

***In situ* tensile testing in SEM of Al-Al₄C₃ nanomaterials**

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

Key words: dispersion strengthened Al, nanomaterials, *in situ* tensile testing in SEM, model of the fracture mechanism.

1. INTRODUCTION

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, thank to which the initiation and development of plastic deformations and fracture can be reliably described.

In our previous papers [1–8], based on papers [9–11], we used *in situ* tensile testing in SEM to analyse deformation processes in various types of Cu and Al based composites. In [9–11] Al-Si-Fe and Al-Si systems were studied by *in situ* tensile

testing in SEM. The result was a design of several models of damage, which considered physical and geometrical parameters of the 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 type with 4 vol% Al₄C₃ was transformed by the equal channel angular pressing (ECAP) method in two passes into a nanocomposite material. The experimental material was pressed through two perpendicular to one another channels of a special die by route "C". The ECAP technology allows obtaining very fine-grained microstructure – nanostructure – by multiple pressings through the die. The mechanical tests of this nanocomposite material in comparison with the not extruded materials show a tensile strength increase of 40% and yield strength increase of 30%. The elongation of extruded nanomaterials decreases in average by 45%.

The aim of this paper is to analyse the fracture mechanism of the Al-Al₄C₃ nanocomposite system and to propose a damage model.

2. EXPERIMENTAL MATERIALS AND METHODS

The starting experimental materials were prepared by mechanical alloying. The Al powder of powder particle size <50 μm was dry milled in an attritor for 120 min with the addition of graphite KS 2.5 thus creating 4 vol% of Al₄C₃. The specimens were then cold pressed using a load of 600 MPa. The specimens had cylindrical shape. Subsequent heat treatment at 550 °C for 10 h induced chemical reaction $4\text{Al} + 3\text{C} \rightarrow \text{Al}_4\text{C}_3$. The cylinders were then hot extruded at 600 °C with 94% reduction of the cross section. Using such a treatment temperature and time of treatment, the complete transformation of graphite takes place [11]. The extent to which C was converted to Al₄C₃ dispersoids was analysed by gas chromatographic measurements in [12]. Due to a high affinity of Al to O₂, the system also contains a small amount of Al₂O₃ particles. The volume fraction of starting Al₂O₃ was low, 1 vol%. Detailed technology description is given in [13–16].

This material with dimensions of Ø10 × 70 mm was deformed by the ECAP technique in two passes at room temperature in a hydraulic press in the equipment described in [17]. In [18], a dislocation model of microcrystalline system with hard nanoparticles has been suggested. Analysis of the evolution of the microstructure and mechanical properties in ECAP is given in [19–23].

For the purposes of investigation, very small flat tensile test pieces (7 × 3 mm, gauge length is 7 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 inside the scanning electron microscope JEM 100 C, which enables direct observation and measurement of the deformation by ASID-4D equipment. From every system five samples were prepared.

3. RESULTS AND DISCUSSION

The microstructure of the starting material with 4 vol% Al_4C_3 was fine-grained (the mean matrix grain size was $0.35\ \mu\text{m}$), heterogeneous, with Al_4C_3 particles distributed in parallel rows as a consequence of extrusion. Beside the phase Al_4C_3 , the systems contained also Al_2O_3 phase (about 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 Al_4C_3 particles, identified by TEM, 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 1 and 2 μm , identified by scanning electron microscopy and on metallographic micrographs; and C – large Al_2O_3 particles with mean size of 1 μm , found on metallographic micrographs and identified by scanning electron microscopy. Morphologically, 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 strain rate of $6.6 \times 10^{-4}\ \text{s}^{-1}$ in the elastic region.

The material after ECAP is on the border of nanostructured materials. The TEM micrographs (Figs. 1 and 2) showed that the mean grain size was 100–200 nm; dislocations are present in nanograins, but mostly on the boundaries. In Fig. 2 these dislocations are weakly visible due to the tilting of the specimen. The nanostructure formation takes place most probably by a three-stage mechanism, described in [22]. This model has been experimentally verified only for several specific materials, but in our case it seems to be usable. The model includes creation of the cell structure, then formation of the transitory cell nanostructure with large angle disorientation, and finally formation of nanostructured grains with size of about 100 nm. However, here one has to consider the retarding effect due to the presence of dispersoid particles.

Deformation process of the loaded layer causes fracture of large, B-type, particles in the middle of the specimen (Figs. 3 and 4), which initializes fracture path roughly perpendicular to the loading direction (Fig. 5). The fracture path is determined also 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 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. 6) a crack propagates along the nanograin boundaries, which has been observed experimentally on the crack line (profile).

