Effect of deformation route on the development of low CN Fe-20%Cr alloy by Equal Channel Angular Pressing

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Abstract. The effect of deformation routes on the microstructure, mechanical, and electrochemical properties of low CN Fe-20%Cr alloys by equal channel angular pressing (ECAP) has been investigated in detail focusing on the anisotropy of the microstructure. This alloy is pressed at 423K up to eight passes via the so-called routes A, Bc and C. The continuous refinement of the microstructure is sustained by ECAP until the sub-grain range. However, the degree of anisotropy of microstructural development was different among the three deformation routes. Materials processed by Route Bc exhibited a comparable micro-hardness value in three orthogonal planes than those processed by routes A and C. Pitting corrosion characteristics of the ECAP processed sample were investigated using an electrochemical potentiodynamic test. The increased pitting potential along with an increased number passes of ECAP were explained by enhanced protective passive layer of ultrafine grain structure, as compared to the coarse grain counterpart.

Keywords: Ferritic Stainless steel, ECAP, Grain Boundary, EBSD, Corrosion

1. Introduction

Low carbon ferritic stainless steel is widely used for corrosive environments as it satisfies the material requirements for high resistance to stress corrosion cracking (SCC), intergranular corrosion, as well as high ductility and formability [1-2]. However, this class of material is of low-strength and this restricts its application to the limited field; hence, grain refinement is often regarded as a useful method for improving its mechanical properties. The method of equal-channel angular pressing (ECAP) is one of the severe plastic deformation (SPD) techniques that produces ultrafine-grained (UFG) materials.

Several ECAP parameters that have an influence on microstructural development and mechanical properties are the die angle, the pressing speed, the die temperature, and the deformation route. The four deformation routes of ECAP have mainly been proposed as follows. In Route A, the sample is pressed without rotation. In Route BA, the sample is rotated by 90 degrees in an alternate direction between consecutive passes. In Route BC, the sample is rotated 90 degrees counterclockwise between each pass. In Route C, the specimen is rotated by 180 degrees between passes [3]. There are many studies concerning the effective deformation routes for grain refinement in face-centered cubic structure (FCC) metals such as pure aluminum [2], nickel [5], copper [6], and titanium [7]; however, there are few studies on body-centered cubic structure (BCC).

Previous studies have attempted to explain the effect of deformation routes on microstructure and mechanical properties, especially in FCC materials [8-10]. They have found that Route Bc is the most efficient for producing isotropic microstructures, due to the crossing of the shear planes in every pass [8-10]. However, the degree of anisotropy on microstructural development has not been carefully evaluated so far, especially in BCC metals [8, 11-13]. Therefore, the effect of deformation routes on the microstructure in the ECAP process as well as the mechanical and electrochemical properties will be further discussed.

2. Experimental procedure

The material used in this experiment had a chemical composition of low carbon nitrogen (CN) Fe-20%Cr alloy with Cr 19.97, C 0.0020, N 0.0015 and Fe balance (in mass percent). This material was machined with dimensions of 7.95 mm×7.95 mm×120 mm for ECAP pressing. ECAP was carried out using a split die with two channels intersecting at an inner angle of 90° and an outer angle of 0° at 423 K (Fig. 1.a). The samples were lubricated with high temperature fluorine lubricating grease and pressed from one pass until eight passes via routes A, Bc and C (Fig.1.b). A transmission electron microscope (TEM, JEM...
2100F) was used to examine the microstructures. Thin foils for TEM were polished using abrasive papers to about 100 µm thick and then thinned by a twin-jet polishing Tenupol 5 facility using a solution of 40% acetic acid, 30% phosphoric acid, 20% nitric acid and 10% distilled water. Mechanical and electrochemical properties were measured by micro hardness and pitting corrosion. The micro hardness experiments were performed on a Vickers hardness testing machine at room temperature. Corrosion testing was carried out at ambient temperature in a flat polarization cell, using platinum counter electrodes and Ag/AgCl reference electrodes to measure the corrosion current and corrosion potential. The pitting corrosion characteristic was obtained in neutral solutions 1 M NaCl.

**Figure 1.** (a) ECAP schema and (b) Deformation routes on ECAP.

### III. Result

3.1. Microstructure

A TEM micrograph including a selected area diffraction pattern (SADP) was observed on one pass as shown in Fig.2. In the ECAP process, after the first pass, 500nm deformation bands can be exhibited clearly on the Y-plane. Therefore, SADP appeared as a network with little dispersion. It indicated small misorientations on the deformation bands. In addition, after one pass, the elongated sub-grain structure was displayed through a TEM. Consequently, the grains were revealed as being finely subdivided after one pass, though the initial structure had larger grain sizes. Several boundaries could then be observed as low angle boundary misorientations, due to having a blurry and thick appearance.

**Figure 2.** TEM micrograph of one pass ECAP (Y-plane).

The microstructure of material processed by ECAP from two until eight passes via routes A, Bc, and C was observed by TEM on the Y-plane as shown in Fig.3. After two passes of ECAP, the microstructure became finer and consisted of elongated grains with rather planar boundaries. Larger grains were also observed within UFG structures. Since the selected area diffraction pattern (SADP) exhibited a rather regular pattern, this microstructure consisted of dislocation cell structures with a similar orientation. After four passes, more homogenous equiaxed UFG structures were observed with sharper boundaries.
Due to grain refinement, the SADP of eight passes of the ECAPed sample displayed a ring diffraction pattern.

