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SUPERPLASTIC DEFORMATION OF Al-Al_4C_3 COMPOSITES

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Deformation of the Al-Al_4C_3 composites with different volume fraction of Al_4C_3 phase was investigated at different temperatures (293 - 723 K) and different strain rates (2,5·10^{-5} s^{-1} - 1,0·10^{-1} s^{-1}). At temperatures 673 - 723 K and at the highest strain rate of 1,0·10^{-1} s^{-1}, a significant ductility increase was observed. TEM analysis suggests the onset of superplasticity may be the result of dynamic grain polygonization, grain slip and rotation, partial recrystallization and dislocation creep in the tested system, which is known as strain induced dynamic recovery. Increase of the volume fraction of secondary phase in the studied composite resulted in a shift from slip on grain boundaries controlled mechanism to the grain rotation controlled deformation mechanism.

Keywords: *composite material, deformation, strain rate, TEM, superplasticity*

Superplastična deformacija Al-Al_4C_3 kompozita. Istraživana je deformacija Al-Al_4C_3 kompozita različitog volumnog udjela Al_4C_3 faze kod različitih temperatura (od 293 do 723 K) i brzina deformacije (od 2,5·10^{-5} s^{-1} do 1,0·10^{-1} s^{-1}). Kod temperatura od 673 do 723 K i najviše brzine deformacije od 1,0·10^{-1} s^{-1} zapaženo je značajno povećanje kovkosti. TEM analiza ukazuje da početak superplastičnosti može biti rezultat dinamičke poligonizacije zrna, klizanja i rotacije zrna, djelomične rekristalizacije i puzanja dislokacija u istraživanom sustavu, što je poznato kao deformacijom inducirano dinamičko oporavljanje. Porast volumnog udjela sekundarne faze u istraživanom kompozitu rezultirao je promjenom kontrolnog mehanizma deformacije od klizanja po granicama zrna do rotacije zrna.

Ključne riječi: *kompozitni materijal, deformacija, brzina deformacije, TEM, superplastičnost*

INTRODUCTION

Mechanical alloying technique, such as dry, high-energy ball milling process, is suitable for producing composite metal powders with a fine controlled microstructure. This method is crucial for obtaining homogeneous distribution of nano-sized dispersoids in a more ductile matrix (e.g. aluminium- or copper based alloys). Dispersoids can be formed in a solid state reaction of materials that react with the matrix during milling or during subsequent heat treatment [1]. Research was focused first on Al and Ti base alloys. Under specific conditions, superplastic deformation was observed in these materials. The details of superplastic behavior in Al based alloys were reported in [2 - 18]. It was concluded that the mechanism of superplastic deformation is a combination of parallel processes such as slip on grain boundaries, dislocation creep and recrystallization [2].

The primary deformation mechanism in such superplastic materials is a slip on grain boundaries with stress accommodation by diffusion or dislocation movement. Subsequently, finer grains would result in higher strain rate at a given stress. Grain below 10 mm with equiaxed morphology and large-angle boundaries are essential for obtaining superplastic deformation. It is possible in such microstructures if the dynamic grain growth is minimized and cavitation at grain boundaries is suppressed [7].

Mechanically alloyed Al-Al_4C_3 composites are the next step in the development of materials with high plasticity [19 - 24]. The advantage is that their strength is higher than that of conventional alloys and it is proportional to the volume fraction and Al_4C_3 particle distribution. These parameters can be controlled by technological conditions of material preparation [19, 20]. The dependence of mechanical properties on strain rate and temperature with regard to fracture micromechanism in a material with low volume fraction of Al_4C_3 was analysed in [22]. The plastic behavior was found to be more favorable with the increase of a strain rate and temperature. The onset of the superplastic behavior was found at 723 K and strain rate of 1,0·10^{-1} s^{-1}.

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The aim of this work is to investigate the Al-Al_4C_3 systems with different second phase particle content under different temperatures and strain rates, and to analyse the corresponding deformation processes.