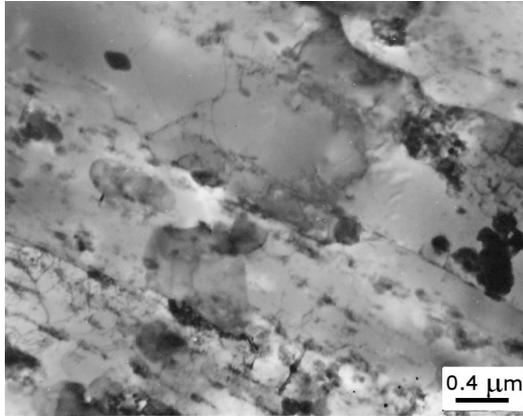


Fig. 1. The microstructure of the starting material.

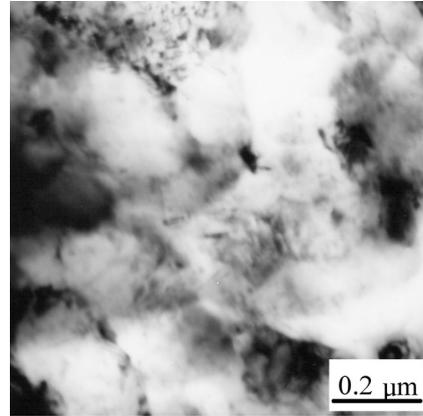


Fig. 2. The microstructure of the material after ECAP.

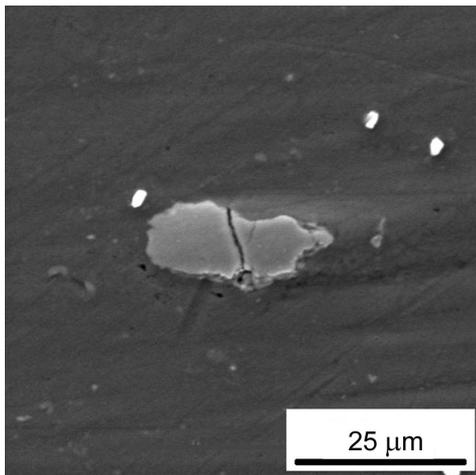


Fig. 3. Fracture of B-type, particles Al_4C_3 in the middle of the layer.

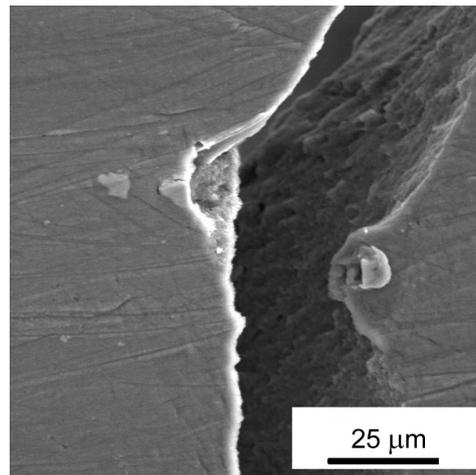


Fig. 4. Final fracture of large particles.

A detailed study of the deformation changes showed that the crack initiation was caused by decohesion and occasionally also by rupture of large particles. Decohesion is a result of different physical properties of different phases of the system. The Al matrix has significantly higher thermal expansion coefficient and lower elastic modulus (from 23.5 to $26.5 \times 10^{-6} \text{ K}^{-1}$, and 70 GPa) than both Al_4C_3 ($5 \times 10^{-6} \text{ K}^{-1}$, and 445 GPa) and Al_2O_3 ($8.3 \times 10^{-6} \text{ K}^{-1}$, and 393 GPa), respectively.

Large differences in the thermal expansion coefficients result in high stress gradients, which arise on the interphase boundaries during the hot extrusion. Since $\alpha_{\text{matrix}} > \alpha_{\text{particle}}$, high compressive stresses can be expected. However,

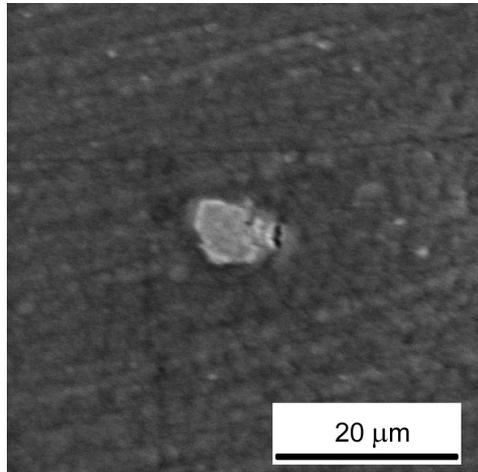


Fig. 5. Decohesion of smaller particles, C-type Al_2O_3 .

