

THE FRACTURE MECHANISM OF “IN SITU” Al-Al₄C₃ NANOMATERIALS

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Abstract

Preparation of dispersion strengthened materials consists of heterogenization of structure. The different methods of how to incorporate the secondary phase particles to the matrix are known. The most optimal one is mechanical alloying during which the matrix particles are fractured and re-welded during the milling process in the attritor and incorporated to the matrix. Following heat treatment, the result is that the dispersion strengthened phase is homogeneously distributed in 3D.

The deformation and fracture mechanisms of Al-Al₄C₃ nanomaterials with 4 vol.% of Al₄C₃ phase have been analyzed using the technique of the “in-situ tensile testing in SEM”. It has been shown that the deformation process causes the break-up of large Al₄C₃ particles and decohesion of smaller ones. The final fracture path is also influenced by boundaries of nanograins, through which the principal crack propagates towards the sample's exterior surface. Based on the experimental observations, a model of the fracture mechanisms of Al-Al₄C₃ nanosystem has been proposed.

Keywords: dispersion strengthened Al, nanomaterials, “in situ” tensile testing in SEM, model of the fracture mechanism

INTRODUCTION

In previous works [1-8] we used “in-situ tensile test in SEM” to analyze deformation processes in various types of Cu and Al based composites. In works [9-11] Al-Si-Fe and Al-Si systems were studied by “in-situ tensile test in SEM”. The result was a design of several models of damage, which considered the physical and geometrical parameters of matrix and particles. The dispersion strengthened alloys Al-Al₄C₃ manufactured by mechanical alloying using powder metallurgy technology are promising structural materials. One microstructured material of such a type with 4 vol.% Al₄C₃ was transformed by the ECAP (Equal Channel Angular Pressing) method in two passes into a nanocomposite material. The ECAP technology enables obtaining a very fine grained microstructure – nanostructure by multiple pressings through the die.

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The aim of the work is to analyze the fracture mechanism of Al-Al₄C₃ nanocomposite system and to propose a fracture model.

EXPERIMENTAL MATERIALS AND METHODS

The experimental materials were prepared by mechanical alloying. Al powder of a powder particle size of <50 μm was dry milled in an attritor for 90 min with the addition of graphite KS 2.5 thus creating 4 vol.% of Al₄C₃, respectively. The specimens were then cold pressed using a load of 600 MPa. The subsequent heat treatment at 550°C for 10 h induced the chemical reaction $4Al+3C\rightarrow Al_4C_3$. Detailed technology preparation is described in [11-14].

This material, with dimensions of Ø10 x 70 mm, was deformed by the ECAP technique in two passes at room temperature in a hydraulic press in the equipment described in [15]. In the work [16] a dislocation model of the microcrystalline system with hard nanoparticles has been suggested. Analyses of microstructure and mechanical properties evolution in ECAP are given in [17-21]. 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. From each one of the systems five samples were prepared.

RESULTS AND DISCUSSION

The microstructure of the starting material with 4 vol.% Al₄C₃ was fine-grained (the mean matrix grain size was 0.35 μm), heterogeneous, with Al₄C₃ particles distributed in parallel rows as a consequence of extrusion. Besides the phase Al₄C₃, the systems also contained Al₂O₃ phase (~ 1 vol.%). Essentially, it was the remnant of oxide shells of the original matrix powder and/or shells formed during the reaction milling in attritors.

When describing microstructures, one has to consider geometrical and morphological factors. According to the microstructure observations, the particles in our materials can be divided into three distinctive groups: A – small Al₄C₃ particles, identified by TEM, with a mean size of approximately 30 nm which made up to 70% of the dispersoid volume fraction; B – large Al₄C₃ particles with a mean size between 1 and 2 μm, identified by scanning electron microscopy and on metallographic micrographs; and C – large Al₂O₃ particles with a mean size of 1 μm, found on metallographic micrographs and identified by scanning electron microscopy. Morphologically, Al₄C₃ particles are elongated and Al₂O₃ particles are spherical. Let us assume that particles of all categories during the high plastic deformation are distributed in rows. The mean distance between the rows is l and between the particles h . The particles are spherical or have only a low aspect ratio, so that they can be approximated as spherical. The experimental materials were deformed at 20°C at a strain rate of $6.6\cdot 10^{-4} s^{-1}$ in the elastic region.