EXPERIMENTAL MATERIALS AND METHODS

Dispersion strengthened Al - Al_4C_3 composites with 4 and 12 vol. % of Al_4C_3 particles have been prepared by reaction milling at ICHTAS, Technical University of Vienna. The mixture of Al powder (grain size under 50 mm) with 1, 2 and 4 % of graphite KS 2,5 grade was milled for 90 minutes. The granulate was compacted under pressure of 600 MPa and annealed at 823 K for 3 hours to transform C into to Al_4C_3. Hot extrusion of rods was performed at 873 K with a 94 % reduction. The residual porosity of this material was less then 1 %.

The microstructure of the as-received materials was investigated by TEM. The size of dispersed phase particles measured on TEM was ~ 30 nm and the matrix grain size was ~ 380 nm. Tensile specimens were oriented in longitudinal direction of extrusion. Transverse test pieces were not produced due to the limited diameter of the extruded rod. The experiments were performed at temperatures from 293 to 723 K and strain rates from $2{,}5 \cdot 10^{-5}$ s^{-1} to $1{,}0 \cdot 10^{-1}$ s^{-1} on a universal testing machine (Tiratest 2300) with a split furnace. Constant crosshead speed was used. The strain rate was calculated from the crosshead speed and prime gauge length.

RESULTS AND DISCUSSION

Stress-strain curves for material Al-12Al_4C_3 at 293 K, 573 K, 673 K and 723 K are illustrated in Figure 1. At 293 K and 573 K, the first part of the curves with deformation strengthening is followed immediately by the loss of plastic stability, localization of plastic deformation and fracture. The curves obtained at high strain rate and 673 - 723 K exhibit linear behavior (part II) with near constant true stress, which corresponds to dynamic recovery. The loss of plastic stability is indicated in part III. According to our experience, ductility, A_5, and reduction of area, Z, are influenced by the differences of deformation and fracture in the last part (III) of the test.

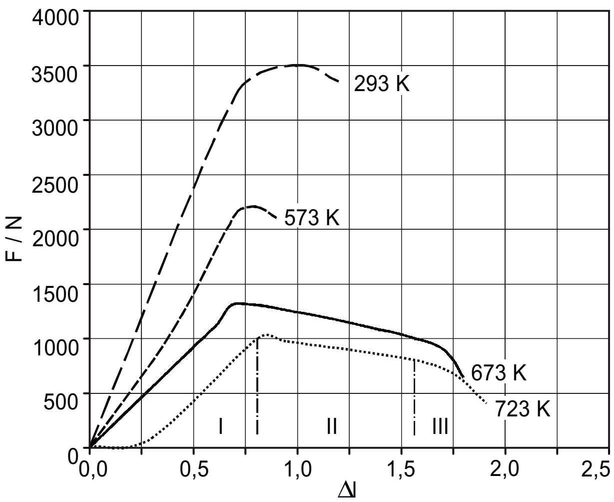

Figure 1. **A comparison of stress-strain curves at 293 to 723 K and strain rate $10^{-1}s^{-1}$ of Al-12Al_4C_3 material**

Slika 1. **Usporedba krivulja naprezanje-deformacija Al-12Al_4C_3 materijala od 293 do 723 K i brzine deformacije $\varepsilon = 10^{-1}$ s^{-1}**