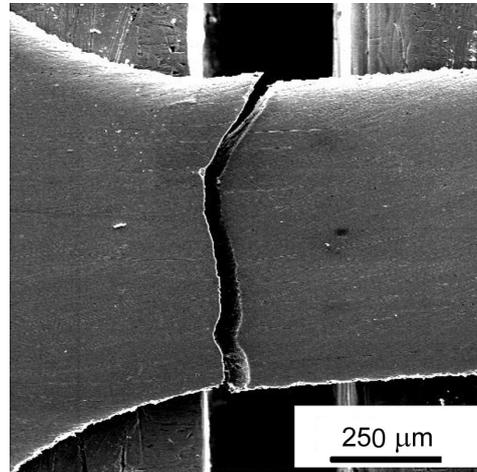


Fig. 6. The final phase of the crack.

because the stress gradients arise due to the temperature changes, during cooling (which results in the 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 in accordance also with the dislocation theories, which argue that the particles in a composite may cause an increase in the dislocation density as a result of thermal strain mismatch between the ceramic particles and the matrix during preparation and/or thermal treatment [18]. 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 a procedure, described in [18].

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. 7).

- 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) depend on their volume fraction (Fig. 7a).
- B. With increasing tensile load, the local cracks, predominantly on specimen side surfaces, are formed by rupture of large (B) and decohesion of smaller (C and/or A) particles (Fig. 7b,c).
- C. With 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, crack propagates preferentially along the nanograin boundaries (Fig. 7d).

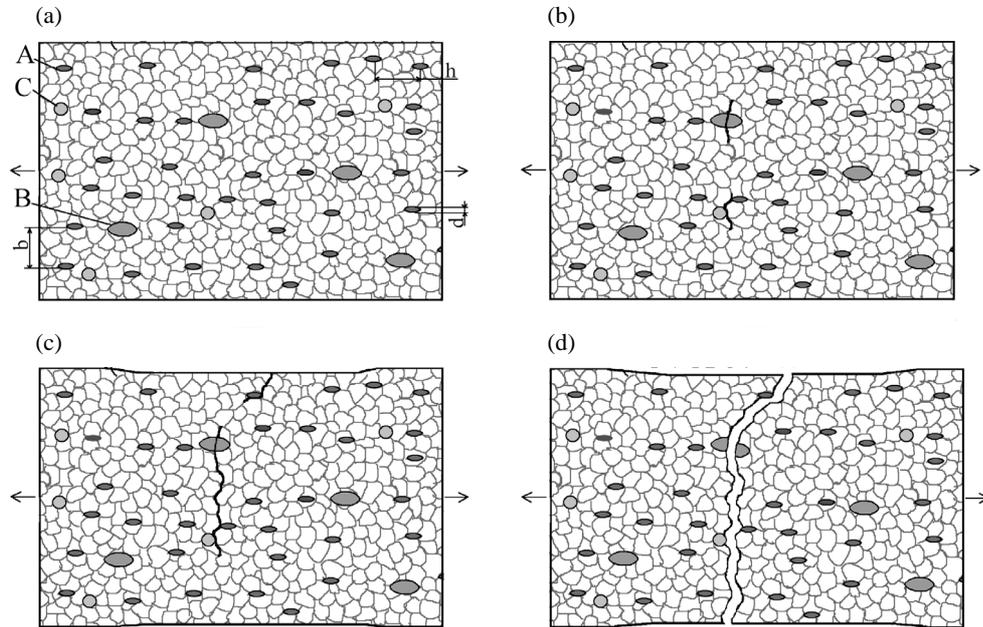


Fig. 7. Model of the fracture mechanism.

4. CONCLUSIONS

The aim of the study was to evaluate the influence of selected 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 testing 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 specimen's side surfaces, are formed by rupture of large and decohesion of smaller particles. The orientation of 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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REFERENCES

