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Microstructure of a low alloyed Cu-Cr-Zr alloy after ECAP-Conform

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Abstract. The microstructure of a Cu-0.1%Cr-0.1%Zr alloy after annealing or aging and equal channel angular pressing with conform (ECAP-C) process was investigated. ECAP-C led to formation of strain induced low-angle boundaries (LAB) which transformed in high-angle boundaries (HAB) during deformation. Deformation microbands developed after 1 ECAP-C pass, leading to new ultrafine grain formation in the microbands upon further processing. Average grain size gradually decreased with strain while dislocation density increased. After 8 ECAP-C passes average grain size achieved 1 µm. Maximum of LAB density occurred after 4 ECAP passes and then LAB density decreased with strain while HAB density gradually increased during ECAP-C. Relationships between dislocation density, grain size, and density of crystallite boundaries were discussed.

1. Introduction.

Cu-Cr-Zr alloys are perspective candidates for electrical application due to combination of high electrical conductivity and strength after appropriate treatment [1-3]. The most investigated and prevalent treatment of Cu-Cr-Zr alloys is severe plastic deformation (SPD) with annealing [4-5]. Ultrafine grained structure with fine particles providing high strength and conductivity can be formed by different deformation methods and heat temperatures. The main disadvantage of SPD technique is production of small samples with volume less 10⁻⁴ m⁻². New SPD technique combining equal channel angular pressing and conform process can help to solve this problem [6]. ECAP-C can produce long rods and wires with unique properties, such as electrodes for point welding, automobile wire, contact wire for high speed trains, etc. [7]. However, there is a deficiency of experimental data about microstructure evolution of Cu-Cr-Zr alloys during ECAP-Conform and effect of microstructure on mechanical properties and electrical conductivity. The aim of the present work is to study the microstructure evolution of a Cu-Cr-Zr alloy during ECAP-Conform.

2. Experiment

A Cu-0.1%Cr-0.1%Zr alloy was chosen as the starting material. The billets were annealed at 920 °C during 1 h and water quenched (ST). Then, a part of the samples was aged at temperature of 550 °C during 4 h (AT). Both types of samples were subjected to ECAP-C at room temperature via route B_C to 1, 2, 4, 8 passes. The intersection angle of matrix channels was 120 °. The microstructures were



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investigated using a Nova NanoSEM 450 scanning electron microscope with an electron backscattered diffraction (EBSD) analyzer. The average grain and subgrain size were estimated as average values between transversal and longitudinal boundary and subboundary intercept. The dislocation density was determinated as function of the Kernel Average Misorientation [8]. The fraction of ultrafine grains (with a size below 2 micron), the density of crystallite boundaries, average misorientation angle, Kernel average misorientation (for scan step of 200 nm) were estimated using the OIM Software. The specimens for microstructure investigations were polished by 25% HNO₃ and 75% CH₃OH electrolyte at temperature of -20 °C and voltage of 10 V by a Tenupol 5 machine.

3. Results and discussion

Microstructure in initial state

After solution treatment at 920 °C, the Cu-Cr-Zr alloy contained low fraction of course particles that crystallized during cooling of ingot. The average grain size was about 140 μ m, and the dislocation density was about 10¹³ m⁻². Aging at 450 °C led to precipitation of fine ellipsoid bcc Cr-rich particles. The size of particles in the longitudinal direction and transverse direction was 10-15 nm and 5-7 nm, respectively. Numerical annealing twins were observed .HAB fractions were above 0.98 and the average misorientation angles comprised 45-48° in both initial states. Kernel Average misorientations over a distance of 3 μ m were about 0.5°.



Figure 1. Microstructure evolution during ECAP-C in the Cu-Cr-Zr alloy. Black and white lines represent HAB and LAB, respectively.

Microstructure after ECAP-C

ECAP-C was accompanied by the formation of strain-induced LAB and elongation of initial grains. The deformation microbands consisting of long parallel HABs splitted initial grains. Chains of crystallites with irregular LAB-HAB were observed in AT samples after 1 ECAP-C pass. Further deformation led to increase in misorientations of LABs with subsequent transformation of LABs into HABs. New

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ultrafine grains formed predominantly in deformation microbands. Fraction of ultrafine grains increased significantly after 4 ECAP-C passes.8 ECAP-C passes led to formation of equiaxed crystallites with a size about 0.5 µm and high fraction of HAB.



Figure 2. Effect of ECAP-C on grain (D) and subgrain size (d), fraction of HAB (F_{HAB}), dislocation density (ρ), Kernel average misorientation (θ_{Ker}), fraction of ultrafine grains (F_{UFG}), average misorientation angle of grain boundaries (θ_{av}), density of HAB (ρ_{HAB}), LAB (ρ_{LAB}) and all crystallite(ρ_{CB}).

ECAP-C at room temperature led to significant grain refinement along with an increase in dislocation density and microstrain. Gradual transformation of strain-induced LAB into HAB during deformation decreased the mean grain size from 120-140 um to 1 um. HAB fraction slowly changed after 1-2 ECAP-C passes followed by significant increase after 4-8 passes. Deformation was accompanied by an increase in HAB fraction to 0.36 and 0.34 in ST and AT condition after 8 ECAP-C passes, respectively. Dislocation densities in ST and AT conditions approached an apparent saturation at 66.5*10¹⁴ m⁻² and 9*10¹⁴ m⁻² after 4 ECAP passes, respectively. Tendencies of change in average boundary misorientation angle and UFG fraction were the same, i.e., a kind of incubation period during 1-4 passes and a drastic increase after 8 ECAP-C passes. ECAP-C promoted an increase in the boundary density. The density of LAB achieved maximum about 4.7-4.8 µm⁻¹ after 4 ECAP-C passes and then decreased to 2.9 µm⁻¹ and 4.7 µm⁻¹ for ST and AT condition, respectively. The density of HAB in AT samples was higher than in ST condition and gradually increased with deformation that testify to the development of LAB to HAB transformation.

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Figure 3. Relationship between grain size (D) and dislocation density (ρ); the density of HAB (ρ_{HAB}), LAB (ρ_{LAB}), and crystallites boundaries (ρ_{CB}) vs the dislocation density.

It is clearly seen that the relationship between the grain size D and the dislocation density ρ can be expressed by logarithmic law:

 $D = -43.2\ln(\rho) + 86 \quad (1)$

An increase in the dislocation density during ECAP-C led to the formation smaller grains. The dislocation density affected the development of strain-induced boundaries. The change in LAB density ρ_{LAB} can be described by sigmoidal law:

 $\rho_{LAB} = 5 + 4 \exp(-3\rho - 19)$ (2) In contrast, the HAB density (ρ_{HAB}) increased with dislocation density according to logarithmic law: $\rho_{HAB} = 2.3 \ln(\rho) - 3.2$ (3)

Equitations (1-3) good described experimental correlation of grain size and grain boundaries density with dislocation density. An increase in dislocation density promoted their rearrangement in strain-induced boundaries, which quickly increased their misorientation, transformed in HAB, leading to new ultrafine grains.

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