Analysis of Al-12Al$_4$C$_3$ Composite

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The yield strength of Al-Al$_4$C$_3$ composite system with high volume fraction of Al$_4$C$_3$ phase is evaluated. The results are compared with the previously obtained ones for lower content of dispersion phase. The validity of the Fisher-Hart-Pry relation between the yield strength and microstructural parameters for Al-Al$_4$C$_3$ system with particle arrangement in rows is confirmed in the paper.

Keywords: composite, additive components of yields strength, microstructure.

INTRODUCTION

In our works [1 – 4], as well as in [5], direct and indirect contributions to strengthening by incoherent secondary phase particles were evaluated. The analysis was done also for the system Al-Al$_4$C$_3$ prepared by mechanical alloying with maximum Al$_4$C$_3$ content of up to 10 vol.%.

Mechanical alloying is a process used to prepare compact systems of powders with fine microstructures. Essentially, it is a high-energy dry milling of powders in intensive attriting during which deformation, repeated disintegration, and welding of powder matrix particles take place together with incorporation of secondary phase particles. This results in extremely fine microstructure usually containing very small inert particles of the secondary phase. The quality of the final product is determined by the starting parameters of the matrix particles and of the disperser, as well as by kinetics of the process of mechanical alloying [6].

The aim of the present work was to evaluate the yield strength of Al-Al$_4$C$_3$ with 12 vol% Al$_4$C$_3$ and to find out whether the previously identified strengthening mechanism is valid also for relatively high degree of matrix strengthening due to the secondary particles.

EXPERIMENTAL MATERIAL AND METHODS

The experimental material Al-Al$_4$C$_3$ with 12 vol% Al$_4$C$_3$ was prepared by mechanical alloying at the Technical University in Vienna. The materials used for the preparation were aluminium powder with the grain size < 100 μm and 3 wt% of graphite with mean particle size 35 nm which, after the carbide transformation, created 12 % volume fraction of Al$_4$C$_3$ particles. The mixture was dry milled in attritor Netsch at 900 rpm for 90 minutes. The granulate was compacted using pressure of 600 MPa and heat treated at 550 °C / 3 h in order to transform C to Al$_4$C$_3$. The obtained material was then hot extruded at 550 °C with cross-section reduction of 94 %. The shape of

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experimental specimen with 3 mm diameter and 40 mm length (length of the measured part was 15 mm) is illustrated in Fig. l. The specimens were tested in static tensile strength test using testing machine Tiratest 2300 at a deformation rate $\varepsilon = 10^{-1}\text{s}^{-1}$.
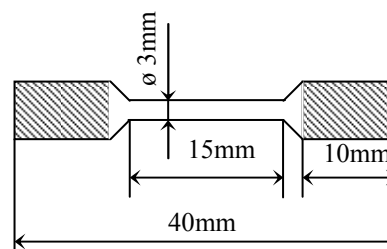

Fig. 1. Shape and dimensions of a specimen

RESULTS AND DISCUSSION

Microstructure of the Al-Al$_4$C$_3$ with 12 vol% Al$_4$C$_3$ was heterogeneous and consisted of fine-grained matrix with Al$_4$C$_3$ particles distributed in parallel rows aligned along the extrusion direction.

Substructure analysis of thin foils by transmission electron microscopy showed (Fig. 2) that the typical size of subgrains was 360 nm – 390 nm with average of $d_{sg} = 380$ nm. The secondary phase particles had needle-like shape and their average size (after approximation to sphere) was 33 nm. They were distributed relatively uniformly both on the boundaries and inside the subgrains.

