

Models of damage mechanism of glidcop Cu-Al₂O₃ micro and nanomaterials

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ABSTRACT

Purpose: of this paper was to analyze the fracture mechanism before and after ECAP in the Glidcop AL-60 grade (with 1.1 wt. % of Al₂O₃) system and to propose damage and/or fracture mechanisms models by means of the method “in situ tensile test in SEM”.

Design/methodology/approach: The method of “in-situ tensile testing in SEM” was used for investigations of fracture mechanisms because it enables to observe and document deformation processes directly, thank to which the initiation and development of plastic deformation and fracture can be reliably described. Analyses of microstructure and fracture surfaces were carried out by means of the scanning electron microscope JEM 100 C.

Findings: The deformation and fracture mechanisms of Glidcop AL-60 grade with 1.1 wt. % of Al₂O₃ phase (1.62 vol. % of Al₂O₃) were analyzed before and after ECAP (Equal Channel Angular Pressing). Before ECAP it was shown that the deformation process causes increasing of pores and formation of cracks. Decohesion of small Al₂O₃ particles and clusters occurs and the final fracture path is influenced by coalescence of cracks originated in such. The principal crack propagates towards the sample exterior surface. After ECAP initial cracks were formed in the middle of the specimen first of all in the triple junctions of nanograins and together with decohesion of Al₂O₃ particles and clusters at small strains lead to the failure.

Research limitations/implications: To develop more complex knowledge about the objective material further studies are necessary to focus also on the other factors which besides the secondary phase amount can influence the failure mechanism, e.g. strain rate, temperature and others. Complex analysis allows better understanding of material behavior at different conditions and possibilities of application of products from these materials will be thereby improved.

Practical implications: This article completes knowledge about damage/fracture mechanisms and processes of the material with 1.1 wt. % of Al₂O₃ phase. Some materials with the different volume fraction of a secondary phase have been studied. This concrete one with 1.1% clarifies the fracture process of Glidcop AL-60 material not only after mechanical alloying process but also after ECAP treatment. An effect of the ECAP process on the final material was crucial because not only microstructure but also failure mechanism have been changed.

Originality/value: Based on the experimental observations original models of damage and/or fracture mechanisms were proposed.

Keywords: Glidcop AL-60 grade; Mechanical alloying; ECAP; Fracture mechanism; In-situ

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ANALYSIS AND MODELLING**1. Introduction**

Copper has the leading role in industrial applications. A variety of Cu alloys has been developed but they exhibit a rather large increase of resistance in both electrical and heat conductivity and low time stability at elevated temperatures. Powder metallurgy can give a solution in dispersing particles in the prepared material with appropriate characteristics [1]. One of the leading candidates for practical application is an industrial material called Glidcop made by SCM Metal Products, Inc. Glidcop is a metal matrix composite alloy (MMC) prepared by mixing copper primarily with aluminum oxide ceramic particles. The addition of small amounts of aluminum oxide has minuscule effects on the performance of the copper at room temperature (such as a small decrease in thermal and electrical conductivity), but greatly increases the copper's resistance to thermal softening and enhances high elevated temperature strength [1]. The addition of aluminum oxide also increases resistance to radiation damage. As such, the alloy has found use in applications where high thermal conductivity or electrical conductivity is required while also maintaining strength at elevated temperatures or radiation levels. Owing to the excellent high-temperature properties and sufficiently high values of electrical and thermal conductivity, the dispersion-strengthened Cu-Al₂O₃ materials, prepared by the methods of powder metallurgy, have found use as conductors in electrical machines employed at high temperatures, in contacts, in electrodes and in vacuum technique parts.

In a work [2] two fracture micromechanisms of nano Cu are identified by the fracture surface analysis (see Fig.1).

Depending on the amount of plastic deformation accumulated by the repeated ECAP passes:

Fracture surfaces up to 14 ECAP passes (90° channel angle) had transcrystalline character with the dimple morphology. With the growing number of ECAP passes, the dimple size decreases and the quantity of dimples increases. Dislocation coalescence and changes in the triple grain junctions manifested by the increasing number of profile vertices with the amount of deformation are the probable fracture mechanisms affecting the fracture initiation, i.e. the first stage of the ductile fracture formation. Void growth and

void coalescence are controlled by usual mechanisms.

