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Investigation of Subsequent Viscoplastic Deformation  
of Austenitic Stainless Steel Subjected to Cyclic Preloading

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Abstract

This paper investigates the effects of cyclic preloading on the subsequent viscoplastic deformation. A series of experiments such as the subsequent creep, subsequent stress relaxation, and cyclic loading with strain rate changes after cyclic preloading were conducted with Type 304 stainless steel at room temperature. The cyclic proportional and non-proportional loadings were conducted as cyclic preloadings. Tension-compression loading was chosen as the cyclic proportional loading, and circular and cruciform loading as the cyclic non-proportional loading. The experimental results showed that the subsequent deformation changes with the number of cycles of cyclic preloading. The differences in the subsequent deformation were examined by transmission electron microscopy (TEM). The observations suggest that changes in the dislocation structure depending on the number of cycles of cyclic preloading affect the subsequent viscoplastic deformation.

*Keywords:* Creep; Dislocations; Stress relaxation; Cyclic loading; Viscoplastic material; Electron microscopy; Mechanical testing

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## 1. Introduction

Recently, extensive research on the subsequent deformations of materials subjected to preloading has been carried out. The stress reversal Bauschinger effect, i.e. the difference in yield stress in tension and compression, is a well known subsequent deformation. This phenomenon must be considered for a precise modeling when forming sheet metals and considering subsequent springback predictions (Chun et al., 2002a, b; Yoshida et al., 2002a, b; Chung et al., 2005; Lee et al., 2005a, b). Mollica et al. (2001) developed a general three dimensional model, which can reproduce the stress-strain response at loading reversals and can be applied to more general changes in loading direction. Deformation induced anisotropy, which leads to different subsequent deformations depending on the loading direction has also been investigated (Ishikawa, 1997; Ishikawa and Sasaki, 1998; Kalidindi, 2001; Yao and Cao, 2002; Tuğcu et al., 2002; Garmestani et al., 2002; Wu, 2002; Chiang et al., 2002; Geng et al., 2002; Wu et al., 2003; Kowalczyk and Gambin, 2004; Tsakmakis, 2004; Häusler et al., 2004; Bron and Besson, 2004; Vincent et al., 2004; Cazacu and Barlet, 2004; Kuwabara et al., 2005; Wu et al., 2005; Barlet et al., 2005). Kalidindi (2001) reviewed and summarized models of anisotropic strain hardening and deformation textures in low stacking fault energy fcc metals and reported a new approach in modeling the deformation behavior of the materials. Garmestani et al. (2002) presented a crystal plasticity based modeling framework for the evolution of anisotropy in Al-Li alloys. Loadings with strain path changes are also an important issue in subsequent deformation (Hiwatashi et al., 1997; Hiwatashi et al., 1998; Kuwabara et al., 2000; Hoc and Forest, 2001; El-Danaf et al., 2001). Hiwatashi et al. (1997) developed a model based on the microstructure and simulated deformation due to the loading with a strain-path change. Hoc and Forest (2001) conducted plane strain tests followed by uniaxial tensile tests on IF-Ti steel to determine the hardening law.

To explain the macroscopic deformation mechanisms from a microscopic point of

view micro/meso scopic observations have been conducted in recent decades (Doong et al., 1990; Jiao et al., 1995; Christ et al., 1995; Feaugas, 1999; Bocher, L. et al., 2001; Gan et al., 2002; Zisman et al., 2002; El-Madhoun et al., 2003; Jia and Fernandes, 2003, Lopes et al., 2003; Haddou et al., 2004; Trivedi et al., 2004, Zuev et al., 2004). Christ et al. (1995) showed the effect of dislocation arrangements on subsequent fatigue tests. Bocher et al. (2001) showed that the over-strengthening due to non-proportional loadings is related to the development of heterogeneous substructures. Zisman et al. (2002) examined a shear microband formed in mild steel after an orthogonal strain-path change. El-Madhoun et al. (2003) concluded that the cellular dislocation structures are low energy structures and that they govern the plastic hardening behavior of commercial purity polycrystalline aluminum.

Extensive researches on the viscoplastic deformations of austenitic stainless steel have been presented (Krempel et al., 1979; Kujawski et al., 1980; Yoshida et al., 1989). However, there are few studies on the subsequent deformations after preloading and the microscopic observations.

This paper reports a series of experiments and observations of the microstructure of austenitic stainless steel (Type 304) at room temperature to determine the effects of cyclic loading with a constant strain range on the subsequent viscoplastic deformation such as the subsequent creep, subsequent stress relaxation, and cyclic loading with strain rate changes. The cyclic proportional and non-proportional loadings were conducted as cyclic preloading. The tension-compression loading was chosen as the cyclic proportional loading, and circular and cruciform loading as the cyclic non-proportional loading. The relationship between the macroscopic stress-strain response and the microscopic dislocation structure is discussed.

## **2. Experimental procedures**

The specimens used in this study were a drawn tube of Type 304 stainless steel

subjected to solution heat treatment. The tube had a 32mm outer diameter and 6mm wall thickness and was annealed by the manufacturer at 1070 °C followed by water quenching for half an hour. The chemical compositions were 0.02C, 0.45Si, 1.09Mn, 0.030P, 0.005S, 8.30Ni, 18.54Cr, and Fe balance in weight percent. Tubular specimens were machined from this tube as shown in Fig.1. The specimens were fabricated by metal cutting and then annealed to ensure an initial isotropic stress state. In the annealing process, the specimens were heated and maintained at 380 °C for 120min followed by air cooling. Young's modulus of the specimen at room temperature was  $E=200\text{GPa}$ .

A servo-controlled axial-torsional testing machine (Shimazu EHF-EB10), with a Shimazu 4825 controller and personal computer, were used for the computerized testing and data acquisition. Strain was measured using two strain gauges applied on opposite sides of the specimen. The axial force was measured using the load cell in the machine.