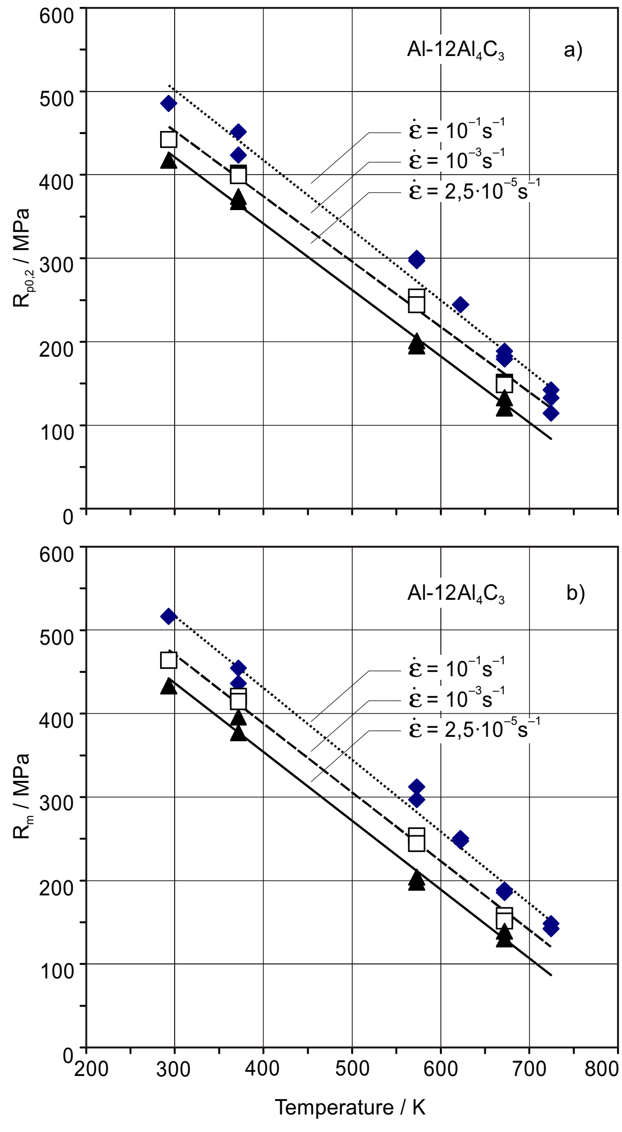

Figure 2. **Temperature dependence of yield point (a) and tensile strength (b) at different strain rates in Al-12Al_4C_3 material**

Slika 2. **Ovisnost granice razvlačenja (a) i vlačne čvrstoće (b) o temperaturi kod različitih brzina deformacije Al-12Al_4C_3 materijala**

Plastic properties of composites with different Al_4C_3 contents are summarized in Figure 2. - Figure 4. Figure

2.a. and Figure 2.b. show that the increase of strain rate in $Al-12Al_4C_3$ caused an increase of $R_{p0.2}$ and R_m, respectively. The increase of Al_4C_3 content from 4 to 12 % result generally in a reduction of elongation (compare Figure 3.a. and

