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Characteristics of an Al-V_2O_5 composite produced by powder metallurgy

Charakterystyka kompozytu Al-V_2O_5 wytworzonego metodą metalurgii proszków

Abstract
The paper presents the results of research on the Al-V_2O_5 composite manufactured by means of the mechanical alloying and powder metallurgy method. Observation of the microstructure and X-ray chemical composition (XRD) analysis of the composite after extrusion were carried out. One of the most desirable features of metallic composites – apart from mechanical properties – is their resistance to increased impact temperature. It was observed that the formation of intermetallic phases due to the chemical reaction between-strengthening particles and the composite matrix depends primarily on the annealing conditions of the material. The hardness of the annealed samples, depending on the annealing temperature, were discussed with respect to the material microstructure transformation. Despite the high-temperature, annealing at 473–773K, the material hardness did not change significantly, and remained approx.125 HV. However, at higher annealing temperatures, i.e. 823K, the chemical reaction between the components leads to the formation of the $Al_{10}V$ particles and increases hardness.

Keywords: Al-V_2O_5 composite, powder metallurgy, mechanical alloying , plastic consolidation

Streszczenie
W pracy przedstawiono wyniki badań kompozytu Al-V_2O_5, który wytworzono metodą mechanicznej syntezy. Przeprowadzono obserwację mikrostruktury oraz rentgenowską analizę składu chemicznego (XRD) kompozytu po procesie wyciskania. Jedną z najbardziej pożądanych cech kompozytów metalicznych – oprócz własności mechanicznych – jest ich odporność na oddziaływanie podwyższonej temperatury. Powstawanie faz międzymetalicznych na skutek reakcji chemicznej pomiędzy cząstkami umacniającymi a osnową kompozytu zależy przede wszystkim od warunków wyżarzania materiału kompozytowego. W pracy przedstawiono zależność twardości od temperatury wyżarzania (473–823 K). Twardość materiału nie zmienia się znacząco w dość wysokiej temperaturze wyżarzania jak w przypadku aluminium (473–773 K), a jej wartość osiąga średni poziom 125HV2. Stwierdzono, że reakcja chemiczna między składnikami podczas wyżarzania w temperaturze 823 K prowadzi do powstania fazy międzymetalicznej Al10V, co powoduje wzrost twardości kompozytu.

Słowa kluczowe: kompozyt Al-V_2O_5, metalurgia proszków, mechaniczna synteza, konsolidacja plastyczna

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1. Introduction

Composites have been used and processed by humans for many hundreds of years and the development of civilization was largely dependent on the development of numerous production technologies. The goal of producing metallic composite materials is to obtain a high strength of the product, which often has to be maintained while maintaining the high resistance of the structure and mechanical properties at an elevated temperature. One of the advantages of mechanical alloying is the possibility to combine components whose synthesis is not possible in a typical metallurgical process because of their high reactivity in the liquid or semi-liquid state. Particular attention is paid to light metal-based composites characterized by high strength properties which are practically unaffected during the product service at high temperatures [1–9].

Numerous works have been done on testing the temperature stability of metallic composites. For example, it was found that chemical reaction between the matrix and reinforcement of Al-CuO composite begins during extrusion at 673K and leads to the growth of equilibrium phase θ (Al_2Cu) particles [4]. For an Al-CeO_2 composite, even prolonged annealing at 773K does not affect the morphology of the strengthening particles. For an Al-Nb_2O_5 composite, the equilibrium phase Al_3Nb is formed practically only after annealing at 873K [2, 4].

