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Characterization of Al matrix composites reinforced with alumina nanoparticles obtained by PM method

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Al-Al_2O_3 metal matrix composite powders with 2%vol.Al_2O_3 nanoparticles were synthesized by high-energy milling of the blended component powders and consolidated via traditional powder metallurgy (PM) route. The evaluation of the milling time from 2 h until 8 h on the microstructures, electrical and mechanical properties was analyzed. Mechanical properties like: hardness, strength and ductility were determined on the specimens with high densification obtained by hot extrusion. The optimum milling time which assured uniform distribution of nano-size alumina particles in the aluminum matrix and best values for the mechanical properties was emphasized.

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1. Introduction

Discontinuously reinforced aluminum matrix composites (DRA) have been attracting attention because of their amenability to undergo deformation processing by conventional metalworking techniques. Extrusion is used in processing of DRA composites for consolidation, redistribution of reinforcements, and shape forming.

Use of nanoparticles to reinforce metallic materials lead to the development of novel composites with unique mechanical and physical properties. In order to achieve desired mechanical properties of composites, reinforcing nanoparticles must be distributed uniformly within metal matrix of the composites.

One of the main challenges towards a homogeneous distribution of ceramic phase in the metal matrix is the selection of the appropriate processing technique.

Mechanical milling (MM) technique was employed in the synthesis of composite powders because of its simplicity, low cost operation and for dispersion nanoparticles more uniformly in metal matrix. MM involves repeated cold welding, fracturing and re-welding of powder particles. This is achieved by repeated collisions between the grinding medium in the milling container. A balance is achieved between the rate of welding that increase the average composite particle size and the rate of fracturing that decreases the average composite particle size [1]. This leads to a steady-state particle size distribution of the composite metal particles. The continuous interaction between the fracture and welding mechanisms tend to refine the grain structure resulting in uniformly distributed particles in the metal matrix.

Lately, literature [2, 3] revealed that size of the particles have a strong effect on the failure mode, strength, and ductility of the Al-based composites. The smallest particles (< 200 nm) usually well bonds to the matrix and do not initiate cavities in the particle. For optimum strength, the second-phase dispersion strengthened particles must be fine and the interparticular spacing small. Tensile strength and ductility decrease with increasing particle size. The mechanical properties [4] of metal-matrix composites can be further enhanced by decreasing not only the sizes of ceramic particulates but also the matrix grains from micro to nanometer level.

In the present work, the fabrication of nanometric particulates reinforced aluminum matrix composite via traditional powder metallurgy (PM) method was performed. The aim has been to study the effect of nanometric reinforcement of Al_2O_3 and the influence of the processing parameters on the microstructure, electrical and mechanical properties.

2. Experimental procedure

Pure aluminum Al_2O_3 and stearic acid powders were obtained from commercial vendors. Pure Al powder of 99.4% purity was obtained from Alba Aluminum Slatna, Romania and Al_2O_3 and stearic acid powders were provided from Alfa Aesar, Germany.

The used raw materials for the obtaining of the homogeneous powders mixtures from the Al-Al_2O_3 system are shown in Table 1.

The following technological flux was used in order to elaborate reinforced Al-2%vol.Al_2O_3 composites of 65 g each charge: powder mixing-homogenizing by MM (2 h, 4 h, 6 h, 8 h) - pressing-sintering-extrusion.

The MM process took place into a mill of Fritsch-Pulverisette 6 type, in the following conditions: balls/powders ratio (BPR) of 10:1, milling speed of 250 rot/min, Ar atmosphere. A combination of different sizes of balls (diameter of 19 mm, 14 mm, 10 mm and 5 mm), was used to obtain higher collision energies.The milling times were 2, 4, 6 and 8 h.

To avoid excessive cold welding of Al particles during milling, 1.5% gr. stearic acid powders have been used as a process-control agent (PCA) [5] The PCA gets adsorbed onto the surface of the powders and minimizes the effect of cold welding and thus inhibits agglomeration.

Table 1. Raw materials for the obtaining of the homogeneous powders mixtures from the Al- Al_2O_3 system.

Materials	Physical and chemical characteristics				Role
	Purity %	Particle size, µm	Density at 20^oC, g/cm^3	Melting Point, ^oC	
Al powders (tip AA 100)	94	< 71	2.7	660	Matrix
Stearic acid powders	98.0	20-45	0.9408	69-71	Milling control agent
Al_2O_3 powders- α phase	99.0	0.60	3.97	2045	Reinforcing element

The uniform distribution of alumina is one of the most important requirements for achieving excellent mechanical and physical properties of the composite. SEM analysis was conducted on the milled powders to check for the uniform distribution of the alumina particles in the aluminum matrix.

