Analysis of Superplastic Deformation of Al-Al$_4$C$_3$ Composites

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Deformation of the Al-Al$_4$C$_3$ composites with different volume fraction of Al$_4$C$_3$ phase was investigated at different temperatures (293 – 723 K) and different strain rates (2.5·10^{-5} s^{-1} to 1·10^{-1}s^{-1}). At temperatures 673 - 723 K and at the highest strain rate 10^{-1} s^{-1}, a significant ductility increase was observed. TEM analysis suggests that it is 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 in the deformation mechanism from more slip on grain boundaries to more grain rotation.
Keywords: composite material, deformation, strain rate, TEM analysis and superplasticity.

1. 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]. Mechanical alloying can produce a variety of dispersion strengthened alloys with improved plastic behaviour after thermo-mechanical treatment [2]. Research was focused first on Al and Ti base alloys. Under specific conditions, superplastic deformation was observed in these materials. The details of superplastic behaviour in Al base 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].

Superplasticity in polycrystalline materials is facilitated by dynamic modifications in the microstructure. The strain rate is defined by Mukherjee equation:

$$\dot{\varepsilon} = \frac{KDGb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{G}\right)^{\frac{1}{m}}, \qquad (1)$$

where K is constant, D is the diffusion coefficient, G is shear modulus, b is the Burgers vector, σ is the deformation stress, T is the temperature, d is the grain size, p is the grain size exponent $(2 – 3)$, and m is the stress exponent $(0.4 – 0.7)$.

The primary deformation mechanism in superplastic materials is slip on grain boundaries with stress accommodation by diffusion or dislocation movement. According to equation (1), finer grains will result in higher strain rate at a given stress. Grains size less than 10 μm, and equiaxed grains with large angle boundaries are essential for obtaining superplastic deformation. Superplastic deformation is possible in such microstructure if the

dynamic grain growth is minimized and cavitations on grain boundaries are suppressed.

Mechanically alloyed Al-Al$_4$C$_3$ composites are the following step in development of materials with higher plasticity [19 – 24]. Their advantage is that their strength is higher than the strength of conventional alloys, and it is proportional to the volume fraction, and Al$_4$C$_3$ particle distribution. These parameters are controlled by the 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$_4$C$_3$ was analysed in [22]. The plastic behaviour was found to be more favourable with the increasing of strain rate and temperature. The onset of the superplastic behaviour was found at 723 K and strain rate of 10^{-1} s^{-1}.

The aim of this work is to investigate deformation process in the Al-Al$_4$C$_3$ systems with different second phase particle content under different temperatures and strain rates, and analyse the corresponding deformation processes.

2. EXPERIMENTAL MATERIALS AND METHODS

Dispersion strengthened Al - Al$_4$C$_3$ composites with 4, 8 and 12 vol. % of Al$_4$C$_3$ particles have been prepared by reaction milling in ICHTAS, Technical University of Vienna. The Al powder (grain size under 50 μm) with 1, 2 and 4 % of KS 2.5 graphite added 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 to Al$_4$C$_3$. Hot extrusion of rods was performed at 873 K with a 94 % reduction. The residual porosity of this material was up to 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 grain size was ~380 nm. The tensile test 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

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temperatures from 293 to 723 K and strain rates from $\dot{\varepsilon} = 2.5 \cdot 10^{-5}$ s^{-1} to $1 \cdot 10^{-1}$ 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.