It is worth noting that a relatively high stability of the composites structural properties can also be obtained by the plastic consolidation of some pre-compressed metallic powders, such as an aluminum-based composite reinforced with a nickel or silver powder addition [10, 11]. During annealing of the Al-Ni composite at 748K, the Al_3Ni intermetallic phase is formed (this phase is not observed after extrusion of Al and Ni powders at 673K). The composite hardness is stable despite the long annealing period (approx. 46HV2) and comparable to the composite hardness immediately after extrusion. An increase in annealing temperature to 823K reduces hardness by approx. 50% [10]. Al_3Ni particle growth usually leads to the increased porosity of the material. The use of the powder pressing method without preliminarily milling of components extends the diffusion process between the components of the composite with respect to composites made by the mechanical alloying method. In the case of refined components in mechanically alloyed composites, the chemical reaction is relatively faster and pores become smaller or practically do not appear in the annealed composite. However, in some composites obtained in the process of the plastic consolidation of metal powders, the intense diffusion of components may occur, leading to the formation of large grains in the intermetallic phase. For comparison, during extrusion of the Ag-Al composite, secondary solutions based on the Ag_2Al intermetallic phase and on the Ag_3Al intermetallic phase are formed. It was reported that intermetallic phase particles effectively strengthened the composite (R_m = 490 MPa, $R_{0,2}$ = 440 MPa, HV2 = 135). Annealing at 673K caused an increase in hardness as a result of diffusion controlled growth of an intermetallic layer between the particles of the composite components [11].

2. Experiments

The Al-V_2O_5 composite was made of a mixture of aluminum powder with an average size of 10 μm and a purity of 99.9%, and an addition of 10% by weight V_2O_5 powder. The powder mixture (approx. 700 g) was ground in an Attritor mill for 30 hours. Alloyed steel balls with a diameter of 9.5 mm were used for the milling process that was carried out at a mixer speed of 120 rpm. A protective argon atmosphere was used to prevent the oxidation of the powders and to prevent the mixture from igniting during milling. The addition of 5 wt.% methanol was used to reduce the formation of agglomerates, which could be deposited on the balls, mixer arms and mill walls. The milled powders were cold pressed under 500 MPa at room temperature and then, after degassing in a vacuum for 1 hour, were hot pressed at 673K under 100 MPa. The last stage of the manufacturing process was extrusion at a temperature of 673K. A composite rod with a diameter of 7 mm was obtained. Composite samples were subjected to Vickers hardness tests according to PN EN ISO 6507-1 standards over a load of 19.61N (HV2). A set of samples prepared from the extruded rod was annealed in a wide temperature range (473–823K) and then the Vickers hardness of the annealed samples was measured.

XRD analysis for extruded and annealed samples was performed using a Rigaku MiniFlex II apparatus. Diffraction reflections were obtained in the 2θ angle range from 20° to 70°. The diffractometer was equipped with a Cu lamp which generated characteristic Kα radiation of 1.5418 Å. Selected microstructures of the samples were observed using an JEM 2010 analytical electron microscope with an accelerating voltage of 200 kV. Thin foils for the microstructure observation were made by means of ion thinning on a GATAN PIPS-691 thinner. A Hitachi SU-70 scanning electron microscope was also used to observe the material microstructure.

3. Results and discussion

Results of the microstructure observations confirm that the process of mechanical alloying, combined with extrusion at elevated temperature, allowed a composite to be obtained with good consistency and a very high degree of fragmentation of the microstructure components. The material is characterized by a lack of porosity, which indicates good plastic consolidation of powders during the extrusion process. These particles are characterized by diverse morphology, with their size changing from a very few nanometers up to 300 nm (Fig. 1).

