INVESTIGATION OF EXTERNAL FACTORS INFLUENCE ON FRACTURE MECHANISM OF Al-Al_4C_3 SYSTEM BY "IN-SITU TENSILE TEST IN SEM"

M. Besterci, O. Velgosová, J. Ivan

Abstract

The method of "in-situ tensile testing in SEM" is suitable for investigations of fracture mechanisms because it enables one to observe and document deformation processes directly, by which the initiation and development of plastic deformation and fracture can be reliably described.

With increasing tensile load, local cracks are formed by the rupture of large particles and decohesion of smaller particles. Further increase of load leads to crack growth by coalescence of cavities in a direction from the surface to the specimen centre. The cracks can be oriented parallel or perpendicular to the loading direction depending on the particle volume fraction. The final rupture takes place in variably dense rows, depending on the volume fractions of carbide (Al_4C_3) and oxide (Al_2O_3) particles.

Keywords: composite material, "in-situ tensile testing in SEM", fracture mechanisms

INTRODUCTION

The dispersion strengthened alloys Al-Al_4C_3 manufactured by mechanical alloying using powder metallurgy technology are promising structural materials enabling significant weight cut for use, first of all in aircraft and automobile industry, and also at elevated temperatures.

In our previous works [1-4] we used the "in-situ tensile test in SEM" to analyze deformation processes in various types of Cu and Al based composites. In works [1,5] were studied strain and fracture on the Al-Al_4C_3 system. The influence of Al_2O_3 vol.% in the Cu-Al_2O_3 system was analysed in works [2,5,6]. Deformation process of the Cu-TiC system was analysed in works [3,4]. In works [8,9] were, by "in-situ tensile test in SEM", studied Al-Si-Fe and Al-Si systems. The result was a design of several models of damage, which considered physical parameters of matrix and particles as well as the geometry and distribution of secondary phases.

The aim of the present study is to evaluate the influence of volume fraction of Al_4C_3 particles (8 and 12 vol. %) on the fracture mechanism.

EXPERIMENTAL MATERIALS AND METHODS

The experimental materials were prepared by mechanical alloying. Al powder of powder particle size <50 μm was dry milled in an attritor for 90 min with the addition of

Michal Besterci, Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 043 53 Košice, Slovak Republic

Oksana Velgosová, Dept. of Non-ferrous Metals and Waste Treatment, Faculty of Metallurgy, Technical University, Letná 9/A, 04200 Košice, Slovak Republic

Jozef Ivan, Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Račianska 75, 838 12 Bratislava, Slovak Republic

graphite KS 2.5 thus creating 8 and 12 vol.% of Al_4C_3, respectively. The specimens were then cold pressed using a load of 600 MPa, the specimens had cylindrical shape. Subsequent heat treatment at 550°C for 3 h induced chemical reaction $4Al+3C \rightarrow Al_4C_3$. The cylinders were then hot extruded at 600°C with 94% reduction of the cross section. Due to a high affinity of Al to O_2 system, there are also contained a small amount of Al_2O_3 particles. The volume fraction of Al_2O_3 phase was low, 1-2 vol.%. Detailed technology preparation is described in [8,9].

For the purposes of investigation, very small flat tensile test pieces (7x3 mm) with 0.15 mm thickness were prepared by electroerosive machining, keeping the loading direction identical to the direction of extrusion. The specimens were ground and polished down to a thickness of approximately 0.1 mm. Finally, the specimens were finely polished on both sides by ion gunning. The test pieces were fitted into special deformation grips in 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 system (8 and 12 vol.% of Al_4C_3) was prepared five samples.

RESULTS AND DISCUSSION

The microstructures of the materials with 8 and 12 vol.% Al_4C_3 were fine-grained (the mean matrix grain size was 0.35 μm), heterogeneous, with Al_4C_3 particles distributed in parallel rows as consequence of extrusion. The average distance between the Al_4C_3 particles, found in thin foils, was 1.1 μm. Besides the phase Al_4C_3, the systems contained also Al_2O_3 phase [8,9]. Essentially, it was a remnant of oxide shells of the original matrix powder and/or shells formed during the reaction milling in attritors.

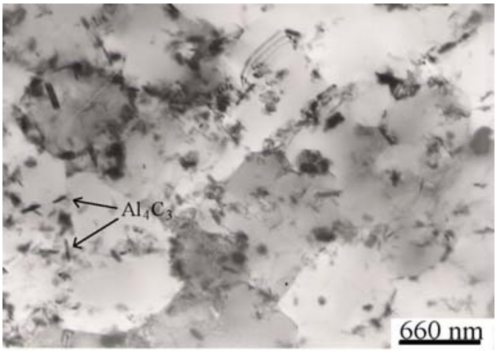

Fig.1. Al_4C_3 particles identified by TEM.

