the spallation neutron source, dislocation might be accumulated heavily during operation by proton and neutron irradiation. If the annihilation and/or rearrangement of the dislocation occurred by cyclic loading by pressure waves, it is expected that the lifetime of the vessel is longer than that of the value which estimated based on the irradiation data under static loading.

![Fig. 7. Change in the dislocation densities as a function of the number of loading cycles.](image)

![Fig. 8. Change in the dislocation arrangement parameter as a function of the number of loading cycles.](image)

4. Summary

Changes of mechanical properties by cyclic loading with a high-strain rate of approximately $10^2$ s$^{-1}$ up to gigacycle for the solution annealed and cold-worked 316L stainless steels were investigated. The cyclic hardening for solution annealed 316L and the cyclic hardening after cyclic softening for cold-worked 316L were recognized from the changes in the microhardness. Furthermore, the change in the dislocation density as a function of the number of loading cycle was investigated by the diffraction profile analysis of the neutron diffractometer measurements obtained in TAKUMI at the MLF of J-PARC using the CMWP procedure. The cyclic hardening and softening phenomena that were recognized as the hardness and residual strength changes are well correlated with the change in dislocation density as a function of the loading cycles. The observation of microstructure using a
Transmission Electron Microscope requires to investigate the change in the cell structure and persistent slip band by cyclic loading are left for a future work.

Acknowledgment

This work was supported by JSPS KAKENHI Grant Number JP17K14565. The neutron diffraction experiments were performed at BL19 in the Materials and Life Science Experimental Facility of the J-PARC with a proposal number of 2014B0321.

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