Mechanical properties of the particles of the mechanically alloyed system Al-Al_4C_3

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Abstract

The contribution deals with estimation of mechanical properties of both particles and matrix in the Al-Al_4C_3 system. Using the depth sensing indentation technique, the instrumented hardness (H_{IT}), elastic modulus (E_{IT}) and indentation deformation work (W) were measured. The $FeAl_3$ particles were identified based on both EDX analysis and measured mechanical properties. Hardness and modulus of $FeAl_3$ particles are 10 and 2.5 times higher than the values of matrix.

K e y w o r d s: mechanical alloying, particle, nanoindentation, hardness, indentation modulus

1. Introduction

Dispersion hardened materials are characteristic by their heterogeneous microstructure. Oxides, nitrides, borides and carbides with the average size of 10–50 nm frequently represent the minority phase. This kind of composite material is mainly prepared via powder metallurgy. The powder reinforcing phase is incorporated into the system during some stage of the preparation or it is formed in-situ during the milling operation – this kind of preparation is also called mechanical alloying [1].

The depth sensing indentation technique (DSI) is the experimental method of hardness measurement based on the continual monitoring of the loading force in dependence on the indentation depth. The result of this measurement is an indentation curve. Nowadays it is possible to use the force of μN and less and to measure the depth in nm. Using the sharp indenter tip following parameters can be evaluated from the indentation curve: Martens hardness (HM), instrumented hardness (H_{IT}) which can be recalculated into the classical Vickers hardness (HV), indentation modulus (E_{IT}), which is comparable with Young's modulus (E), yield strength (R_e), indentation deformation work (W) and others [2].

The aim of this work is to evaluate mechanical properties of the particles and matrix in mechanically alloyed system Al-Al_4C_3 using the depth sensing indentation technique.

2. Material and experimental work

Experimental material was prepared via mechanical alloying. Al powder with average size < 50 μm and 1 wt.% of the graphite KS 2.5 were milled together in the attritor for 90 min. The formation of Al_4C_3 in amount of 4 wt.% is expected under this process condition. Then granulate was cold pressed into the rods using the pressure of 600 MPa. Next heat treatment (550 °C/3 h) causes the chemical reaction $4Al + 3C \rightarrow Al_4C_3$. Samples in rods were heat extruded (600 °C) on 94 % of the initial diameter [1].

Microstructural analysis of the system shows that the average size of the grain is 0.8 μm. Three size categories of particles are present in system distributed in rows (1. and 2.) or statistically (3):

1. Al_4C_3 < 0.03 μm
2. Al_4C_3, Al_2O_3, $FeAl_3$ < 1.5 μm
3. Al_4C_3, Al_2O_3, $FeAl_3$ > 1.5 μm [1].

The particles of the first category are shown in Fig. 1,

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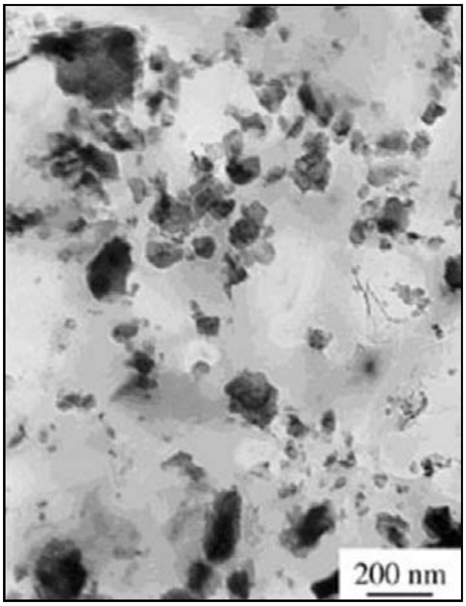

Fig. 1. The particles of the 1st size category of Al-A_4C_3.