Mixed fracture surfaces with intercrystalline facets and fine dimple ductile fracture surface are typical after more than 14 ECAP passes. It is assumed that microcracks on high angle boundaries and plasticity exhaustion are the reasons of the different deformation mechanism.

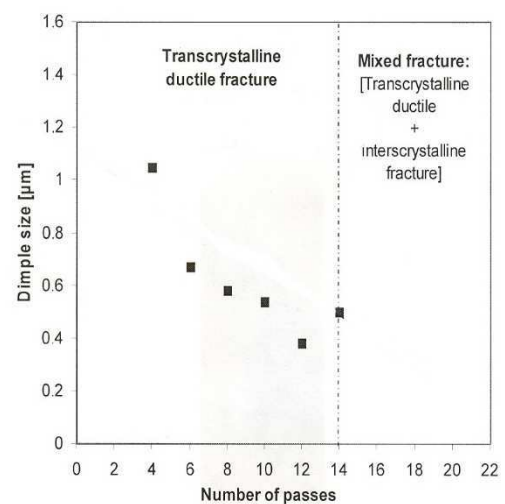


Fig. 1. Dimple size in dependence on number of ECAP passes

The fracture of experimental Cu-Al₂O₃ and Cu-TiC systems by direct monitoring of the strain and fracture in a scanning electron microscope by “in situ tensile test in SEM” was analyzed in [3-5]. Both systems were prepared by different powder metallurgy technologies. The dispersed oxides and carbides in the matrix were not coherent. Differences in the particle size and distribution caused differences in the fracture mechanism, although both fractures were ductile transcrystalline with dimples. In the present work we extend our activities to a commercially available industrial product with a goal to relate its properties to those of the previously investigated materials. The method of the in situ tensile test in SEM [6] is a powerful method to investigate mechanism of the initiation and propagation of microcracks in materials and was used for other materials, too [7-19]. It is a useful technique allowing the direct observation of crack formation and propagation on microscopical scales. Though a number of works focused on the MMC have been suggested in the

literature the works focused on the fracture of micro- and especially nano-grain MMC are still limited.

The purpose of this paper is to study the fracture mechanism in the Glidcop AL-60 grade system (1.62 vol. % of Al_2O_3) before and after ECAP (micro and macro scale) and to propose damage models.

2. Experimental material and methods

A Glidcop AL-60 grade with 1.62 vol. % of Al_2O_3 prepared by mechanical milling was used for all experiments, as the material before ECAP (A) and after ECAP (B). More details on the preparation and properties of the experimental material are described in [1].

For the purposes of investigation very small flat tensile test pieces (7x3 mm) were prepared, keeping the loading direction identical to the direction of extrusion. They were ground and polished mechanically. The final operation consisted in double-sided final polishing of specimens

in an ion thinning machine to a thickness of approximately 0.1 mm.

The test pieces were fitted into special deformation grips inside the scanning electron microscope JEM 100 C, which enabled direct observation and measurement of the deformation by ASID-4D equipment.

3. Results

3.1. Status before ECAP

Microstructure of the experimental material contained, besides the Al_2O_3 dispersoid secondary phase, also a low volume fraction, approx. 0.5 vol. %, of closed sharp edged pores (Fig. 2a). Size of Al_2O_3 particles was less than 10 nm, the particles had a globular shape and occurred separately and also in clusters. Clusters of particles were randomly distributed in the matrix. The size of pores was significantly higher than the particles size, approximately 1-3 μm . The approximate size of matrix grains was $\sim 5 \mu\text{m}$.

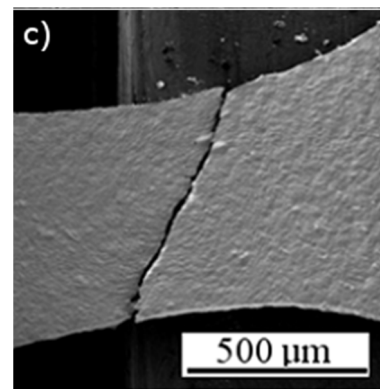
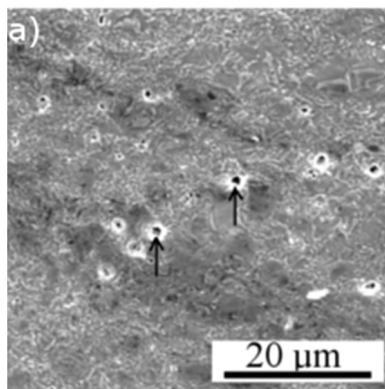