Four types of tests were conducted at room temperature: (1) the tension-compression cyclic loading at strain rate 0.01%/sec with strain range 1%; (2) the subsequent creep tests after 10 and 30 cycles of tension-compression cyclic loading at strain rate 0.01%/sec with strain range 1%; (3) the subsequent stress relaxation tests at 0.5% tensile strain after 10, 30, or 100 cycles of tension-compression cyclic loading at strain rate 0.01%/sec with strain range 1%; (4) the cyclic loading with strain rate changes for measurement of the deviation of stresses between fast and slow strain rates; and (5) the subsequent creep tests after non-proportional cyclic loading at the equivalent strain rate 0.01%/sec with an equivalent strain range 0.5%. The circular loading and cruciform loading were chosen as the non-proportional loading paths.

The observations of dislocations were performed with a JEOL JEM-2000ES (200kV) TEM. Foil specimens for TEM observations were prepared by cutting thin slices from the gauge length of the specimen parallel to the stress axis. The slices were mechanically polished on both sides and jet thinned in a solution.

### **3. Results and discussion**

#### **3.1 Cyclic hardening and softening during cyclic tension-compression loading**

Figure 2 shows the relationship between the maximum peak stress and the number of cycles during the tension-compression cyclic loading at the strain rate 0.01%/sec with the strain range 1%. It can be seen that the maximum peak stress initially increases and that it decreases after the 20<sup>th</sup> cycle in this experimental condition. Cyclic hardening and softening depends on the strain range as shown in the researches (Ohno, 1982; Mizuno et al., 2000; Kang et al., 2003). The cyclic loading with large strain range of type 304 stainless steel shows no cyclic softening as shown in Ohno (1982). However, in the experimental condition chosen in this study, the cyclic hardening and cyclic softening processes would result in the same maximum peak stresses. For example the maximum peak stresses of the 10<sup>th</sup> and 30<sup>th</sup> cycles are almost the same (315MPa). Figure 3 shows a comparison of the stress-strain curves of the 10<sup>th</sup> cycle with that of the 30<sup>th</sup> cycle. The stress-strain curve of the 10<sup>th</sup> cycle agrees well with that of the 30<sup>th</sup> cycle.

#### **3.2 Subsequent creep and stress relaxation tests after tension-compression cyclic loading**

In Fig.4 the open circles and the solid line show the creep curves at the peak stress of 315MPa in tension after 10 and 30 cycles of tension-compression cyclic loading at the strain rate of 0.01%/sec with the strain range of 1%. The creep curve after 30 cycles of preloading is smaller than that after 10 cycles although the stress-strain curves of the preloading at the 10<sup>th</sup> and 30<sup>th</sup> cycles are the same as shown in Fig.3. This different subsequent creep curves after the preloading can not be described by classical phenomenological constitutive models. Those models predict the same subsequent deformations if the stress-strain behavior just before the subsequent loading were same. Yoshida et al. (1989) conducted the similar subsequent experiments after the cyclic preloading. However, the cyclic loadings were carried out under the stress control and

they discussed the accumulated strain during the cyclic loading and the effects on the subsequent loading. On the other hand, in our study the cyclic loadings were carried out under strain control and there is no accumulation of strain during cyclic loading.

Figure 5 shows the relationship between the relaxed stress and time caused by the stress relaxation tests at 0.5% tensile strain after tension-compression cyclic preloading at the strain rate 0.01%/sec with the strain range 1%. In Fig.5,  $\circ$ ,  $\square$ ,  $\times$ , and  $\bullet$  represent the subsequent stress relaxation curves after 10, 30, 60, and 100 cycles of cyclic preloading, respectively. The stress relaxation curves after 10 and 30 cycles of preloading are clearly different as is the subsequent creep deformation, i.e., the relaxed stress decreases with increases in the number of cycles of preloadings.

### **3.3 Deviation of stresses between fast and slow strain rates after cyclic preloading**

Figure 6 shows a schematic diagram of stress-strain curves due to cyclic tension-compression loading with strain rate changes. After 10, 30, 60, and 100 cyclic preloadings, the strain rate changed during the cyclic loading. In Fig.6, the solid lines shows the stress-strain curves at a strain rate of 0.1 and the broken line is at 0.001%/sec. Figure 7 shows the stress-strain curve of cyclic loading with variations in strain rate. It can be seen that the stress-strain curve changes abruptly just after the strain rate changes. The deviation of stresses between the strain rate 0.1 and 0.001%/sec at the 0.3% strain were measured from points A and B in Fig.7. Table 1 shows the deviations in stress after cyclic loading of 10, 30, 60, and 100 cycles. The deviation of stress decreases with the increase in the number of cycles. In most macroscopic constitutive models (e.g. Krempl, 1987; Chaboche, 1989), the stress is ordinarily divided into kinematic hardening stress (or back stress), isotropic hardening stress (or quasi-static yield stress), and time-dependent stress (or over stress). The decrease in the deviations in the stress corresponds to the decrease of the time-dependent stress. A large time-dependent stress leads to a large creep deformation and the stress relaxation is due to the process of decreases in the time-dependent stress from the macroscopic model concepts. Therefore

the experimental results in Figs.4 and 5 are also explained by the decrease in the time-dependent stress.

### **3.4 Subsequent creep after non-proportional cyclic loading**

Subsequent creep tests after non-proportional cyclic loading were also carried out. The strain paths were circular and cruciform loadings as shown in Fig.8 (a) and (b) because it is well known that the loading can be classified into three groups due to the non-proportionality (Tanaka et al., 1985). Proportional loading, circular loading, and cruciform loading are classified different categories. In Fig.9 the open circles and open squares show the relationship between the maximum peak stresses and the number of cycles with circular and cruciform loadings. In both cases, the maximum peak stresses initially increase for several tens of cycles, and then decrease. Compared to the result of the proportional loading shown in Fig.2, the non-proportional loading shows greater cyclic hardening than the proportional loading. Moreover, the number of cycles, where cyclic softening starts, is larger than that of the proportional loading.

The peak stresses at the 25<sup>th</sup> and 300<sup>th</sup> cycles are almost the same with the circular loading, and the peak stresses at the 30<sup>th</sup> and 300<sup>th</sup> cycles are almost the same with cruciform loading. The stress trajectories at these two cycle numbers are shown in Fig.10 (a) and (b). It may be concluded that there are no differences in the trajectories of the hardening and softening processes when the peak stresses are the same.

