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Investigation of Texture in ECAP Materials Using Neutron Diffraction

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Abstract. The use of severe plastic deformation techniques such as equal channel angular pressing, has been shown to refine metal microstructures giving advantageous mechanical properties. Metals and alloys subjected to ECAP procedures can have very high yield strengths while maintaining substantial ductility, a unique and attractive combination. However, the implicitly large deformations (the application of repeated shear strains of ~1 are typical) make prediction of the resulting mechanical properties difficult. In particular, modeling the polycrystalline texture evolution and microstructural strain response is challenging. In this paper, results are presented from a neutron diffraction study on aluminum, copper, nickel and beryllium processed by ECAP. Specific attention is given to the evolution of the bulk texture after one pass and the effect of the initial texture. The neutrons probed volumes on the order of cubic centimeters and therefore provided texture and strain information averaged over the bulk of the sample. The results are discussed in the context of a visco-plastic self-consistent model.

Introduction

Significant interest has developed recently in processing materials by severe plastic deformation (SPD) [1]. The principle is to repeatedly subject a material to a high shear strain without changing the cross-sectional dimensions. Several different processes have been developed but the most promising appear to be High-Pressure Torsion (HPT) [2], Equal-Channel Angular Pressing (ECAP) [3] or possibly the new technique of Repetitive Corrugation and Straightening (RCS) [4]. All of these procedures are capable of producing materials with ultrafine grain sizes (10 to 1000 nm) without modifying the chemistry or leaving any residual porosity. To date, most attention has been devoted to ECAP, which has the greatest potential for cost effective production of reasonably large bulk materials. During the ECAP process, ultra-fine grain sizes are achieved through the process of simple shear by pressing a sample through a die with two intersecting channels, equal in cross-section. As the cross-section of the billets is preserved, the process can be repeated and various routes comprising longitudinal billet rotations between pressings have been developed.

The importance of the grain orientation distribution, the texture, on materials properties is well established [5], and motivated the present study. This investigation addressed samples prepared by a single ECAP cycle of pure Ni, Cu, Al and Be. Previous studies of texture evolution during ECAP utilized OIM/EBSD [6] or X-Rays [7], all of which probe small volumes close to the sample surface. In contrast, we perform quantitative bulk texture analysis utilizing the deep penetration depth of neutrons into most materials to study the effects of the initial texture on texture evolution in ECAP. A complementary objective was to benchmark texture predictions made by a visco-plastic self-consistent

(VPSC) model, which models each individual grain in a homogeneous effective medium representing the average properties of all other grains. The VPSC model therefore ignores local effects, making neutrons a perfect verification as local effects can be also neglected.

Experimental materials

Three face-centered cubic (Ni, Cu, Al) and one hexagonal closed-packed specimen (Be) were prepared. A single ECAP pass was applied to all four rod-shaped materials at room temperature using a solid die containing two channels having a diameter of 10 mm and intersecting at an angles of $\Phi = 90°$ and $\Phi = 120°$ for the fcc and hcp metals, respectively. The 90° die had an outer curvature spanning $\Psi = 20°$ and for the 120° die the value of Ψ was 0° (see [8] for a definition of these angles). The magnitude γ of the simple shear along the shear plane, $\Phi/2$, calculated as a function of die geometry is [8]:

$$\gamma = 2\cot(\Phi/2 + \Psi/2) + \Psi\csc(\Phi/2 + \Psi/2) \quad (1)$$

Thus, for the 90° die used the accumulated shear strain per pass is $\varepsilon = \gamma/2 = 0.914$. For the neutron diffraction measurements, specimens of 19 mm length and 8 mm diameter were cut from the centers of the processed rods using electrical discharge machining (EDM), thereby removing the surface layer which exhibits an inhomogeneous microstructure due to friction effects during the pressing. For all materials, measurements were taken on the unprocessed material and after one pass of ECAP.