Fig. 2 a,b. Glidcop before ECAP a) microstructure, b) fracture of the material

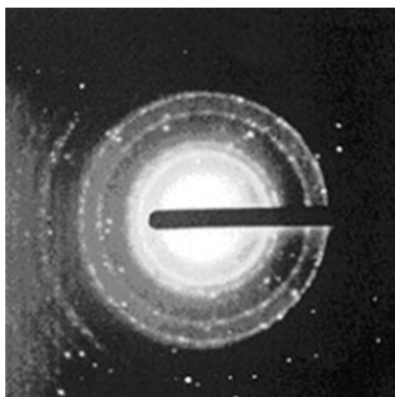


Fig.3 Diffractogram of Al_2O_3 particles

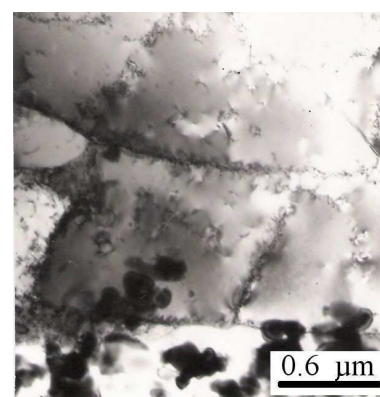


Fig. 4. Substructure of Glidcop before ECAP

The experimental material was deformed at 20°C at a strain rate of $6.6 \times 10^{-4} \text{ s}^{-1}$ in the elastic region. During deformation of samples the first cracks were created on particle clusters and then on pores within the sample. Further deformation of the samples caused the crack propagation due to the internal effect of the particle clusters (decohesion of small clusters of spheroid Al_2O_3 particles with size $< 100 \text{ nm}$) and pores.

Initiation, crack propagation and fracture before ECAP were in the plane of maximum shear stresses e.i. cca 45° angle to the direction of tensile loading, Fig. 2b. Macroscopic deformation was too small. Resulting fracture was the transcrystalline ductile with dimples with size $< 1 \text{ }\mu\text{m}$. Dimples had a regular Poisson-type distribution, unlike the pores which were distributed irregularly.

Presence of Al_2O_3 particles was confirmed by the electron diffraction, Fig. 3. The substructure of the material before ECAP is shown in Fig. 4.

3.2. Status after ECAP

ECAP was realized at room temperature by two passes by route C. The experimental material was pressed through two right angled (90°) channels of a special die [2]. Substructure obtained by TEM in Fig. 5 shows Al_2O_3 particles sized $\sim 10 \text{ nm}$ as well as nanograins with the size of 100–200 nm. It was found that ECAP refined the grain size of the matrix material. Electron diffraction confirmed presence the both Cu and Al_2O_3 phases, Fig. 6.

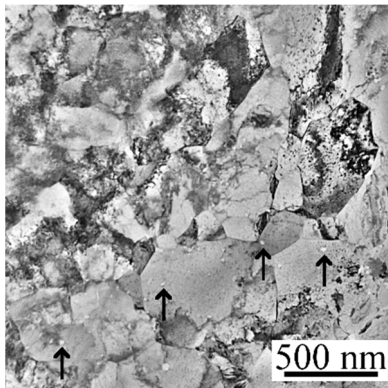


Fig. 5. Substructure of Glidcop after ECAP

The experimental material was deformed at the same conditions as the material before ECAP. Fig. 7 shows the fractured specimen without significant plastic deformation. A fracture surface is perpendicular to the direction of loading and crack propagation, what is caused by normal stresses. Initiation of cracks is likely in the triple junctions

resp. in nanograin boundaries, what is in agreement with the work [12]. Decohesion of Al_2O_3 particles and clusters contributed to the crack initiation, too. It is visible in Fig. 8 where transcrystalline ductile fracture of the material is showed.

