THE INFLUENCE OF VOLUME FRACTIONS OF PARTICLES ON ENHANCED PLASTICITY OF Al-Al₄C₃ COMPOSITES

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Deformation process of the Al-Al₄C₃ composites with different volume fraction of Al₄C₃ phase was tested at different temperatures and different strain rates. As shown, at temperatures 673–723 K and the highest strain rate applied 10^{-1} s⁻¹, a significant increase of ductility was observed. According to the results of transmission electron microscopy analysis (TEM), this behaviour is supposed to be the result of dynamic grain polygonization, grain slip and rotation, partial recrystallization and dislocation creep in the tested system, known as the strain induced dynamic recovery. Increase of the volume fraction of second phase in the investigated composite changed the deformation mechanism (from more grain boundary sliding to predominantly grain rotation).

Key words: composite material, strain rate, TEM analysis, superplasticity

1. Introduction

As known, the plasticity of $Al-Al_4C_3$ composites decreases with the increase of temperature. Any final reshaping is hard and demanding on the tools used. It is also known and has been frequently reported, that in these very fine dispersion strengthened composites, some combination of elevated temperature and high strain rate results in superplastic behaviour. Savings in tool and energy costs can be obtained by the exploitation of the phenomenon of the superplastic deformation.

Superplasticity in polycrystalline materials is facilitated by dynamic modifications in the microstructure. Retrieval of the superplastic microstructure at elevated

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temperatures rests on the ability to maintain dynamic recovery. The strain rate is defined by the Mukherjee equation (1):

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

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

The primary deformation mechanism in superplastic materials is grain boundary sliding with stress accommodation by diffusion or dislocation movement. According to the Eq. (1) the finer the grains the higher the strain rate to obtain superplasticity at a given stress. Grain size less than 10 μ m, and equiaxed grains with large angle boundaries are essential. Superplasticity is maintained if the dynamic grain growth is minimized. However, superplasticity can be limited by cavitations on grain boundaries.

Superplasticity in Al base alloys is reported in [1-17]. Contribution of materials produced by mechanical alloying is significant. The kinetics and mechanisms of deformation are described. As reported, the mechanism of superplastic deformation is a combination of simultaneous processes as grain boundary sliding, dislocation creep, and dynamic recrystallization.

We have described in our works [18-21] the production technology, the microstructure of produced Al-Al₄C₃ materials and their properties. The composites have been prepared by mechanical alloying. Strength of the composites is higher than the strength of conventional alloy, and both the strength and plasticity of the composites are decreasing with the increase of temperature.

The aim of this work is to analyse the deformation of the $Al-Al_4C_3$ system at different temperatures, strain rates and second phase particle content, and describe the deformation process.

2. Experimental materials and methods

Dispersion strengthened Al-Al₄C₃ composites with different volume fraction of Al₄C₃ (4 and 12 vol.%) have been prepared by powder metallurgy, namely by the method of reaction milling. The milling of the Al powder (grain size less than $50 \ \mu\text{m}$) with 1 and 4 % of graphite lasted 90 minutes. The granulate was compacted under the pressure of 600 MPa and annealed at 823 K during 3 hours to transform Al and C to Al₄C₃. Hot extrusion of rods was performed at 873 K with 94% reduction. The residual porosity of this material was up to 1 %. The experimental materials were produced at the Technical University of Vienna, Austria.

The specimen for tensile tests is in Fig. 1. From its gauge thin slices for TEM foils were cut. The size of dispersed phase particles measured by TEM was of

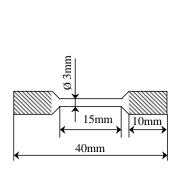


Fig. 1. The specimen for tensile tests.

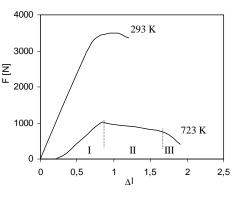


Fig. 2. $F-\Delta l$ curves comparison for Al--12Al₄C₃ material by temperatures 273 and 723 K, strain rate 10^{-1} s⁻¹.