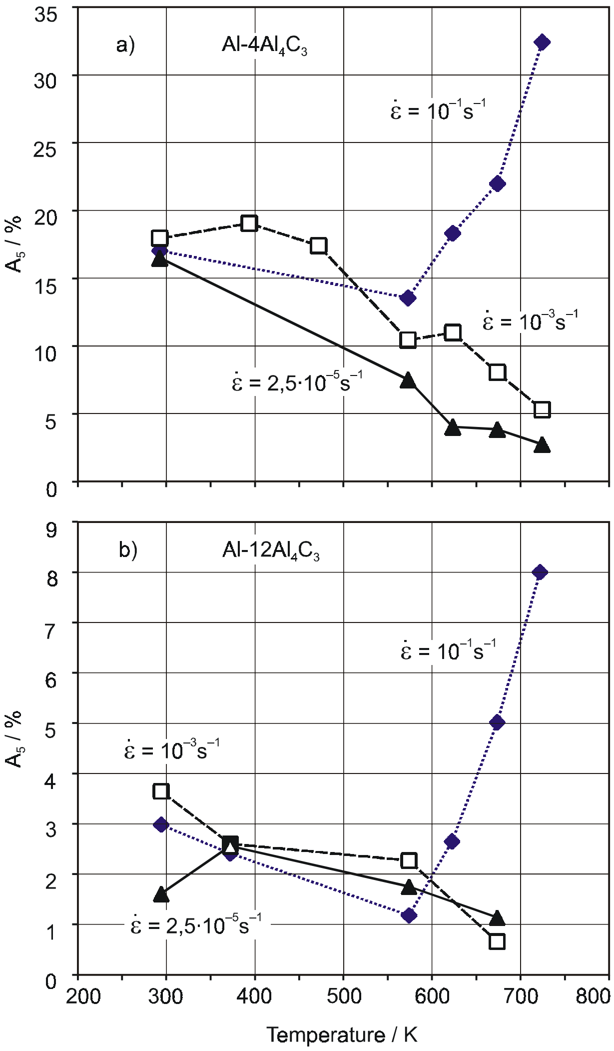

Figure3. **Temperature dependence of elongation, A_5, on strain rate for different materials a) $Al-4Al_4C_3$ and b) $Al-12Al_4C_3$. The onset of superplastic deformation is observed at strain rate $\varepsilon = 1{,}0 \cdot 10^{-1}$ s^{-1} and 573 K**

Slika 3. **Ovisnost istezanja A_5 o temperaturi i brzini deformacije za različite materijale. Početak superplastične deformacije zapažen je kod $\varepsilon = 1{,}0 \cdot 10^{-1}$ s^{-1} i 573 K a) $Al-4Al_4C_3$ materijal, b) $Al-12Al_4C_3$ materijal**

Figure 3.b.) and the decrease of reduction of area, *Z*, (compare Figure 4.a and Figure 4.b.). These parameters usually decrease with decreasing strain rate and testing temperature. However, the increase of A_5 and Z at higher temperatures and strain rate, $1{,}0 \cdot 10^{-1}$ s^{-1}, are evident. An increase of A_5 and *Z* at 673 and 723 K in composites deformed under this strain rate is considered to be the onset of superplasticity.

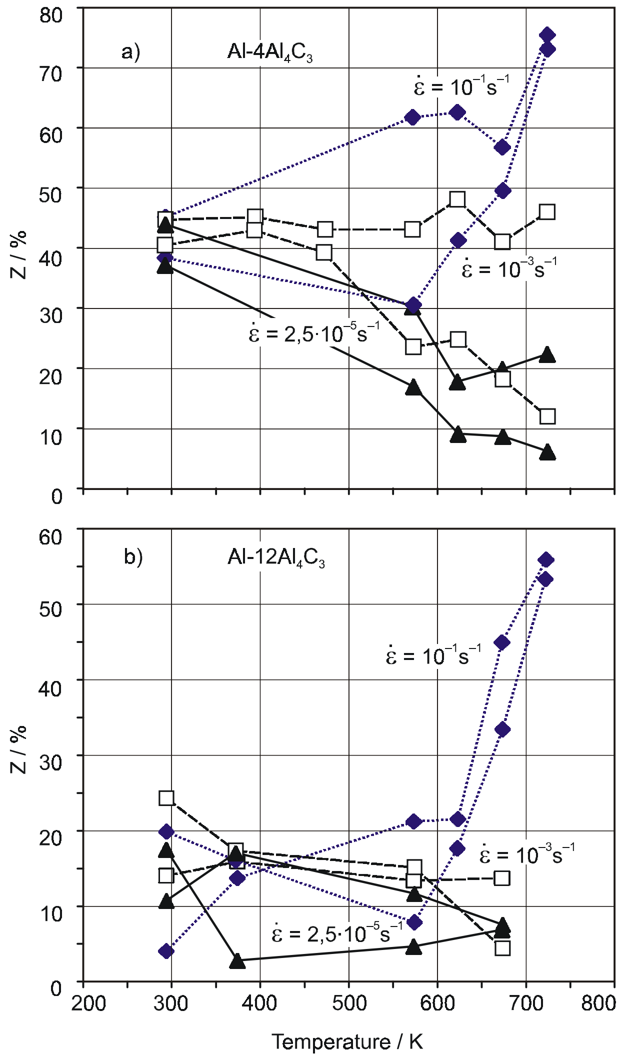

Figure 4. **Temperature dependence of the reduction of area *Z* at different strain rates for different material a) $Al-4Al_4C_3$ and b) $Al-12Al_4C_3$**

Slika 4. **Ovisnost kontrakcije *Z* o temperaturi kod različitih brzina deformacije za različite materijale a) $Al-4Al_4C_3$ materijal, b) $Al-12Al_4C_3$ materijal**

