Lattice Rotation in Fe-20%Cr Alloy Single Crystals Subjected to Sliding Wear[†]

by

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Sliding wear tests were conducted on Fe-20%Cr alloy single crystals having (001), (110) and (111) surfaces, using the ball-on-disk type friction machine under the condition of 10N vertical load and 30m wear distance. After the wear tests, the single crystals were cut to investigate microstructures developed below the worn surface. The cross-sectional observation revealed that subsurface regions of all the worn single crystals were composed of fine grains which were generated by severe plastic deformation introduced by the sliding wear process. It was confirmed from EBSD analyses that, below the fine-grained regions, all the single crystals rotated around the axis which is almost perpendicular to the wear direction. Such lattice rotation was expected to be a preceding event for the grain boundary formation in the fine-grained region. The lattice rotations were evaluated by the misorientation angles from the single crystal matrices. Extent of the lattice rotation depended on crystallographic orientation of the single crystals. The depth where the misorientation angle is 10° increased in ascending order of (001), (110) and (111) single crystals, suggesting that the lattice rotation was promoted favorably at the (111) single crystal. In order to understand the orientation dependence of the lattice rotation, we considered three slip deformation models (horizontal shear, vertical shear and resolved shear stress models) which incorporated the geometrical relationship between a slip system and wear direction.

Key words:

Grain refinement, Severe plastic deformation, Wear, Stainless steel, Single crystal

1 Introduction

When a ductile metallic material is subjected to severe plastic deformation (SPD), component grains are significantly refined¹⁾. This kind of grain refinement has been achieved by special plastic-work techniques including the equal channel angular pressing (ECAP), the high-pressure torsion (HPT) and accumulated roll bonding (ARB)²⁾. According to the Hall-Petch relation, yield strength of a metallic material increases with decreasing grain size. Thus, if average grain size is reduced down to nanometer scale, we could obtain the materials having very high strength³⁾⁻⁵⁾.

It has been known that surface friction often causes the grain refinement locally near surface⁶⁾⁻⁸⁾. Such grain refinement can be associated with local SPD induced by frictional force. Frictions of materials have long been considered as the phenomena to avoid, because they frequently result in surface degradation such as wear loss and cracking. However, the surface friction has recently been utilized as the surface modification techniques that enable local grain refinement⁹⁾⁻¹¹⁾. The surface-processed materials must contain the gradient fine-grained structure near the worn surface. The sample having such gradient fine-grained structure reveals both high strength and high ductility¹²⁾, and thereby the surface grain refinement seems a promising route to improve the tensile property. For the further improvement of the gradient microstructure, it is desirable to clarify microscopic aspect of the grain refinement.

Early investigations on the microstructures of the worn materials have been conducted by transmission electron microscopy (TEM). The TEM observation in worn copper has shown that a matrix grain was significantly rotated below the fine-grained region¹⁾. Such lattice rotation below the fine-grained region was clearly manifested by the electron backscatter diffraction (EBSD) analyses in our previous study on pure iron¹³⁾. The observed lattice rotation should be a preceding event for the grain refinement, because occurring at the region where plastic deformation is insignificant in comparison with the worn surface vicinity. Hence, for the investigation of initial stage of the grain refinement during the frictional process, we should understand the lattice rotation.

In a polycrystalline material, plastic flow near worn surface should be affected by crystallographic orientation of each grain. Moreover, dislocation glides are certainly inhibited by grain boundaries. Hence, to simplify plastic deformation process causing the lattice rotation, an experiment employing a single crystal is preferable. This is because the single crystals have well defined orientations and the effect of grain boundaries is eliminated. Indeed, in the course of our studies on the worn copper single crystals¹⁴⁾⁻¹⁷⁾, we have shown that low-angle boundaries were generated as a result of dislocation rearrangement and the lattice rotation axis had significant impact on the grain boundary character distribution of high-angle boundaries. In the present study, we

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prepared single crystals of Fe-Cr alloy having a bcc lattice structure. The sliding wear tests were conducted on the single crystals having various surface planes. In particular, we investigated orientation dependence of the lattice rotation occurring at deep region from surface.

