Low-cycle fatigue of Fe-20%Cr alloy processed by equal-channel angular pressing

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Abstract. Low-cycle fatigue properties were investigated on Fe-20%Cr ferritic stainless steel processed by equal channel angular pressing (ECAP). The Fe-20%Cr alloy bullets were processed for one to four passes via Route-Bc. The ECAPed samples were cyclically deformed at the constant plastic strain amplitude $\varepsilon_{pl}$ of $5 \times 10^{-4}$ at room temperature in air. After the 1-pass ECAP, low-angle grain boundaries were dominantly formed. During the low-cycle fatigue test, the 1-pass sample revealed the rapid softening which continued until fatigue fracture. Fatigue life of the 1-pass sample was shorter than that of a coarse-grained sample. After the 4-pass ECAP, the average grain size reduced down to about 1.5 $\mu$m. At initial stage of the low-cycle fatigue tests, the stress amplitude increased with increasing ECAP passes. At the samples processed for more than 2 passes, the cyclic softening was relatively moderate. It was found that fatigue life of the ECAPed Fe-20%Cr alloy excepting the 1-pass sample was improved as compared to the coarse-grained sample, even under the strain controlled fatigue condition.

1. Introduction

Prior to fatigue rapture, a metallic material undergoes several stages including fatigue crack initiation and propagation. An intragranular fatigue crack in a coarse-grained ductile material is often nucleated along a slip band where cyclic plastic deformation is localized [1,2]. The slip band formed under fatigue loading is often referred to as the persistent slip band (PSB). The microstructure of the PSB has intensively been studied with transmission electron microscopy (TEM) [3]. The PSB formed in copper is composed of array of dislocation dipolar walls whose average spacing was of 1.3 $\mu$m. Similar dislocation wall structure with micron scale have been found in iron-based alloys such as Fe-Cr-Ni alloy [4] and Fe-Cr alloy [5, 6]. Accordingly, the slip band formation —— which precedes to fatigue cracking —— would require the dislocation self-organizations having the micron length scale.

Severe plastic deformation (SPD) can achieve grain refinement down to submicron scale. This grain size is smaller than the spacing between the dislocation walls which have been observed within the PSBs in coarse-grained materials fatigued. Thus, one can expect that the dislocation self-organization is suppressed by the presence of the fine grains. In this sense, the cyclic strain localization would require grain coarsening of the fine-grained material.
In TEM observation on fatigued ultrafine-grained (UFG) copper, local grain coarsening was reported [8]. The UFG copper samples have often shown cyclic softening behaviour in low-cycle fatigue test where axial strain amplitude is constant [7-9], while cyclic hardening normally occurs in an annealed copper having coarse-grained structure. It seems probable that the microstructural change such as the grain coarsening affected the cyclic softening. If we aim for the engineering application of the SPD material, stabilization of microstructure against the cyclic straining is strongly desired.

Low-cycle fatigue behavior of iron-based SPD materials exhibited different cyclic stress-strain responses. Significant cyclic softening were observed in the iron-based alloys such as Fe-Ni invar alloy [10] and Fe-Ni-Cr alloy [11,12]. In contrast, the cyclic softening was almost negligible in the interstitial-free steel processed by the SPD [13, 14]. However, detailed mechanism explaining such a difference is still unclear.

In the present study, we conducted the ECAP processing on Fe-20%Cr ferritic stainless steel having bcc lattice structure. Low-cycle fatigue properties under a constant plastic strain amplitude were conducted on the Fe-20%Cr alloy proceed for different number of ECAP passes. Particularly, the cyclic softening was investigated in conjunction with the microstructures analysed with electron backscatter diffraction (EBSD) technique.

2. Experimental Procedure

The Fe-20%Cr ferritic stainless steel with the chemical composition listed in Table 1 was processed by the ECAP. Before the ECPA processing, the material was water-quenched from 1323K. The average grain size of the as-quenched material was of 1mm. The quenched material was shaped to the bullets having $4 \times 4 \times 40$mm$^3$ shape. The ECAP processing was conducted using the die having the $4 \times 4$mm$^2$ channel and the 90° corner angle. During the ECAP, the die temperature was kept at 423K using a surrounding heater. The samples were processed for selected number of the ECAP passes $N$, via Route Bc. The maximum number of the ECAP pass was $N=4$. In addition to the ECAPed samples, an as-quenched sample was prepared for the low-cycle fatigue test.

The ECAPed samples were shaped to small tensile specimens with a gauge shape of $1.5 \times 1.5$mm$^2$ cross section and 2mm length, using a spark erosion cutter. The tensile axis was parallel to the extrusion direction. The tensile specimens were mechanically and electrolytically polished. Low-cycle fatigue tests were carried out using a servo-hydraulic machine (Shimadzu Servo Pulser EHF-LB10kN-10N). Axial strain was measured with a strain gauge cemented at the gauge part. The fatigue tests were conducted in air at room temperature, under the constant plastic strain amplitude of $5 \times 10^{-4}$. Triangular strain wave was applied at the frequency of 0.2Hz.