Texture measurements

Bulk texture measurements were recorded on the new neutron time-of-flight diffractometer HIPPO (*HI*gh *P*ressure *P*referred *O*rientation) at the pulsed neutron source LANSCE. Thirty detector panels were used for the texture measurements, providing 30 probed sample directions. The panels are at nominal diffraction angles of 40° (12 panels), 90° (10 panels) and 150° (8 panels). The forward (40°) and back-scattering (150°) panels cover full rings around the incident neutron beam, the 90° panels leave a gap of about 90° for access to the sample chamber. The samples were oriented with their cylinder axis vertical and perpendicular to the beam. By measuring four orientations (0°, 45°, 67.5° and 90° rotations around the sample cylinder axis), a total of 120 sample directions were probed. Total measurement times were 80, 60, 120, and 40 minutes for Ni, Cu, Al and Be, respectively. The neutron beam spot was collimated to 10 mm diameter, resulting in a probed volume of ~0.5 cm^3.

Quantitative texture analysis was performed using the spherical harmonics representation of the orientation distribution function (ODF) implemented in the Rietveld package GSAS [9-12] and extended for the out of plane detector panels on HIPPO. Ninety histograms were chosen for the Rietveld refinement. The coverage of the pole figures is shown in figure 1. In this figure, each symbol represents a diffraction vector for which the multiple reflections were probed. All 90 histograms were refined simultaneously. Histogram scale factors, background parameters, lattice parameter, peak profile parameters and isotropic thermal motion parameter were added sequentially to the refinement. The texture was described by a spherical harmonics representation of the ODF (assuming no sample symmetry) in which the order of the spherical harmonics representation was increased sequentially and all parameters were varied until convergence was achieved for each order. The maximum order used during this analysis was 12. Further increase of the order did not reduce the reduced χ^2 of the refinement. An example of a refinement is shown in figure 1. Pole figures were exported from GSAS

into popLA [13,14], a package for texture analysis, to generate discrete representations of the ODF as input for the modeling. Using popLA, also a small (<10°) experimental azimuthal misalignment was estimated and corrected for all samples prior to plotting pole figures presented in this paper. The strength of the texture is assessed using a texture index having a value of 1 for a random orientation distribution and infinity for a single crystal and which can be calculated from the spherical harmonics coefficients.

VPSC texture model

A visco-plastic self-consistent (VPSC) computational model for predicting the visco-plastic deformation, microstructural evolution (distributions of grain shape and size) and texture evolution in polycrystalline materials during the ECAP process has been previously reported [15]. In the VPSC model [16] elastic deformation is neglected and single crystals in the aggregate deform by slip and twinning. The slip response is described by a rate-sensitive constitutive law, in which a threshold stress τ_s drives slip activation. Each grain is allowed to deform and re-orient independently in a manner that depends on its shape and orientation with respect to the applied deformation in the effective medium.

The model permits a distinct evolution of τ_s for each individual slip system. Therefore VPSC, unlike the Taylor model, can be applied to low-symmetry materials, such as hexagonal Zr, Be, and Mg, as well as materials with cubic structure such as Cu, Ni, and Al. In principle, any hardening law can be used and for this work we used the following Voce law,

$$\tau_s = \tau_0 + (\tau_1 + \theta_1 \Gamma)\left(1 - e^{-\frac{\theta_0 \Gamma}{\tau_1}}\right) \tag{2}$$

where Γ is the accumulated shear in each grain, τ_1 and θ_1 are adjusted to fit the macroscopic flow curve. The sample orientation of model and measurement with respect to the presented pole density plots are identical apart from experimental error. Therefore, the pole figures are directly comparable. Figure 1 shows the sample orientation in the pole figures with respect to the ECAP die. The sample is oriented with its axis horizontal exiting the ECAP die to the right. The shear plane of the ECAP process is also shown for the case of the 90° die.

In [15,17], we employed in the VPSC model a scheme to model grain-grain interactions [18] and analyze their influence on texture. Specifically, orientations are paired at random and their associated