2 Experimental Procedure

2.1 Preparation of Single Crystal Specimens

The single crystals of the Fe-Cr alloy (a ferritic stainless steel) were prepared by the Bridgman method. Chemical composition of the material is listed in Table 1. The single crystals were molten at 1813K in an alumina mold having 30mm inner diameter in 99.999% Ar gas, and then a furnace was raised at a constant speed of 2×10^{-6} mm/s.

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

C	S	O	N	Cr	Fe
0.003	0.0004	0.175	0.0016	20.25	bal.

Crystallographic orientations of the grown single crystals were analyzed by the Laue x-ray backscatter diffraction technique. After the orientations were adjusted using a goniometer, the single crystals were cut along (001), (110) and (111) planes by a spark-erosion cutter. Thickness of the single crystal disk was 2mm. The single crystal disks were annealed at 1373K in vacuum for 24 hours, and then

Fig.1 Stereographic projections of crystallographic orientations of the Fe-Cr alloy single crystals having (a) (001), (b) (110) and (c) (111) surfaces.

quenched in water. Before the sliding wear tests, the specimen surfaces were mechanically and electrolytically polished to obtain mirror-like surface. The electrolytical polishing was conducted at 27V in the solution consisting of 60% perchloric acid and acetic acid, mixed at the ratio of 1:9. Fig.1 shows stereographic projections of crystallographic orientations of the prepared single crystal disks. Surface of a single crystal disk is parallel to the y - z plane indicated in the figure.

2.2 Sliding Wear Tests and Microstructural Observations

Fig.2(a) is a schematic illustration of the sliding wear test. The wear tests were carried out in air at room temperature using a ball-on-disk type testing machine (Rhesca FPR-2000), under unlubricated condition. A SUJ2 steel ball of 3.8mm diameter was put at the specimen surface. Static load of 10N was applied vertically to the ball. The specimen was fastened at the rotating table. By rotating the specimen, we could form a circular wear track on the surface. Sliding speed was set at 10 mm/s. The sliding wear tests were interrupted when total sliding distance reached 30m.

After the sliding wear tests, the specimens were cut perpendicular to the worn surface to obtain a cross section, as shown in Fig.2(b). Before the cross sectioning, the worn surface was covered with electrodeposited nickel to protect microstructures generated near the worn surface. The cross sections were electrolytically polished. Wear-affected zone below the worn surface was observed with the JEOL JSM-6500F scanning electron microscope (SEM), under electron channelling contrast (ECC) imaging mode. Lattice rotation in the wear-affected zone was analyzed by the EBSD technique with the EDAX-OIM system.

Fig.2 Schematic illustrations of (a) the sliding wear test and (b) subsequent cross sectioning to observe microstructure below the worn surface.

3 Results and Discussion

3.1 Sliding Wear Tests

Fig.3 shows changes in friction coefficient during the sliding wear tests. In the figure, the testing time of 3000s corresponds to the wear distance of 30m. In the initial stage of all the wear tests, the friction coefficients decreased rapidly, and then increased moderately. The friction coefficients changes from 0.65 to 0.85. There was no significant orientation dependence of the friction coefficient.

Fig.3 Friction coefficients of the single crystals during the wear tests.

3.2 Microstructure and Lattice Rotation

After the sliding wear tests, the single crystal disks whose surfaces were covered with the electrodeposited nickel were cut perpendicular to the frictional direction. The cross section orientations were the (100), $(\bar{1}11)$ and $(11\bar{2})$ for the single crystal disks having the (001), (110) and (111) surfaces, respectively. Low-magnification ECC images of the cross sections are presented in Fig.4. We could recognize the gradient microstructures which were introduced apparently by the sliding wear processes. Modifications in microstructure are visible notably in the vicinities of the worn surfaces. Below the subsurface region, there is a layered region where the intensity of the ECC gradually changes. In the region where the depth is more than 50μm, the change in microstructure is no longer detectable.

Fig.5 shows the ECC images of the close vicinities of the worn surfaces. From the ECC images, the grains near the worn surfaces were refined down to less than 1μm at all the single crystals. The grain sizes of the (110) and (111) single crystals were smaller than that of the (001) single crystal.

