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Microstructural characteristics of pure gold processed by equal-channel angular pressing

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Experiments were conducted to evaluate the microstructural characteristics of pure (4N) gold processed by equal-channel angular pressing using routes A or B_c. Using atomic force microscopy and X-ray diffraction, it is shown that, although these two routes lead to similar dislocation densities of $\sim 1.5–1.7 \times 10^{15}$ m^{-2} and similar average grain sizes of ~460–490 nm, there are significant differences in the shearing patterns and in the densities of planar faults.

Keywords: Atomic force microscopy (AFM); X-ray diffraction (XRD); Deformation structure; Equal-channel angular pressing (ECAP); Gold (Au)

Severe plastic deformation (SPD) is an effective tool for producing ultrafine-grained (UFG) metals and alloys [1,2]. At the present time, the SPD procedure of equal-channel angular pressing (ECAP) is used most frequently because it is relatively easy to establish in any materials laboratory, it has a potential for scaling up to large samples and it produces reasonably homogeneous microstructures without any reduction in the cross-sectional dimensions of the samples [3]. To date, the effect on the microstructure of the various ECAP strain paths has been investigated for several face-centered cubic (fcc) metals, including Al [4–9], Ni [10] and Cu [11]. However, only limited information is available for Au [12,13], and for this metal there are no direct measures of either the dislocation densities after ECAP or the densities of any planar faults. Accordingly, the present research was undertaken to evaluate the microstructural characteristics of 24 carat (99.99% purity) Au processed by ECAP for four passes using either route A, in which the sample is pressed repetitively without rotation, or route B_c, in which the sample is rotated by 90° in the same sense between each separate pass [14].

The processing by ECAP was conducted at room temperature using samples having lengths of ~70 mm and cross-sectional areas of 10×10 mm^2. Pressing was conducted at a velocity of 0.1 mm s^{-1} using an ECAP die containing an internal channel bent through an abrupt angle of 90° and with an outer sharp corner so that the angle denoting the arc of curvature was 0°. It can be shown that these conditions give an equivalent strain of ~4.6 after a total of four passes [15].

Following ECAP, specimens were prepared for examination using atomic force microscopy (AFM) and X-ray diffraction. All observations were taken on the plane parallel to the side face at the point of exit from the die, generally designated the *Y* plane [3]. This plane was first mechanically polished to a mirror-like finish in three steps using 1 μm and 60 nm diamond paste and a 20 nm colloidal silica suspension. The surface was etched for 3 min at room temperature (20 °C) in a solution of 17 g potassium cyanide, 3.75 g potassium ferrocyanide, 3.75 g potassium sodium tartrate, 3.5 ml phosphoric acid and 1 ml ammonia in 250 ml water. The surface topographies were examined using an atomic force microscope (Solver P47H) operating in the contact mode with conductive silicon cantilevers having a resonant eigenfrequency of 14–27 kHz. Additional microstructural observations were also undertaken

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using X-ray diffraction line profile analysis. The X-ray line profiles were measured using a high-resolution diffractometer (Nonius, FR 591) with Cu $K_{\alpha 1}$ radiation and the line profiles were evaluated through use of the extended convolutional multiple whole profile fitting procedure [16]. In this method, the experimental profiles are fitted using theoretical profile functions calculated on the basis of a model microstructure where it is assumed that the lattice strains are caused by dislocations and planar faults. Through the use of this method, it is possible to obtain information on both the density of dislocations, ρ, and the density of planar faults, β.

The larger micrographs in Figures 1 and 2 show typical AFM images of the microstructures of the Au samples processed by ECAP using routes A and B_c, respectively, where the pressing direction is horizontal, the billet is moving from left to right and the bottom edges of the images correspond to the pressing, or X, direction. It is apparent from these micrographs that processing by ECAP produces a relatively homogeneous grain structure and, from inspection of the magnified smaller images, the average grain sizes for both processing routes are of the order of ~500 nm. It is apparent also that the grains have a slightly elongated configuration after processing through route A but that they are reasonably equiaxed after processing through route B_c; a similar trend was reported earlier in the processing of pure aluminum [5,6]. The AFM images reveal the formation of distinctive shear bands during ECAP. These mesoscopic traces, labeled Band 1 in Figures 1 and 2, are reasonably aligned with the ideal shearing plane when the sample passes through the die. Similar bands have been reported in experiments on other polycrystalline fcc metals processed by ECAP, including Al [17], Cu [18,19] and Ni [19], and the results are also consistent with data reported for the pressing of Al single crystals [20]. During subsequent deformation in tensile testing, earlier experiments on pure Au processed by ECAP showed that this shear localization leads to the development of macroscopic deformation bands [12].

