EFFECT OF ANNEALING TEMPERATURE ON THE STRUCTURE OF MECHANICALLY ALLOYED Al-AgO COMPOSITE

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Abstract

Mechanical alloying (MA) of light metal powders with addition of other metal-oxides is a very promising method for production of high strength metallic composites. Of particular interest are the aluminum matrix composites reinforced by heavy metal oxides (*MeO*), which cannot be produced by means of common metallurgical processes due to the high reactivity of components. In this work, mechanical alloying and hot extrusion method was used to manufacture Al-based composite reinforced by 12.8 wt.% addition of AgO. Structure observations of as extruded Al-AgO composite revealed well-consolidated material having nanoscale structure. Annealing experiments were performed to test the material thermal stability and to analyze the effect of chemical reaction between components on the material structure. The annealing of as extruded samples at 773 K results in the reduction of the materials hardness from 178 HV to 120 HV for as-extruded material and sample annealed for 7 days, respectively. Received results are discussed on the basis of SEM and TEM observations, which have revealed specific structural processes responsible for the material hardness vs. annealing time reduction.

Keywords: Mechanical alloying, aluminum composite, powder metallurgy, powder's consolidation

1. INTRODUCTION

Manufacture of light metal-based composites is a competitive method for the production of high-strength materials with respect to common metallurgy technologies. The properties of composite materials such as: electrical conductivity, thermal expansion, mechanical properties, abrasion resistance, can be controlled by appropriate selection and refining of structural components resulting from used conditions of processing procedures. These materials are in wide demand of aviation and car industry [1]. Mechanical alloying (MA) gives the possibility of successful production of composites based on light-metal matrix such as aluminum, which are strengthened with fine metal-oxides particles. It is worth stressing that similar composites reinforced with heavy metal-oxide particles cannot be manufactured by means of a common metallurgy method because of high reactivity of their components. The light-metal matrix assures relatively low density and high strength of the product [2-8]. Technique of consolidation allows obtain solid material from different kinds of materials like powders, RS ribbons and metal chips [9-12]. Studies of structural and mechanical properties of mechanically alloyed Al-CeO_2 and AlMg-CeO_2 composites confirmed high strengthening effects and relatively high structural stability of the composite structure at 773K, in particular, for Al-CeO_2 composite [2, 3]. Al-CeO_2 characterized by thermal stability during annealing at 773K. Development of Ce_7O_{12} particles in as annealed samples was ascribed to the effect of chemical reaction in aluminum matrix. Material properties, which are related to the structural components morphology, depend on both heat treatment conditions and the composite system. Therefore, consecutive experiments, described below, were performed on mechanically alloyed Al-AgO composite.

2. EXPERIMENT

Experiments were performed on mechanically alloyed aluminum-matrix composite reinforced with addition of 12.8 wt.% silver oxide (AgO). The mixture of air-atomized powder of aluminum and high-purity AgO powders were milled using an Attritor ball mill with addition of methanol as the process control agent (PCA). The powders were milled under argon atmosphere in order to avoid the oxidation of powders and reduce the risk of self-induced explosive reactions. Mechanically alloyed powders were then consolidated by cold pressing and vacuum degassing, followed by the hot extrusion at ~ 673. As a result, rods of 7 mm in diameter were received. Microstructural analyses were performed using Hitachi SU-70 scanning electron microscope (SEM) and JEOL JEM2010 transmission electron microscopy (TEM) equipped with X-ray energy disperse analysis system (EDS). Samples for SEM were prepared by means of common metallographic technique. Thin foils for TEM were prepared using mechanical grinding and final ion-thinning with Gatan PIPS 691 machine. Hardness measurements were performed using Shimadzu hardness tester and an indenter load of 19,6N. Average hardness value was calculated for at least 10 measurement results. X-ray diffraction phase analysis (XRD) was carried out by means of Rigaku MiniFlex II diffractometer and Cu $K_{\alpha 1}$ radiation.

3. RESULTS AND DISCUSSION

SEM microstructure, of as extruded Al-AgO composite is shown in **Fig. 1a** to present a uniform distribution of reinforcements. Very-fined-grained material did not practically contain any voids or cracks.