Due to the low density and small size of the Al$_4$C$_3$ particles it was not possible to obtain their diffraction patterns. However, we obtained diffractograph of a large spherical particle of Al$_2$O$_3$ (Fig. 3) which proves that besides the intentionally created Al$_4$C$_3$ phase some oxide particles were present, too. This is in agreement with [7] where, in materials Al-Al$_4$C$_3$ with 1 vol% – 10 vol% Al$_4$C$_3$, chemical analysis of oxygen showed presence of Al$_2$O$_3$ phase. In those cases they were remnants of oxide shells of the original matrix powder grains disintegrated during milling or the shells formed during the milling process. The authors of [7] assumed that the fraction of Al$_2$O$_3$ phase was 2 vol% – 3 vol%. In work [8] a great

variety of the grain sizes in Al-Al$_4$C$_3$ system were found. In thin foil only particles of about 200 nm sizes were identified. These particles represented approximately 70 % of the secondary phase. We assume that, because of the same composition and kinetics of the process, the distribution in our system will be similar and that the remaining 30 % are large Al$_4$C$_3$ and Al$_2$O$_3$ particles.
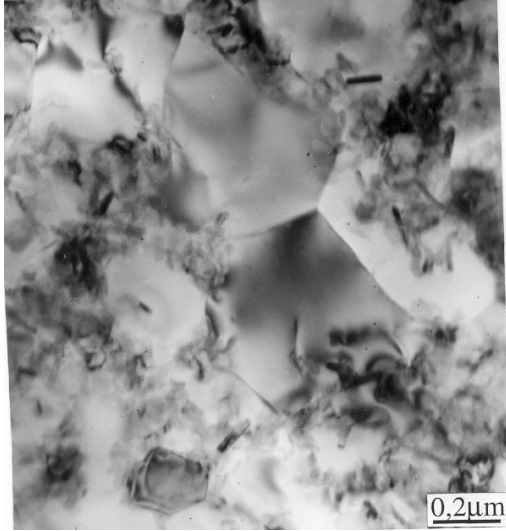

Fig. 2. Substructure of the Al-Al$_4$C$_3$ material
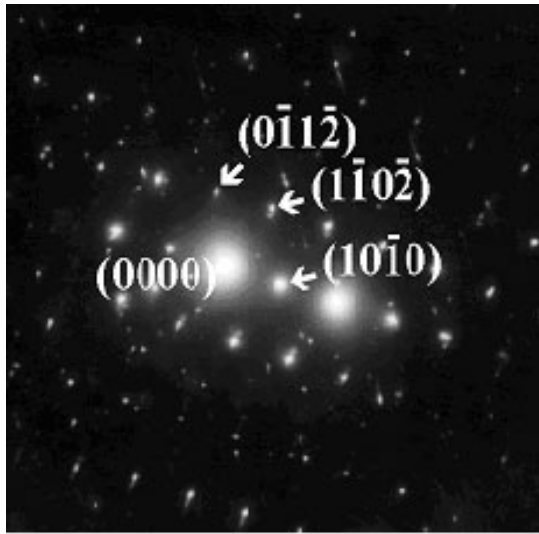

Fig. 3. Diffraction pattern from a thin foil of Al-Al$_4$C$_3$ material – [02$\bar{2}\bar{1}$] α-Al$_2$O$_3$

Among the basic parameters necessary for analysis of the yield strength of dispersion strengthened materials belong: particles size, grain and/or sub grain size, volume fraction of secondary phase, and average particle distance. Other parameters are: modulus of elasticity in shear G, Burgers vector b, and dislocation density ρ.

Using the static tensile test we found that at deformation rate 10^{-1} s^{-1} and 20 °C the average yield strength was 487 MPa. By simplification of contributions to dispersion strengthening various relationships had been formulated. Assuming their additive character the yield strength can be expressed by equation

$$R_{p0.2} = R_{PN} + R_s + R_{DD} + R_{LC} + R_p , \qquad (1)$$

Each contribution can be calculated from measured values and physical quantities, or the minor ones can be estimated.

The contributions R_{PN} (Peierls-Nabarro strengthening) and R_s (substitution strengthening) are relatively small. According to work [4] we estimate their values as $R_{PN} = 10$ MPa and $R_s = 5$ MPa.

The R_{LC} (sub grain strengthening) can be calculated from the size of features found in thin foils using relation of Langford-Cohen [9]:

$$R_{LC} = k_{LC} d_s^{-1} , \qquad (2)$$

here $k_{LC} = 6Gb = 0.0454$ N mm^{-1}, where $G = 27 \cdot 10^3$ N mm^{-2}, $b = 2.8 \cdot 10^{-7}$ mm, and $d_s = 0.38$. This leads to $R_{LC} = 120$ MPa.