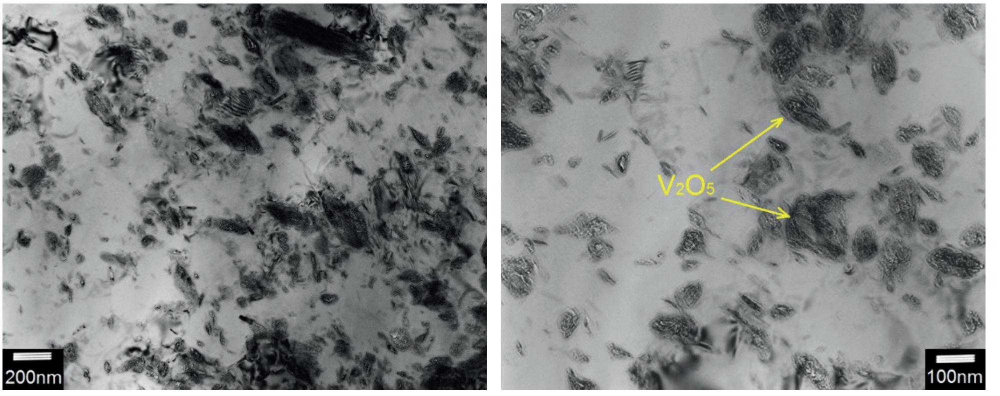


Fig. 1. TEM microstructure of as-extruded Al-V_2O_5 composite

X-ray analysis of as extruded composite suggests (Fig. 2), that V_2O_5 particles were not reduced during extrusion at 673K. The remaining V_2O_5 oxides were identified in both the extruded and annealed samples, which confirms the high stability of the composite microstructure. Similar research results can be found in the literature. In experiments on the Al-CeO_2 [6] composite (a material also made by mechanical synthesis), the presence of CeO_2 and Ce_4O_7 oxides was identified after the extrusion process. However, in most cases, the strengthening particles are reduced (totally or partially) within Al matrix at the extrusion process and the development of intermetallic phase was detected already at the extrusion stage. Such results can be observed for example in the Al-AgO composite [12]. The Ag_2Al intermetallic phase was formed during hot extrusion (673K), and the authors of the work were unable to find AgO particles that suggests the complete reduction of AgO particles in the Al-matrix occurs during the manufacturing process.

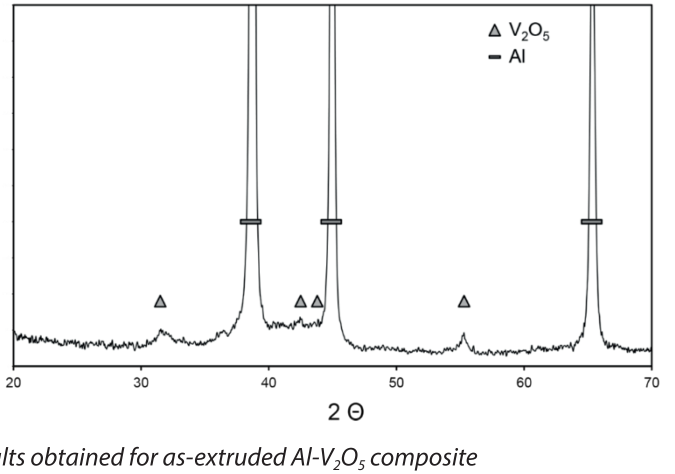
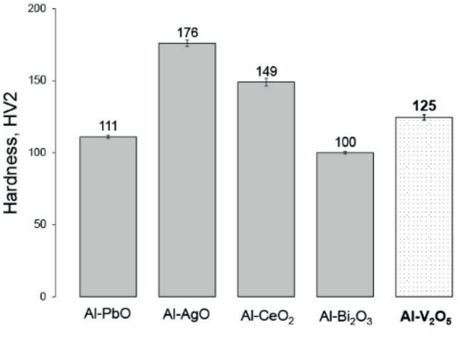


Fig. 2. XRD results obtained for as-extruded Al-V_2O_5 composite

The hardness of the extruded Al-V_2O_5 sample is 125 HV. The hardness value for the Al-V_2O_5 and some other composites reinforced with different oxides under similar manufacturing conditions were compared in Figure 3a. The composite reinforced with Bi_2O_3 oxide has a hardness lower by 25 HV than Al-V_2O_5, while the Al-AgO composite has a hardness higher by over 50 HV than the tested material.

The stability of the mechanical properties resulted from the stability of the composite microstructure at high temperature and this is one of the most desirable features of composite materials. The formation of intermetallic phases due to the chemical reaction between strengthening particles and the matrix of the composite depends primarily on the annealing conditions of the material. The effect of temperature on the hardness of some composites annealed for 2 hours is shown in Figure 3b for comparison purposes. Annealing at 473–773K does not significantly change the hardness of the Al-V_2O_5 composite, however, a slight hardness increase was noticed when the annealing was carried out at 823K. Similar stability can be observed for Al-CeO_2 and Al-Ta_2O_5 composites [4, 6]. However, in the case of Al-PbO, Al-AgO and Al-SnO composites, annealing at a temperature higher than 673K leads to a significant reduction in hardness (Fig. 3b).

a)



b)