Figure 11 shows the subsequent creep curves at the peak stress (375MPa) in tension of the 25<sup>th</sup> and 300<sup>th</sup> cycles after circular loading and Fig. 12 is at the peak stress (333MPa) in tension of the 30<sup>th</sup> and 300<sup>th</sup> cycles after cruciform loading. Different subsequent creep curves are observed for the different numbers of cyclic non-proportional preloadings as same as the proportional loading case in Fig.4. Comparing the subsequent creep strain at 1000seconds, the subsequent creep strain after the 30<sup>th</sup> cycle of proportional preloading is the largest, that after the 30<sup>th</sup> cycle of cruciform preloading is smaller, and that after the 25<sup>th</sup> cycle of circular preloading the

smallest.

#### **4. Microscopic observations**

To establish the reasons for the changes in time-dependent deformation due to the cyclic preloading, microscopic observations were conducted by TEM. Figures 13 (a), (b), (c), and (d) show the dislocation structures of the specimens at the initially annealed state, and after 10, 30, and 100 cycles of tension-compression cyclic loadings. Initially there are few dislocations in the specimen as shown in Fig. 13 (a). After 10 and 30 cyclic preloadings, dislocation tangles become more common as shown in Fig. 13 (b) and (c). After 100 cycles, there are dislocation cell structures as shown in Fig. 13 (d). These observations suggest that the cyclic loadings resulting in the formation of dislocation cell structures. The dislocation tangles are transient features occurring between the low dislocation density of the initially annealed state and the formation of the developed cell structures.

Many models of deformation due to cyclic loadings are based on the concept that a high dislocation density leads to only hardening. According to these models, the phenomenon that the peak stress decreases with increases in dislocation density is contradictory, and the TEM observations here suggest that both the dislocation density and the dislocation structure must be considered in a constitutive model to explain cyclic hardening and cyclic softening. The observations here suggest that cyclic hardening is mainly caused by increases in the dislocation density while cyclic softening is mainly caused by the formation of dislocation structures.

Figures 14 (a), (b), (c), and (d) show the dislocation structures after 25 and 300 cycles of circular loadings and after 30 and 300 cycles of cruciform loadings, respectively. As for the tension-compression cyclic loading, cell structures are observed during cyclic softening as shown in Fig. 14 (b) and (d), while there are no apparent cell structures but tangles are observed during the cyclic hardening as shown in Fig. 14 (a)

and (c). Comparing Fig.13 with Fig.14, it may be suggested that more dislocation tangles are present with increases in non-proportionality, i.e., the highest incidence of dislocation tangles occur in the circular preloading. The cell structures during cyclic softening (Fig. 13(d), Fig. 14(b) and (d)) depend on the cyclic preloading, larger non-proportionality of the cyclic preloading leads to smaller cell structures. Overall, the size of the cell structure correlates with the subsequent time-dependent deformation.

The creep deformation occurs due to both intra-grain sliding and inter-grain dislocation slips. The TEM observations suggest that the dislocation structures affect the differences in subsequent viscoplastic deformation.

Consequently, the subsequent viscoplastic deformation depends on the dislocation structure caused by the cyclic preloading. The dislocation structure works as an obstacle to the subsequent viscoplastic deformation such as creep, stress relaxation, and cyclic loading under different strain rates. El-Danaf et al. (2001) demonstrated a similar concept termed ‘limited barriers of twinning’ for the reduced strain hardening in shear deformation.

## **5. Conclusions**

In this study, experimental and micro-structural investigations of subsequent viscoplastic deformation after the cyclic proportional and non-proportional loading at a constant strain rate with a constant strain range were conducted using Type 304 stainless steel at room temperature. As a result the following may be concluded:

- (1) The creep curves are different for different numbers of cyclic preloadings although the stress-strain curves of the cyclic preloading just before the creep are the same. The subsequent creep strain decreases with increases in the number of cycles of cyclic preloading. This phenomenon was observed with both the proportional and non-proportional loading states.
- (2) The stress relaxation curves are different for different numbers of cyclic preloadings

although the stress-strain curves of the cyclic preloading just before the stress relaxation tests are the same. The relaxed stress decreases with more cycles of cyclic preloading.

(3) The deviation in stress with fast and slow strain rates at the same strain were measured in the cyclic loading tests with variations in strain rates. The deviation decreases with increases in the number of cycles of cyclic preloading.

(4) Dislocation structures are different with different numbers of cyclic preloadings though the stress-strain curves coincide. The dislocation structures affect the viscoplastic deformation such as creep, stress relaxation, and strain rate dependence of cyclic loading.

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### **Captions of figures and tables**

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Fig.14 Dislocation structures after cyclic non-proportional loading