3. RESULTS AND DISCUSSION

$F - \Delta l$ curves for material Al – 12 Al$_4$C$_3$ at 293 K, 573 K, 673 K and 723 K are illustrated in Fig. 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. $F - \Delta l$ curves obtained at high $\dot{\varepsilon}$ and 673 – 723 K exhibit linear behaviour (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, both A_5 and Z are influenced by differences of deformation, and fracture in the last part (III) of the test.
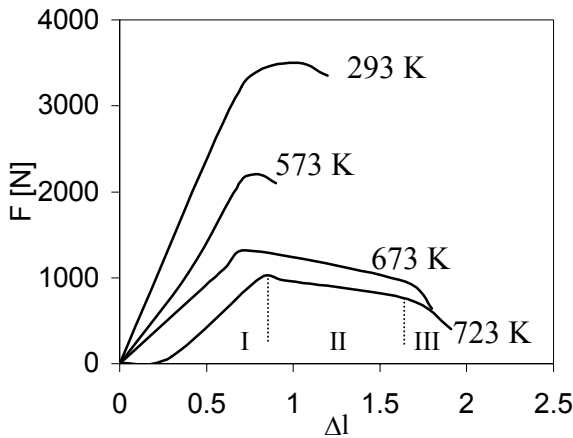

Fig. 1. A comparison of $F - \Delta l$ curves at 293-723 K and $\dot{\varepsilon} = 10^{-1}$s^{-1} in the case of material Al-12Al$_4$C$_3$
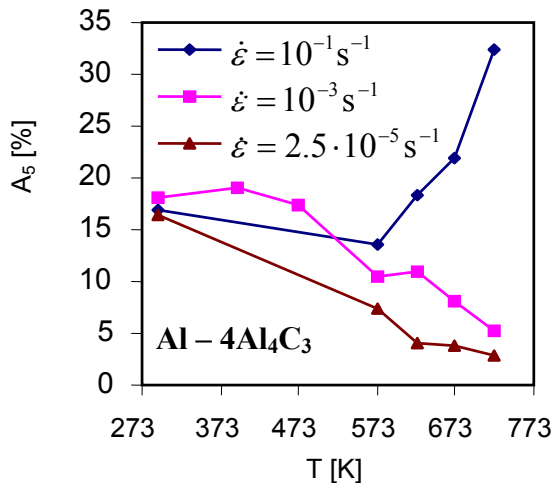

Fig. 2. Dependence of ductility A_5 on temperature and strain rate for material Al–4Al$_4$C$_3$

The plastic properties of composites with different Al$_4$C$_3$ content are expressed by ductility A_5 and reduction of area Z in Fig. 2. – Fig. 5. The increase of Al$_4$C$_3$ content from 4 to 12 % caused generally a reduction of ductility

and the reduction of area. An increase of A_5 and Z at 673 and 723 K in composites deformed under the highest strain rate, $\dot{\varepsilon} = 1 \cdot 10^{-1}$ s^{-1}, we may consider to be an onset of superplasticity. However, because the values of plasticity are not ultra high process is called quasi-superplasticity.
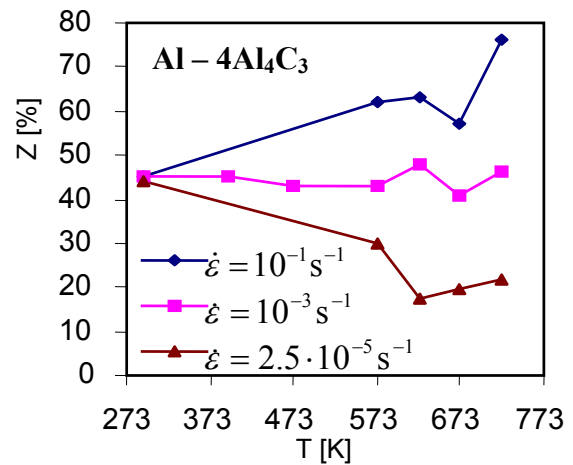

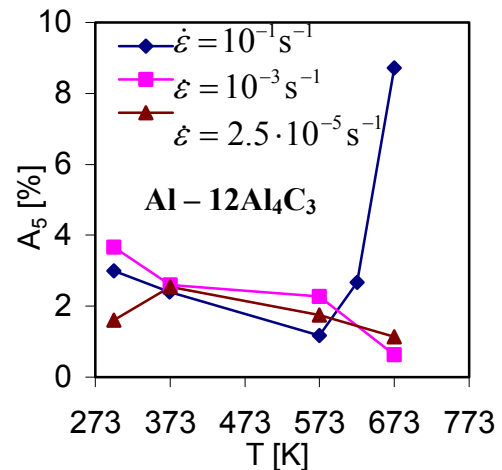**Fig. 3.** Dependence of reduction of area Z on temperature and strain rate for material Al–4Al$_4$C$_3$

