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

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ABSTRACT

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

The advances in the technology of mechanical alloying allow us to incorporate very fine particles in a fairly uniform distribution into often oxidation- and corrosion-resistant metal matrices in accordance with the earlier developed theoretical concepts. A mechanical alloying technique, such as the 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's equation:

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

where D – is the diffusion coefficient, T – temperature, d – grain size, p – 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 would result in

higher strain rate at a given stress. Grains of a 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 next step in development of materials with higher plasticity /19-23/. 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 an increase 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 the 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 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

Stress-strain curves for material Al-12Al$_4$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. Stress-strain curves obtained at high strain rate and temperatures 673 K and 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_s and Z are influenced by differences of deformation, and fracture in the last part (III) of the test.
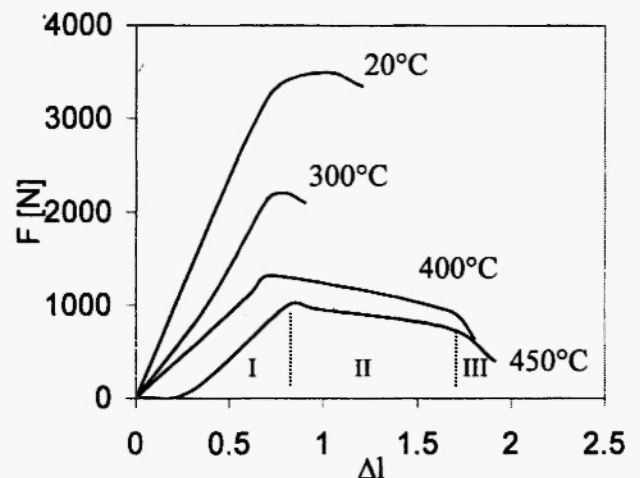

Fig. 1: A comparison of stress-strain curves at 293 K to 723 K and strain rate 10^{-1}s^{-1} in the case of material Al-12Al$_4$C$_3$.

The plastic properties of composites with different Al_4C_3 content are expressed by ductility A_5 and reduction of area Z in Fig. 2 – Fig. 4. The increase of Al_4C_3 content from 4 to 12% caused a general reduction of ductility and a 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, the process is called quasi-superplasticity.

The results suggest that at high strain rate of $1 \cdot 10^{-1}$ s^{-1} and high dispersed phase content (12 vol.% of Al_4C_3), dynamic recovery is driven by the high accumulated deformation energy. It occurs at first by dynamic polygonization of grains and their re-positioning by rotation (elongated grains were not observed). Partial recrystallization and dislocation creep can also take place. Clusters of particles identified as Al_4C_3 in Fig.5 and Fig.6 on grain boundaries suggested that rotation would be easier than slip of the grains.
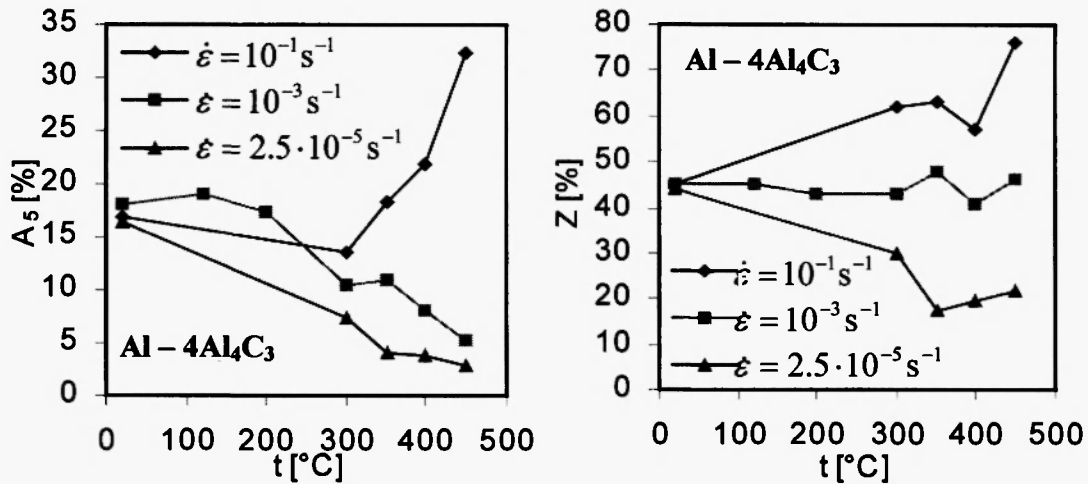

