THE FRACTURE MECHANISM OF Al-Al_4C_3 SYSTEM 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, by which the initiation and development of plastic deformation and fracture can be reliably described.

With increasing tensile load, local cracks are formed by rupture of large particles and decohesion of smaller 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 in 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.

1 Introduction

Dispersion strengthened materials belong to the group of composite materials, which are made mainly by the techniques of powder metallurgy. Their structure is formed with a polycrystalline matrix, in which dispersion particles are incorporated, mainly of the oxide, carbide and nitride types. The strengthening effect of dispersoids is both direct, based on the braking of the matrix dislocation movement, and indirect, resulting from a. forming, dispersoids increase the density of dislocations and refine the grain and subgrain structure. The effectiveness of the strengthening effect of dispersoids depends on their type, size, morphology, volume fraction and distribution. Their resistance against dissolution and coalescence is an important factor in their strengthening effect, mainly at high temperatures.

The polycrystalline matrix is formed from both metallic and non-metallic powders. The most important methods of powder production include mechanical milling, reduction of oxides, carbonization, water and gas spraying in air, in a protective atmosphere or in vacuum, and also electrolysis. The method of rotary electrode, rotary disk and plasma spraying belong among the most modern methods of powder production. The method of evaporation and condensation is used for very fine powders. The basic characteristics of powders include their chemical composition, purity, granulometric composition, shape, specific surface, bulk density and density after jarring, compressibility, sintering capacity and others. Metallurgical purity, as well as changes taking place during the treatment process is of key significance in the production of dispersion-strengthened materials. The oxides formed, foreign particles and gases, which can be a cause of the origin of technological defects of the microcrack type, separation of layers, etc. plays the decisive role. It has been shown, from theoretical considerations and experiments, that the maximum effect of strengthening is achieved at the following structural parameters:

a) the size of the strengthening particles of secondary phases (dispersoids) should not exceed 50 nm. The particles of higher sizes are of little effectiveness from the point of view of strengthening.
b) the mean distance between strengthening particles should be of the range from 0.1 to 0.5 μm, and their distribution should be uniform, without any heterogeneities and clusters.

The real volume fraction of dispersoids follows from the above-mentioned parameters. This depends on the required properties of materials, however, does not exceed, as a rule, 10 vol.%.

Mechanical alloying is the process of production of macroscopically homogeneous materials from heterogeneous mixtures. The process is implemented in laboratories in attritors, and on the industrial scale in high-energy ball mills. It is based on deformation, repeated disintegration and welding of powder particles during intensive dry milling.

Originally, this technology was developed for production of Ni and Fe super alloys. It has since been shown that mechanical alloying is able to produce several stable and metastable phases including solid solutions, metastable crystalline and quasicrystalline phases, as well as amorphous phases. It is remarkable that the crystalline phases are usually of nanometric sizes. Because these effects are similar to other ones, obtained by non-equilibrium techniques, such as quick solidification of the metal, the system is characterized by the increased mechanical properties. The advantage of preparing amorphous materials by mechanical alloying in comparison with the technology of quick solidification is represented by the fact that it makes it possible to produce larger quantities of materials, and extends the possibilities for alloying. The technology of mechanical alloying is used for metals, ceramic and polymers and considerable attention is paid to it throughout the world. In the literature, the term for mechanical alloying is “mechanical alloying” if elementary powders are used, and “mechanical milling”, if prealloyed powder is used as the starting material. A series of material systems has been developed using mechanical alloying, mainly for high-temperature applications on the basis of Ag, Al, B, Ba, Bi, Co, Cr, Cu, Fe, Ge, Hf, La, Li, Mg, Mn, Nb, Nd, Pb, Re, Si, Sm, Sn, Ta, Ti, V, W, Y and Zr. The materials with low specific weight on the basis of Al, Li and Mg represent an important group.

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 [1-7] following [8-10] we used “in-situ tensile test in SEM” to analyze deformation processes in various types of Cu and Al based composites. In works [1, 7] were studied the strain and fracture on Al-Al_4C_3 system. The influence of Al_2O_3 vol.% in Cu-Al_2O_3 system was analyzed in works [2, 5 and 6]. Deformation process of Cu-TiC system was analyzed in works [3 and 4]. In works [8-10] 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 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.