Consolidation of the milled powders was carried out with an optimum specific pressure of 5 tf/cm^2 at automatic cold press. Before extrusion, compacts were sintered at 620^oC, 1 h maintaining time and then, cooled in Ar atmosphere [6]. Sintered cylindrical compacts were hot extruded at 220 ± 10^oC by direct method at a horizontal extrusion press on Fogg & Young type. Before extrusion, the sintered compacts were heated for homogenization at 450^oC for 30 min., in a Carbolite type furnace.

Specimens sampled from extruded bars of Ø=8.3 mm were characterized from microstructural, electrical and mechanical point of view.

3. Results and discussion

In order to refine the aluminum matrix, the Al powders with particulate size between 45-71µm were mechanical milled for 22 h in Ar atmosphere.

The morphological aspects of the Al powder before and after MM are presented in fig. 1 a and b.

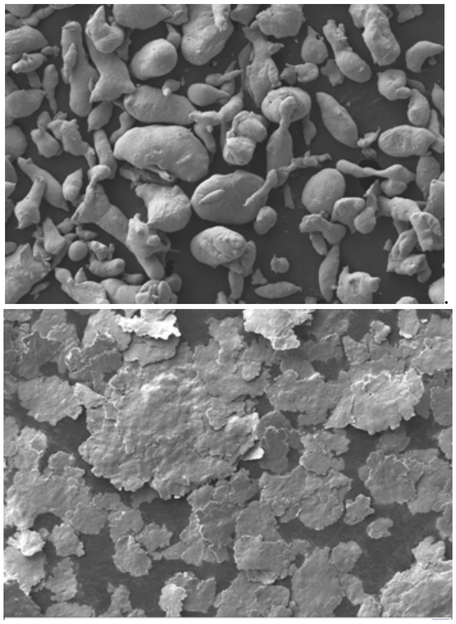

Fig.1. a) SEM images of initial Al powders (<71µm) and b) of Al powders after22 of MM, x 500.

By adding of 2%vol. of hard Al_2O_3 nanoparticles in the matrix MM for 22 h, and by continuing milling for 2h, 4h, 6h and 8h, fragmentation and welding processes occurs and the particles changes the initial morphologies. After 6h and 8h of MM (fig. 2a and b), a steady state condition is installed and an uniform granulation with particle sizes ranging between 4-10µm is obtained.

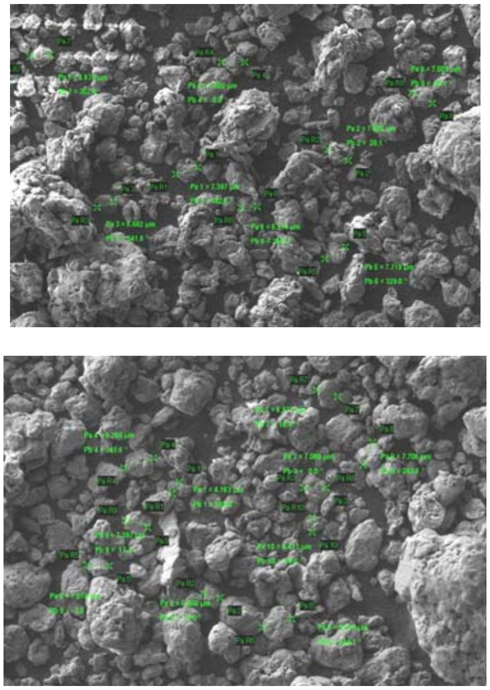

Fig.2.SEM image of Al-2%vol.Al_2O_3 powders after MM of 6h (a) and 8h (b), x 2000.

In order to obtain extruded bars, 3 cylindrical compacts of each composite obtained by different MM were realized with an automatic cold press of Meyer type.

In Table 2 the pressing parameters and extrusion criteria verification are presented for compacted Al-2%vo.Al_2O_3 composites.