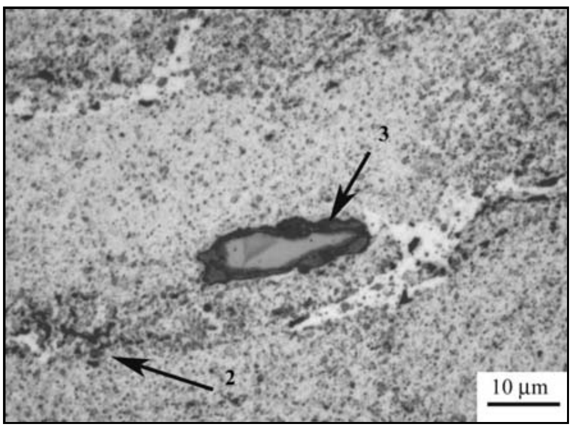

Fig. 2. The particles of the 2nd and 3rd size category distributed in rows and statistically, respectively.

particles of the second and third category are shown in Fig. 2.

Mechanical properties of the Al_2O_3 and Al_4C_3 particles were presented in [3] and [4], properties of the Fe-based particles will be presented in this work. Based on the results of works [1, 5], Fe in the Al-Al_4C_3 system is present because of contact with the agglomerate and the mill-walls, milling arms and balls of the mill. Fe with Al form brittle intermetallic compounds – particles of the second and third category. These particles are less effective in the reinforcing mechanism of the matrix because of their cleavage failure and they represent the stress concentrators [1].

Depth sensing indentation measurements were realized on Nano Indenter XP in West Bohemian University, Pilsen. Three-sided pyramidal diamond Berkovich indenter tip with radius less than 100 nm was used. Continuous Stiffness Measurement (CSM) mode was chosen for the experiment. In this mode the contact stiffness (S) (between indenter and specimen) during the entire test is continuously measured.

The principle is based on the superimposing of low load (p) with defined amplitude (p_0) on the tip of the indenter and measurement of the harmonic response with exciting frequency (ω) and phase angle (Φ). Following from both the measured stiffness and known tip geometry the reduced modulus (E_r) can be determined and then the indentation modulus E_IT can be calculated:

$$\frac{p_0}{\Delta h_0}\cos\phi = E_\mathrm{r}\frac{2\sqrt{A}}{\sqrt{\pi}} = S, \qquad (1)$$

$$\frac{1}{E_\mathrm{r}} = \frac{1-\nu_\mathrm{i}^2}{E_\mathrm{i}} + \frac{1-\nu^2}{E}, \qquad (2)$$

where E_i, ν_i are Young's modulus and Poisson's ratio of the tip material (1140 GPa and 0.07) and E and ν are Young's modulus and Poisson's ratio of the investigated material, respectively, A is the contact area.

Samples were prepared in the form of metallographic sections. Measurements of the particles and matrix properties were realized under defined conditions: CSM, maximal indentation depth 1 μm, exciting frequency 45 Hz, amplitude 2 nm, thermal drift $< 0.05\ \mathrm{nm\ s^{-1}}$, number of indents 10. In the case of the particles one indent was positioned into one particle. Particles with average size ≥ 2 μm were chosen for measurements.

3. Results and discussion

Indentation curves of the matrix of the system Al-Al_4C_3 are shown in Fig. 3. CSM method is controlled by indentation depth, therefore for the comparison with the measurements the scatter of the loading force must be regarded. This scatter is ±15 %. Al-Al_4C_3 is the material with heterogeneous microstructure. So the scatter can be explained by placing the indents in the areas with different content of the minority phase Al_4C_3. The same scatter was recorded for the values of the indentation hardness (H_IT) and for the indentation modulus (E_IT), Fig. 4. Minimal value of the standard deviation E_IT in the indentation depth < 0.2 μm corresponds with values for the pure Al. Average value of E_IT does not change with increasing indentation depth, $E_\mathrm{IT} = 77 \pm 3$ GPa. Average value of H_IT increases by 15 % with increasing indentation depth. The scatter of the hardness values is constant. Average value of H_IT and E_IT was evaluated in the maximum depth h_max, $H_\mathrm{IT} = 1.25 \pm 0.15$ GPa.

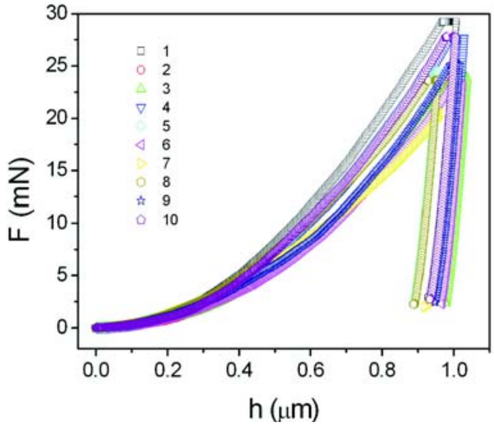

Fig. 3. Indentation curves, Al-Al_4C_3 matrix.