2 Experimental materials and methods

The experimental materials were prepared by mechanical alloying. Al powder of 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 8 and 12 vol.% of Al_4C_3, respectively. The specimens were than 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 Al to O_2 system also contains 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 [11-15].

Experiments were considered also with material Al-Al_4C_3 with nano-matrix. It was tested by SPD using ECAP (equal channel angular pressing). The tested material was compact only after one pass at the angle 90°, however at the next passes cracks appeared, so we had to finish the testing. In (Fig.1) is the dependence of mechanical properties on volume fraction of Al_4C_3 is shown the R_m and A_5 values, too. The mean grain size decreased for 1 μm to 0.6 μm.

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 system (8 and 12 vol.% of Al_4C_3) was prepared five samples.

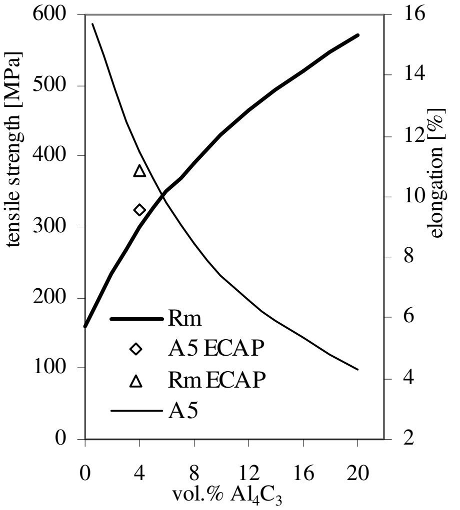

Fig. 1. The dependence of tensile strength and ductility on Al-Al_4C_3 system.

3 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 in consequence of extrusion. The average distance between the Al_4C_3 particles, found in thin foils, was 1.1 µm.

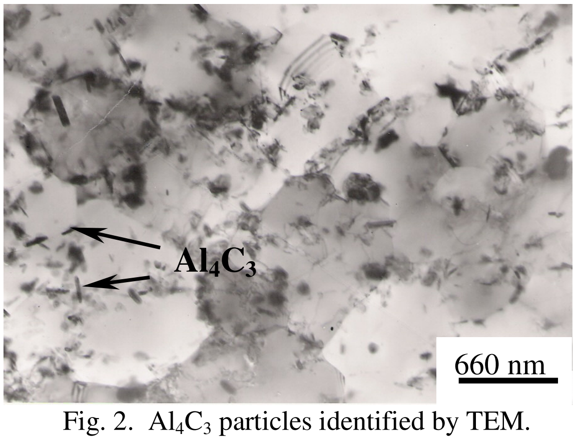

Fig. 2. Al_4C_3 particles identified by TEM.

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 distinctive groups: A – small Al_4C_3 particles, identified by TEM, (Fig. 2), 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 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.3).

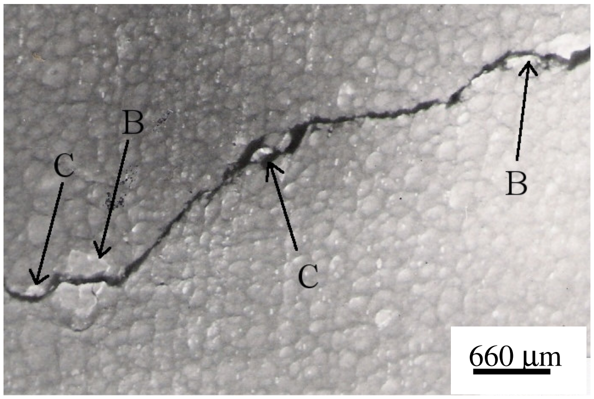

Fig. 3. Fracture path in the material with 8 vol.% Al_4C_3. Rupture of a large Al_4C_3 grain and decohesion of the smaller particles (ε=0.12).