Fig. 2: Dependence of ductility A_5 and reduction of area Z on temperature and strain rate for material $Al-4Al_4C_3$.
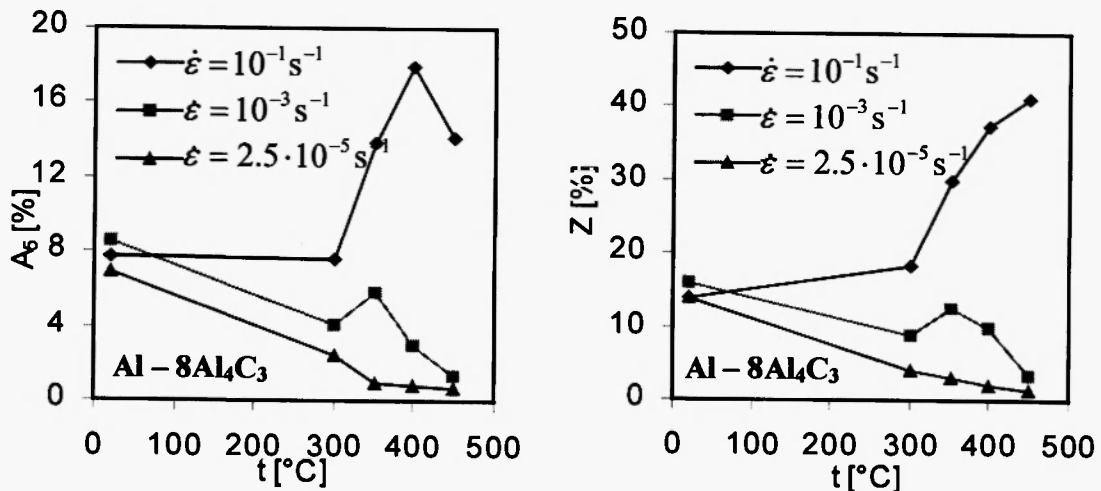

Fig. 3: Dependence of ductility A_5 and reduction of area Z on temperature and strain rate for material $Al-8Al_4C_3$.
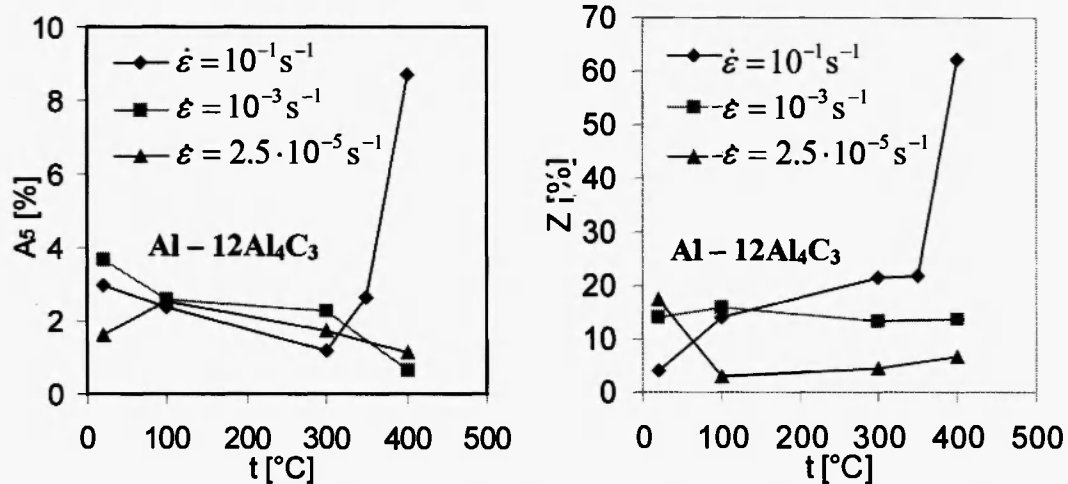
Fig. 4: Dependence of ductility A_5 and reduction of area Z on temperature and strain rate for material Al-12Al₄C₃.