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| Pass number | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|-------------|---|---|---|---|---|---|---|
| Route A     | ![Image](image1.png) | ![Image](image2.png) | ![Image](image3.png) | ![Image](image4.png) | ![Image](image5.png) | ![Image](image6.png) | ![Image](image7.png) |
| Route Bc    | ![Image](image8.png) | ![Image](image9.png) | ![Image](image10.png) | ![Image](image11.png) | ![Image](image12.png) | ![Image](image13.png) | ![Image](image14.png) |
| Route C     | ![Image](image15.png) | ![Image](image16.png) | ![Image](image17.png) | ![Image](image18.png) | ![Image](image19.png) | ![Image](image20.png) | ![Image](image21.png) |

**Figure 3.** TEM micrograph after ECAP from two passes until eight passes by route A, Bc and C (Y-plane).

The increase in the number of ECAP passes resulted in a decreased boundary spacing on the Y-plane as shown in Fig.4. The boundary spacing of the material processed by routes A and Bc decreased more significantly than those processed by Route C, while the boundary spacing of Route C saturated after four passes. These results corresponded with the mechanical properties. As a result, boundary spacing achieved 150nm on materials processed by Route A.

**Figure 4.** Boundary spacing of ECAP passes number on different deformation routes (Y-plane)
3.2. Mechanical properties

Micro hardness from one to eight passes of ECAP can be observed in three orthogonal planes as shown in Fig.5. A significant increment of micro-hardness can be observed as the number of passes increases, but the degree of hardening was different among the three planes, namely, the hardening was relatively low in Z-plane, especially in routes A and C. After eight passes, materials processed by routes A and Bc grew three times higher than as-received. On the other hand, the micro-hardness of Route C saturated after four passes. This anisotropic strain hardening seems to be characteristics to BCC metals. The isotropic hardening in the material processed by route Bc reflected the isotropic configuration of high angle grain boundary. This hardening may suggest that the 45-degree rotation of billets in route Bc resulted in cross hardening by interactions of mobile dislocation.

Figure 5. Microhardness after ECAP process with different planes on (a) route A, (b) route Bc, (c) route C and (d) average of three orthogonal planes

3.3. Electrochemical properties

Pitting corrosion testing was carried out in 1M of NaCl solution for the as-received, first, second, fourth, and eighth passes of ECAP via Route Bc as shown in Fig.6. The pitting potential of the ECAPed sample increased with the number of passes and shifted to a positive value. After eight passes of ECAP, the pitting testing graph exhibited the most stabilized pitting corrosion. This happened as the microstructural parameters of eight passes on the Y-plane performed higher than any other number of passes.

Figure 6. Pitting corrosion resistance of as received and one, two, four and eight passes via route Bc ECAP sample on Y-plane.
IV. Discussion

The microstructual evolution of extremely low CN Fe-20%Cr alloy as low carbon steel can be examined in terms of crystal slips that are intrinsic to BCC structures. The Peierls barrier of screw dislocations is higher than that of edge dislocation in BCC crystals; thus, slip by screw character is more predominant than by edge dislocation. When plastic straining increases by the formation and extension of dislocation loops, edge dislocation characters with high mobility slip faster than screw dislocation characters with lower mobility, resulting in extended lines of screw dislocations. Therefore, the nature of the slip, which is intrinsic to screw dislocation, has an influence on the macroscopic behavior of plastic deformation.

The micro hardness exhibited an increase by ECAP as was reported before in many metals and alloys. However, the degree of hardening of the ECAPed sample was different among three orthogonal planes in different routes. This anisotropic strain hardening seems to be a characteristic of BCC metals. The isotropic hardening in the material processed by route Bc was reflected the isotropic configuration of high angle grain boundary (HAGB). Namely, this isotropic hardening tendency was observed due to high dislocation density in route Bc. This hardening was suggested that the 45°rotation of billets in route Bc, which resulted in cross hardening. Since screw dislocations are predominant in BCC metals, positive and negative screw dislocation pairs tend to meet by cross-slip and disappear in both the forward-forward and forward-reverse shear in successive passes.

Electrochemical properties can be determined by pitting corrosion testing. The pitting potential increased as the number of ECAP passes increased. An increase in the pitting potential can be explained by a shift in the boundary between imperfect passivity and the pitting region [14]. It indicated that the protective passive layer in the ECAPed sample is more stable than the as-received sample [14]. In the percolation model for passivation in stainless steels, high Cr concentration on the surface is required to for passivation [15,16]. Preferential dissolution of less noble Fe elements accelerate the passivity [15,16]. In UFG structures by SPD, there are high density of grain boundaries and dislocations on the surface, and they should enhance the diffusion of Cr elements from the interior to the surface. Therefore, when the passivated surface is locally attacked by aggressive species such as Cl-, then the passivation can be recovered by faster supply of Cr elements from the interior part through enhanced diffusion. In summary, the increased pitting resistance may also be attributed to the enhanced diffusion of Cr elements due to the high density of grain boundaries and dislocations.

V. Conclusion

The microstructural evolution of extremely low CN Fe-20%Cr alloy processed by ECAP from one to eight passes via routes A, Bc, and C were quantitatively analyzed by focusing on the degree of their anisotropy. The degree of anisotropy of microstructures was different among the three deformation routes. They were generally isotropic in route Bc, namely the grains were equiaxial, and the hardness was comparable in the three orthogonal planes. The improvement of the corrosion resistance of nanocrystallized materials by surface nanocrystallization processes was commonly interpreted as chromium enrichment in the passive film.

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