(a) After 25 cycles of cyclic circular loading

(b) After 300 cycles of cyclic circular loading

(c) After 30 cycles of cyclic cruciform loading

(d) After 300 cycles of cyclic cruciform loading

Table 1 Deviation in stresses with the fast and slow strain rate changes

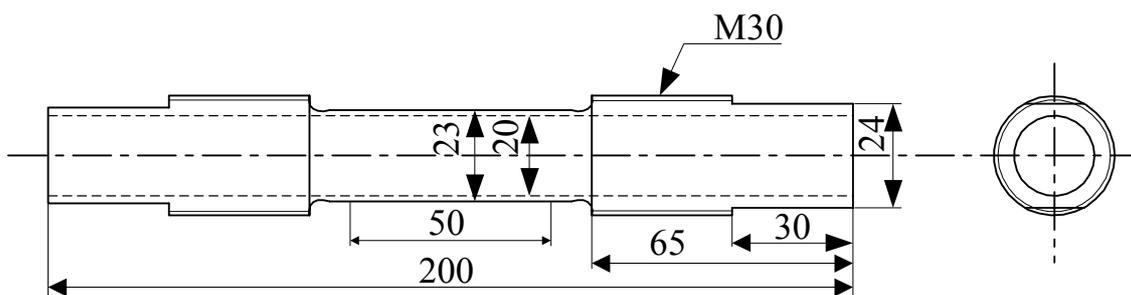


Fig.1 The geometry of specimens

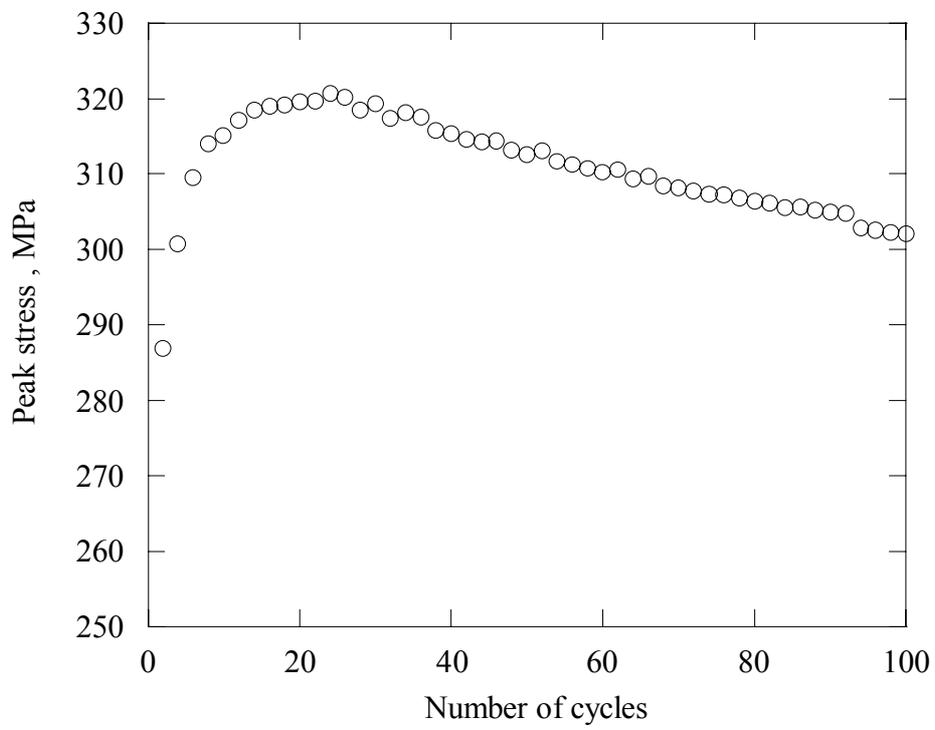


Fig.2 Relationship between peak stress and number of cycles in cyclic tension-compression loading under strain rate 0.01%/sec with strain range 1.0%

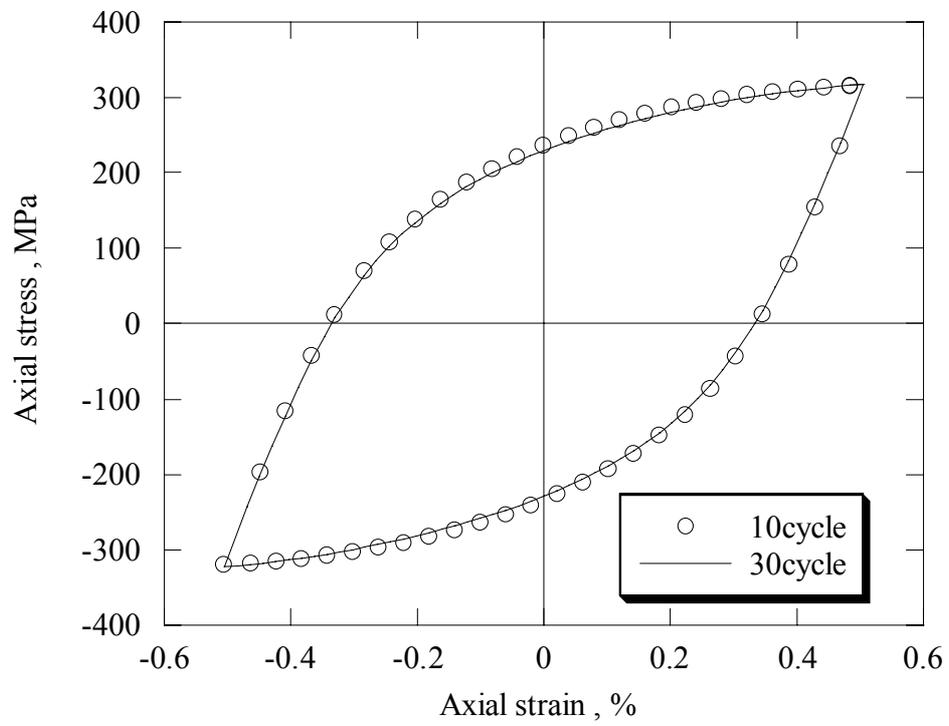


Fig.3 Stress-strain curves of 10th and 30th cycles

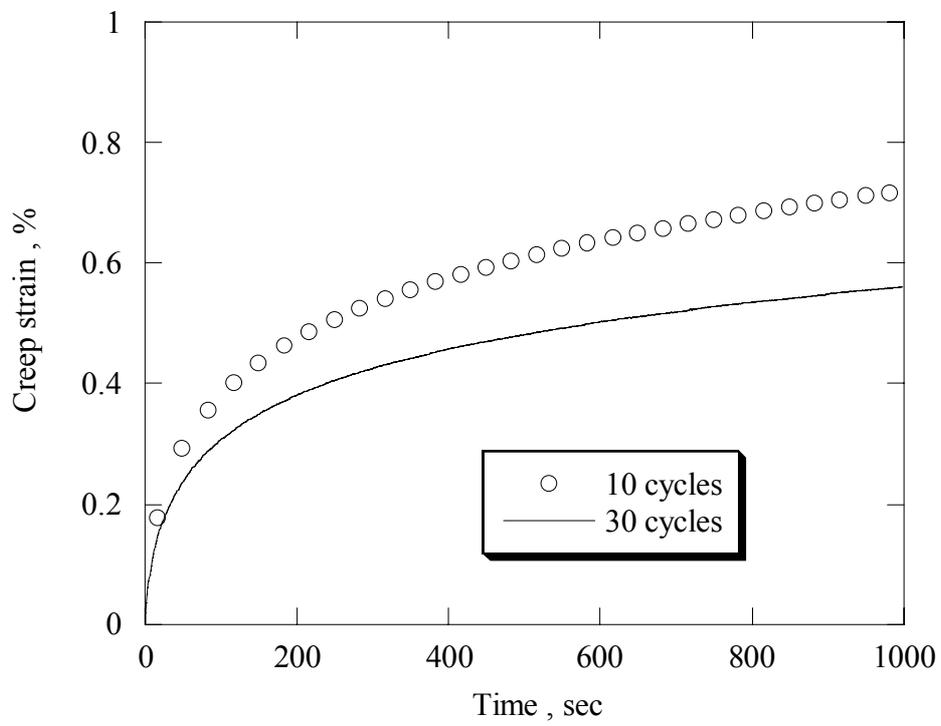


Fig.4 Subsequent creep curves at the peak stress 315MPa in tension after 10 and 30 cycles of tension-compression cyclic loading

