TEM AND SEM STUDY OF PRECIPITATION HARDENED CU-CO ALLOYS AFTER SEVERE PLASTIC DEFORMATION

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Abstract

The paper reports on preliminary results of microstructural study of Cu-2wt.%Co alloy processed by ECAP. The experimental alloy was cast and annealed at 1273 K for 10 hours followed by water cooling, resulting in the solid solution (the initial state). Further heat treatment was then applied to produce two-phase material with fine distribution of precipitates. ECAP was then conducted at room temperature on both the initial and the precipitation hardened states. Resulting microstructures were studied and characterized by microhardness testing, TEM observations and texture analysis by means of electron backscatter diffraction (EBSD) technique in SEM.

Keywords: microstructure, severe plastic deformation, electron microscopy

1. INTRODUCTION

Various ways of severe plastic deformation (SPD) of bulk metallic materials are thoroughly (re)examined in the last two decades. They aim at production of ultrafine-grained (UFG) structures with superior mechanical properties. Production and application of UFG materials require exploring efficient manufacturing techniques, theoretical modelling of manufacturing processes and materials performance, and investigation of the link between microstructure and enhanced properties in UFG materials.

The technique of equal-channel angular pressing (ECAP) is one of the most developed SPD techniques, capable of achieving a significant grain refinement and, at the same time, producing a large bulk billet with low porosity suitable for further mechanical testing. Fabrication of homogeneous UFG microstructures by ECAP is a complex process since the number of passes and the selected processing route can be very important parameters. The primary microstructural change associated with ECAP is the introduction of a very high density of dislocations into the crystalline lattice. ECAP may also influence the microstructure in other significant ways. The high pressure involved in the processing may lead to the fragmentation and decohesion of precipitates. Furthermore, depending upon the processing regimes, ECAP may lead also to precipitate dissolution and/or precipitate formation and coarsening. The interactions between these various processes will depend in practice upon both the alloy composition and the pressures and temperatures used in the pressing operation. In this work we report on our preliminary results of microstructural study of Cu-2wt.%Co alloy processed by ECAP after various heat treatment.

2. EXPERIMENTAL

The experimental alloy was cast and annealed at 1273 K for 10 hours followed by water cooling. Both scanning and transmission electron microscopy (SEM and TEM) confirmed homogeneous coarse grained microstructure in the initial state, with low dislocation density and with all Co dissolved in the matrix. Elastic modulus measured by depth sensing indentation test at load 100 mN was (135±5) GPa [7] and microhardness HV1 = 56. Further heat treatment was then applied in the range from 773 to 973 K to produce two-phase material with fine distribution of precipitates. ECAP was then conducted on both the initial and the
precipitation hardened states at room temperature with a die having internal angle of 90°. Up to twelve subsequent extrusion passes were performed at speed of 10mm/min. The billet was rotated by 90° in the same direction after each pass (route B<sub>C</sub>) [5]. Selected samples underwent creep test after ECAP. Polished sections perpendicular to extrusion (and creep) direction were studied after 1, 2, 4, 8 and 12 passes using electron backscatter diffraction (EBSD) technique in a TESCAN LYRA 3 XMU FEG/SEM×FIB scanning electron microscope equipped with Nordlys EBSD detector by Oxford Instruments with Channel5 and Aztec HKL software.

3. RESULTS AND DISCUSSION
Starting from the grain size well above 1mm in the initial state, ECAP subsequently reduces grain size down to the submicron level. Significant changes in the grain size as well as in the relative amount of high angle grain boundaries were observed between 2<sup>nd</sup> and 4<sup>th</sup> ECAP pass of the initial state [7]. Further increasing number of passes mainly improved the microstructure homogeneity. The mean grain size after 12 ECAP passes reached 0.3µm. Pure Cu 99.99% processed in the same way and reported in our previous work [6] ended at about 0.6µm. Creep tests conducted at 673 K and 50 MPa confirmed a substantial improvement of mechanical properties due to ECAP processing. In comparison with the initial state, time to fracture increased as well as fracture elongation. Fig. 1 shows the results of microstructural studies of samples after ECAP and creep by means of EBSD in SEM.

![EBSD orientation maps of samples after ECAP and creep](image)

**Figure 1.** EBSD orientation maps of samples after ECAP and creep. Samples differ in the ECAP processing prior to creep: 2 passes (a), 4 passes (b) or 8 passes (c) were applied.

Grain growth during the creep tests at 673 K was observed, the final average grain size reaching 4-5 µm after creep fracture. A detailed quantitative analysis of EBSD results in Fig. 1 showed decreasing proportion of low angle boundaries and increasing amount of special high angle boundaries (namely Σ3 and Σ9) with increasing number of ECAP passes prior to creep testing. In all studied cases low Σ grain boundaries form the majority of all high angle grain boundaries detected by EBSD. Special high angle boundaries are often reported [8] to be strengthened against sliding and deformation, thus improving the creep resistance.

Thermal processing was applied to the initial state aiming at preparation of various forms of precipitation strengthened material. In the whole range of applied temperatures 773-973 K the heat treatment of the initial state resulted in the homogeneous distribution of fine coherent Co precipitates differing mainly in their size.
and density (Fig. 2). Measured values of microhardness HV1 changed correspondingly; reaching 91 after annealing 873 K / 20 min and 93 after annealing 973 K / 20 min (longer annealing at 973 K resulted in the precipitate coarsening and a decrease of HV1).

**Figure 2.** TEM micrographs of Cu-2wt.%Co alloy annealed at 873 K for 20 min (left) and at 973 K for 80 min (right).

**Figure 3.** EBSD orientation maps. The microstructure of the initial state with colour legend (a), the microstructure after four ECAP passes of the sample annealed at 873 K for 20 min at low and medium magnification (b) and the microstructure after four ECAP passes of the sample annealed at 973 K for 80 min at low and medium magnification (c).

The first ECAP experiments on heat treated samples reveal an inhomogeneous grain size distribution with high proportion of low angle boundaries. The microstructure after four ECAP passes is shown in Fig. 3 for two selected annealing regimes together with the microstructure of the initial state. By comparison of these results with previous ones [7] obtained on single phase Cu-2wt.%Co alloy we can conclude that dispersion of
small coherent precipitates slows down the grain refinement and grain size homogenization during ECAP processing. Markedly bimodal grain size distribution (see Fig. 3b,c) of samples ECAPed after heat treatment may in general improve deformation behaviour of nano- and UFG materials as the stress concentrations can be relaxed through plastic deformation in larger grains [9].

4. SUMMARY

The paper reports on preliminary results of microstructural study of Cu–2wt.%Co alloy processed by ECAP either in the initial state of solid solution or after various heat treatments. Resulting microstructures were studied and characterized by microhardness testing, TEM observations and texture analysis by means of electron backscatter diffraction (EBSD) technique in SEM. ECAP of the initial state substantially improves the creep properties. The heat treatment in the range from 773 to 973 K was shown to produce two-phase material with fine distribution of coherent Co precipitates. ECAP conducted at room temperature on the precipitation hardened materials shows that fine dispersion of precipitates slows down the grain refinement and grain size homogenization during ECAP processing. Combination of ECAP with prior thermal treatment leads to the formation of bimodal grain size distribution with possible improvement of deformation behaviour.

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REFERENCES