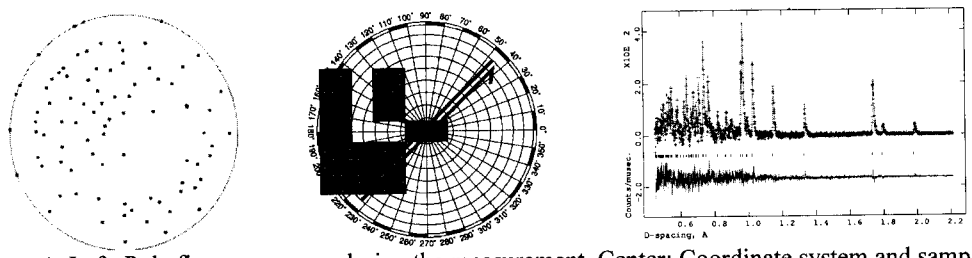

Figure 1: Left: Pole figure coverage during the measurement. Center: Coordinate system and sample orientation for the pole figures. Right: Histogram of unprocessed Be sample.

ellipsoids are forced to co-rotate during deformation. When this interaction scheme is implemented, the texture evolution is weakened [17] and the model better agrees with the maximum intensities of the texture measurements. The code also employs an empirical grain subdivision law which accounts for the changes in grain size induced by ECAP [15], imposing a direct relationship between the ellipsoidal shape of the deformed grain (or grain elongation) and grain subdivision.

For the fcc materials, Ni, Cu, and Al, we assume deformation on the (111)[110] slip systems and a constant τ_s, so $\tau_0 = \tau_s = 1$ and the other three parameters are zero in (2). Accordingly there is no difference in the model calculations for the fcc materials other than their initial textures. Adding hardening behavior by (2) will not significantly alter the fcc texture results because (2) and the grain refinement criteria would not be linked in the model and all modes of slip harden equally. For the Be, on the other hand, we assumed *a priori* that the active slip systems were basal, prismatic, and pyramidal <a+c>. The associated Voce law parameters used in the simulation are listed in Table 1 and were determined by fits to compression tests on hot pressed (no initial texture) and rolled Be at 425°C. As in the experiment, we consider $\Phi = 90°$ with $\Psi = 20°$ for fcc materials and $\Phi = 120°$ with $\Psi = 0°$ for Be. We make the simple assumption that all deformation takes place on a shear plane parallel to the die intersection plane.

Slip Mode	τ_0	τ_1	θ_0	θ_1
Basal	100	130	3500	0
Prismatic	150	150	4500	70
Pyramidal <c+a>	420	1100	100,000	0

Table 1. Voce law parameters for each active slip mode assumed in ECAP of Be at 425°C.

Results and Discussion

Pole density figures were re-calculated from the spherical harmonics description of the ODF derived from the measurement using the Rietveld method. For initial and one ECAP pass on Ni, Cu and Al, (111) pole density figures are shown in equal area projection in figure 2. For Be, the (001) pole density figures are presented. In the same figure, corresponding modeled pole density figures are shown, based on predictions using the measured initial textures as input. We used about 1900 distinct orientations for the model to represent the initial textures. Texture indices for all eight experimental samples are listed in the first column of figure 2. Except for Al, the initial materials show almost random texture with some indication of a weak fiber texture. The initial Al shows a strong fiber texture pattern. These qualitative findings are supported by the texture indices of the initial materials.

The experimental texture after one pass ECAP for Ni and Cu show A and D partial fibers of almost the same strength while the B fiber texture component is relatively weak (see [5], sect. 5.1.3 for a discussion of texture components resulting from simple shear). Consequently, these pole figures have symmetry about the shear plane direction and normal (see figure 1 for the orientation of the shear plane). For Al, this symmetry is broken and the texture can be described by A and B fibers only. For all fcc metals, the maximum measured density is not strong, being less than four times random as expected for simple shear textures [5]. For all fcc materials, modeling predicts well the A fiber component and location and strength of the density maxima. However, contrary to the measurement, a strong B fiber is predicted for Ni and Cu, while it is relatively weak for Al. Considering this result and noticing that the strength of the initial wire drawn texture increases from Ni over Cu to Al, we suggest that this initial texture favors the A component while decreasing the B fiber component during simple shear.