Figure 5. and Figure 6. show principal differences in the microstructure in longitudinal and transverse directions in the materials with 4 and 12 % of Al_4C_3 observed by transmission electron microscopy. The microstructures of both materials are approximately identical in perpendicular direction and consist of equiaxed grains. In confront to that, the microstructure of deformed material with low content of Al_4C_3 (Figure 5.a.), consist of elongated grains, which are parallel to the applied stress. In the case a composite with 12 % Al_4C_3 no preferential grain orientation was observed. Grains were approximately equiaxed and similar to those, seen on the perpendicular cross sections. The size of the grains in the perpendicular direction remains approximately the same in both composites (1,0 to

1,5 μm). However, the size of elongated grains in Al-Al_4C_3 material in longitudinal direction is more than 3,5 μm. Evidently, oriented dynamic recrystalisation has to take

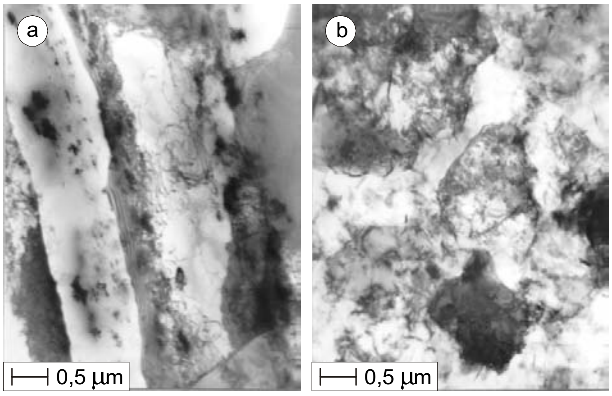

Figure 5. **TEM micrographs of the substructure of Al-4Al_4C_3 material in longitudinal (a) and transverse direction (b), after deformation at 673 K. Large elongated grains form along tensile direction.**

Slika 5. **TEM mikrografije substrukture Al-4Al_4C_3 materijala u uzdužnom (a) i poprečnom smjeru (b) nakon deformacije kod 673 K. Velika izdužena zrna su u smjeru vlačnog naprezanja**

place to obtain oriented elongated grains whereas this process is absent or suppressed in the composite with high content of Al_4C_3.

Thus, the deformation mechanism for high and low volume fraction of Al_4C_3 differs. It should be noted that cavities were not found and their role is negligible in all materials studied.

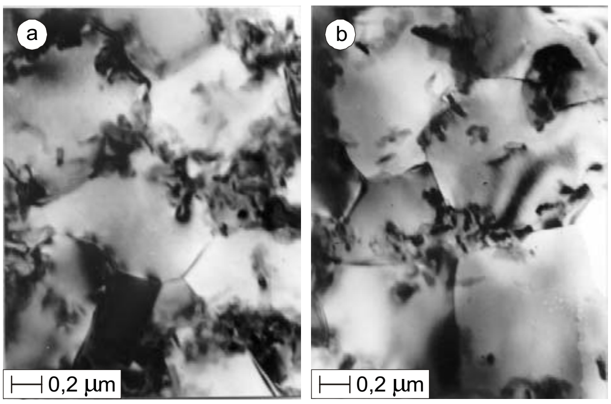

Figure 6. **TEM micrographs of the substructure of Al-12Al_4C_3 material in longitudinal (a) and transverse direction (b). No differences between both directions were observed**

Slika 6. **TEM mikrografije substrukture Al-12Al_4C_3 materijala u uzdužnom (a) i poprečnom smjeru (b). Nisu zapažene razlike ovisno o smjeru ispitivanja**

According to the analysis of Mishra and Mukherjee [2], the deformation of the system Al-Al_4C_3 includes the following mechanisms:

a) dynamic polygonization by dislocation migration and annihilations,
b) slip on grain boundaries,
c) displacement of grains by rotation,
d) partial recrystallization causing grain boundary movement of polygonized grains and
e) dislocation creep, resulting in accommodations of defects at grain boundaries (first in triple points).