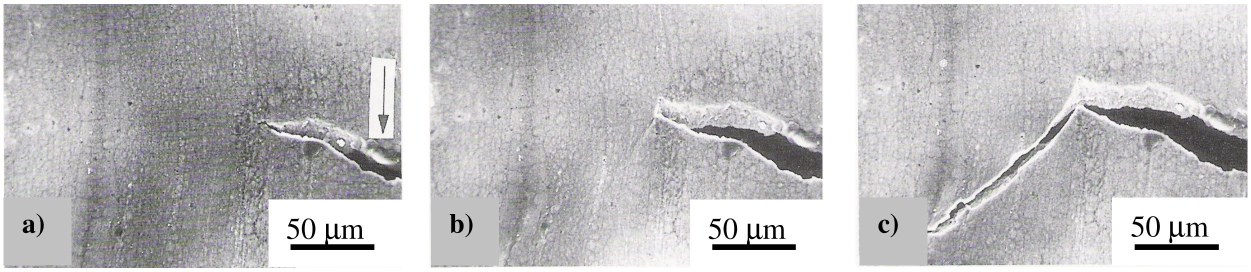

Fig. 4. Propagation of the fracture toward the specimen interior: a) elongation 0.12 mm; b) elongation 0.18 mm; c) elongation 0.185 mm.

The fracture may be initiated on the surface of a specimen where large particles undergoing the 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.4a, 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.5) and became irregular.

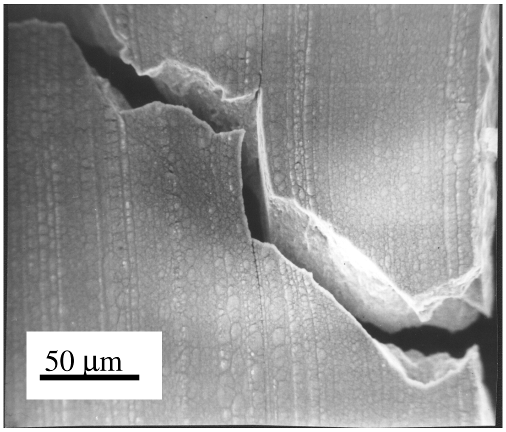

Fig. 5. Irregular fracture formed by a crack growing alternatively along the particle rows and between them in the material with 8 vol. % Al_4C_3 (ε=0.15).

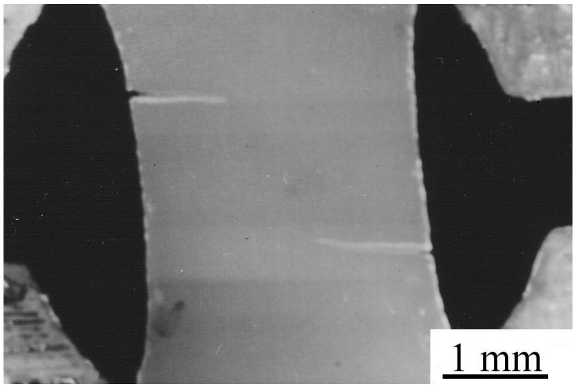

Fig. 6. Two cracks initiated on the opposite sides of a specimen. Surface morphology and initiation of cavities in the matrix–particle interphase in the material with 12 vol% Al_4C_3 (ε=0.04).

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.6). Coalescence of the final fracture progressed along densely populated rows of Al_4C_3 (A, B) particles parallel to the load direction (Fig.7). The morphology and size of the deformed surface and three categories of particles on fracture surface can be seen in Fig.8.

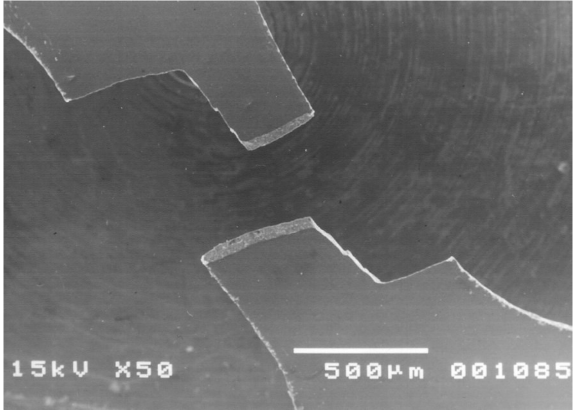

Fig. 7. Final fracture by interconnecting the two opposite side cracks in the material with 12 vol% Al_4C_3 (ε=0.05).

