DOI: 10.1017/S1431927607075459
Microsc Microanal 13(Suppl 2), 2007

EBSD Investigations of Equal Channel Angular Extruded Copper

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Development of nano- and submicron-structured materials has attracted significant research interest in the last ten years [1, 2]. Most recently, an innovative technology called the Equal Channel Angular Extrusion (ECAE) process has demonstrated its capability of producing nano- and submicron-structured metallic alloys with substantial strength improvement [3-8]. ECAE adopts the principle of mechanical attrition and imposes very heavy shear deformation on bulk materials without causing major dimensional changes of the extruded products [3]. It has been suggested that this technology has great advantages over the conventional mechanical attrition of ball milling because it can produce large sized samples free of any residual porosity.

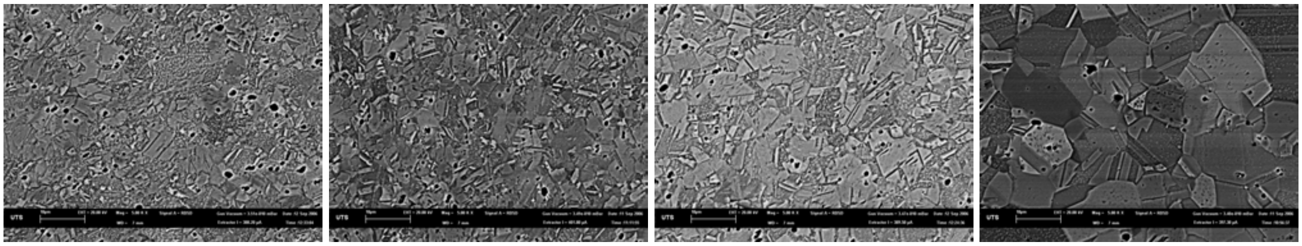
The ECAE process is different to conventional extrusions such that no major changes occur on the external dimensions between the feed and the extruded billet. The extrusion die is constructed with two channels of equal cross-section intersecting at a design angle. During the extrusion process, a section of metal with dimensions tightly fitted within the channel is pressed through the die to achieve a large plastic strain. Shear deformation occurs in the material when it passes through the angular intersection connecting the two channels. A high plastic strain is therefore imposed on the material but without changing its external dimensions. The extrusion process is repeated to increase the magnitude of the imposed strain on the material until a very high deformation is achieved.

In studying the structural development of metallic alloys in the ECAE process, Gholinia et al. [6] reported that as the shear strain increased, the banded structure vanished and the microstructure became largely isotropic and comprised of ultrafine grains/subgrains with a built-up of high angle boundary areas. As large deformation is involved in the ECAE process, it is expected that high strain energy is stored in the ECAE materials, which may provide a large driving force to substantial grain growth of the nanostructures. Growth of the nanograin structure may reduce the strength of the materials and restrict the application of these high performance materials. The present study aims to investigate the structural stability of nanostructured copper produced by the ECAE process, and to determine the transition temperature if substantial change of the nanostructures occurs.

A 15 mm diameter copper billet of commercial purity and an initial grain size of 20-50μm was extruded at room temperature by the ECAE process. An equal channel die of 120°, which corresponds to a shear strain of 1.15 for each pass, was used in this process. After extrusion of 15 passes, a total strain of ~17 was imposed on the deformed metal. Subsequent heat treatments at temperatures ranging from 27 to 500°C for 30 minutes were applied to the extruded metal for structural stability study. Scanning electron microscopy and electron back-scattered diffraction (EBSD) were performed to evaluate the microstructural changes in the metal. The specimens were prepared for EBSD by manual grinding on progressively finer grades of silicon carbide paper, until all visible damage from the previous step was removed, followed by a 3μm diamond polish on a nap cloth. Specimens were then electropolished at 18V for 18 seconds, then electrolytically etched at 40V for one second. 20% Phosphoric acid at a temperature of 12-16°C was used as the electrolyte for the electrolytic polish and etch procedures.

It was found that the nanostructures remained stable at temperatures up to 300°C. SEM investigations confirmed that at low annealing temperatures, the fine grain structures were stable and indeed became more equiaxed and well defined.

The extruded metal generally consisted of fine grained structures as those shown in Figure 1a. The grain sizes were in the order of 0.6 – 2.4 µm with an average of 1.0µm. After subsequent heat treatments, the grain structures of the metal became well defined. A fine grained structure was generally maintained at annealing temperatures up to 300°C (see Table 1), but coarse grains were also occasionally observed. The typical grain structure after annealing at 150°C and 300°C is shown in Figure 1b-c. At 500°C, a much larger grain size was observed with subsequent lowering of mechanical properties.



a. b. c. d.

Figure 1: Scanning electron microscopy showing fine grained structure of a) as extruded, b) heat treated copper at 150°C, c) heat treated copper at 300°C and d) heat treated copper at 500°C.

Table 1. Grain size measured for copper heat treated under different conditions.

Copper	Average Grain Size	Grain Size Range
As -received	30µm	10-50µm
After ECAE	1.1µm	0.1-2.4µm
150°C	1.5µm	0.3-10µm
300°C	3.4µm	0.5-11µm
500°C	12µm	2.0-30µm

A high volume of high angle grain boundaries (>15°) were identified in the extruded material. Orientation distribution of the grains was also investigated, showing no strong textures. Development of weak {110} components was however observed.

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