Fig. 4. Dependence of ductility A_5 on temperature and strain rate for material Al–12Al$_4$C$_3$
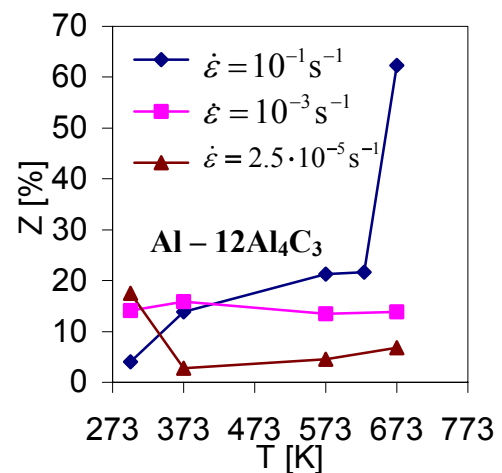

Fig. 5. Dependence of reduction of area Z on temperature and strain rate for material Al–12Al$_4$C$_3$

The results suggest that at high strain rate of $1 \cdot 10^{-1}$ s^{-1} and high dispersed phase content (12 vol.% of Al$_4$C$_3$), dynamic recovery is driven by the high accumulated deformation energy. It occurs at first by dynamic polygonization of grains and their repositioning by rotation (elongated grains were not observed). Partial recrystallization and dislocation creep can also take place. Clusters of particles identified as Al$_4$C$_3$ in Fig. 6 and Fig. 7 on grain boundaries suggested that rotation would be easier than slip of the grains.

Fig. 6. Submicrostructure (foil) material Al–12Al$_4$C$_3$ - transverse direction
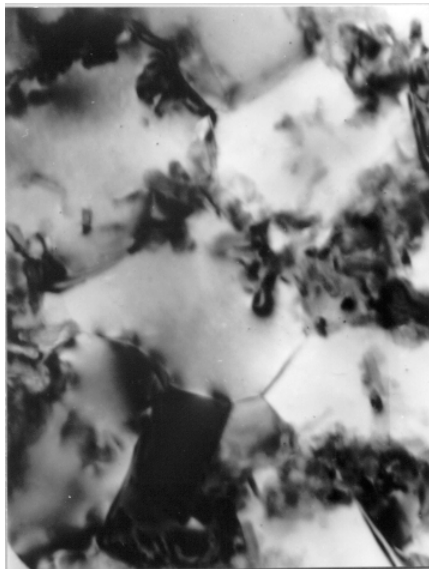

Fig. 7. Submicrostructure (foil) material Al–12Al$_4$C$_3$ longitudinal direction

In the case of materials with 4 and 8 vol.% of Al$_4$C$_3$ the increase of A$_5$ and Z are evident at higher temperatures and high strain rate. These parameters decrease with decreasing strain rate and increase of Al$_4$C$_3$ content. Fig. 8 and Fig. 9 show the microstructure of material with 4 vol.% of Al$_4$C$_3$. Obviously the slip on grain boundaries is easier in the microstructure with elongated grains than in the equiaxed microstructures. The probability of