Microstructures of the samples were investigated with the EBSD technique with JEOL JSM-6500F, before and after the low-cycle fatigue tests. Evolution of dislocation structure after the fatigue was observed by electron channelling contrast imaging (ECCI) technique [15].

Table 1 Chemical composition of the Fe-20%Cr alloy. (wt.%)

| Element | Composition (wt.%) |
|---------|-------------------|
| Fe      | 20.25              |
| Cr      | 0.0016             |
| O       | 0.175              |
| N       | 0.0004             |
| C       | 0.003              |

3. Results and Discussion

3.1. Microstructure of as-ECAPed samples

After the ECAP processing, the microstructures at the flow surface were investigate using the EBSD system. Figure 1 shows inverse pole figure (IPF) maps of the as-ECAPed samples. In the 1-pass sample, the orientations of analyzed area are essentially the same direction. Low-angle boundaries were dominantly formed after the 1-pass ECAP: the fraction of the high-angle boundaries in all the grain boundaries was about 10%. In the IPF map of the 4-pass sample, refined grains with various orientations are recognized. The fraction of high-angle boundaries increased up to about 40%. An
average grain size of the 4-pass sample was 1.5µm, when the grain boundaries having misorientation angle more than 5° are counted.

![IPF maps of the Fe-20%Cr alloy after (a) 1-pass and (b) 4-pass ECAP processing.](image)

3.2. Low-cycle fatigue test
Figure 2 shows hysteresis loops of the as-quenched and the ECAPed samples at 1,000 cycles. The hysteresis loop of the as-quenched sample exhibits a typical shape of an annealed metallic material, where nonlinear plastic part is distinguished from linear elastic one. In the ECAPed samples, the increase of stress amplitude is appreciable. The hysteresis loops show lenticular shape and a transition point from elastic to plastic deformation is unclear. The width of hysteresis loop of the 4-pass sample is thin, although all the plastic strain amplitudes are equal among the three loops. The thin loop shape should owe to very early onset of reverse plastic deformation. For example, just after the tensile peak, the 4-pass sample seems to start compressive plastic deformation although tensile stress is still exerted. Since the ECAPed samples have such high Bauschinger effect and small loop area, it can be said that the cyclic deformation of the ECAPed samples contained more reversible processes, compared to that of the as-quenched sample.

![Hysteresis loops at 1,000 cycles in (a) the as-quenched, (b) the 1-pass and (c) the 4-pass samples.](image)
Figure 3 is cyclic softening curves of the ECAPed and the as-quenched samples. A plotted stress amplitude is a mean value of tensile and compressive peak stresses of a hysteresis loop. The initial stress amplitude of the as-quenched sample was approximately 200MPa. The stress amplitude of the as-quenched sample increased with increasing cycles, as well as cyclic hardening of an annealed ductile material. After gradual reduction of the stress amplitude, the sample was fractured at 8300 cycles.

The initial stress amplitude of the ECAPed samples increased with increasing ECAP passes. For the 4-pass sample, the initial stress amplitude amounted to 580MPa. During the fatigue tests, all the ECAPed samples revealed cyclic softening behavior. Particularly in the 1-pass sample, the cyclic softening of was very rapid. The fatigue life of the 1-pass sample was shorter than that of the as-quenched sample. This degradation of low-cycle fatigue durability is the same result as the UFG copper [9]. The cyclic softening was suppressed in the ECAPed samples of $N_E \geq 2$. It should be emphasized that fatigue lives of the ECAPed sample of $N_E \geq 2$ were longer than that of the as-quenched sample, even under the condition of the constant plastic strain amplitude.

![Cyclic softening curves of the Fe-20%Cr alloy processed for different number of ECAP passes under the constant plastic strain amplitude of 5x10^-4.](image)

As seen in Fig.3, after the initial rapid softening during which the softening rate decreased gradually, the stress amplitudes of the ECAPed samples showed secondary softening. Figure 4 shows the cyclic hardening rate of the 1-pass sample, together with the stress amplitude. The cyclic hardening rate increased during the initial stage of fatigue, and then attained a local maximum. This local maximum point corresponds to an inflection point in the cyclic softening curve.

In order to estimate the initial softening, we calculated the cyclic softening ratio which is defined as $(\sigma_{\text{max}} - \sigma_{\text{inf}})/\sigma_{\text{max}}$, where $\sigma_{\text{max}}$ is the maximum stress amplitude and $\sigma_{\text{inf}}$ is the stress amplitude at the inflection point. In the present experiment, the maximum stress amplitude corresponds to the stress amplitude at the beginning of fatigue test. The stress amplitudes and the cyclic softening ratio of the ECAPed samples are presented in Fig.5, as a function of the ECAP passes. The maximum stress amplitude increased with increasing number of ECAP passes. The cyclic softening ratio of the 1-pass sample was 0.13. In spite of the high stress amplitude, the softening ratios of the samples of $N_E \geq 2$
were reduced to less than 0.08. Accordingly, the stability against the cyclic plastic deformation was certainly improved by increasing ECAP passes.