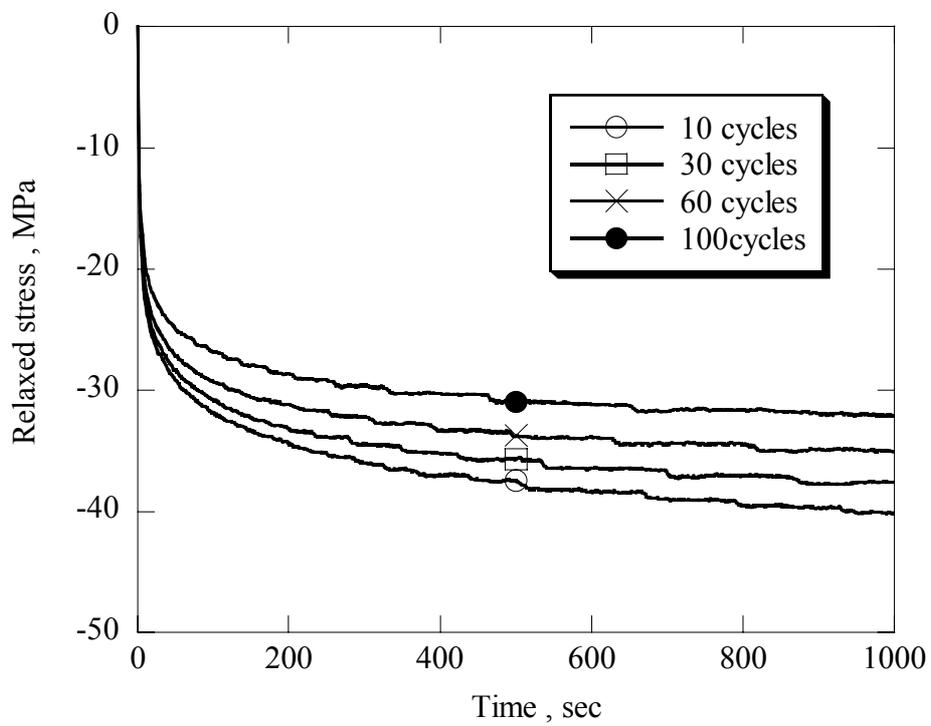


Fig.5 Subsequent stress relaxation curves at 0.5% tensile strain after 10, 30, 60, and 100 cycles of tension-compression cyclic preloading