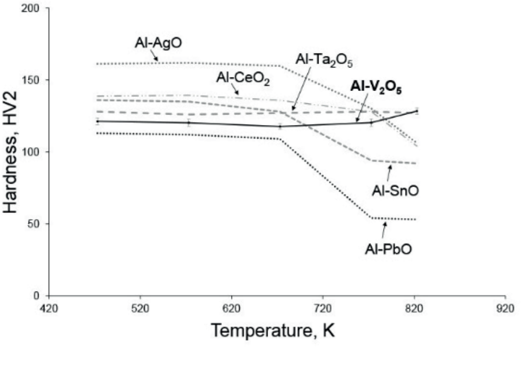


Fig. 3. Hardness (HV2) of some mechanically alloyed composites reported in literature: a) as-extruded; b) samples annealed at 473–823K for 2 hours [2–6]

The hardness increase for Al-V_2O_5 composite annealed at 823K can be ascribed to the formation of small $Al_{10}V$ particles, the presence of which is confirmed by structural analysis performed using scanning electron microscopy SEM (Fig. 4, Tab. 1) and transmission electron microscopy (Fig. 5). The formation of the $Al_{10}V$ intermetallic phase occurs as a result of a chemical reaction between the components of the composite as follows:

$$70Al + 3V_2O_5 > 6Al_{10}V + 5Al_2O_3$$

It is worth noting that observations of the Al-V_2O_5 composite microstructure after annealing at 823K for 168 hours did not reveal the presence of V_2O_5 oxide, which indicates that during the annealing practically all of the $3V_2O_5$ particles completely reacted with Al-matrix, which led to the formation of $Al_{10}V$ intermetallic phase precipitates and Al_2O_3 aluminum oxide particles in the surrounding Al-matrix (Fig.4).

a) b)

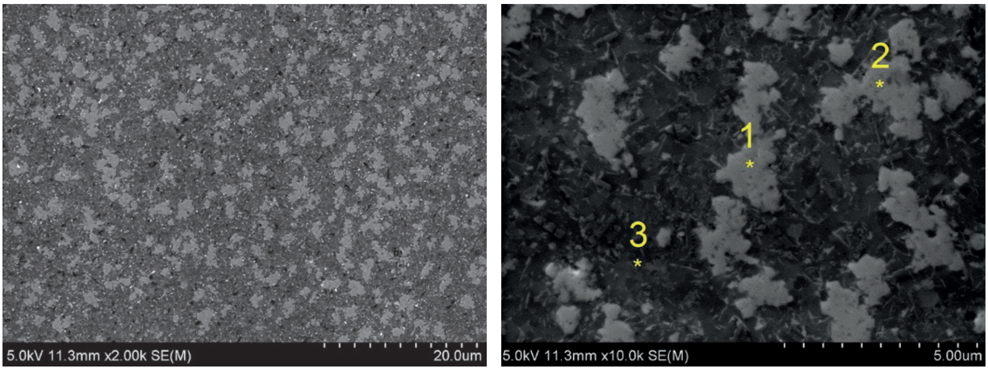
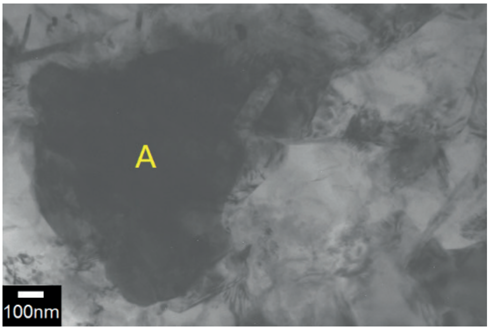


Fig. 4. SEM microstructure of Al-V_2O_5 composite annealed at 823K for 168 hours: a) magnification ×2000; b) magnification ×10000. EDX analysis results obtained for the grains marked 1, 2, 3 are shown in Table 1

Table 1. Results of X-ray chemical analysis (EDX) for selected grains marked 1, 2, 3 in Figure 4b

Point number	**O-K**	**Al-K**	**V-K**
		atom%	
Al-V_2O_5_pt1	10.21	81.19	8.60
Al-V_2O_5_pt2	10.21	81.43	8.35
Al-V_2O_5_pt3	8.09	91.36	0.55
		weight%	
Al-V_2O_5_pt1	5.85	78.46	15.69
Al-V_2O_5_pt2	5.87	78.86	15.27
Al-V_2O_5_pt3	4.94	94.00	1.06

a)



b)