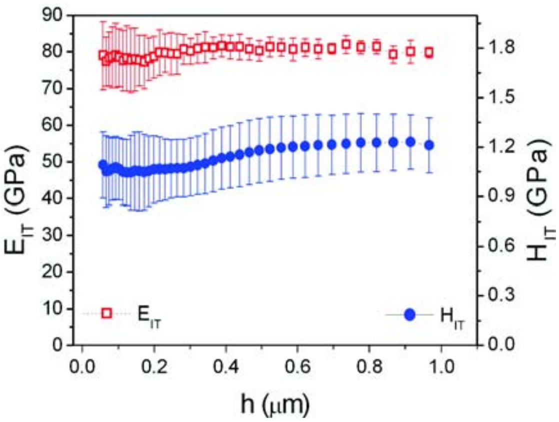

Fig. 4. Dependence of E_{IT} and H_{IT} on the indentation depth, Al-Al_4C_3 matrix.

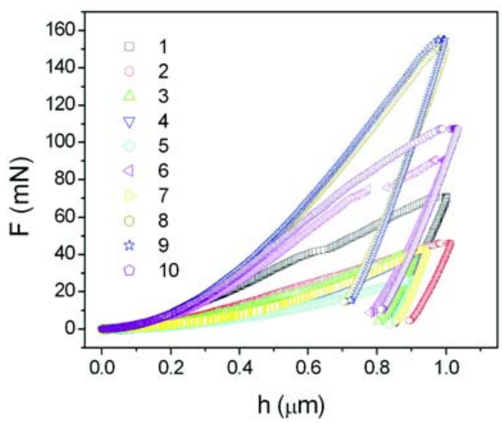

Fig. 5. Indentation curves, particles $FeAl_3$ embedded in Al-Al_4C_3 matrix.

The particles were identified on the basis of EDX analysis and the data from [5] were used. The average size of $FeAl_3$ particles is 5 μm. Area ratio of the particles is < 1 %. Indentation curves measured on these particles are in Fig. 5. The scatter of $F_{\max}$ is multiple compared with other measurements in the matrix. The quantitative comparison between particles and matrix should be possible in the case if all the particles have the same volume under the surface. But this volume is unknown thus the depth sensing indentation measurements such as EDX analysis are influenced by the matrix properties. Figure 6 shows the dependence of H_{IT} and E_{IT} on the indentation depth. Trend for both is the same, with increasing indentation depth values of both parameters decrease. In the work [4] the assumption that the properties of the particles respond to the maximal measured values was presented. Based on this assumption only the measurements with $H_{\mathrm{IT}} > 8$ GPa in the depth of 100–200 nm were taken into account. Thus the values evaluated in this interval are $H_{\mathrm{IT}} = 12 \pm 2$ GPa and $E_{\mathrm{IT}} = 220 \pm 30$ GPa, recalculated HV $= 1110 \pm 185$. Following the work [6] and the ratio $E/H < 40$ yield strength $R_{\mathrm{eIT}} = 5.8$ GPa for the particles was calculated. Measured values are in good accordance with the properties of $FeAl_3$ phases incipient during the welding process of Fe and Al [7].

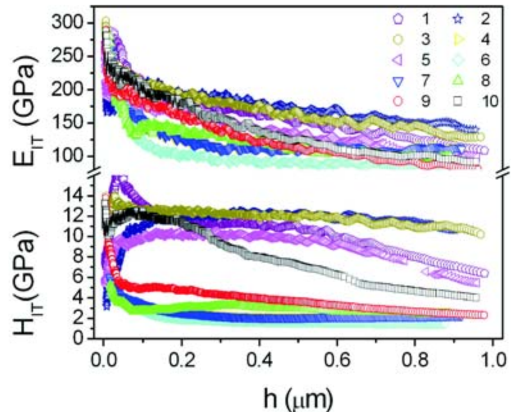

Fig. 6. Dependence of E_{IT} and H_{IT} on indentation depth, $FeAl_3$ particles embedded in Al-Al_4C_3 matrix.