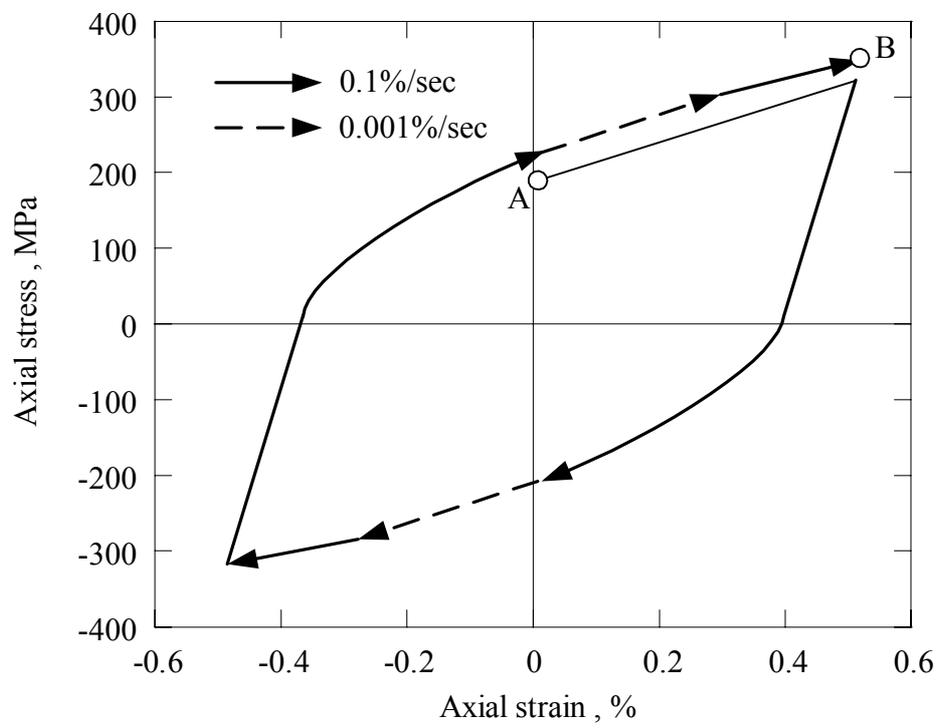


Fig.6 Stress-strain curves of cyclic loading with variations in the strain rate

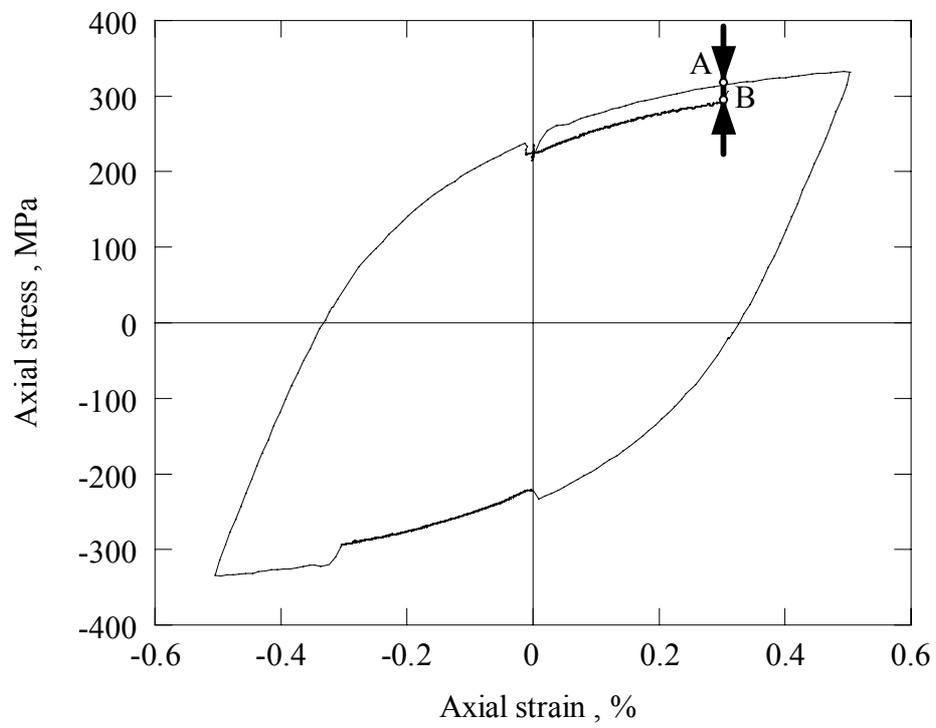
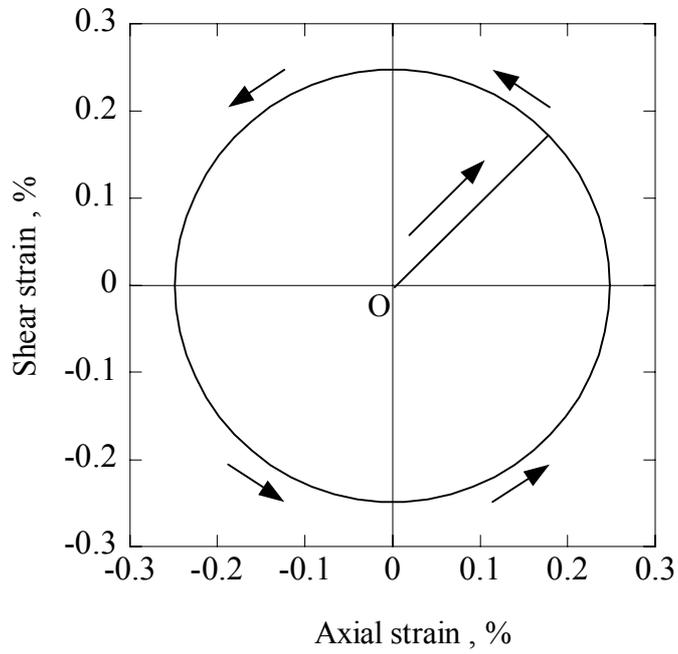
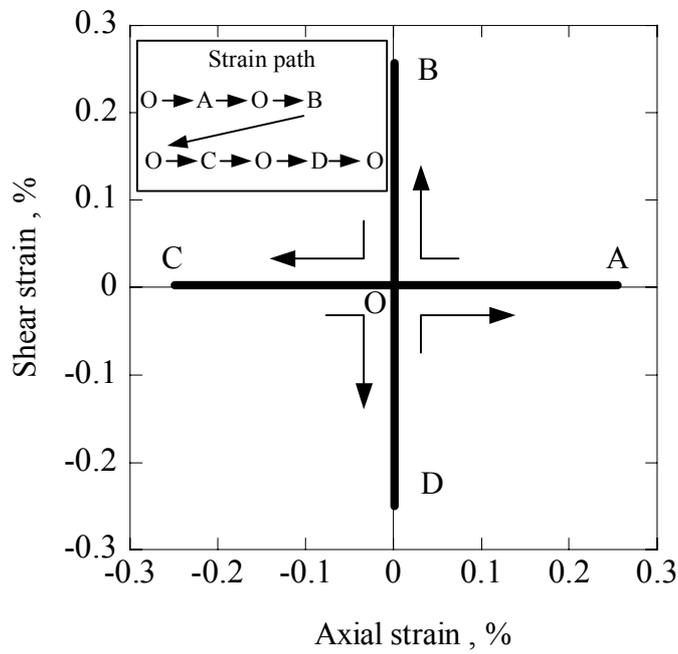