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 distinct groups: A – small Al_4C_3 particles, identified by TEM, (Fig.1), with mean size approximately 30 nm, which made up to 70% of the dispersoid volume fraction; B – large Al_4C_3 particles with mean size between 0.4 and 2 μm, found on metallographic micrographs; and C – large Al_2O_3 particles with mean size of 1 μm. By morphology, Al_4C_3 particles are elongated and Al_2O_3 particles are spherical. Let us assume that particles of all categories during the high plastic deformation are distributed in rows. 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 rate of $6.6x10^{-4}$ s^{-1} in the elastic region.

In the material with lower volume fraction (8 vol.%) of Al_4C_3, with an increase of the deformation load the initiation of microcracks on the large Al_4C_3 particles (B) was observed to occur by their rupture simultaneously with decohesion of the smaller Al_4C_3 and Al_2O_3 particles (C and B - Fig.2). The fracture may be initiated on the surface of a specimen where large particles undergoing damage are located. Cases of crack initiation by decohesion of large particles from the matrix and propagation of cracks towards the interior of the specimen was also observed, Fig.3a, b, c. The crack then propagated from the surface into the bulk of the specimen. On further deformation, as a result of higher concentration of smaller Al_4C_3 particles (A), the perpendicular fracture trajectory partially deviated toward the load direction (Fig.4) and became irregular.

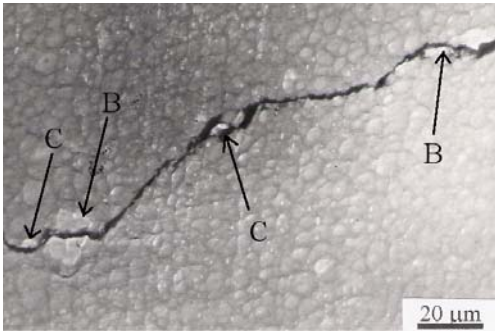

Fig.2. Fracture path in the material with 8 vol.% Al_4C_3. Rupture of a large Al_4C_3 grain and decohesion of the smaller particles (ε=0.12).

In the case of the higher volume fraction (12 vol.%) of Al_4C_3 the deformation process was very rapid due to the low plasticity of the material. Cracks were initiated on the surface and propagated approximately perpendicularly to the tensile load direction (Fig.5). Coalescence of the final fracture progressed along densely populated rows of Al_4C_3 (A, B) particles parallel to the load direction (Fig.6). The morphology and size of the deformed surface and three categories of particles on the fracture surface can be seen in Fig.7.

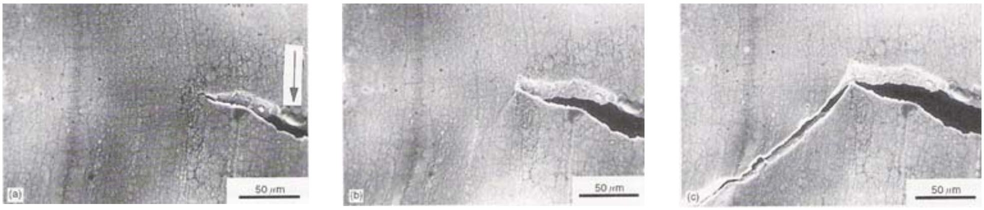

Fig.3. Propagation of the fracture toward the specimen interior: a) elongation 0.12 mm; b) elongation 0.18 mm; c) elongation 0.185 mm.

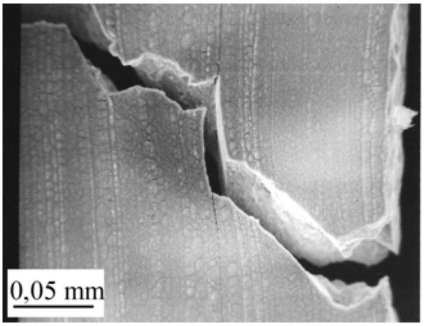

Fig.4. Irregular fracture formed by a crack growing alternatively along the particle rows and between them in the material with 8 vol.% Al_4C_3 (ε=0.15).

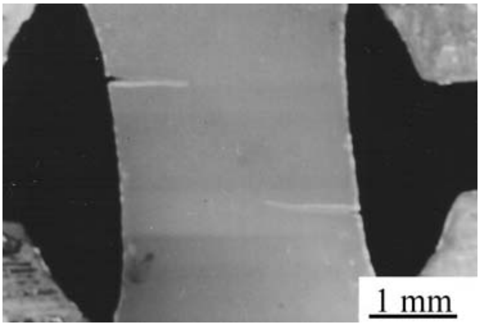

Fig.5. Two cracks initiated on the opposite sides of a specimen. Surface morphology and initiation of cavities in the matrix–particle interphase in the material with 12 vol% Al_4C_3 (ε=0.04).

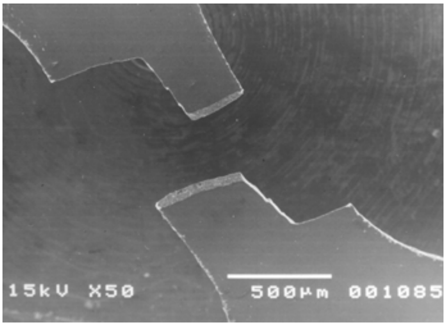

Fig.6. Final fracture by interconnecting the two opposite side cracks in the material with 12 vol% Al_4C_3 (ε=0.05).