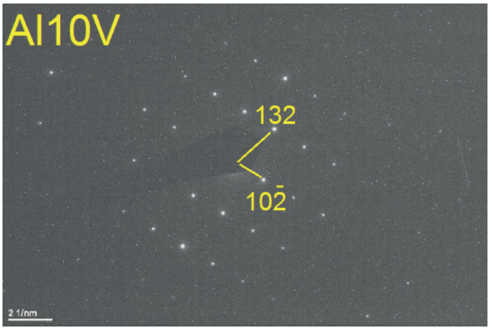


Fig. 5. TEM microstructure of Al-V_2O_5 composite annealed at 823K for 168 hours (a) and diffraction patterns for A particle marked A in TEM pictures, confirm $Al_{10}V$ microstructure (b)

4. Conclusions

Mechanical alloying and plastics consolidation of powders by hot extrusion is a convenient method of production for a Al-V_2O_5 composite with low porosity and a high refining microstructure.

Annealing in 473–823K does not significantly change the hardness of the Al-V_2O_5 composite. It has been found that the hardness of the material increases slightly during annealing at 823K. Microstructure observations and hardness measurements of Al-V_2O_5 samples revealed relatively good thermal stability of the examined composite up to a temperature of 823K.

The hardness increase for Al-V_2O_5 composite annealed at 823K can be ascribed to the formation of small Al10V particles, the presence of which is confirmed by structural analysis performed using scanning electron microscopy SEM.

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References

[1] Błaz L., Kaneko J., Sugamata M., Kamei R.: Structural features of mechanically alloyed Al-PbO and Al-PbO-WO_3 composites. Materials Science and Engineering, 2003, A349 (1–2), 111–119

[2] Błaż L., Kaneko J., Sugamata M., Sierpiński Z., Tumidajewicz M.: Structural aspects of annealing and hot deformation of Al-Nb_2O_5 mechanically alloyed composite. Materials Science and Technology, 2005, 6(21), 715–721

[3] Błaz L., Kaneko J., Sugamata M.: Microstructural evolution in mechanically alloyed Al-heavy-metal oxide composites. Materials Chemistry and Physics, 2003, 81, 387–389
[4] Kim D.G., Kaneko J., Sugamata M.: Structures and properties of mechanically alloyed aluminum-metal oxide powders and their P/M materials. Journal of Japan Institute of Metals,1993, 57(6), 679–685
[5] Seimiya K., Sugamata M., Błaż L., Kaneko J.: Structures and properties of P/M materials of Al-Mg-oxide (GeO2, SnO2, PbO) systems processed by mechanical alloying. Journal of the Japan Society of Powder and Powder Metallurgy, 2006, 53(12), 899–908
[6] Skrzekut T., Kula A., Blaz L., Wloch G., Sugamata M.: High-strength and thermally stable Al-CeO_2 composite produced by means of mechanical alloying. International Journal of Materials Research (formerly Zeitschrift für Metallkunde), 2014, 105 (3), 282–287
[7] Skrzekut T., Kula A., Blaz L., Wloch G., Sugamata M.: Structural characterization of mechanically alloyed AlMg-CeO2 composite. Key Engineering Materials, 2015, 641, 10–16
[8] Zygmunt-Kiper M., Blaż L., Sugamata M.: Effect of magnesium addition and rapid solidification procedure on structure and mechanical properties of Al-Co alloy. Archives of Metallurgy and Materials, 2013, 58(2), 399–406
[9] Kaneko J., Sugamata M., Blaż L., Kamei R.: Aluminium-low melting metal alloys prepared by mechanical alloying with addition of oxide. Key Engineering Materials, 2000, 188, 73–82
[10] Wloch G., Skrzekut T., Sobota J., Blaz L., Woźnicki A., Cisoń J.: The structure and mechanical properties of plastically consolidated Al-Ni alloy. Key Engineering Materials, 2016, 682, 245–251
[11] Wloch G., Skrzekut T., Sobota J., Woźnicki A., Blaż L.: Silver matrix composite reinforced by aluminium--silver intermetallic phases. Archives of Metallurgy and Materials, 2017, 62, 1, 259–266
[12] Skrzekut T., Blaz L.: Examination of aluminium matrix composites obtained by powder metalurgy and strengthened by AgO and CeO2 particles. In: METAL 2018: 27th International Conference on Metallurgy and Materials. Brno: TANGER, 2018, 1722–1728