From Table 2, the compacts had porosities included in the interval of 14...17%. The dimensions of the cylindrical compacts are in concordance with the requirements imposed for extrusion on the horizontal presses: $L_s/D_s\in(1.5...3)$ in order to produce a uniform deformation. Because $L_s/L_c\notin(0.5...0.7)$, three cylindrical compacts have been extruded at the same time, with an unitary degree of deformation, ε_u = 78.98% and an extrusion ratio, λ = 1.58.

In Table 3 the physical and mechanical characteristics of the sintered semi finished parts of Al-2%vol.Al_2O_3 composites are presented.

Table 2. Pressing parameters and extrusion conditions.

Composite material of Al-Al_2O_3/MM	Pressing parameters					Imposed criteria for extrusion	
	Height h_p,[mm]	Diameter, [mm]	Mass [g]	Density ρ,[g/cm^3]	Porosity [%]	L_s/D_s	L_s/L_c
Al-2%Al_2O_3/2 h	31.21	18.11	18.74	2.331	14.47	1.72	0.24
Al-2%Al_2O_3/4 h	31.65	18.11	19.03	2.334	14.36	1.75	0.24
Al-2%Al_2O_3/6 h	32.28	18.11	19.02	2.287	16.08	1.78	0.25
Al-2%Al_2O_3/8 h	33.37	18.11	19.53	2.272	16.63	1.84	0.26

$h_p = L_s$; $d_p = D_s$; $L_c = 130$ mm, h_p= compact height, L_s= semi finished length for extrusion, L_c = extrusion die container length.

Table 3. Physical and mechanical characteristics of the sintered semi finished parts of Al-2%vol.Al_2O_3 composites.

Composite material of Al-Al_2O_3/MM	Sintering parameters					Hardness, HV_{med} 0.3/15 [daN/mm^2]
	Height h_s,[mm]	Diameter d_s,[mm]	Mass [g]	Density ρ,[g/cm^3]	Porosity[%]	
Al-2%Al_2O_3/2 h	31.28	18.14	18.54	2.402	11.86	66.56
Al-2%Al_2O_3/4 h	31.73	18.11	18.86	2.396	12.08	74.64
Al-2%Al_2O_3/6 h	32.22	18.07	18.87	2.322	14.80	56.96
Al-2%Al_2O_3/8 h	33.16	18.05	19.41	2.324	14.72	71.36

h_s= sintered semi finished height, d_s= semi finished diameter.

Comparing with the pressed state (Table 2), after sintering treatment of the Al-2%volAl_2O_3 compacted composites, the porosity is decreasing due to reduced size of the particles obtained during MM, which favors a better welding during sintering (Table 3).

In Table 4 the physical and mechanical characteristics of the extruded semi finished parts of Al-2%vol.Al_2O_3 composites are presented.

Table 4. Experimental results obtained after extrusion of sintered semi finished parts of Al-2%vol Al_2O_3.

Composite material of Al-Al_2O_3/MM	Extrusion parameters					Hardness, HV_{med} 0.3/15 [daN/mm^2]	Extrusion Force, [KN]
	Length L_e,[mm]	Diameter d_e,[mm]	Mass [g]	Density ρ,[g/cm^3]	Porosity[%]		
Al-2%Al_2O_3/2 h	105	8.3	13.52	2.694	1.15	128.4	324
Al-2%Al_2O_3/4 h	100	8.3	14.01	2.712	0.49	132.2	324
Al-2%Al_2O_3/6 h	110	8.3	15.91	2.684	1.51	133.4	324
Al-2%Al_2O_3/8 h	120	8.3	16.89	2.668	2.10	140.6	324

L_e= extruded semi finished length, d_e= extruded semi finished diameter.

After extrusion, the Al-2%vol.Al_2O_3 composites had a high degree of densification, the remanent porosities being under 3%. The porosity decreased from 11-14% (sintered state in Table 3) until to1-2% (extruded state in Table 4) while the Vickers hardness values increased from 128.4 HV until to 140.6 HV, when milling time is increasing from 2h until to 8h.

The micrographic aspects of Al-2%vol.Al_2O_3 extruded bars are presented in fig. 3.

In fig. 4 a and b, the optical and electronic microscopy images of the Al-2%vol.Al_2O_3 are presented.

After extrusion, all studied composites had presented homogeneous microstructures with a very fine and low remanent porosity.

At the lowest MM time (2h), from fig. 4b, it is evidenced at a very high magnification (x 50.000), an uniform distribution of Al_2O_3 nanoparticles of 50…60nm in the Al matrix and a random distribution of some pores of 0.1… 0.4µm.