Fig.7 Stresses at strain 0.3%. A:0.1%/sec ; B:0.001%/sec



(a) Circular loading



(b) Cruciform loading

Fig.8 Loading paths of non-proportional cyclic loading tests

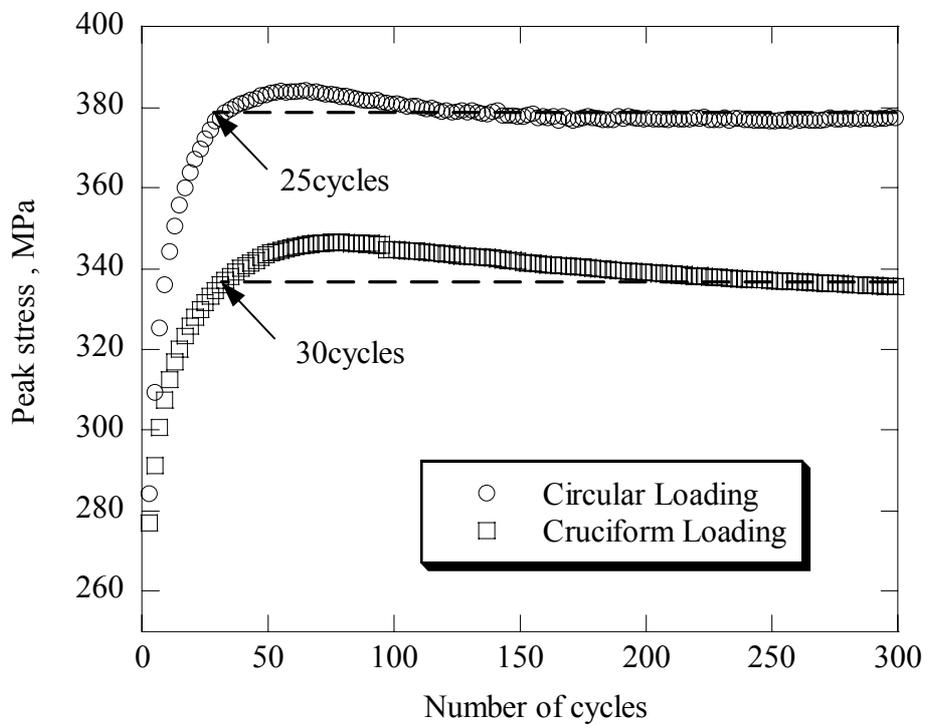
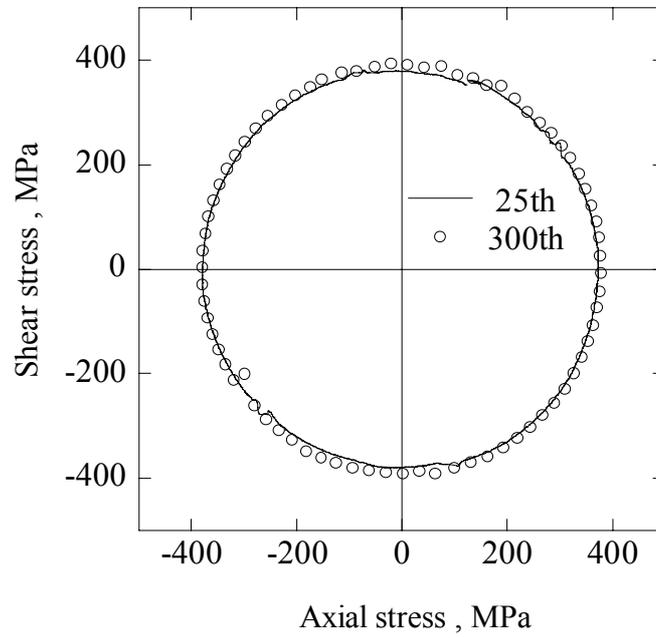
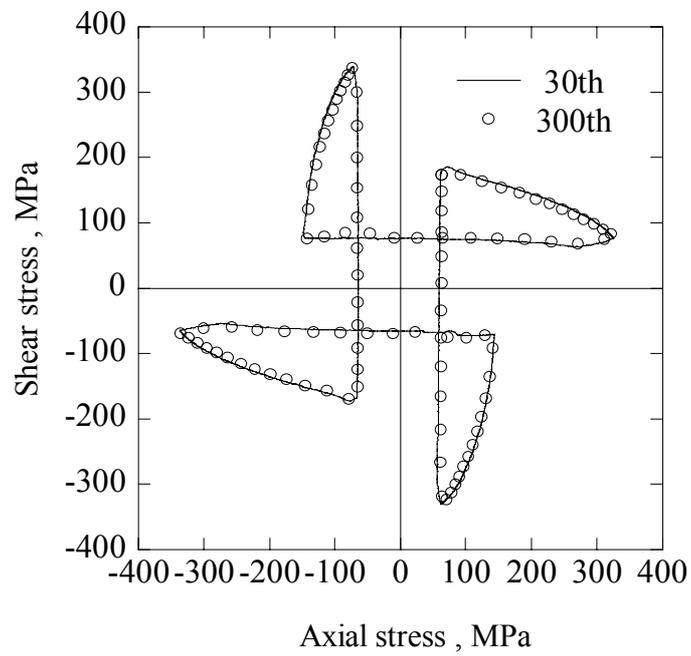


Fig.9 Peak stress versus number of cycles in non-proportional cyclic loading



(a) Circular loading



(b) Cruciform loading

Fig.10 Stress trajectories at two cycle numbers of non-proportional loading tests