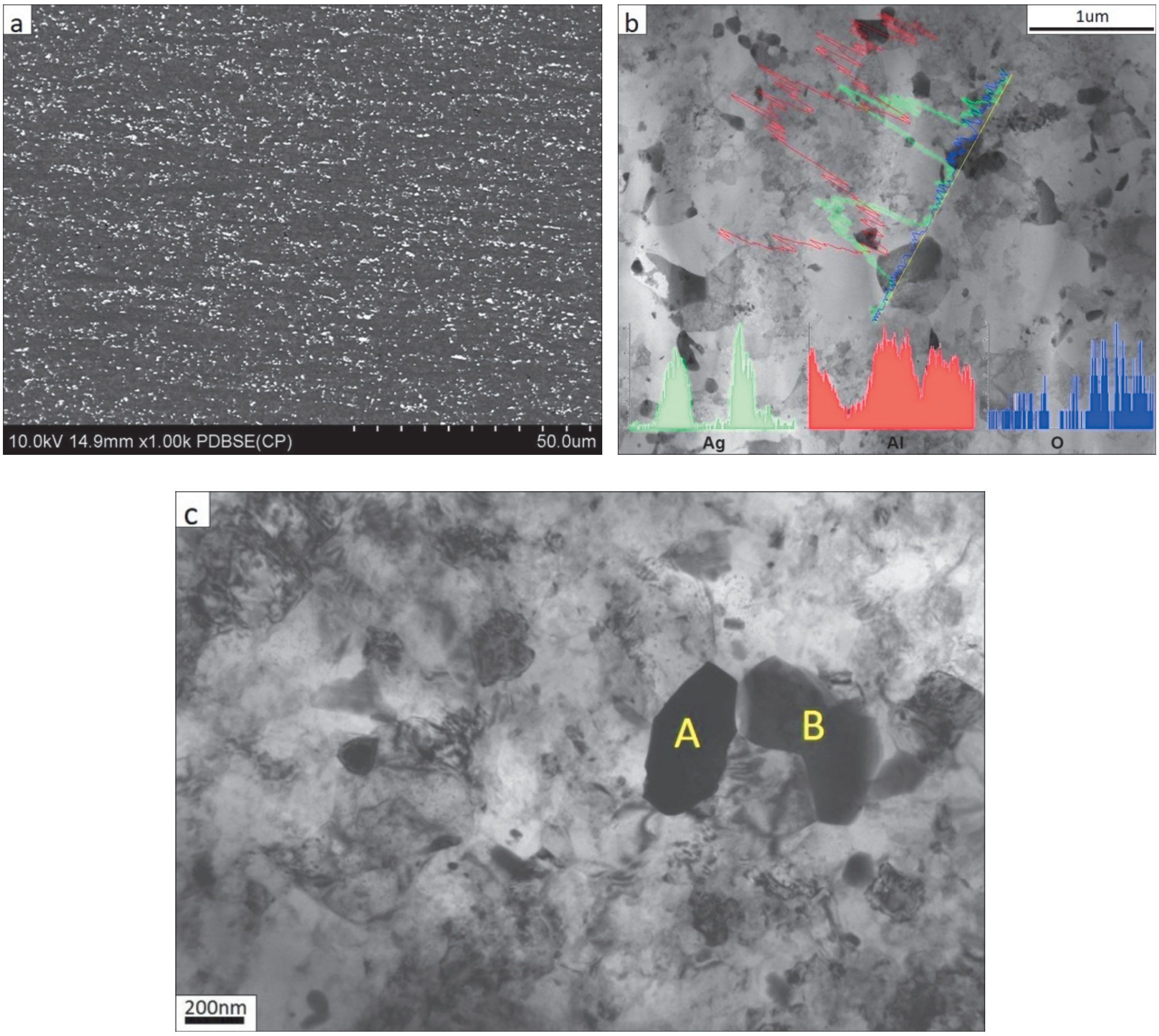

Fig. 1 Microstructure of as-extruded Al-AgO composite revealed by means of (a) SEM, (b) STEM and (c) TEM. EDS line analysis across the intermetallic particles is shown in (b). Results of EDS point analysis, performed at A and B, are shown in **Table 1**.

Table 1 Results of EDS analysis performed at particles marked A and B in **Fig. 1c**

Particle:	Al		Ag		O	
	wt.%	at.%	wt.%	at.%	wt.%	at.%
A	24.8	**56.9**	75.2	**43.1**	0	**0**
B	14.6	**40.7**	85.4	**59.3**	0	**0**

Low porosity of the composite resulted from well-performed mechanical consolidation of MA-powders at applied processing method. Preliminary identification of structural components in tested composite was performed by means of TEM/EDS analysis (**Fig. 1b** and **Fig. 1c**). The distribution of aluminum, silver and oxygen along the line marked on STEM image is shown **Fig. 1b**. Relatively dark particles revealed in **Fig. 1b** are enriched with silver and aluminum. More detailed analysis of such particles is based on EDS point analysis. The results of chemical analysis of the particles are shown in **Table 1**. Particles marked A and B in the **Fig. 1c** were found to be a binary intermetallic compounds that consist of aluminum and silver that suggest the aluminum-silver phase development. Further identification of structural components is based on X-ray phase analysis as shown in **Fig. 2**. The intensity peaks ascribed to Ag_2Al phase are marked in this **Fig. 2**.