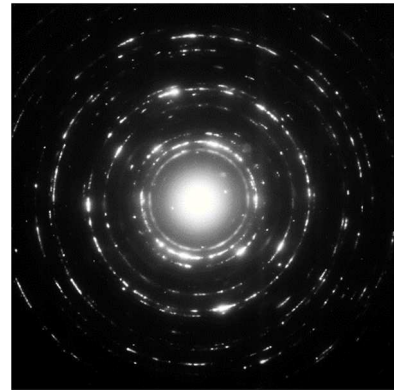


Fig. 6. Electron diffraction of both Cu and Al_2O_3 phases

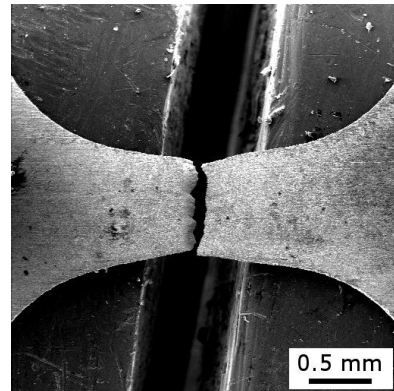


Fig. 7. Fractured specimen

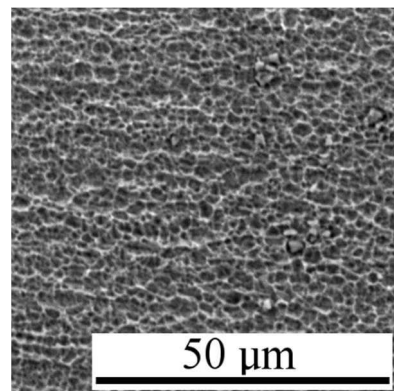


Fig. 8. Transcrystalline ductile fracture

2. Discussion and fracture models

A detailed study of the deformation changes showed that the crack initiation was caused by decohesion of the particles, and occasionally also by the coalescence of pores. Decohesion is a result of different physical properties of different phases of the system. The Cu matrix has significantly higher thermal expansion coefficient and lower elastic modulus $\alpha = 17.0 \times 10^{-6} \text{ K}^{-1}$, $E = 129.8 \text{ GPa}$ than Al_2O_3 $\alpha = 8.3 \times 10^{-6} \text{ K}^{-1}$ and $E = 393 \text{ GPa}$. Large differences in the thermal expansion coefficients result in high stress gradients, which arise on the interphase boundaries during the hot extrusion. Since $\alpha_{\text{matrix}} > \alpha_{\text{particle}}$, high compressive stresses can be expected. However, because the stress gradients arise due to the temperature changes, during cooling (which results in increase of the stress peaks) their partial relaxation can occur. The weak interface cannot withstand the external load and the internal stresses which are large enough to cause microcracks, so microcracks are mainly initiated by interface decohesion and the failure mechanism of the composite is interface-controlled.

Physical properties of the reinforcement as well as presence of their clusters influence formation of microcracks. According to Clyne and Withers [20], two distinct processes of the reinforcement clusters are probably

present during plastic deformation. First, the clusters can deform collectively as a group somewhat like a single hard particle, so that the deformation within the matrix at the heart of the cluster is much less than in the composite as a whole. Such process leads to the formation of dimples. On the other hand, tensile hydrostatic stresses in the matrix proposed by Prangnell et al. [21] will be relaxed by diffusion and void nucleation, resulting in the ductile fracture. While in the second process, the particles behave independently, so that the deformation within the cluster is much greater than in the composite overall.

4.1. Fracture model before ECAP

Based on the microstructure changes observed in the process of deformation, the following model of the fracture mechanism before ECAP was proposed, Fig. 9 a, b, c:

- the microstructure in the initial state is characterized by Al_2O_3 particles their clusters and pores in matrix,
- with increasing of tensile load local cracks on pores occurred, there are cracks formed by decohesion of Al_2O_3 particles and clusters,
- with further increase of deformation of the material crack propagates preferentially along the pores and decohesed particles and clusters in a 45° angle.