The material after ECAP is on the border of nanostructured materials. The mean grain size is 100-200 nm, dislocations are present in nanograins, too, but mostly on the boundaries [8]. The nanostructure formation takes place most probably by a three-stage mechanism described in [20]. This model has been experimentally verified only for several specific materials, but in our case it seems to be probably usable. The model includes creation of cell structure, then formation of transitory cell nanostructure with large angle disorientation, and finally formation of nanostructured grains with a size of approx. 100 nm. However, here one has to consider the retarding effect due to the present dispersoid particles.

The deformation process of the loaded layer causes fracture of large, B-type, particles in the middle of the specimen (Fig.1 and 2) which initializes a fracture path

roughly perpendicular to the loading direction (Fig.3). The fracture path is also determined by decohesion of smaller particles (type A and/or C). Since the volume fractions of Al_4C_3 and Al_2O_3 particles are small, their distribution in lines does not influence the trajectory of the fracture which has low relative deformation $\varepsilon = 0.135$. Unlike the microstructured Al based composites, in this case it has been shown that the nanograin boundaries play an important role. In the final phase (Fig.4) a crack propagates along the nanograin boundaries, which has been observed experimentally on the crack line (profile).

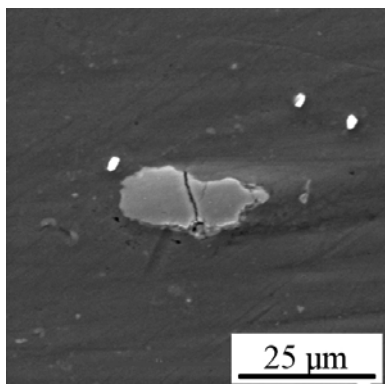


Fig.1. Fracture of B-type, particles Al_4C_3 in the middle of the layer.

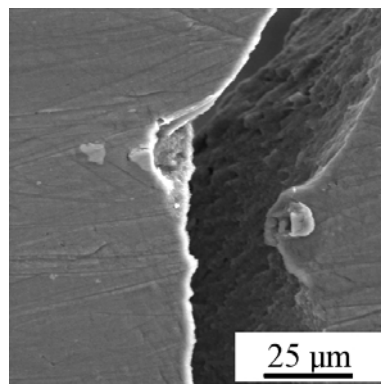


Fig.2. Final fracture of large particles.

A detailed study of the deformation changes showed that the crack initiation was caused by decohesion, and also occasionally by rupture of the large particles. Decohesion is a result of different physical properties of different phases of the system. The Al matrix has a significantly higher thermal expansion coefficient and lower elastic modulus (from 23.5 to $26.5 \cdot 10^{-6} \text{ K}^{-1}$, and 70 GPa) than both Al_4C_3 ($5 \cdot 10^{-6} \text{ K}^{-1}$, and 445 GPa) and Al_2O_3 ($8.3 \cdot 10^{-6} \text{ K}^{-1}$, and 393 GPa), respectively. Large differences in the thermal expansion coefficients result in high stress gradients, which arise on the interphase boundaries during 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 an increase of the stress peaks) their partial relaxation can occur. Superposition of the external load and the internal stresses can initiate cracking at interphase boundaries. This is also in accordance with the dislocation theories which argue that the particles in composite may cause an increase in the dislocation density as a result of the thermal strain mismatch between the ceramic particles and the matrix during preparation and/or thermal treatment. In our case, the coefficient of thermal expansion of the matrix is much higher than that of the secondary particles and the resulting thermal tension may relax around the matrix-particle interfaces by emitting dislocations, whose density can be calculated according to the procedure described in [16].