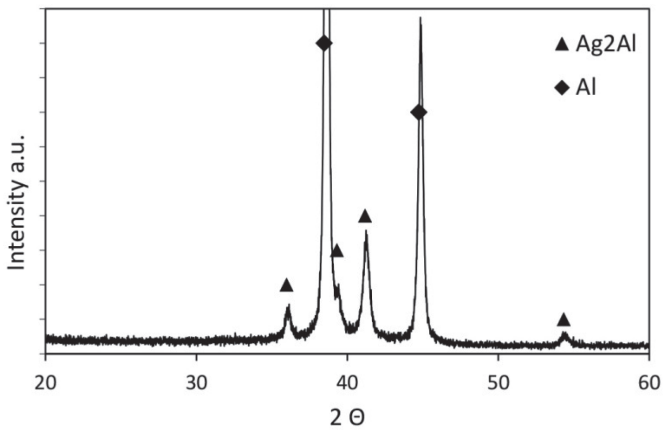

Fig. 2 X-ray diffraction analysis results for as extruded Al-AgO composite. Identified peaks of the intermetallic phase Ag_2Al-type are marked in the figure.

X-ray results revealed also typical peaks for aluminum matrix, however characteristic peaks for Ag-oxides were not detected (**Fig. 2**). Results described above lead to the conclusion that mechanical alloying and/or hot extrusion of the material at elevated temperature have induced chemical reaction between aluminum matrix and Ag-oxides. As a result, intermetallic Ag_2Al-type grains at the stage of the composite manufacturing are observed. Similar effects are reported for composites reinforced with addition of CuO or Fe_2O_3 oxides [13]. Thermal stability of MeO-reinforcements significantly depends on the standard free energy of the component formation (ΔG^o). Metal oxides such as CuO, Fe_2O_3 are characterized by low ΔG^o value, hence they are unstable in aluminum matrix. Formation of Me-aluminide as well as very-fine aluminum oxides due to solid state chemical reaction during and after mechanical alloying can also be detected.

In order to determine the effect of temperature on the microstructure and the composite hardness, annealing tests at 773 K were performed. The effect of annealing time on the material hardness is shown in **Fig. 3**. Initial hardness of as extruded composite is also marked in the figure for comparison. High hardness of the material results from fine-grained structure of the material. It is worth stressing that grain coarsening is practically retarded and very-fine structure of the sample annealed 7 days at 873 K is still retained (**Fig. 4**). A so, one may conclude that the hardness reduction during annealing can mostly depend on structural softening process such as recovery and local recrystallization of the matrix. Manufacturing conditions were favor for the development of chemical reaction between aluminum matrix and silver oxides. Therefore, intermetallic grains

Ag_2Al were grown. However, fine intermetallic grains are less than 1 µm in diameter in spite of prolonged annealing time. It is worth mentioning that similar annealing conditions for Al-V_2O_5 MA composite were found to result in enormous coarsening of needle like $Al_{10}V$ grains up to ~80 µm in length [7].

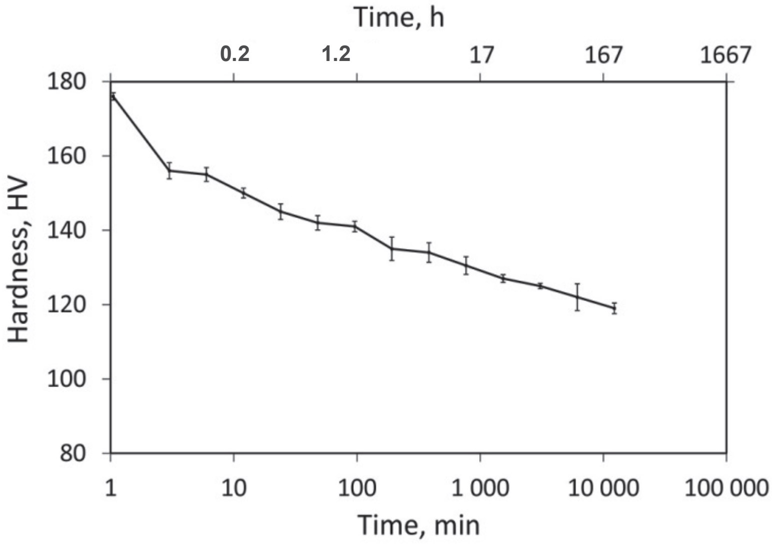

Fig. 3 Effect of annealing time on hardness of Al-AgO composite annealed at 773 K

