Fracture mechanisms of Glidcop Cu-Al₂O₃ composite before and after ECAP observed by "in-situ tensile test in SEM"

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Abstract

The method of "in-situ tensile testing in SEM" is suitable for investigations of fracture mechanisms because it enables to observe and document deformation processes directly, thanks to which the initiation and development of plastic deformation and fracture can be reliably described. 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) using technique of the "in-situ tensile testing in SEM". Before ECAP it was showed that the deformation process caused increasing of pores and formation of cracks. Decohesion of small Al₂O₃ particles and clusters occurred and the final fracture path was influenced by coalescence of originated cracks. The principal crack propagated towards the sample exterior surface. After ECAP initial cracks were formed in the middle of the specimen first of all in triple junctions of nanograins and together with decohesion of Al₂O₃ particles and clusters at small strains led to the failure. Based on the experimental observations a model of damage and/or fracture mechanisms has been proposed.

 ${\rm K\,e\,y}$ words: Glidcop AL-60 grade, mechanical alloying, ECAP, fracture mechanism, "in-situ"

1. Introduction

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 aluminium oxide ceramic particles. The addition of small amounts of aluminium oxide has minuscule effects on the performance of the copper resistance to thermal softening and enhances high elevated temperature strength [1]. The addition of aluminium oxide also increases resistance to radiation damage.

We have analyzed, as reported in [2–4], the frac-

ture 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". Both systems were prepared by different powder metallurgy technologies. The dispersed oxides and carbides in the matrix were not coherent. Differences in particle size and distribution caused differences in 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 in situ tensile test in SEM [5] is a powerful method to investigate the mechanism of initiation and propagation of microcracks in materials and was used for other materials, too [6-18]. It is a useful technique allowing the direct observation

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of crack formation and propagation on microscopical scales. Though a number of the 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 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, and after ECAP. More details on the preparation and properties of experimental material are presented in [1].

For the purposes of investigation, very small flat tensile test pieces $(7 \times 3 \text{ mm}^2)$, thickness of approximately 0.1 mm) were prepared, keeping the loading direction identical to the direction of extrusion. The test pieces were fitted into special deformation grips inside the scanning electron microscope JEM 100 C, which enables direct observation and measurement of the deformation by ASID-4D equipment.

3. Results

3.1. Status before ECAP

Microstructure of the experimental material contains, besides the Al₂O₃ dispersoid secondary phase, also a low volume fraction, approx. 0.5 vol.%, of closed sharp edged pores (Fig. 1a). Size of Al₂O₃ particles is less than 10 nm, the particles have a globular shape and occur separately and also in clusters. Clusters of particles are randomly distributed in the matrix. The size of pores is significantly higher than the particles, approximately 1–3 μ m. The approximate size of matrix grains was ~ 5 μ m.

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 inside the sample. Further deformation of samples causes the propagation of crack due to internal effect of the particle clusters (decohesion of small clusters of spheroid Al₂O₃ particles with size < 100 nm) and pores, Fig. 1b.

Initiation and crack propagation before ECAP is in the plane of maximum shear stresses, i.e., ca 45° angle to the direction of tensile loading, Fig. 1c. Macroscopic deformation is too small. Resulting fracture is transcrystalline ductile with dimples with size $< 1 \ \mu m$. The dimples have a regular Poisson-type distribution,

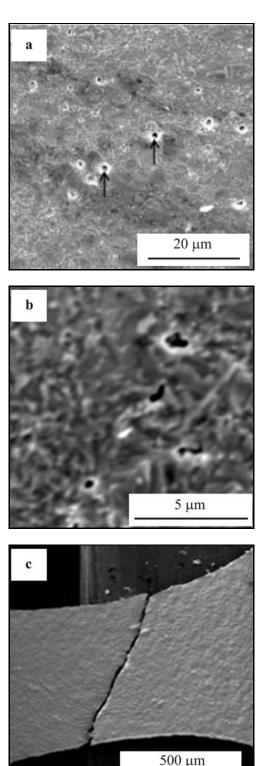


Fig. 1a,b,c. Glidcop before ECAP.

unlike the pores which are distributed irregularly.