3.3. Microstructure of after fatigue test

After the low-cycle fatigue tests, surface morphologies were observed with the scanning electron microscope (SEM). Band-like surface relieves were detected at all the fatigued samples. The band-like relief at the as-quenched sample exhibited a typical extrusion morphology of the PSBs in the fatigued Fe-Cr alloy [5,16]. For the ECAPed samples, the band-like relieves were inclined to the tensile axis at about 45°, as shown in Fig.6. Hence, the band-like relieves of the ECAPed samples can be classified into shear band. In a ductile metallic material, an intragranular fatigue crack is normally nucleated along a well-developed slip band [1,2]. It is thus likely that the fatigue fractures in the present ECAPed samples were originated from the shear bands. Indeed, we can find microcracks along the shear bands in the 1-pass sample (Fig.6(a)). Morphology of the shear bands depended on the ECAP
passes. As seen in Fig.6, length and width of the shear bands of the 4-pass sample were evidently shorter than those of the 1-pass sample.

After the SEM observation, the surfaces of the fatigued samples were polished again, and then the microstructures were examined by the EBSD analysis. Figure 7 presents the grain boundary maps of the fatigued 1-pass and 4-pass samples. The analysed region of the 1-pass sample contains the shear band which is inclined to the tensile axis. It is apparent that the low-angle boundaries at the shear band almost disappeared after the fatigue test.

As shown in Fig.1(a), the microstructure of the 1-pass sample was composed mainly of the low-angle boundaries, which can be expressed as dislocations arrays. In the shear bands where the cyclic deformation is localized, the dislocation arrays are possibly dissociated such that the individual dislocations can glide to accommodate the imposed plastic strain. Thus, the disappearance of the low-angle boundaries in the 1-pass sample is interpreted by low stability of the low-angle boundaries against cyclic shear strain localization. The rapid cyclic softening of the 1-pass sample is attributable to the dissociation of the low-angle boundaries which can trap gliding dislocations.

Before the fatigue test, the 4-pass sample had the high fraction of high-angle boundaries. Even though we analysed several regions in the gauge part, no drastic grain coarsening was found as seen in Fig.7(b), in contrast to the low-angle boundary dissociations at the 1-pass sample. It can be said that
the grain coarsening under the cyclic plastic deformation was inhibited in the 4-pass sample which has the fine grains and the large fraction of high-angle boundaries.

To detect changes in dislocation microstructure after the fatigue tests, the polished surfaces of the fatigued samples were examined by the ECCI method. The microstructure below a thick slip band at the as-quenched sample is presented in Fig.8(a). We can recognize the formation of dislocation cell structure having average cell size of 1.4µm. Each cell is separated by the dislocation walls which are characterized as bright boundary lines in the ECC image. The TEM observation on the fatigued Fe-13%Cr alloy has shown that the final internal structure of the PSBs --- where intragranular fatigue cracks are preferentially nucleated --- is the cell structure [3]. The evolution of the surface relief in the present Fe-Cr alloy can be associated closely with the internal microstructure of the dislocation cell shown in Fig.8(a). The microstructure below the shear bands of the 1-pass sample was essentially the same as Fig.8(a).

In the ECC image of the 4-pass sample (Fig.8(b)), the fine grains are still visible even after the fatigue. As well as in the grain boundary map, no significant grain coarsening is recognized. In addition, no evidence of dislocation self-organization such as the vein structure was found in the present observations. It should be pointed out that a typical size of the large grain in the 4-pass sample after the fatigue is almost equal to the dislocation cell size of the as-quenched sample. Hence, the dislocation self-organization leading to the surface relief was unexpected inside such small grains. Accordingly, it is most likely that good stability of the highly-densified grain boundaries in the fine-grained microstructure contributed to the improved fatigue performance of the ECAPed samples of $N_E \geq 2$.

![Fig.8 ECC images showing the microstructures of (a) the as-quenched and (b) the 4-pass samples after fatigue.](image)

4. Summary

(1) After the 4-pass ECAP processing at 423K, the grains of the Fe-20%Cr alloy were refined down to average size of about 1.5µm.

(2) In the low-cycle fatigue tests under the constant plastic strain amplitude of $5 \times 10^{-4}$, the initial stress amplitude increased with increasing number of ECAP passes. The initial stress amplitude of the 4-pass specimen amounted to 580MPa. The 1-pass specimen revealed rapid cyclic softening from the beginning of fatigue test. However, the cyclic softening was suppressed in the ECAPed samples of $N_E \geq 2$.

(3) The low-cycle fatigue life of the 1-pass sample was shorter than that of the as-quenched sample having coarse grains. In contrast, when the ECAP passes was more than 2, the fatigue performance was improved with compared to that of the as-quenched sample.
(4) At the 1-pass sample where the low-angle boundaries were dominantly formed, the low-angle boundaries disappeared along the shear band after the fatigue test. No significant grain coarsening was detected at the fatigued 4-pass samples having the fine grains.

Acknowledgments
This study was financially supported by the Grant-in-Aid for Scientific Research on Innovative Areas “Bulk nanostructured metals” through MEXT Japan under contract No. 22102006, and by that on Challenging Exploratory Research through JSPS under contract No. 24656091.

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