1. Besterčí, M. and Ivan, J. Failure mechanism of dispersion strengthened Al-Al₄C₃ systems. *J. Mater. Sci. Lett.*, 1996, **15**, 2071–2074.
2. Besterčí, M. and Ivan, J. The mechanism of the failure of the dispersion-strengthened Cu–Al₂O₃ system. *J. Mater. Sci. Lett.*, 1998, **17**, 773–776.
3. Besterčí, M., Ivan, J., Kováč, L., Weissgaerber, T. and Sauer, C. Strain and fracture mechanism of Cu–TiC. *Mater. Lett.*, 1999, **38**, 270–274.
4. Besterčí, M., Ivan, J., Kováč, L., Weissgaerber, T. and Sauer, C. A model of deformation mechanism of the system Cu–TiC. *Kovové Mater.*, 1998, **36**, 239–244.
5. Besterčí, M., Ivan, J. and Kováč, L. Influence of volume fraction of Al₂O₃ particles on fracture of the Cu–Al₂O₃ system. *Kovové Mater.*, 2000, **38**, 21–28.
6. Besterčí, M., Ivan, J. and Kováč, L. Influence of Al₂O₃ particles volume fraction on fracture mechanism in the Cu–Al₂O₃ system. *Mater. Lett.*, 2000, **46**, 181–184.
7. Besterčí, M., Ivan, J., Velgosová, O. and Pešek, L. Damage mechanism of Al–Al₄C₃ system with high volume fraction of secondary phase. *Kovové Mater.*, 2001, **39**, 361–367.
8. Besterčí, M., Velgosová, O., Ivan, J., Hvizdoš, P. and Kohútek, I. Influence of volume fraction on fracture mechanism of Al–Al₄C₃ system studied by “in-situ tensile test in SEM”. *Kovové Mater.*, 2008, **46**, 139–143.
9. Mocellin, A., Fougerest, F. and Gobin, P. F. J. A study of damage under tensile loading in a new Al–Si–Fe alloy processed by the Osprey route. *Mater. Sci.*, 1993, **28**, 4855–4861.
10. Velíšek, R. and Ivan, J. Mechanism of “in-situ deformation in SEM” Al–Si system. *Kovové Mater.*, 1994, **32**, 531–539.
11. Jangg, G., Šlesár, M., Besterčí, M., Ďurišín, J. and Schröder, K. Influence of heat treatment during manufacturing of Al–Al₄C₃ materials on microstructure and properties. *Powder Metallurgy Int.*, 1989, **5**, 25–30.
12. Jangg, G., Zbiral, J. and Wu, S. Einfluß des Mahlens und der Wärmebehandlung bei Strangpreßprodukten aus dispersionsverfestigten Al–Al₄C₃ Werkstoffen. *Aluminium*, 1992, **68**, 238–246.
13. Broutman, L. V. and Krock, R. H. Analysis of deformation of Al–Si system. *Compos. Mater.*, 1974, **5**, 27–38.
14. Jangg, G., Vasgura, H., Schröder, K., Šlesár, M. and Besterčí, M. In *Proc. Int. Conference on Powder Metallurgy PM 86*. Düsseldorf, 1986, 989–999.
15. Korb, G., Jangg, G. and Kutner, F. Dispersionsverfestigte Al–Al₄C₃ Werkstoffe. *Draht*, 1979, **30**, 318–327.
16. Šalunov, J., Šlesár, M., Besterčí, M., Oppenheim, H. and Jangg, G. Einfluss der Herstellungsbedingungen auf die Eigenschaften von dispersionsverfestigten Al–Al₄C₃ Werkstoffen. *Metall*, 1986, **6**, 601–609.
17. Besterčí, M., Šilleiová, K. and Kvačkaj, T. Fracture micromechanisms of copper nanomaterials prepared by ECAP. *Kovové Mater.*, 2008, **46**, 309–311.
18. Lukáč, P. and Trojanová, Z. Deformation and damping behaviours of microcrystalline Mg reinforced with ceramic nanoparticles. *Kovové Mater.*, 2006, **44**, 243–249.
19. Valiev, R. Z. Approach to nanostructured solids through the studies of submicron grained polycrystals. *Nanostruct. Mater.*, 1995, **6**, 73–85.
20. Valiev, R. Z. In *Proc. Metallic Materials with High Structural Efficiency. NATO Science Series*, Kiev (Senkov, O. N., Miracle, D. B. and Firstov, S. A., eds.). IOS Press, Amsterdam and Kluwer Acad. Publ., Dordrecht, 2003, 79–89.
21. Zhu, Y. T., Huang, J. Y., Gubicza, J., Ungár, T., Wang, Y. M., Ma, E. and Valiev, R. Z. Nanostructures in Ti processed by severe plastic deformation. *J. Mater. Res.*, 2002, **18**, 1908–1917.
22. Valiev, R. Z. and Alexandrov, I. V. *Nanostrukturnye Materialy Poluchennye Intensivnoj Plasticheskoj Deformaciej*. Logos, Moscow, 2000 (in Russian).
23. Kozlov, E. V. and Koneva, M. V. Features of work hardening of polycrystals with nanograins. *Mater. Sci. Forum*, 2008, **35**, 584–586.

Al-Al₄C₃ nanomaterjalide *in situ* SEM-tõmbeteim

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Uurimisobjektiks on Al-Al₄C₃ (4 mahuprotsenti) nanomaterjalide deformatsiooni ja purunemise mehhanismid, kasutades *in situ* skaneerivat elektronmikroskoopiat (SEM). On välja selgitatud, et deformatsiooniprotsess kutsub esile suurte Al₄C₃ osakeste purunemise ja väiksemate osakeste dekohesiooni. Lõplik praod areng on mõjutatud ka nanoteradevahelistest piiridest, läbi mille areneb põhipragu tagasi katsekeha välispinnale. Tulenevalt eksperimentaalsetest uuringutest on välja selgitatud kahjustuste ja purunemise mehhanismid.