~ 30 nm and grain size was of ~ 380 nm. The material was tested at temperatures from 293 to 723 K and initial strain rates from $\dot{\varepsilon} = 2.5 \times 10^{-5} \text{ s}^{-1}$ to $1 \times 10^{-1} \text{ s}^{-1}$. The tensile test specimens were oriented in longitudinal direction with respect to the direction of extrusion. Transverse specimen could not be produced owing to the limited diameter of the extruded rod.

3. Results and discussion

 $F-\Delta l$ curves for material Al-12Al₄C₃ are shown in Fig. 2. At 293 K the first part characteristic by the deformation strengthening is followed immediately by the loss of plastic stability, plastic deformation localization and fracture. On the other side, curves for high strain rate and temperatures 673 and 723 K showed after the part I a linear part II with almost constant true stress (sometimes called "plateau"), with signs of dynamic recovery, and only after that the part (III), with the loss of plastic stability.

Plastic properties are characterized by the ductility A_5 and the reduction of area Z in Figs. 3, 4 for different Al₄C₃ contents. For lower strain rates the decrease of plastic properties is characteristic (both A_5 and Z) for an increase of temperature up to 723 K. The increase of the Al₄C₃ dispersed phase particles content (4 and 12 %) lowered the values of both the ductility A_5 and the reduction of area Z. A peak-like significant increase of plastic properties, both ductility A_5 and reduction of area Z is visible at 673 and 723 K in all plots (Figs. 3, 4) for the highest strain rate $\dot{\varepsilon} = 1 \times 10^{-1} \text{ s}^{-1}$.

According to our experience, both the ductility A_5 and the reduction of area Z (likely the latter more) are influenced by microstructure parameters. TEM micrographs of thin foils were produced from the fracture area. The values of ductility

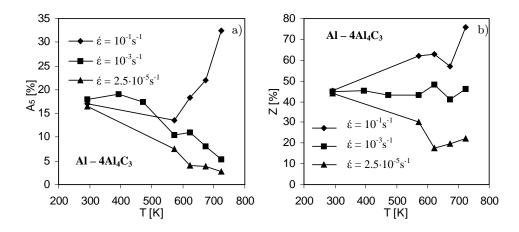


Fig. 3. a) Temperature dependence of ductility A_5 and b) the reduction of area Z of Al-4Al₄C₃ for different strain rates.

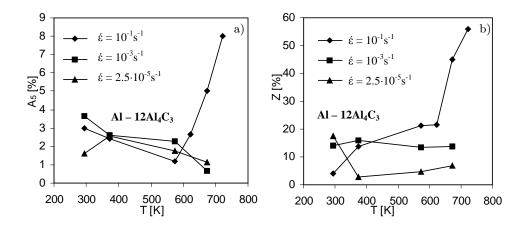


Fig. 4. a) Temperature dependence of ductility A_5 and b) the reduction of area Z of Al-12Al₄C₃ for different strain rates.

and reduction of area are much lower than those for known superplastic materials. According to our previous results [22] and results described in [1], we suppose that the deformation behaviour of the system $Al-Al_4C_3$ consists of a combination of the following mechanisms: dynamic polygonization by dislocation glide and annihilation, grain boundaries sliding, displacement of grains by rotation, partial recrystallization due to grain boundary movement of polygonized grains, disloca-

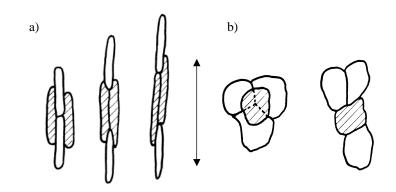


Fig. 5. Supposed reorientation mechanisms: a) grain boundary sliding, b) rotation.

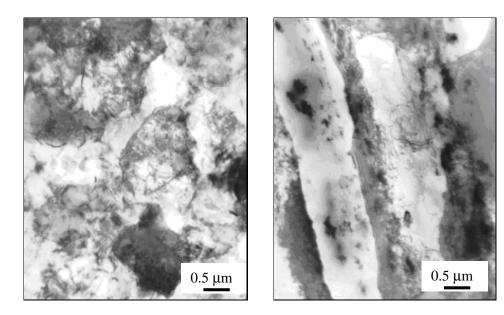


Fig. 6. TEM micrograph of the substructure of Al-4Al₄C₃ in the transverse direction by 673 K.