polygonization and partial recrystallization would by also lower. Thus, the deformation mechanism for high and low volume fraction of Al$_4$C$_3$ is differs. It should be noted that cavities were not found and their role is negligible in all materials studied.
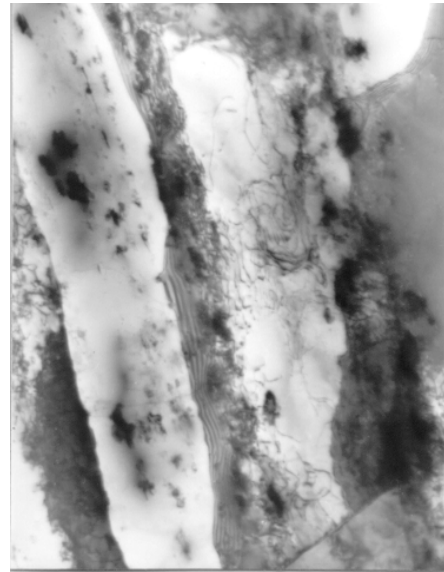

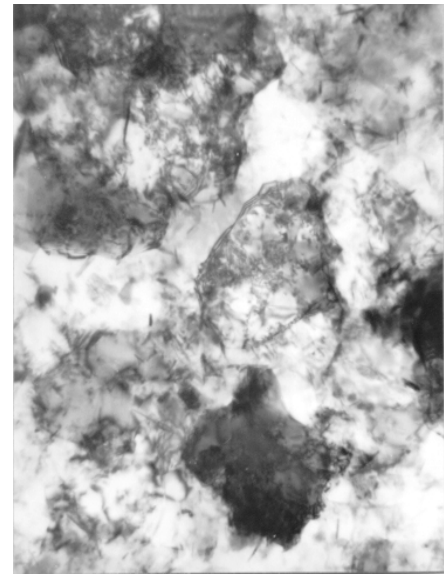**Fig. 8.** Submicrostructure (foil) material Al–4Al$_4$C$_3$ longitudinal direction

Fig. 9. Submicrostructure (foil) material Al–4Al$_4$C$_3$ transverse direction

According to current results and analysis [2], the deformation of the system Al–Al$_4$C$_3$ seems to include 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).
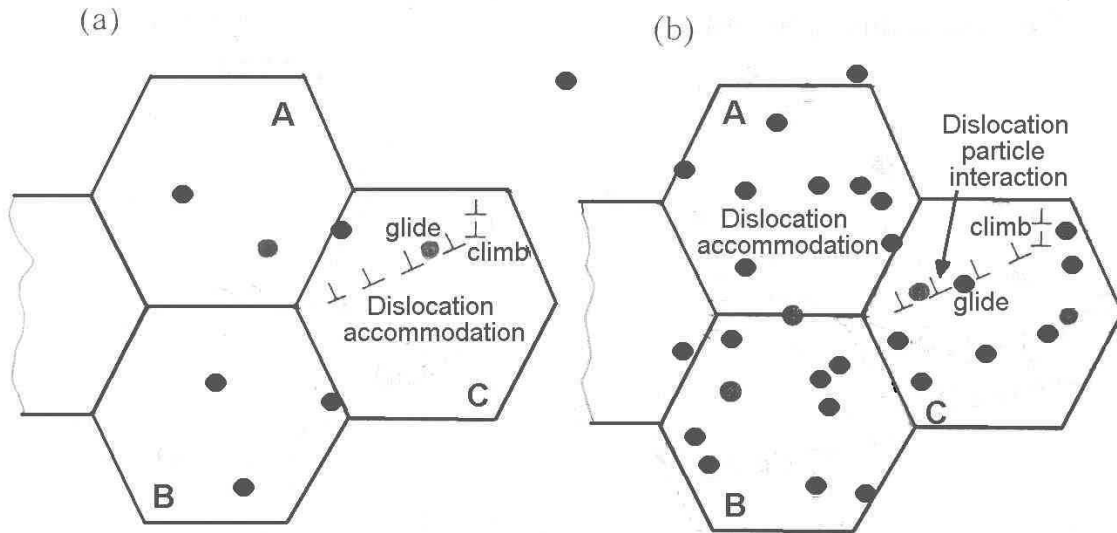
Fig. 10. Tensile deformation: a – in the case of low volume fraction of dispersoides, b – in the case of high volume fraction of dispersoides
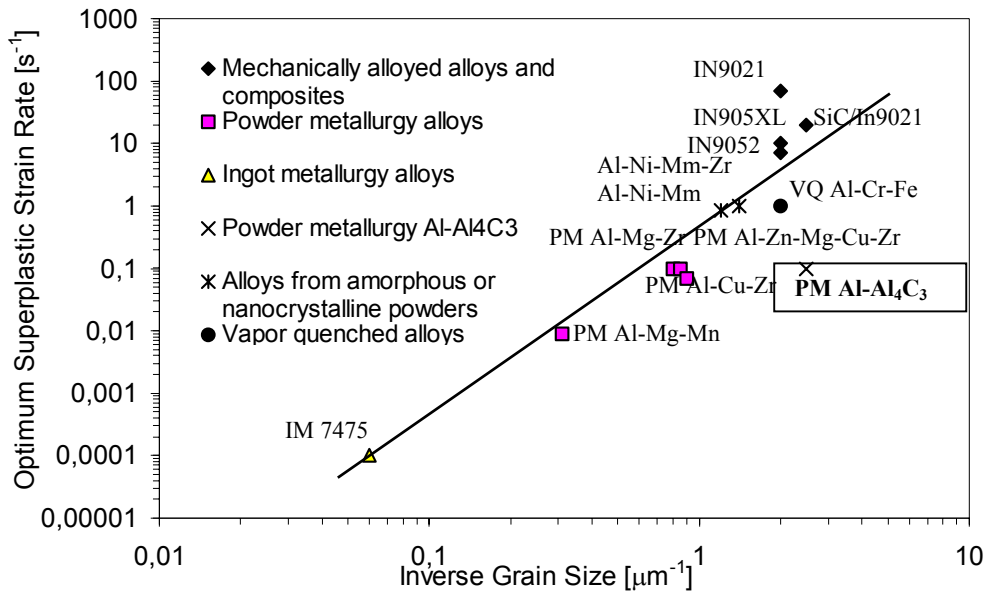