During superplastic deformation, grain boundary sliding and related grain shape accommodation, which occurs either by diffusion or by dislocation mechanisms, play dominant role. Because of extremely high deformation rates, dif-

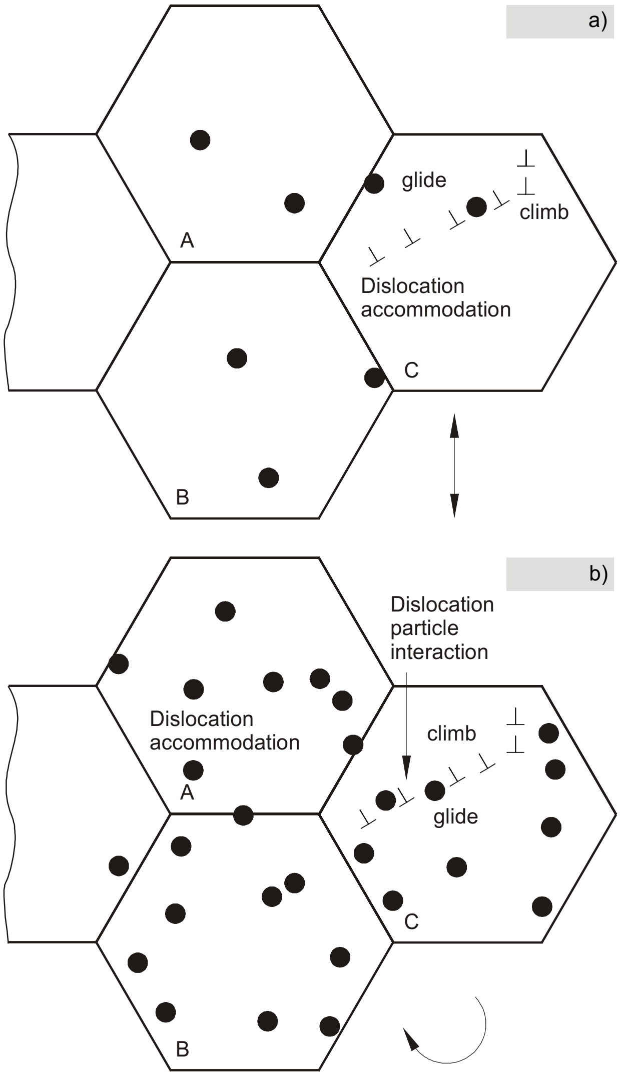

Figure 7. **Schematic illustration of the microstructure and possible dislocation mechanism in the case of low volume fraction of dispersoides (a) and high volume fraction of dispersoides (b)**

Slika 7. **Shematski prikaz mikrostrukture i mogućeg dislokacijskog mehanizma u slučaju niskog (a) i visokog volumnog udjela dispergenata (b)** fusion mechanism for the stress relaxation at the grain boundaries and/or phase interface between the matrix and dispersoides is unprobable. Therefore, it can by assumed that grain boundary sliding and grain shape accommodation result from dislocation mechanisms.