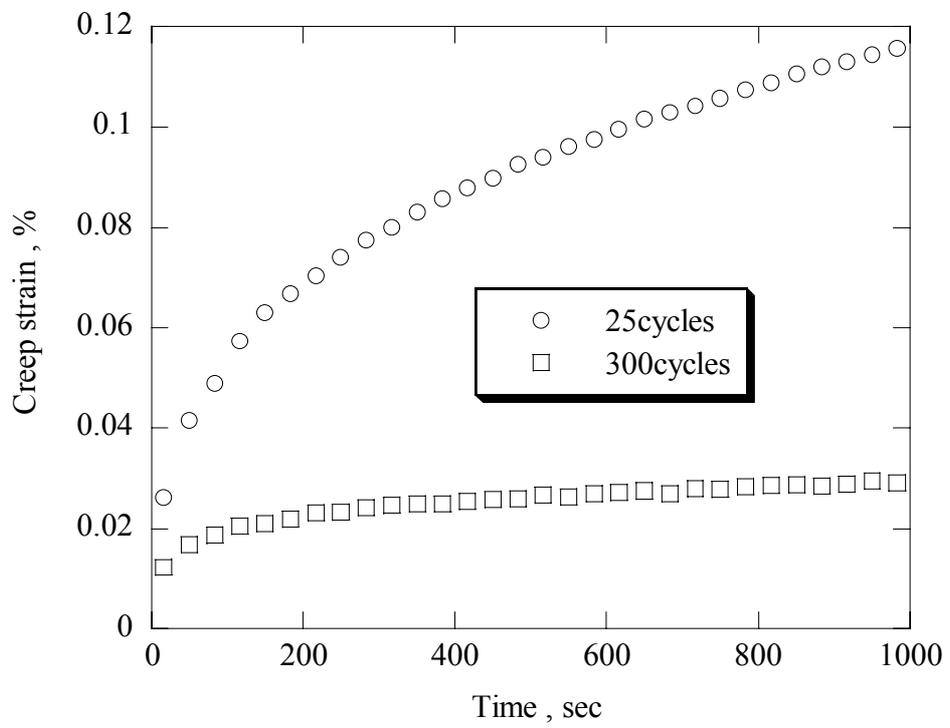


Fig.11 Subsequent creep curves after 25 and 300 cycles of circular loading

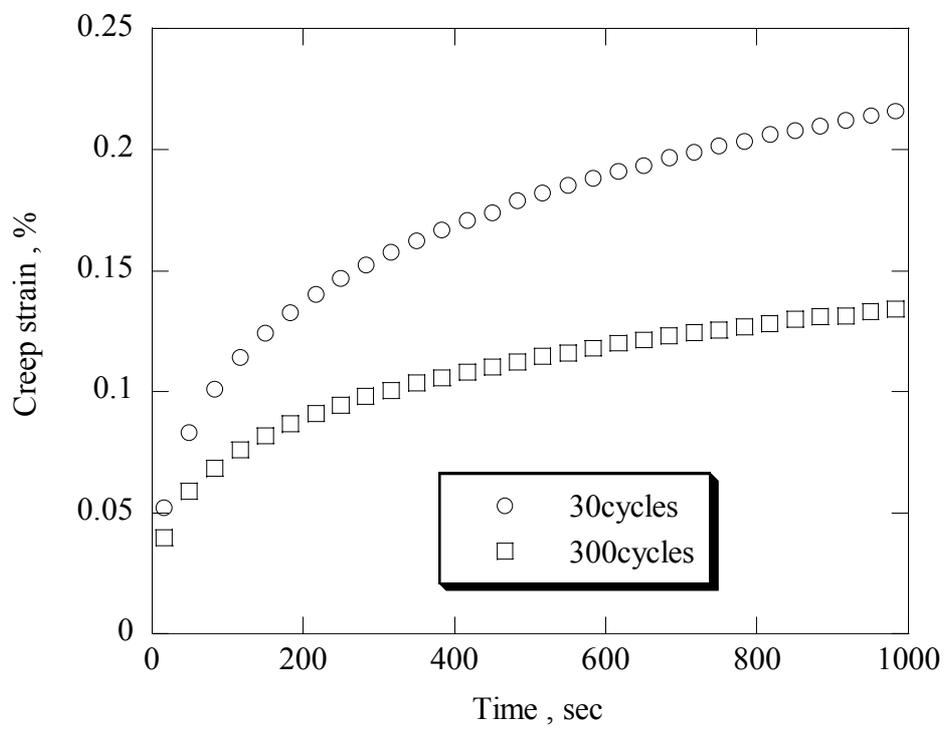
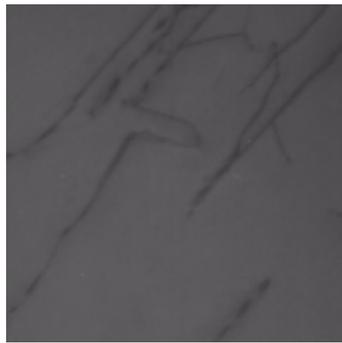
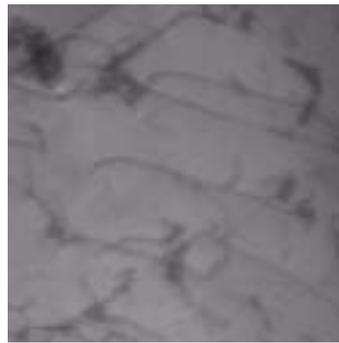


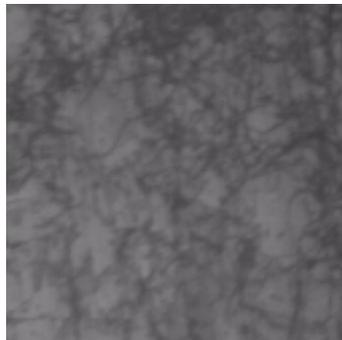
Fig.12 Subsequent creep curves after 30 and 300 cycles of cruciform loading



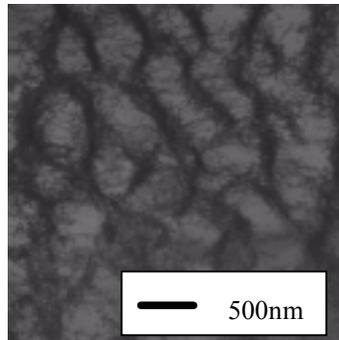
(a) As annealed specimen



(b) After 10 cycles of tension-compression cyclic preloading

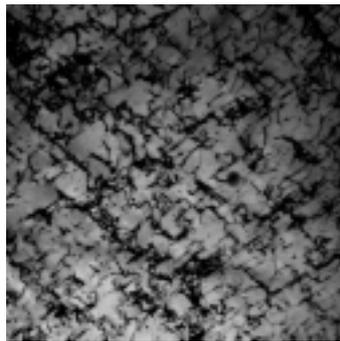


(c) After 30 cycles of tension-compression cyclic preloading

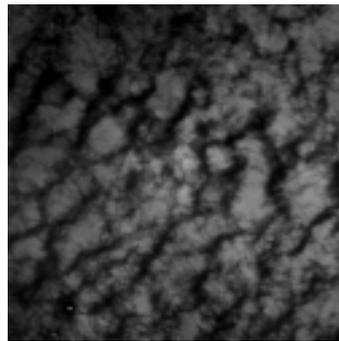


(d) After 100 cycles of tension-compression cyclic preloading

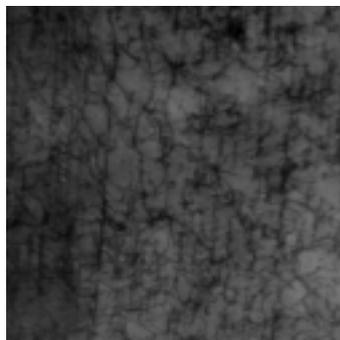
Fig.13 Dislocation structures of the initially annealed and tension-compression cyclic loaded specimens



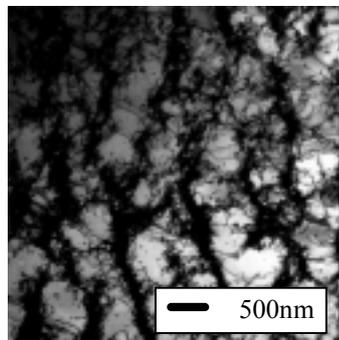
(a) After 25 cycles of cyclic circular preloading



(b) After 300 cycles of cyclic circular preloading



(c) After 30 cycles of cyclic cruciform preloading



(d) After 300 cycles of cyclic cruciform preloading

Fig.14 Dislocation structures after cyclic non-proportional loading

Table 1 Deviation in stresses with the fast and slow strain rate changes

Number of cycle	10	30	60	100
Over stress [MPa]	22.9	18.5	16.9	15.2