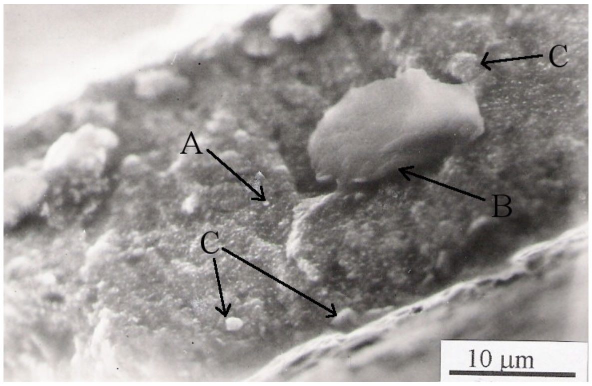

Fig. 8. Surface morphology of the material with 12 vol% Al_4C_3 (ε=0.05).

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 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 the hot extrusion. Since $\alpha_{matrix} > \alpha_{particle}$, high compressive stresses can be expected. However, because the stress gradients arise due to the temperature changes, during cooling (which results in 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.

In view of the dislocation theories the particles in 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. The difference between the coefficients of thermal expansion of the particles and the matrix may create the thermal residual stresses after cooling from the processing temperature to room temperature. The coefficient of thermal expansion of the matrix is much higher than that of the secondary particles. The thermal tension may relax around the matrix-particle interface by emitting dislocations. An increase in the dislocation density reinforcement has been calculated as [16].

$$\Delta\rho = \frac{Bf\Delta\alpha\Delta T}{b(1-f)} \cdot \frac{1}{t} \tag{1}$$

where $\Delta\alpha$ is the difference of the coefficient of thermal expansion between matrix and particles, ΔT is a temperature change, t is the minimum size of reinforcement, f is the volume fraction of particles, b is the magnitude of the Burgers vector of dislocations and B is a geometrical constant (depending on the aspect ratio). The newly formed dislocations are obstacles for the motion of dislocations in the matrix. Therefore a higher stress for the moving dislocations is necessary in comparison to materials without secondary particles.

From Eq. 1 is obvious that the density of the newly created dislocations increases with an increase in the volume fraction. Therefore the number of obstacles for the dislocation motion increases and the stress necessary for the motion of dislocations increases too. It should be mentioned that the effect of different types of secondary particles (Al_4C_3, Al_2O_3) on the reinforcement and damage is depended not only from the coefficients of thermal expansion difference but also from the properties of the matrix/particle interface.

The fractures of the studied materials started at the side-rims of the deformed samples. When compared to the material with 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.

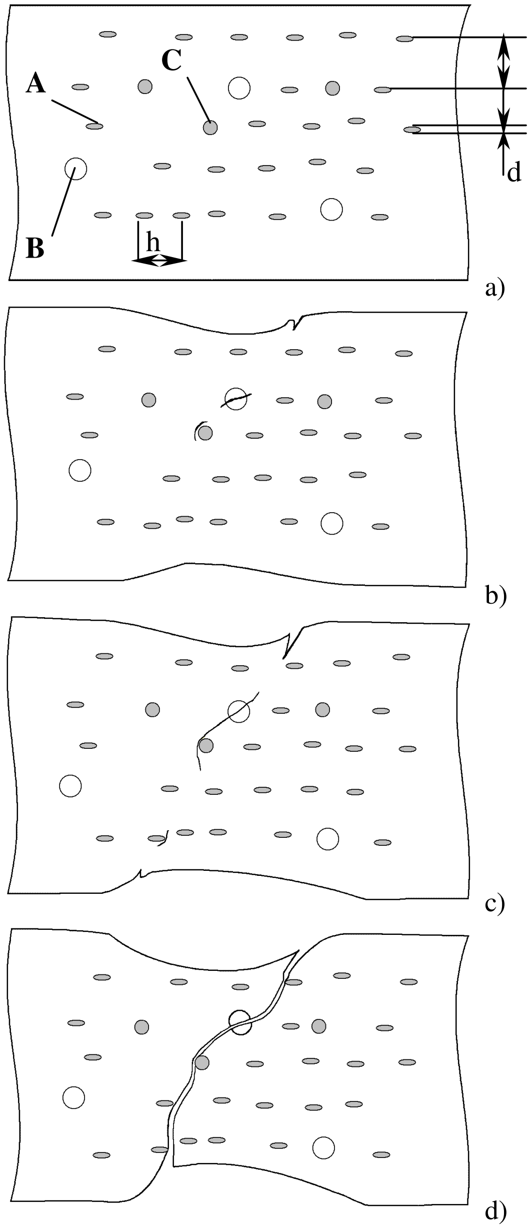

Fig. 9. Model of the fracture mechanism.