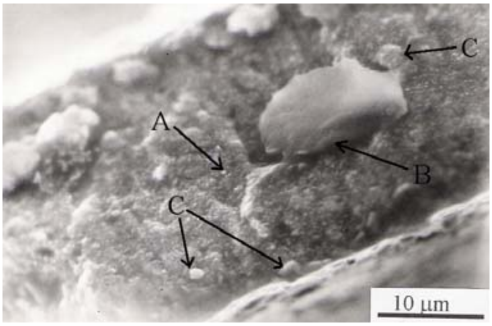

Fig.7. Surface morphology of the material with 12 vol.% Al_4C_3 (ε=0.05).

A detailed study of the deformation changes showed that the crack initiation was caused by decohesion, and occasionally also 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 26.5×10^{-6} K^{-1}, and 70 GPa) than both Al_4C_3 (5×10^{-6} K^{-1}, and 445 GPa) and Al_2O_3 (8.3×10^{-6} 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. Hence $\alpha_{matrix} > \alpha_{particle}$, 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. The fractures of the studied materials started at the side-rims of the deformed samples. When compared to the material with a lower volume fraction of Al_4C_3, in the present system the development of slip bands in the bulk was inhibited. This fact, and the absence of long-range slip in the matrix, implies that the fracture is not inclined to the applied load but is perpendicular to it. This is caused by the high volume fraction of the strengthening particles and by their short distance. Considering the sample width (0.1 mm), the crack grew at 45° with respect to the sample surface. The fracture was transcrystalline, ductile.

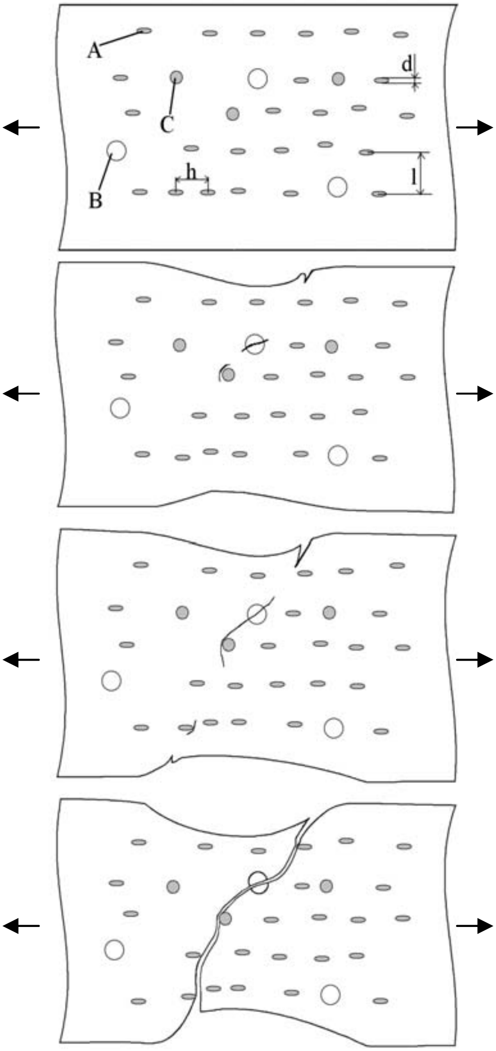

Fig.8. Model of the fracture mechanism.

Based on the microstructure changes observed in the process of deformation, the following model (it is not a general model but it is a consequent model on our experiments) of fracture mechanism is proposed (Fig.8):

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 specimen side surfaces are formed by the rupture of large (B, C) and decohesion of smaller (A) particles.

c) Further increase of load leads to the crack growth by coalescence of cavities in the direction from the surface to the specimen centre. The cracks can be oriented parallel or perpendicular to the loading direction in depending on the particle volume fraction.
d) The final rupture, i.e. interconnection of the side cracks along the loading direction, takes place in variably dense rows, depending on the volume fractions of carbide (Al_4C_3) and oxide (Al_2O_3) particles.

CONCLUSION

The aim of the study was the evaluation of volume fraction of Al_4C_3 (8 and 12 vol.%) and Al_2O_3 (1-2 vol.%) particles on the fracture mechanism of the method "in situ tensile test in SEM".

Based on the microstructure changes obtained in the process of deformation the dispersion strengthened Al-Al_4C_3 alloys were the model of the fracture mechanism proposed. With increasing tensile load, local cracks, predominantly on specimen side surfaces are formed by rupture of large (B, C) and decohesion of smaller (A) particles. Further increase of load leads to the crack growth by coalescence of cavities in the direction from the surface to the specimen centre. The cracks can be oriented parallel or perpendicular to the loading direction, depending on the particle volume fraction. The final rupture, i.e. interconnection of the side cracks along the loading direction, takes place in variably dense rows depending on the volume fractions of carbide (Al_4C_3) and oxide (Al_2O_3) particles.

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