The direct contribution of strengthening by particles R_P for materials with line-like microstructure was determined using Fisher-Hart-Pry relation [10]:

$$R_P = kf^{3/2}/r , \qquad (3)$$

where k is a constant, value of which for Al was determined to be approximately 115 MPa [7]. Its analytical calculation from $k \sim 3NGb$ is complicated because finding the number of dislocation loops N inside particles in technical systems is practically impossible. The f is the disperser volume fraction, in this case 12 vol %, r is the mean disperser particle radius, in this case 16.5 nm. The resulting value was $R_P = 289$ MPa.

In the work [7] the authors determined the dependence of yield strength on parameter $kf^{3/2}r^{-1}$ for material Al-Al$_4$C$_3$ with 1 vol % – 10 vol % of secondary phase and mean particle diameter $d = 10$ nm to 35 nm. The particle size increased with increase of the disperser volume fraction. This dependence, measured for various volume fractions of the secondary phase using theory of Fisher-Hart-Pry, is plotted in Fig. 4 (dashed lines).

Extrapolating to 12 vol% Al$_4$C$_3$ we can estimate the value of R$_{p0.2}$. For volume fraction of the disperser $f = 12$ vol % and mean particle size $d = 33$ nm we got value of $f^{3/2}/r^{-1} = 2.5$ µm^{-1}. The extrapolated value obtained from the plot is $R_{p0.2} = 472$ MPa which corresponds well to the measured value of 487 MPa (denoted in Fig.4 as ×). The lines AB, BC, CD, DE correspond to particular strengthening contributions as they are denoted in the plot legend.

The contribution from the deformation induced dislocations R_{DD} ought to be relatively high. However, since it was difficult to prepare a thin foil and to find a region with dislocation contrast at such a high volume fraction of Al$_4$C$_3$, it was not possible to calculate R_{DD} because the dislocation density ρ was unknown.

Assuming that R_{DD} estimated from the graph will not be very different from the real value it can be assumed that $R_{DD} = 53$ MPa. Using this value the dislocation density was determined from equation

$$R_{DD} = 2\alpha Gb\rho^{1/2} , \qquad (4)$$

where α is the strength coefficient of the dislocation network $\alpha = 0.3$, G is the shear modulus, b is the Burgers vector, and ρ is the dislocation density. This gives the dislocation density $\rho = 1.36 \cdot 10^{10}$ cm^{-2}.
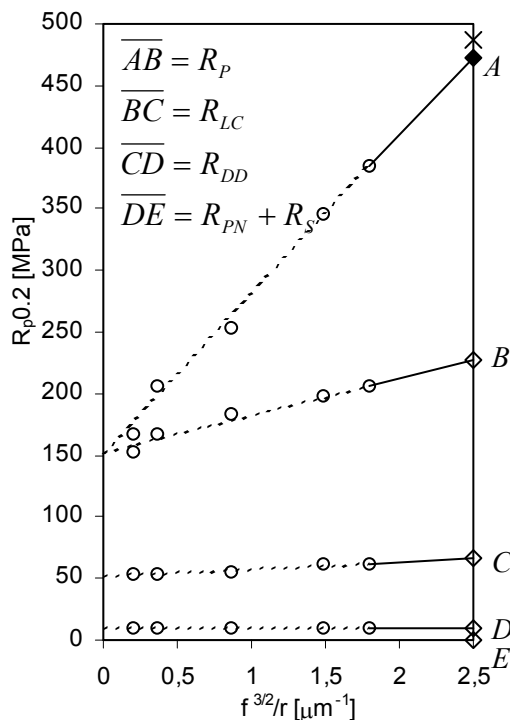
Fig. 4. Plot of yield strength vs. microstructural parameters $f^{3/2}/r$ ($t = 20\,°C$ and $\acute{\varepsilon} = 10^{-1}\,s^{-1}$): × – experimentally obtained $R_p0.2$ for Al-Al$_4$C$_3$, ♦ – extrapolated value of $R_{p0.2}$ for Al-12Al$_4$C$_3$, ◊ – extrapolated values of hardening contributions for Al-12Al$_4$C$_3$, ○ – values of hardening contributions for Al-12Al$_4$C$_3$, with 1 vol% – 10 vol% Al$_4$C$_3$ [6].