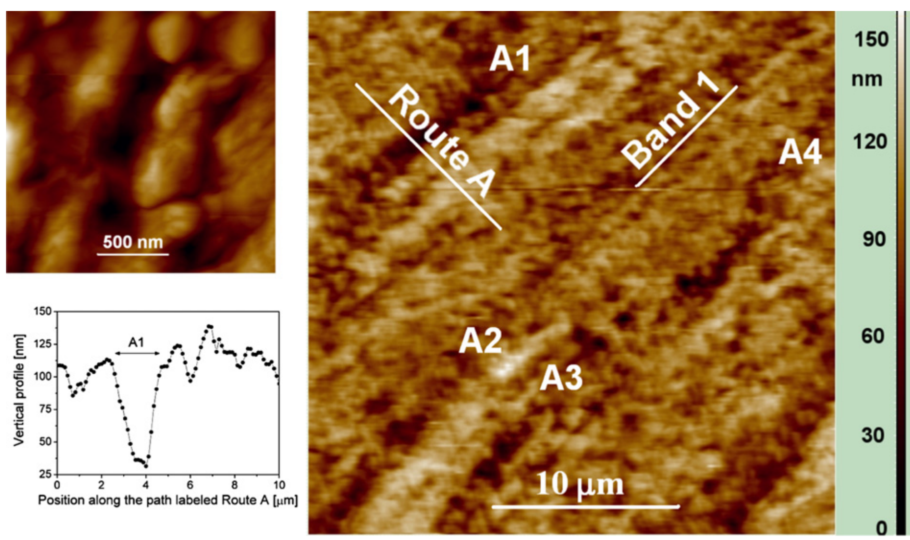

Figure 1. Typical AFM images and a representative surface vertical profile taken on the Au sample processed by ECAP using route A.

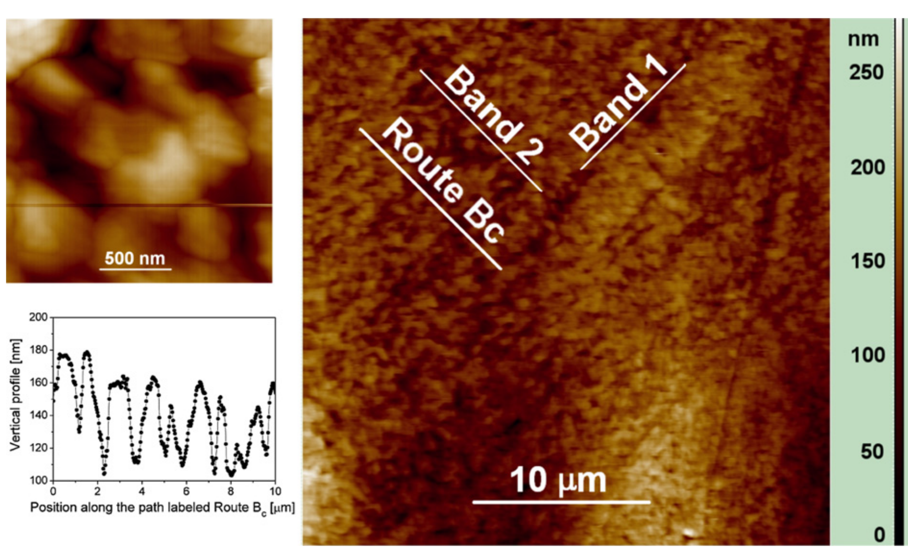

Figure 2. Typical AFM images and a representative surface vertical profile taken on the Au sample processed by ECAP using route B_c.