Fig. 7. TEM micrograph of the substructure of Al-4Al₄C₃ in the longitudinal direction 673 K.

tion creep, causing accommodation of defects at grain boundaries (first in tripple points).

Grains can move or be reordered as shown in Fig. 5 by two mechanisms:

a) grain boundaries sliding,

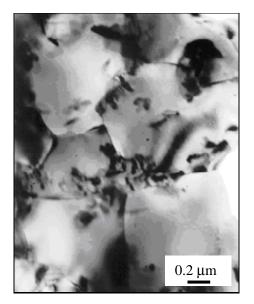


Fig. 8. TEM micrograph of the substructure of $Al-12Al_4C_3$ in the transverse direction 673 K.

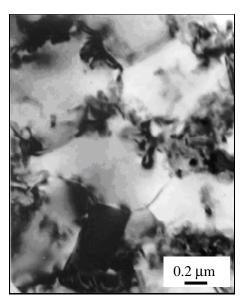


Fig. 9. TEM micrograph of the substructure of $Al-12Al_4C_3$ in the longitudinal direction 673 K.

b) rotation.

We have analysed the partial contributions of different mechanisms to the superplastic deformation. For low particle content (4 vol.% of Al_4C_3) the elongated grains after the grain boundaries sliding are more frequent, which means a lower probability of polygonization and partial recrystallization, Figs. 6 and 7.

We suppose that at highest strain rate $1 \times 10^{-1} \text{ s}^{-1}$ and high dispersed phase content (12 vol.% of Al₄C₃), dynamic recovery starts with the high accumulated deformation energy. First dynamic polygonization of grains, repositioning by rotation (no elongated grains seen), partial recrystallization and dislocation creep may take place, too. Clusters of particles identified as Al₄C₃ in Figs. 8 and 9 at grain boundaries indicate more rotation than slip.

The recovery is not as perfect as known for superplastic materials. Local deformation with neck formation and fracture with limited reduction of area are the signs showing the limits of these materials. Consequently, it would be more precise to define the deformation process as a quasi-superplastic behaviour.

For the description of the overall effect of temperature and strain rate on the conventional yield point, the parameters of the mechanical equation of the state used for materials with 12 vol.% of Al_4C_3 subjected to high stresses according to

Poirier [23] were determined:

$$\dot{\varepsilon} \cdot \exp(Q/RT) = 3.15 \times 10^{13} \cdot \exp(-0.152 \cdot R_{\rm p} 0.2),$$
 (2)

where $\dot{\varepsilon}$ is strain rate, Q is activation energy, R is universal gas constant, T is abs. temperature and $R_{\rm p}0.2$ is yield strength.

The presented Eq. (2) includes the value of Zener-Hollomon parameter in the temperature region 573–773 K in the investigated interval of deformation rate. Its correlation coefficient r = 0.953 suggests a certain scatter of material properties. The apparent activation energy reached the value of 325 kJ·mol⁻¹. The value of the activation energy for the deformation controlled diffusion of atoms in the matrix, for example with the mechanism of by-passing of particles by climbing dislocations, should reach the theoretical value of 142 kJ·mol⁻¹ [24]. The value of 325 kJ·mol⁻¹ indicates, that the deformation process in the entire investigated range of temperature and strain rate, consists of several deformation mechanisms.

The given interpretation of our experimental results is in good agreement with the "superplastic mechanism map" designed by Mishra and Mukherjee [1] as shown in Fig. 10. Our experimental results for the material $Al-Al_4C_3$ in the dependence of reinforcement size on temperature are in the area of grain boundary sliding.

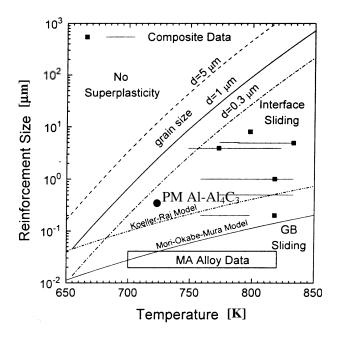


Fig. 10. The "superplastic mechanism map" for dispersion strengthened Al materials [2].