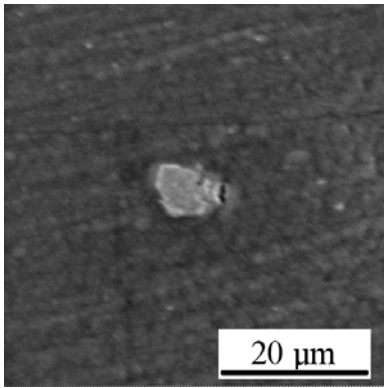


Fig.3. Decohesion of smaller particles, C-type Al_2O_3 .

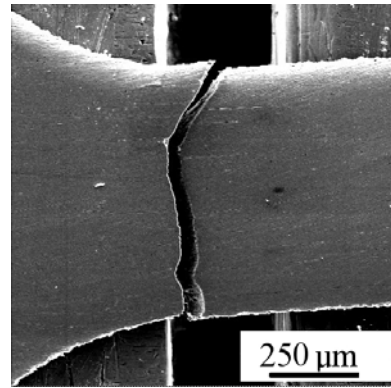


Fig.4. The final phase of the crack.

Based on the microstructure changes observed in the process of deformation, the following model (it is not a general model but one that resulted from our experiments) of fracture mechanism is proposed (Fig.5):

- a) The microstructure in the initial state is characterized by Al_4C_3 and Al_2O_3 particles, categorized as A, B and C, whose geometric parameters (l , h and d) depends on their volume fraction.
- b) With increasing tensile load, local cracks, predominantly on the specimen's side surfaces, are formed by the rupture of large (B) and decohesion of smaller (C and/or A) particles.
- c, d) In further increasing deformation of nanocomposite materials the nanograin boundaries start to play an important role. Since the volume fraction of these boundaries is high and the size of the B and C particles is equal to the matrix grain size, the crack propagates preferentially along the nanograin boundaries.

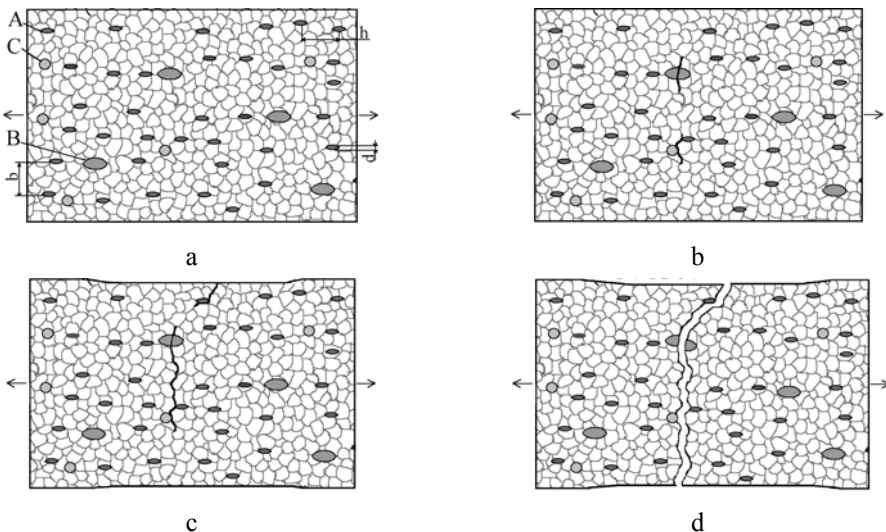


Fig.5. Model of the fracture mechanism.

CONCLUSIONS

The aim of the study was to evaluate the influence of volume fraction of Al_4C_3 (4 vol.%) and Al_2O_3 (1 vol.%) particles on the fracture mechanism by means of the method "in situ tensile test in SEM".

Based on the microstructure changes obtained in the process of deformation of the dispersion strengthened Al- Al_4C_3 alloys, a model of fracture mechanism was proposed. With increasing tensile load, the local cracks, predominantly on the specimen's side surfaces, are formed by the rupture of large (B) and decohesion of smaller (C, A) particles. The orientation of the cracks tends to be perpendicular to the loading direction. The final rupture, i.e. interconnection of the side cracks along the loading direction, takes place at nanograin boundaries.

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