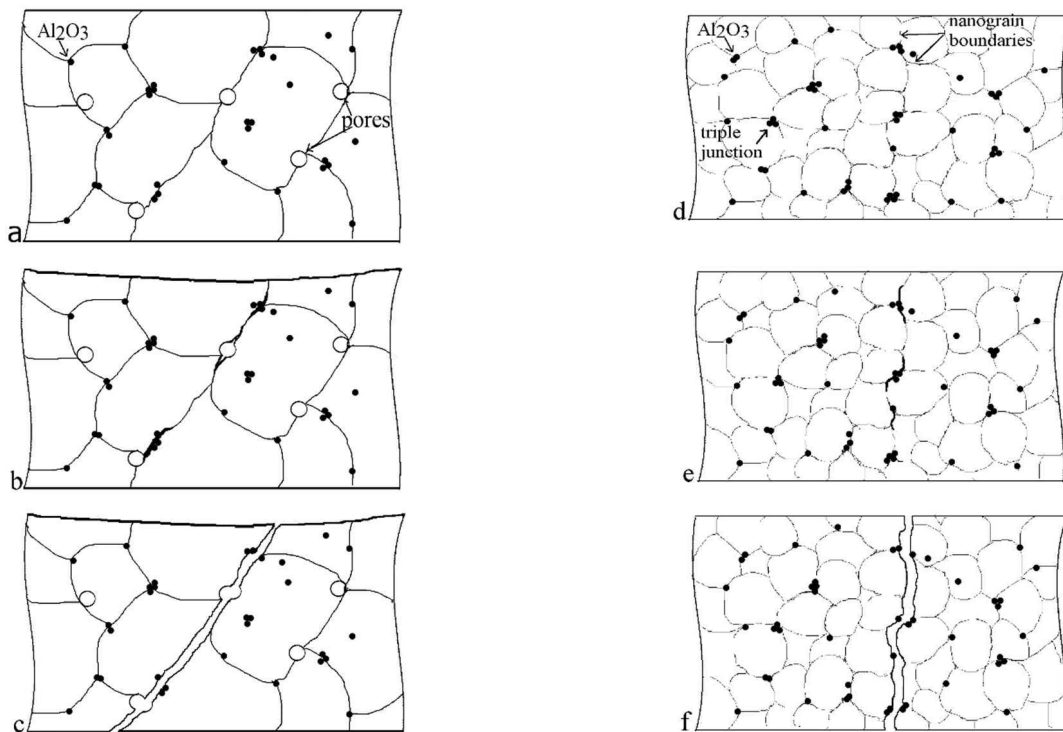


Fig 9. Model of the fracture mechanism before ECAP (a, b, c) and after ECAP (d, e, f)

4.2. Fracture model after ECAP

The following model of the fracture mechanism of the ECAPed material was proposed, Fig. 7d, e, f:

- initiation of fracture is caused by cracks in triple junctions as well as by decohesion of Al_2O_3 particles and clusters in the middle of the specimen,
- fracture is propagated very quickly at minimum plastic deformation,
- fracture is perpendicular to the tensile loading of the specimen.

Fracture is uniform, transcrystalline ductile with the dimple morphology. Dimples on the fracture surface can be divided into two categories: small dimples ($\sim 0.1\ \mu\text{m}$) initiated by the discrete Al_2O_3 particles and large ones ($\sim 2.5\ \mu\text{m}$) initiated by clusters of Al_2O_3 particles. Fracture process takes place in three stages: initiation, growth and coalescence of dimples.

5. Conclusions

The aim of the study was to analyze the fracture mechanism before and after ECAP in the Glidcop AL-60 grade (with 1.1 wt. % of Al_2O_3) system and to propose the damage models by means of the method "in situ tensile test in SEM".

Based on the microstructure changes, obtained in the process of deformation, a model of fracture mechanism of the material before ECAP was proposed. With increasing tensile load the local cracks formed predominantly along the pores and decohesion of smaller Al_2O_3 particles and clusters occurred. Fracture after ECAP was initiated by cracks in triple junctions of nanograins and by decohesion of Al_2O_3 particles and clusters.

Acknowledgements

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Additional information

Selected issues related to this paper are planned to be presented at the 22nd Winter International Scientific Conference on Achievements in Mechanical and Materials Engineering Winter-AMME'2015 in the framework of the Bidisciplinary Occasional Scientific Session BOSS'2015

celebrating the 10th anniversary of the foundation of the Association of Computational Materials Science and Surface Engineering and the World Academy of Materials and Manufacturing Engineering and of the foundation of the Worldwide Journal of Achievements in Materials and Manufacturing Engineering.

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