Fig. 11. Dependence of optimum superplastic strain rate on the grain size for superplastic Al alloys produced by different routes

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, diffusion 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.

In the case of low volume fraction of dispersoides (Fig. 10 a), large number of independent slip systems, which satisfy on Misses criterion, exists in the grains. Grain elongation during tensile deformation can therefore occur by dislocation slip mechanism. In the case of high volume fraction of dispersoides (Fig. 10 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. However, it is possible that the relaxation of the external and local tensile stresses occurs by a collective rearrangement of grains, e. g. enhanced rotation of the whole grains, when grain shape changes only in very localised contacts are necessary. These processes should occur via dislocation mechanism, otherwise cavities would form, which is contrary to the experimental observations. Another argument supporting dislocation mechanism is that deformation takes place at relatively low homological temperature.

Fig.11 [2] shows the dependence of optimum superplastic strain rate on the inverse grain size for a large variety of superplastic aluminium alloys produced by

different routes. The best experimental values are close to the line characterize with $n = 3$ (see equation (1)). The experimental conditions for Al-Al$_4$C$_3$ materials studied are under this line. This suggests that the applied strain rates up to 10^{-1} s^{-1}, limited by the testing equipment, were out of the optimum conditions to get superplastic behavior.

4. CONCLUSIONS

1. The increase of Al$_4$C$_3$ content from 4 to 12 % resulted in reduction of ductility and the reduction of area. At temperatures $673 - 723$ K and strain rate $\dot{\varepsilon} = 1 \cdot 10^{-1}$ 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$_4$C$_3$ particles and equiaxed grains at higher content of Al$_4$C$_3$. This suggests that the deformation mechanism is changing from slip of grains to rotation of grains with the increase of Al$_4$C$_3$ content.

3. The dynamic recovery in this material is limited compared to other superplastic materials.

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