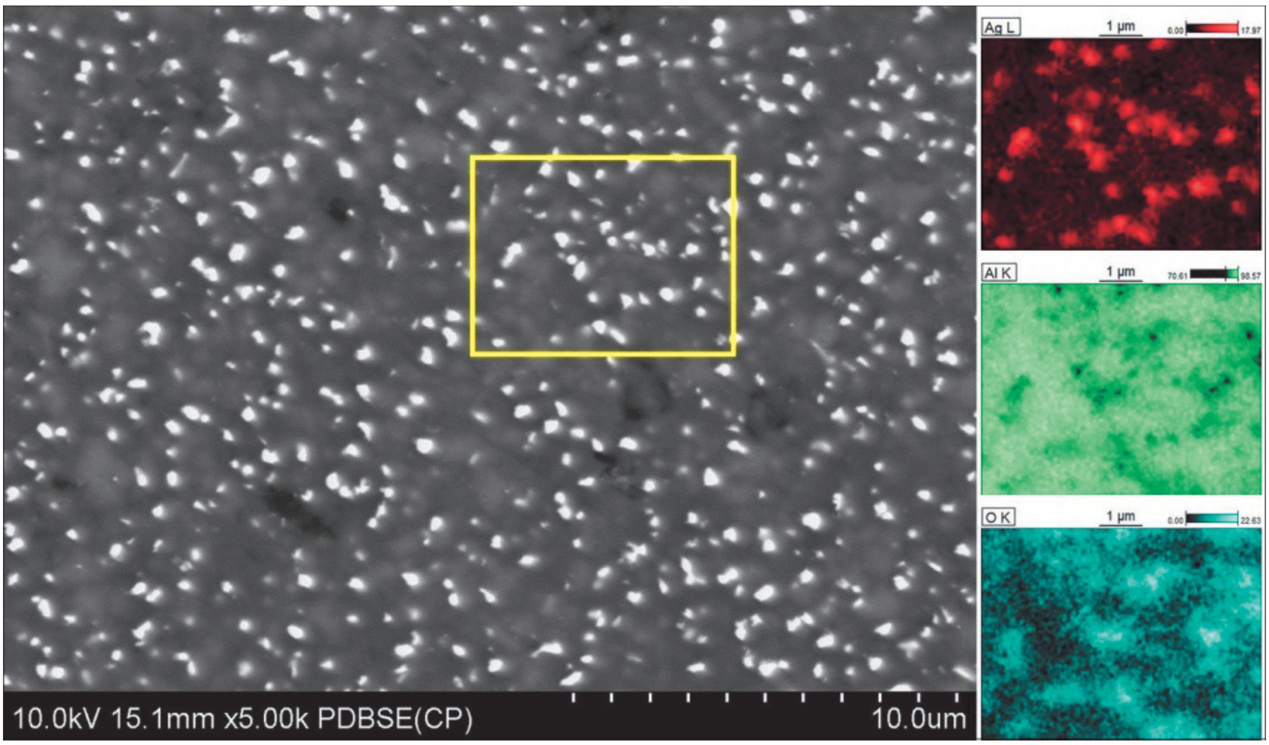

Fig. 4 SEM microstructure of Al-AgO sample annealed at 873 K for 7 days and element map distribution of silver, oxygen and aluminum

CONCLUSIONS

1) Hot extrusion of mechanically alloyed Al-AgO composite was found to be very convenient method for manufacturing of well-consolidated material. Very low porosity and highly refined structure resulted in high hardness of as extruded material (HV 178).
2) Chemical reaction between AgO particles and aluminum matrix that was initiated during manufacturing procedures was found to result in a formation of aluminide intermetallic phase. Therefore, vey-fine Ag_2Al particles were observed for both as extruded and annealed samples.
3) Material hardness was found to decrease during annealing at 773 K. In spite of prolonged annealing time coarsening of the intermetallic phase was limited. Therefore, the material softening was ascribed to recovery of Al-matrix rather than Ag_2Al particles coarsening.

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