Fig. 5: Submicrostructure (foil) material Al-12Al₄C₃ – transverse direction.
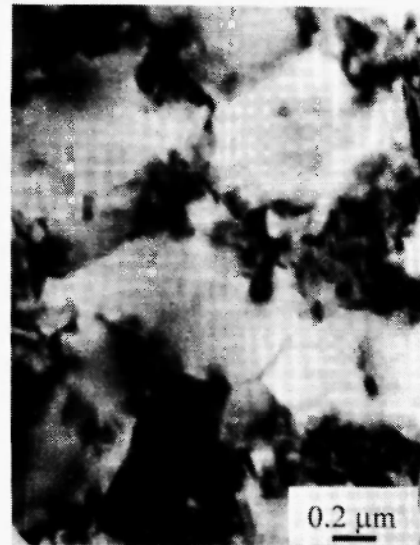

Fig. 6: Submicrostructure (foil) material Al-12Al₄C₃ – longitudinal direction.

In the case of materials with 4 and 8 vol.% of Al₄C₃ the increase of A_5 and Z is evident at higher temperatures and high strain rate. These parameters decrease with decreasing strain rate and increase of Al₄C₃ content. Fig. 7 and Fig. 8 show the microstructure of material with 4 vol.% of Al₄C₃. 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 also be lower. Thus, the deformation mechanism for high and low volume fraction of Al₄C₃ is different.

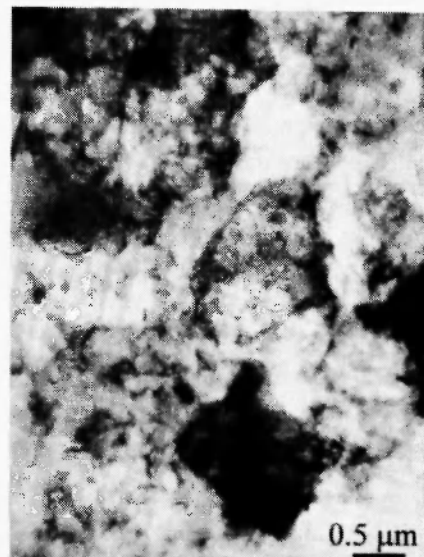**Fig. 7:** Submicrostructure (foil) material Al-4Al$_4$C$_3$ – longitudinal direction.

Fig. 8: 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).

Grains can move or can be reoriented by two mechanisms – Fig.9:

a) slip along grain boundaries,

b) rotation

The given interpretation of our experimental results is in good agreement with the "superplastic mechanism map" designed by Mishra and Mukherjee /2/ shown in Fig. 10. The deformation of Al-Al$_4$C$_3$ materials studied in the dependence of particle size and temperature would be in the area of grain boundary sliding. Fig. 11 /8/ shows the dependence of optimum superplastic
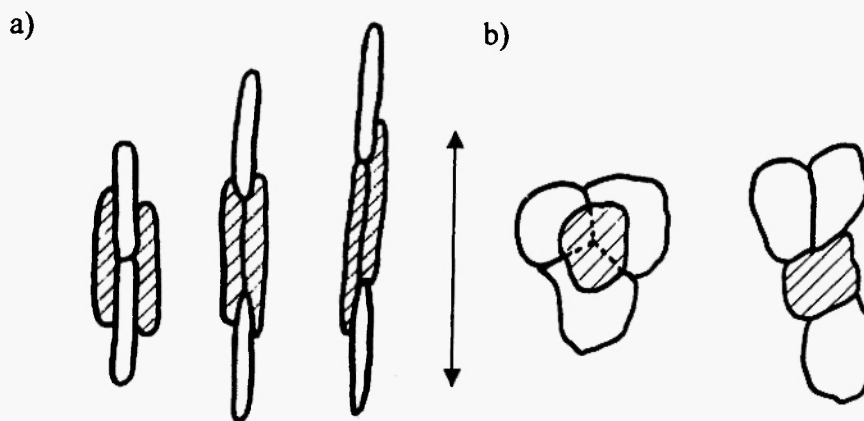

Fig. 9: Supposed reordering mechanisms: a) slip, b) rotation.