3.2. Status after ECAP

ECAP was realized at room temperature by two

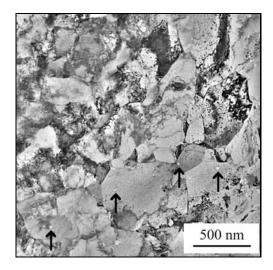


Fig. 2. Substructure of Glidcop after ECAP.

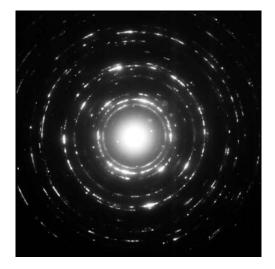


Fig. 3. Electron diffraction of both Cu and Al₂O₃ phases.

passes by route C [18]. Substructure obtained by TEM in Fig. 2 shows Al_2O_3 particles sized ~ 10 nm as well as nanograins with size 100–200 nm. It was found that ECAP refined the grain size of matrix material. Electron diffraction confirms presence of both Cu and Al_2O_3 phases, Fig. 3.

The experimental material was deformed at the same conditions as the material before ECAP. Figure 4 shows 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 triple junctions, resp. in nanograin boundaries, what is in agreement with the work [11]. Decohesion of Al_2O_3 particles and clusters contributes to crack initiation, too. It is visible in Fig. 5, where transcrystalline ductile fracture of material is showed.

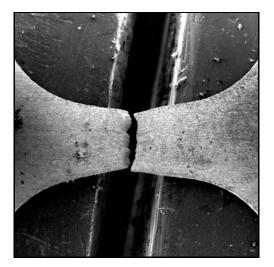


Fig. 4. Fractured specimen.

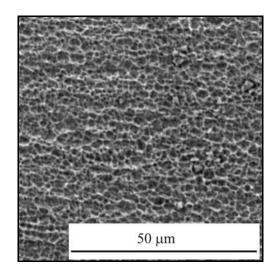


Fig. 5. Transcrystalline ductile fracture.

4. 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 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 GPa than Al₂O₃, $\alpha =$ $8.3 \times 10^{-6} \text{ K}^{-1}$ and E = 393 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

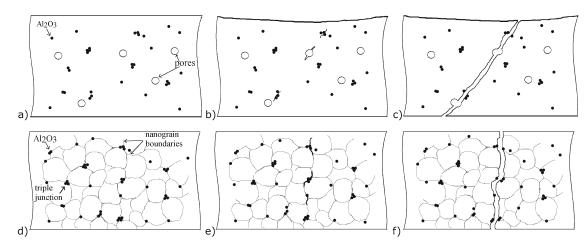


Fig 6. Model of the fracture mechanism before ECAP (a, b, c) and after ECAP (d, e, f).

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 [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, the 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. 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 fracture mechanism before ECAP is proposed, Fig. 6a,b,c:

a) the microstructure in the initial state is characterized by Al_2O_3 particles, their clusters and pores in matrix;

b) with increasing tensile load local cracks on pores occur, there are cracks formed by decohesion of Al_2O_3 particles and clusters;

c) with further increase of deformation of the material crack propagates preferentially along the pores and decohesed particles and clusters in a 45° angle.

4.2. Fracture model after ECAP

The following fracture mechanism of the ECAPed material was proposed, Fig. 6d,e,f:

a) 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;

b) fracture is propagated very quickly at minimum plastic deformation;

c) fracture is perpendicular to the tensile loading of the specimen.

Fracture is uniform, transcrystalline ductile with the dimple morphology. Dimples on fracture surface can be divided into two categories: small dimples ($\sim 0.1 \ \mu m$) initiated by the discrete Al₂O₃ particles, and large ones ($\sim 2.5 \ \mu m$) initiated by clusters of Al₂O₃ particles.

5. Conclusion

The aim of the study was to analyze fracture mechanism before and after ECAP in the Glidcop AL--60 grade (with 1.1 wt.% of Al_2O_3) system and propose 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 material before ECAP was proposed. With increasing tensile load, the local cracks formed predominantly on pores and decohesion of smaller Al_2O_3 particles and clusters occurred. Initiation of fracture after ECAP is in triple junctions of nanograins and decohesion of Al_2O_3 particles and clusters.

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