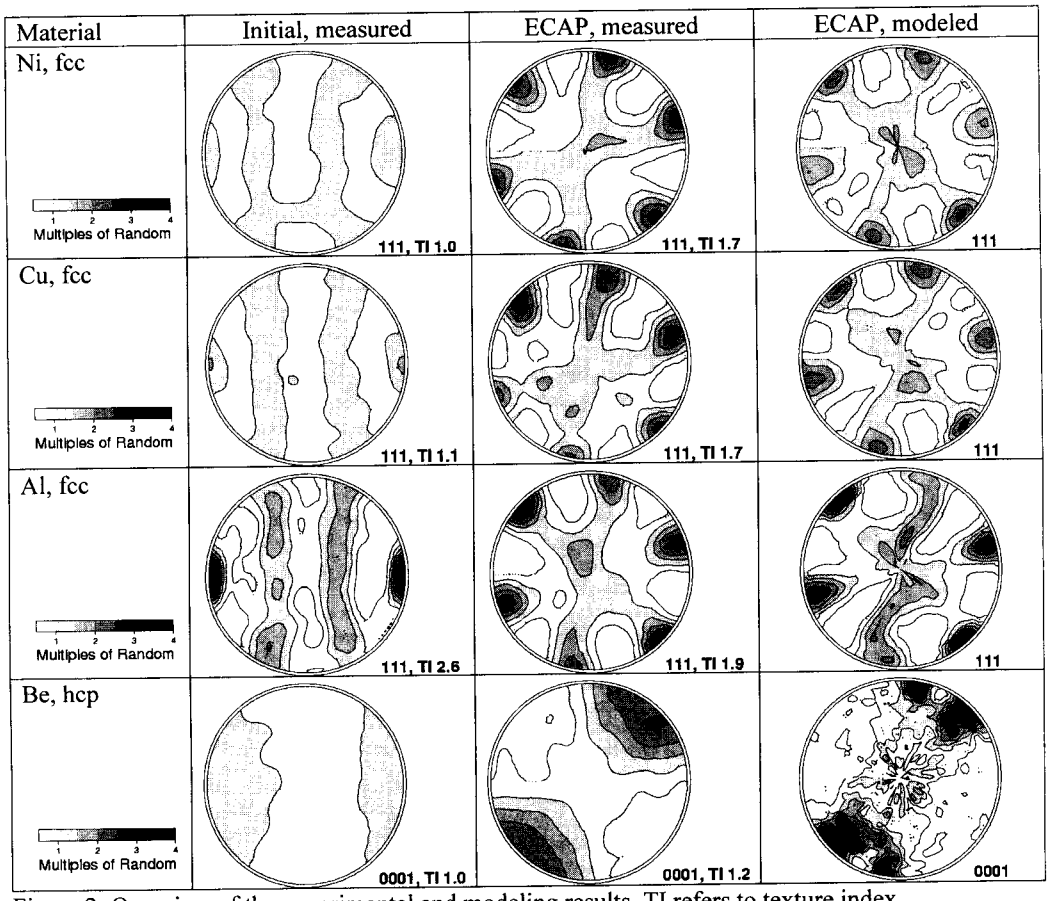

Figure 2: Overview of the experimental and modeling results. TI refers to texture index.

For the Be, on the other hand, a broad accumulation of c-axes is observed experimentally around the intersection between shear plane and equator. Note that the shear plane for the 120° die, used for ECAP of Be, is rotated 15° counterclockwise from that in figure 1. There is some experimental uncertainty with respect to the sample orientation for the Beryllium which had to be processed while encased in a nickel sheath (removed prior to the measurement). The maximum observed density is ~2.6 times random. Contrary to the measurement, less broad but split maxima of much higher density up to ~7.3 are predicted. For hcp materials, the model prediction is sensitive to the slip modes selected and their associated hardening laws. Better characterization of the τ_i and θ_i parameters at ECAP strain levels, strain rate and temperature will be required to eliminate any discrepancy with the measurement, such as the splitting of the maxima.

Summary

Textures after a single pass of ECAP were measured for three fcc metals, Ni, Cu and Al, respectively and one hcp metal, Be. Using neutron diffraction for the texture measurements allowed us to measure the texture averaged over a volume of 0.5 cm^3. Comparison of measured textures with modeling

predictions showed that the applied VPSC model captures the mechanisms governing texture at shear strain levels up to ~1.0 well for fcc metals, but more efforts to establish appropriate hardening parameters are required to model Be. Our work also emphasizes the importance of the initial texture on subsequent texture evolution during ECAP. To improve the predictions, the grain refinement process and texture evolution need to be coupled.

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