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 characterized by $n = 3$ (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 outside the optimum conditions to obtain superplastic behavior.

CONCLUSION

1. The increase in 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 $\dot\varepsilon = 1\cdot10^{-1}$ s^{-1}, a significant increase in plastic properties was observed. This increase in plasticity is considered to be the 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 in Al$_4$C$_3$ content.

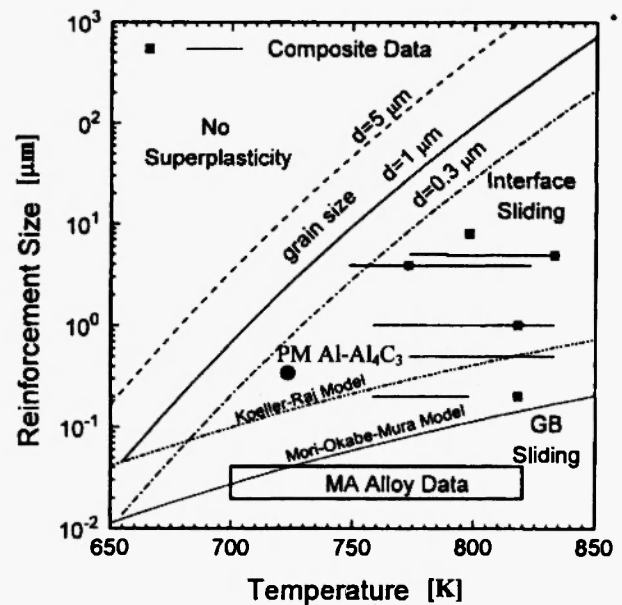3. The dynamic recovery in this material is limited compared to other superplastic materials.

Fig. 10: The "superplastic mechanism map" for dispersion strengthened Al materials.
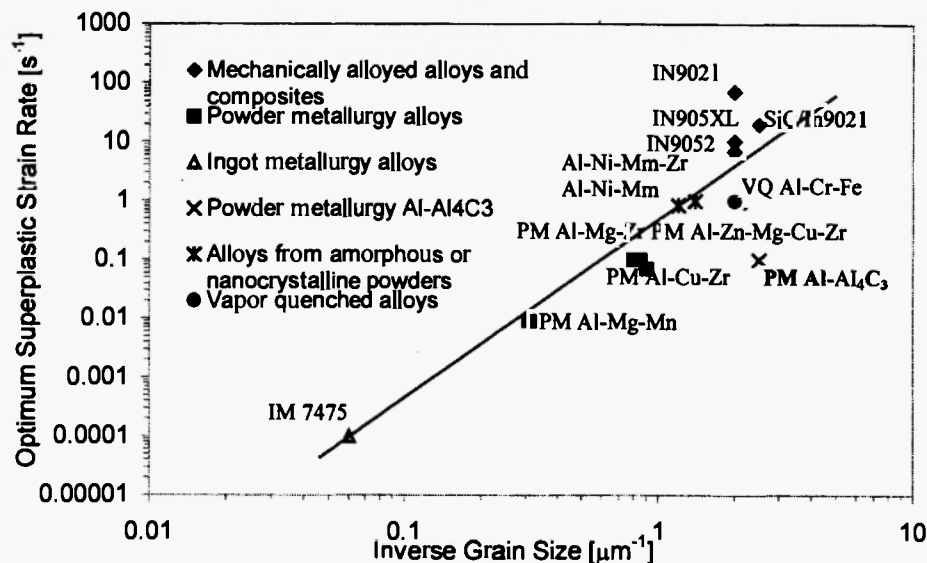
ACKNOWLEDGEMENT

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

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