After the ECC observations, the cross sections were analyzed by the EBSD technique. Fig.6 shows inverse pole figure (IPF) maps of the cross sections, where the wear

Fig.4. ECC images of the cross sections of the worn single crystals.

Fig.5 ECC images showing the fine-grained structures.

direction is along the y-axis. In addition, (100) pole figures at the subsurface and the deep regions are drawn. At the deep region, the IPF map and the pole figure indicated that the crystallographic orientations were identical to those of the single crystal matrices, and thus there is no lattice rotation. At the subsurface region, the orientations expressed by color gradually changes as an analyzed position approaches surface. In the pole figures of this depth, the {100} orientations distributed from the single crystal matrix orientations to downward side. As a consequence of this result, it is concluded that the lattices near the surface rotated around the z-axis.

In order to estimate the lattice rotation quantitatively, we calculated a misorientation angle θ from the single crystal matrix, along a line parallel to the x-axis. The analysis line was set at the center of z-position in the corresponding IPF map in Fig.6. Fig.7 shows the misorientation angles plotted against the depth from the worn surface. Since the lattice rotated around the z-axis, the calculated misorientation would be almost identical to the rotation angle around the z-axis. When the distance from surface is deep enough, the misorientation angles were zero because no lattice rotation occurred. As the depth decreases, the misorientation angle continuously increased at all the single crystals. This continuous lattice rotation region corresponds to the layered

Fig.6 IPF maps and (100) pole figures of the cross sections of the worn single crystals.

region in Fig.4. When the analyzed position approaches the worn surface, discontinuous changes in the misorientation frequently appeared. A generated grain boundary coincides with the discontinuous change. There was no significant z -position dependence of the misorientation curves, except near the worn surface where the high-angle boundaries were

Fig.7 Misorientation angles from the single crystal matrices, plotted against the depth from surface.

densely generated.

It is confirmed that the misorientation curve in Fig.7 depended on the worn surface orientation of the single crystals. The misorientation angle rapidly increased near the worn surface in the (111) single crystal, while the increase was moderate in the (001) single crystal. To compare the extent of the lattice rotation, we read off the depth, x_{10} , where the misorientation angle is 10° . If a single crystal exhibits large-scale lattice rotation, the x_{10} value will become high. The x_{10} values read from the misorientation curves were $5.7\mu\text{m}$, $11.8\mu\text{m}$ and $15.1\mu\text{m}$ for the (001), (110) and (111) single crystals, respectively. On the basis of the x_{10} value, it can be said that the (111) single crystal easily induced the lattice rotation.

3.3 Orientation Dependence of Lattice Rotation

The orientation dependence of the lattice rotation should be attributed to the slip system geometries relative to both the wear direction and the worn surface. To investigate the effect of the slip system geometry on the lattice rotation, we considered two extreme models of slip deformation below the worn surface, as schematically shown in Fig.8. One is the horizontal shear model (Fig.8(a)). This model possesses the slip plane parallel to the worn surface and the slip direction parallel to the wear direction, and assumes that the slip deformation is activated owing to the frictional force. The other is the vertical shear model (Fig.8(b)). In this model, the subsurface deformation is accommodated by the slip deformation where the slip plane is vertical to the wear direction and the slip direction is parallel to the worn surface normal. To estimate the similarity between the models and the slip systems of the tested single crystal, we defined two geometrical parameters, s_h and s_v , which are given by the following equations.

$$s_h = |(\mathbf{n}_p \cdot \mathbf{e}_x)(\mathbf{v}_b \cdot \mathbf{e}_y)| \quad (1)$$

$$s_v = |(\mathbf{n}_p \cdot \mathbf{e}_y)(\mathbf{v}_b \cdot \mathbf{e}_x)| \quad (2)$$