The area under the loading segment of the indentation curve represents total indentation deformation energy (W). The area under the unloading segment of the indentation curve represents elastic indentation work (W_{e}). Difference between total and elastic energy represents plastic indentation energy (W_{p}). Ratio W_{p}/W characterizes the ability of the material for the plastic deformation. The Al-Al_4C_3 matrix absorbs 90 % of total indentation energy. Total work evaluated from the indentation curves for the particles includes apart from particles properties also the ratio of the work absorbed for the damage of these particles (pop-in) and also the influence of the matrix. For the evaluation of the deformation ability of the particles it is necessary to use the dependence between H/E_{r}

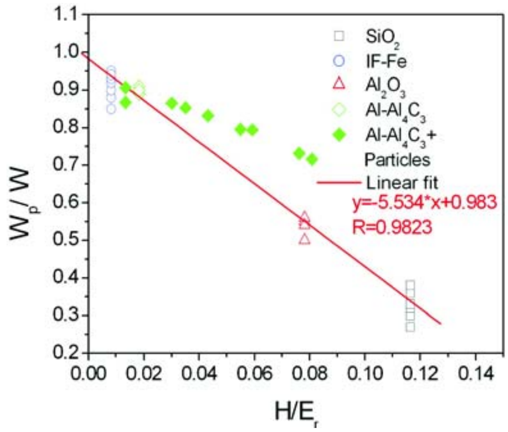

Fig. 7. Dependence of W_p/W on H/E_r represents plastic ability of materials.

and W/W_p for different kind of bulk materials measured with Berkovich tip (Fig. 7). The relation: $W/W_\mathrm{p} = 1 - 5.5H/E_\mathrm{r}$ is known from [8]. Indentation curves used for the evaluation of the average values of H_IT and E_IT were also used for the deformation work determination. The occurrence of the first discontinuities on these curves is observed for values ≥ 10 mN. Thus the total deformation work was determined from indentation curves for this load. Particles absorb only 63 % of total deformation work as plastic deformation.

4. Conclusions

In the present study we demonstrated the following:

– Depth sensing indentation technique is a suitable method for determination of mechanical properties of $FeAl_3$ particles in the Al-Al_4C_3 system. Mechanical properties of the composite matrix were experimentally evaluated using this method: $H_\mathrm{IT} = 1.25 \pm 0.15$ GPa, $E_\mathrm{IT} = 77 \pm 3$ GPa.

– Hardness of $FeAl_3$ particles is ten times higher than the hardness of the whole Al-Al_4C_3. Measured values of hardness and modulus are in good accordance with the literature data.

– During the loading with sharp Berkovich tip the matrix absorbs 90 % of total deformation work in the form of the plastic deformation, the particles absorb only 63 %.

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References

[1] BESTERCI, M.: Progress in powder metallurgy: Dispersal aluminium prepared by mechanical alloying 2. Šumperk, VÚPM 1996.
[2] FISCHER-CRIPPS, A. C.: Nanoindentation. New York, Springer 2002.
[3] BESTERCI, M.—VELGOSOVÁ, O.—IVAN, J.: In: 16th Inter. Conf. on Composite Mat. Ed.: Ishikawa, T. Kyoto, Japan Soc. of Composite Mat. 2007, p. 184.
[4] BESTERCI, M.—PEŠEK, L.—ZUBKO, P.—HVIZDOŠ, P.: Materials Letters, *59*, 2005, p. 1971.
[5] SCHALUNOV, J.—ŠLESÁR, M.—BESTERCI, M.—OPPENHEIM, H. —JANGG, G.: Metall, *40*, 1986, p. 601.
[6] MENČÍK, J.: In: Proc. Local Mechanical Properties. Ed.: Pešek, L. Košice, TU v Košiciach 2005, CD-ROM.
[7] POTESSER, M.—SCHOEBERL, T.—ANTREKOWITSCH, H.—BRUCKNER, J.: In: Proc. of 135th TMS Annual Meeting & Exhibition. Ed.: Howard, S. M. San Antonio, USA, TMS 2006, p. 167.
[8] MALZBENDER, J.: J. Mater. Res., *20*, 2005, p. 1090.