Based on the microstructure changes observed in the process of deformation, the following model (it is not general model but it is a consequent model on our experiments) of fracture mechanism is proposed (Fig.9):

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 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.

3 Conclusions

The aim of the study was 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 was model of 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 in 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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References

[1] Besterci M. and Ivan J. “Failure mechanism of dispersion strengthened Al-Al_4C_3 systems”. *Journal of Mater. Sci. Lett.*, Vol. 15, pp 2071-2074, 1996.

[2] Besterci M. and Ivan J. “The Mechanism of the Failure of the Dispersion-strengthened Cu–Al_2O_3 Systém”. *Journal of Mater. Sci. Lett.* 17, 773-776 (1998).

[3] Besterci M., Ivan J., Kováč L., Weissgaerber T. and Sauer C. “Strain and fracture mechanism of Cu–TiC”. *Mat., Letters,* Vol. 38, pp 270-273, 1999.

[4] Besterci M., Ivan J., Kováč L., Weissgaerber T. and Sauer C. “A model of deformation mechanism of the system Cu-TiC”. *Kovové Mater.*, Vol. 36, pp 239-244, 1998.

[5] Besterci M., Ivan J. and Kováč L. “Influence of volume fraction of Al_2O_3 particles on fracture of the Cu-Al_2O_3 systém”. *Kovové Mater.*, Vol. 38, pp 21-25, 2000.

[6] Besterci M., Ivan J. and Kováč L. ”Influence of Al_2O_3 particles volume fraction on fracture mechanism in the Cu–Al_2O_3 systém”. *Materials Letters* Vol. 46, pp 181-184, 2000.

[7] Besterci M., Ivan J., Velgosová O. and Pešek L. ”Damage mechanism of Al-Al_4C_3 system with high volume fraction of secondary phase”. *Kovové Mater.* Vol. 39, pp 361-367, 2001.

[8] Mocellin A., Fougerest F. and Gobin P.F. ”A study of damage under tensile loading in a new Al-Si-Fe alloy processed by the Osprey route”. *J. Mater. Sci.* Vol. 28, pp 4855-4861, 1993.

[9] Velísek R. and Ivan J. ”Mikromechanizmus porušovania materiálov Al-Si-Fe metódou “in situ tensile test””. *Kovové Mater.* Vol. 32, pp 531-540, (1994).

[10] Broutman L.V. and Krock R.H. ”Analysis of deformation of Al-Si system”. *Compos. Mater.* Vol. 5, pp 27-38, 1974.

[11] Besterci. M., Šlesár. M. and Jangg G. “Structure and properties of dispersion hardened Al-Al_4C_3 materials” *Powder Met. Int.* Vol. 24, No.1, pp 27-34, 1972.

[12] Korb G., Jangg G. and Kutner F. ”Dispersionsverfestigten Al-Al_4C_3 Werkstoffen”. *Draht* Vol. 30, pp 318-327, 1979.

[13] Šalunov J., Šlesár M., Besterci M., Oppenheim,H. and Jangg,G. ” Einflus der Herstellung bedingungen die Eingeschaften von dispertionsverfestigten Al-Al_4C_3 Werkstoffen”. *Metall* Vol. 6, pp 601-609, 1986.

[14] Besterci,M. ”Preparation, microstructure and properties of Al–Al_4C_3 system produced by mechanical alloying”. *Materials and Design* Vol. 27, pp 416-421, 2006.

[15] Ďurišín J., Ďurišínová K., Orolínová M. and Besterci M. ”Microstructure stability of Al_4C_3 materials at elevated temperatures”. *High Temp. Mat. and Processing*, Vol. 25, pp 149-155, 2006.

[16] Lukáč. P. and Trojanová. Z. “Deformation and dampling behavioursr of microcrystaline Mg reinforced with ceramic nanoparticles”. *Metall. Mat.* Vol 44, No.5, pp 243-249, 2006