Summing up all the calculated strengthening contributions according to the Eq (1) we obtain the yield strength $R_{p0.2} = 477$ MPa. This is in a good agreement with the measured value $R_{p0.2} = 487$ MPa as well as with the value $R_{p0.2} = 472$ MPa estimated by graphical extrapolation from the graph.

From the results it follows that the yield strength value determined with help of measured microstructural parameters was in a good agreement with the value $R_{p0.2}$ calculated from the Fisher-Hart-Pry equation at the volume fractions of Al$_4$C$_3$ up to 10 vol% as well as with the measured value. This suggests that the strengthening mechanism did not change with increasing volume fraction of the Al$_4$C$_3$ phase and the incoherent Al$_4$C$_3$ particles, besides the direct interaction with dislocations, influenced also the substructure formation (parameters d_{sg}, ρ), increasing thus the strengthening of the material. Using linear regression the Fisher-Hart-Pry equation was modified for the whole range of volume fraction (1 % – 12 %) as follows

$$R_{p0.2} = 153 + 129\,(f^{3/2}/r)\,. \tag{5}$$

CONCLUSIONS

1. It has been confirmed that for the yield strength of the Al-Al$_4$C$_3$ system with 12 vol% of Al$_4$C$_3$ at 20 °C the modified Fisher-Hart-Pry equation (5) is valid.

2. The additive strengthening contributions (R_{PN}, R_S, R_{DD}, R_{LC} R_P) agreed with linear tendency of their dependence on the microstructural parameters and modify its overall final value.

Acknowledgments

REFERENCES

1. **Besterci, M.** Structure Analysis of Dispersion Strenthening *Scripta Materialia* 30 1994: pp. 1145 – 1149.

2. **Besterci, M.** Kompozity Cu-Al$_2$O$_3$ pripravené práškovou metalurgiou *Pokroky práškovej metalurgie* 37 1999: pp. 5 – 56 (in Slovakian).

3. **Besterci, M.** Štruktúrna analýza disperzného spevňovania *Kovové Mater* 32 1994: pp. 85 – 89 (in Slovakian).

4. **Šlesár, M., Jangg, G., Besterci, M., Ďurišin, J., Miškovičová, M.** Mechanické vlastnosti disperzne spevnených materiálov typu Al-Al$_4$C$_3$ *Kovové Mater* 20 1982: pp. 328 – 332 (in Slovakian).

5. **Hazzledine, M.** Analysis of Dispersion Strengthening *Scripta Materialia* 26 1992: pp. 57 – 60.

6. **Korb, G., Jangg, G., Kutner, F.** Mechanism der Dispersionenvestigten Al-Al$_4$C$_3$ Werkstoffen *Draht* 5 1 1979: pp. 318 – 324.

7. **Jangg, G., Kutner, F., Korb. G.** Dispersionsgehärteten Al-Al$_4$C$_3$ Werkstoffen *Aluminium* 51 1975: pp. 641 – 645.

8. **Besterci, M., Orolínová, M., Šlesár, M.** Stanovenie skutočného objemového podielu disperzných častíc materiálu Al-Al$_4$C$_3$ kvantitatívnym mikroskopickým rozborom *Kovové Mater* 3 1983: pp. 73 – 76 (in Slovakian).

9. **Langford, G., Cohen, M.** Subgrains Strengthening of Materials *Trans. ASM* 62 1969: pp. 623 – 635.

10. **Fisher, J. C, Hart, E., Pry, R. M.** Strengthening of Composites by Secondary Particles *Acta Metall* 1 1953: pp. 336 – 342.