where $\mathbf{n}_p$ and $\mathbf{v}_b$ are the normalized vectors of a slip plane normal and a slip vector, $\mathbf{e}_x$ and $\mathbf{e}_y$ are the basis vectors of the x - and y -axes. These vectors are schematically shown in Fig.9. The s_h and s_v values give the degrees of coincidence with the horizontal and vertical shear models, respectively. In the ideal horizontal shear model where the $\mathbf{n}_p = \mathbf{e}_x$ and $\mathbf{v}_b = \mathbf{e}_y$, the s_h value is unity. Likewise, the s_v value is unity in the ideal vertical shear model. In the Fe-Cr alloy having a bcc lattice structure, no defined slip plane exists because of frequent cross slips, while the slip vector is exactly along the $\langle 111 \rangle$ direction. Hence, in the calculation of the s_v and s_h values, the slip planes were chosen so as to maximize these values, under the condition that the slip plane normal is vertical to the $\langle 111 \rangle$ slip vector.

Fig.8 (a) Horizontal and (b) vertical shear models of slip deformation near the worn surfaces.

Fig.9 A schematic model of a slip system, which is drawn with reference to the worn surface and the wear direction.

Beside the slip geometry models shown in Fig.8, we considered the effect of resolved shear stress on the lattice rotation. In the vicinity of the worn surface, the frictional force should exert in addition to vertical force from the steel ball. Thus, it is postulated that, under the normal stress σ_x , the surface is suffered additionally from shear stress τ_{xy} ($=\mu\sigma_x$), where μ is the friction coefficient. We calculated the resolved shear stress τ_r of respective slip systems under the condition of the unit compressive stress $\sigma_x=-1$ and $\mu=0.75$. In the present study, we refers the τ_r values as the normalized resolved shear stress. This is a parameter analogous to the Schmid factor used under uniaxial deformation. The slip plane of each slip direction was chosen to be the maximum resolved shear stresses plane (mrssp), such that the normalized resolved shear stress τ_r was maximized.

Fig.10 shows the depths x_{10} at the 10° lattice rotation, plotted against the s_h , s_v and τ_r values. The plotted s_h , s_v and τ_r values are the maximum ones among the slip systems in the respective single crystals. Among the single crystals, the s_h and s_v values were maximized at the (110) and (111) single crystals, respectively. The (110) single crystal had the

maximum normalized resolved shear stress τ_r , while the τ_r value of the (111) single crystal showing the highest lattice rotation was the minimum among the single crystals. As seen in Fig.10, the depth x_{10} — expressing the ease of the lattice rotation — apparently had good correlation ($R=0.997$) with the s_v value. The correlations with the s_h and the normalized resolved shear stress τ_r were relatively poor. Accordingly, it is most probable that the lattice rotations during the sliding wear were favorably promoted when the single crystals had the slip geometry like the vertical shear model, as long as the lattice rotation is estimated with the x_{10} value. If the calculated slip planes are limited to the low-index planes of {110} and {112}, the values plotted against the horizontal axis of Fig.10 became somewhat smaller. However, the maximum discrepancy with data in Fig.10 was only 8.5%, and the s_v value still have the best correlation with the x_{10} value even for the calculation on the low-index slip planes.

The vertical shear model is compatible also with the dislocation model which can explain the lattice rotation^{13),14)}. Strain gradient in a grain, which can result in the lattice rotation, has been explained by a concept of geometrically-necessary (GN) dislocations¹⁸⁾. The vertical shear model involves the process of edge dislocation emission from the worn surface. The emitted dislocations possibly distribute over the wear-affected zone. Because having a single sign, such distributed dislocations can behave as the GN dislocations. In the vertical shear model, the edge dislocations having the Burgers vector along the x -axis are assumed to glide along z - x plane and consequently the lattice would rotate around the z -axis.

Fig.10 Depth at the misorientation angle θ of 10° , plotted against s_v value, s_h value and normalized resolved shear stress τ_r .

4 Conclusions

1. Under the sliding wear condition of the 10N vertical load and the 30m wear distance, the submicron fine grains were generated near the worn surfaces of the Fe-Cr alloy single crystals.
2. The EBSD analyses of the cross sections revealed that the lattice rotations occurred below the fine-grained regions. It is found that the lattice rotation depended on the crystallographic orientation of the single crystal. The depth at the 10° lattice rotation increased in ascending order of the (001), (110) and (111) single crystal.

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