Fig.3. Micrographic aspect of the Al-2%vol.Al_2O_3 extruded bars.

Comparing with Al electrical conductivity (σ_{Al} = 3.80 x 10^7 (Ω x m)$^{-1}$) [7], the analyzed composites had electrical conductivities values included in the interval $\sigma \approx 1.4 \div 1.9$ x 10^7 (Ω x m)$^{-1}$.

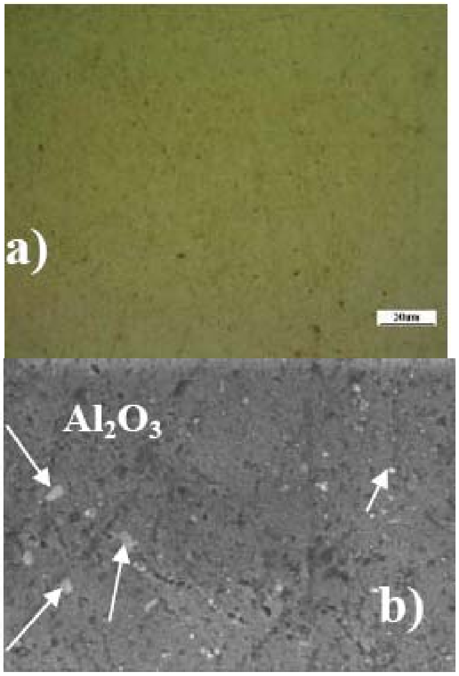

Fig. 4. a) OM image of extruded Al-2%vol.Al_2O_3 (4 h MM) b) SEM image of extruded Al-2%vol.Al_2O_3 (2 h MM), x 50000.

In fig. 5 the mechanical properties of the Al-2%vol.Al_2O_3 extruded composites resulted from tensile test of specimens, is graphically represented in compare with the mechanical properties of the Al 6061which is a precipitation hardening aluminum alloy, containing magnesium and silicon as its major alloying elements. This 6061 alloy is widely used for construction of aircraft structures, such as wings and fuselages, in homebuilt aircraft and yacht construction, in the construction of bicycle frames and components and also, in automotive parts, such as wheel spacers [7].

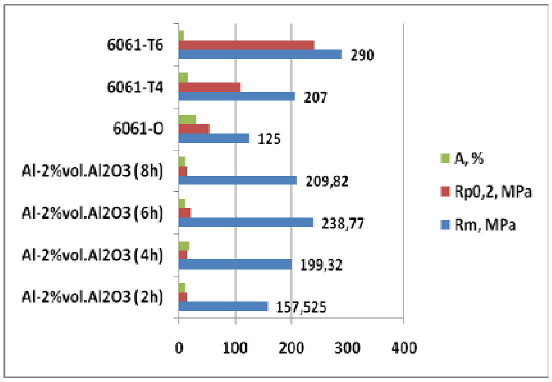

Fig.5. Tensile mechanical properties of the Al-2%vol.Al_2O_3 extruded composites.

In fig. 6, the mechanical properties of the Al-2%vol.Al_2O_3 extruded composites resulted from compression test of specimens, is graphically represented.

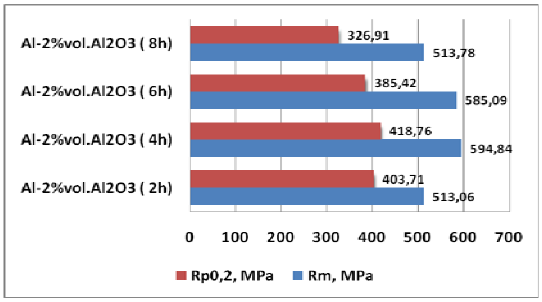

Fig.6. Compression mechanical properties of the Al-2%vol.Al_2O_3 extruded composites.

4. Conclusions

A homogeneous distribution of the Al_2O_3 nanoparticles reinforcement phase in the Al matrix was obtained by high-energy milling of Al-2%vol.Al_2O_3 blends. Characterization of the mechanically milled powders confirmed uniform distribution of the reinforcement phase.

Increasing milling time leads the composite towards steady-state condition in which, all microstructure properties such as powder size, powder shape and distribution of Al_2O_3 within Al matrix remain fixed.

An optimum milling period of time (4h - 6h), is established for the processing of the Al-2%vol.Al_2O_3 composites, that assures mechanical strength and elongation close to those of the Al 6061.

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