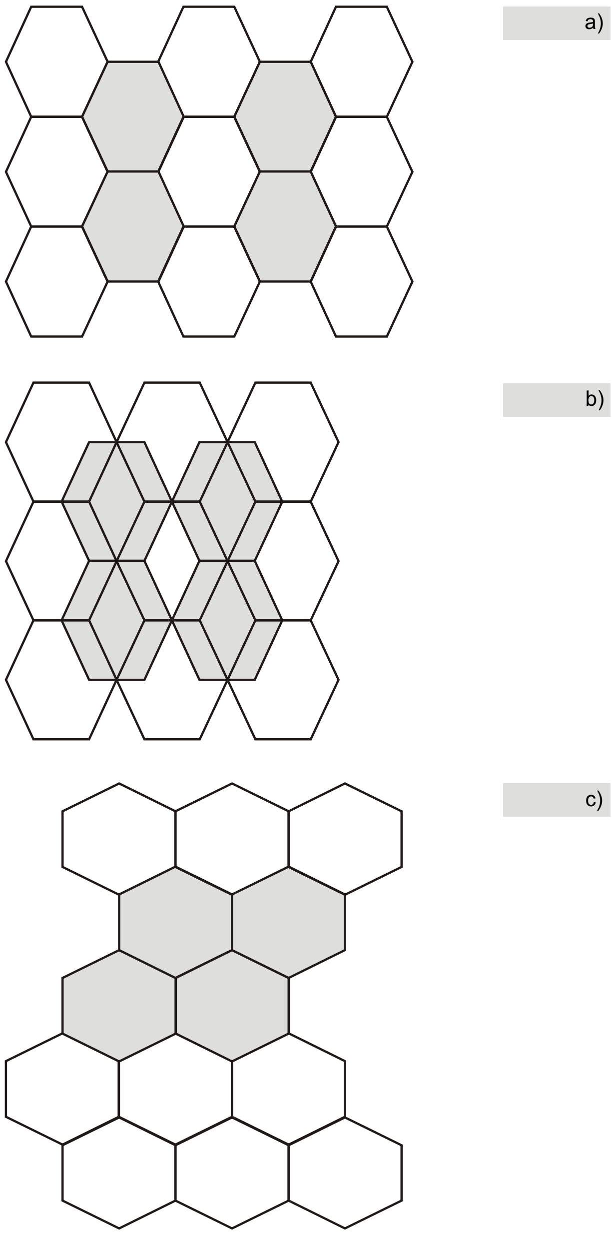

Figure 8. **Schematic illustration of possible superplastic deformation mechanism via grain sliding accompanied by dynamic recrystalisation/polygonisation in the materials with low content of dispersoids**

Slika 8. **Shematski prikaz mogućeg superplastičnog deformacijskog mehanizma putem klizanja zrna praćenog dinamičkom rekristalizacijom/poligonizacijom u materijalu s nisikm sadržajem dispergenata**

The current results suggest that at high strain rates (10^{-1} s^{-1}) and high dispersed phase content (12 vol. % of Al_4C_3), dynamic recovery may occur at first by dynamic polygoni-zation of grains and their repositioning by rotation (elongated grains do not form). Partial recrystallization and dislocation creep can also take place, (see Figure 5. - 6.). Clusters of particles identified as Al_4C_3 in Figure 6. at the grain boundaries suggested that rotation would be easier than slip of the grains.

In the case of low volume fraction of dispersoides (Figure 7.a.), large number of independent slip systems, which satisfy on Misses criterion, exists in the grains. Grain elongation during tensile deformation occurs by dislocation slip mechanism accompanied by dynamic recrystallization. In the case of high volume fraction of Al_4C_3 (Figure 7.b.), the movement of dislocations in the grains is suppressed due to the pinning effect of dispersoides. Because of more restricted shape accommodation of individual grains by dislocation mechanism, mutual sliding of the grains is also limited. The relaxation of the external and local tensile stresses could eventually occur by a collective rearrangement of grains, e.g. enhanced rotation of the whole grains, when only very localized grain shape changes in the contacts are necessary. These processes occur via dislocation mechanism, otherwise cavities would form, which is contrary to the experimental observations. The difference in the microstructure of both composites strongly supports the model of grain sliding, dynamic recrystallization/ polygonisation mechanisms in the case of Al-4Al_4C_3 materials, as it is shown in Figure 8. In the case of Al-12Al_4C_3, the possible mechanisms include grain rotation and very limited dislocation sliding.

CONCLUSION

1. The increase of Al_4C_3 content from 4 to 12 % resulted in a reduction of elongation and decrease of the reduction of area. At temperatures 673 - 723 K and strain rate $1{,}0 \cdot 10^{-1}$ s^{-1}, a significant increase of plastic properties was observed. This increase of plasticity is considered to by an onset of superplasticity.
2. TEM observation showed the formation of elongated matrix grains in the composites with low content of Al_4C_3 particles and equiaxed grains at higher content of Al_4C_3. This suggests that the deformation mechanism is changing from slip of grains to rotation of grains with the increase of Al_4C_3 content.

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