4. Conclusions

The influence of temperature and strain rate on mechanical properties of Al_4C_3 composites was tested for different volume fraction of the dispersed phase. Dependences of ductility A_5 and reduction of area Z on temperature and strain rate were estimated.

The analysis of fracture surfaces and microstructure results showed signs of dynamic recovery processes: dynamic polygonization, grain boundary sliding and rotation of grains, dislocation creep, and partial recrystallization. Micrographs showed elongated grains at lower dispersed phase particle content and equiaxed grains at higher content. We suppose the corresponding deformation mechanism is changing also because of prevailing sliding to rotation of grains with the increase of Al_4C_3 secondary phase content.

Acknowledgements

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REFERENCES

- [1] MISHRA, R. S.—MUKHERJEE, A. K.: Mater. Sci. Eng., A234–236, 1997, p. 1023.
- [2] MISHRA, R. S.—BIELER, T. R.—MUKHERJEE, A. K.: Scripta Metall., 26, 1992, p. 1605.
- [3] BIELER, T. R.-MUKHERJEE, A. K.: Mater. Sci. Eng., A128, 1990, p. 171.
- [4] HIGASHI, K.—OKADA, T.—MUKAI, T.—TANIMURA, S.: Scripta Metall., 25, 1991, p. 2053.
- [5] BIELER, T. R.—MUKHERJEE, A. K.: Materials Trans. JIM, 32, 1991, p. 1149.
- [6] HIGASHI, K.—OKADA, T.—MUKAI, T.—TANIMURA, S.—NIEH, T. G.—WADS-WORTH, J.: Scripta Metall., 26, 1992, p. 185.
- [7] HIGASHI, K.: Mater. Sci. Eng., A166, 1993, p. 109.
- [8] HIGASHI, K.—OKADA, T.—MUKAI, T.—TANIMURA, S.: Mater. Sci. Eng., A159, 1992, L1-L4.
- [9] JIN, I.—BIELER, T. R.: J. Mater. Sci., 28, 1993, p. 2413.
- [10] NIEH, T. G.—WADSWORTH, J.—IMAI, T.: Scripta Metall., 26, 1992, p. 703.
- [11] ASHBY, M. F.—VERRALL, R. A.: Acta Metall., 21, 1973, p. 149.
- [12] GIFKINS, R. C.: J. Mater. Sci., 13, 1978, p. 1926.
- [13] SAKAI, M.—MUTO, H.: Scripta Mater., 38, 1998, p. 909.
- [14] HIGASHI, K.—OKADA, T.—MUKAI, T.—TANIMURA, S.: Scripta Metall., 26, 1992, p. 1605.
- [15] LI, F.—ROBERTS, W. T.—BATE, P. S.: Acta Mater., 44, 1996, p. 217.
- [16] ZELIN, M. G.—BIELER, T. R.—MUKHERJEE, A. K.: Metall. Trans. A, 24A, 1993, p. 1208.
- [17] UREÑA, A.—GÓMEZ DE SALAZAR, J. M.—QIUÑONES, J.—MARTÍN, J. J.: Scripta Mater., 34, 1996, p. 617.
- [18] JANGG, G.—ZBIRAL, J.—ŠLESÁR, M.—BESTERCI, M.: Aluminium, 68, 1992, p. 238.

- [19] ŠLESÁR, M.—JANGG, G.—BESTERCI, M.—ĎURIŠIN, J.—OROLÍNOVÁ, M.: Kovove Mater., 27, 1989, p. 476. [20] BESTERCI, M.—ŠLESÁR, M.—JANGG, G.: Powder Metall. Int., 24, 1992, p. 27. [21] PEŠEK, L.—BESTERCI, M.—ZUBKO, P.: Kovove Mater., 42, 2004, p. 415.

- [22] BESTERCI, M.—VELGOSOVÁ, O.—KOVÁČ, L.: High Temperature Materials and Processes, 21, 2002, p. 79.
- [23] POIRIER, J. P.: Plasticité a haute température des solides cristallins. Paris, Eyrolles 1976.
- [24] ČADEK, J.: Creep kovových materiálů. Praha, Academia 1984.