Fabrication through P/M of ecological aluminum based composite materials. Part 1-Characterization and densification of mixture powders

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Abstract: The paper presents the experimental results concerning fabrication through powder metallurgy (P/M) of new ecological aluminum matrix composites (AMC) at a low cost for automotive industry applications. The difficult compaction and sintering of aluminum powder products and especially of powders from aluminum composite mixtures reinforced with hard ceramic particles require complex phenomenon's analysis which occurs during materials' fabrication. Thus was realized a complex experimental program, where have been varied proportion of SiCp (5-20%wt.) and process parameters: applied pressure (100-450MPa), sintering temperature (520-620)^{o}C and secondary heat treatment applied after sintering. In the first part of the research activities was determined the effects of varied amount of silicon carbide particles (SiCp) addition in the mixture on the characteristics and densification of powders of Al-Cu alloys matrix composites, using analysis' techniques specific to P/M processes and also Environmental Scanning Electron Microscopy and Electron Probe Micro Analyzer.

Key-Words: ecological AMC obtained by P/M**,** physical, chemical and technological characterization of elemental and mixture powders, densification of powders

1 Introduction

The continuous developing of techniques in automotive industries requires intensive researches in obtaining of new performing materials at a low cost and ecological for the environment. These conditions are accomplished in many parts of the light composites discontinuous reinforced with ceramic particles, like aluminum based composites. These composites combine the characteristics of aluminum based matrix (low density in comparison with ferrous materials, good corrosion resistance and machinability) with the characteristics of ceramic particles (e.g. SiC, TiC, B_4C, Al_2O_3, SiO_2, etc.) which improve in special mechanical, tribological and thermal expansion characteristics [1, 2].
The processing of AMC it is realized by a diversity of methods that are classified in function of aggregation state (liquid, semisolid or solid) of matrix in the processing route. Among the various manufacturing technologies, powder metallurgy (P/M) is the most advantageous techniques to fabricate isotropic distribution of particles in matrix, good dimensional accuracy in an economical manner. The conventional P/M process can easily formulate different composition by simply mixing elemental or premixed powders, consolidate and sinter the powder mixtures to the near shape [1-5].

2 Problem Formulation

Despite of the advantages of processing by P/M of powders, in aluminum matrix composites, the powder mixtures are more difficult to compact and sinter than other composites, for example in comparison with the iron or copper based matrix, owing to presence of the stable Al_2O_3 layers who covered particles of aluminum and which obstruct the consolidating processes.
In addition, the presence of hard ceramic particles in aluminum ductile matrix increases this processing difficulty. The researching studies realized until now, solved in part the problems of difficult compaction and sintering of aluminum alloys matrix with specific composition [6 -10], but for the rest of them, in function of the nature of components and processing condition of composites, still require many investigations.

In this paper, we have developed new materials in terms of composition (Al-4Cu/SiC) and manufacturing process and were determined the optimal technological parameters of densification of composites.

2.1. Materials Selection

The objective of the present researches was to determine the effect of silicon carbide particles (SiCp) on the characteristics of composite mixtures Al-Cu/SiCp and compaction behavior of them. First of all it is necessary a good selection of powder materials and processing parameters. Thus, a commercial air atomized aluminum and electrolytic copper powders, both with particles size less than 100 µm were selection as the matrix powders. The copper choose for alloying aluminum, four weight percent in composition, forms at sintering temperature an eutectic liquid who allow a good sintering of materials and as a result good characteristics of the materials [1, 6]. As the reinforcing phase, β - SiC particles from import (Norton type, sort M400) with particle size less than 60 µm were used.

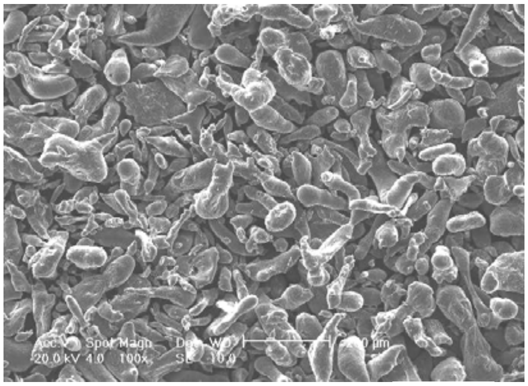
2.2. Experimental Procedure

The determined characteristics of elemental powders and mixtures was following: particle size and shape, distribution and surface area (the physical characteristics), chemical macroanalysis and microanalysis, respectively the technological characteristics (apparent and tap density). Chemical characterization consisted in chemical macro-analysis of samples which were made in according with standards for analysis of melted and casting metals and alloys. The morphology of raw powders (size and shape of elemental powders) was made with Eniviromental Scanning Electron Microscopy (ESEM), FEI XL-30 type (Philips).
The apparent density of the powder is a very important parameter that depends on the physical characteristics and the degree of porosity of the particles [2]. The apparent density was determined by flowing mass of powder into a container of know volume and measuring the weight of powder which completely fills the space, according to SR EN 23923-1: 1998 standards.
The tap density is a mass of loose powder that is mechanically or taped (SR EN 23923-2: 1999). The flow rate of powder was determined in concordance with the standard method SR EN ISO 3953: 1998, who measuring the time necessary for 50 mgs of powder to flow through a prescribed small orifice using the Hall Flow meter. The area surface was determined with BLAINE permeabilimeter, who consist in measure of permeability in air of a quality know of powders, in stationary flow condition, described by the standard ISO 10070-1991.
The green density of the compacts was determined by physical measurements. Before the manufacturing of the composite, the electrolytic copper powders was reduced in Siemens - Plania furnace in presence of hydrogen gas at 280°C for 60 min and the β - SiC powders was heated for 400 °C, holding 120 min, for eliminate of adsorbed gases, moisture and organic contaminants.
The dosage of mixtures was made gravimetric and the elemental powder of the matrix and composite mixtures was dry blended using the Double Cone Blender (10kg capacity) at a rotation speed of 20 r.p.m, between 3 and 9 hours. The minimum time of mixing powders was used for Al-Cu matrix powders and, in function with increasing of quantities of SiCp, we increased the time of mixing of powders (the maximum time of mixing was applied for 20%SiCp).
Were realized five mixture at each 2 kg weight: a first mixture (aluminum with 4wt.%Cu) without the SiCp, which consist the Al-Cu matrix and other four mixtures of Al-4Cu mixtures in addition with respectively 5, 10, 15 and 20wt.% SiCp. For all mixtures 2 wt. % zinc stearat powder lubricant was added and homogeny blended to reduce friction between the powder mass and the surface of the die and obtain a good compaction. The obtained mixtures were homogeny at macroscopically level. The mixed homogenous powders were compacted at room temperature in a double action hardened steel die with a automate hydraulic press of 30 tone force, Meyer Type. The compaction pressures were varied from 100 to 450 MPa. The compacts were dewaxed 420oC for 30 min and sintered at 520-620 for 60 min in presintered-sintered furnace (Siemens-Plania type) in a protective atmosphere.

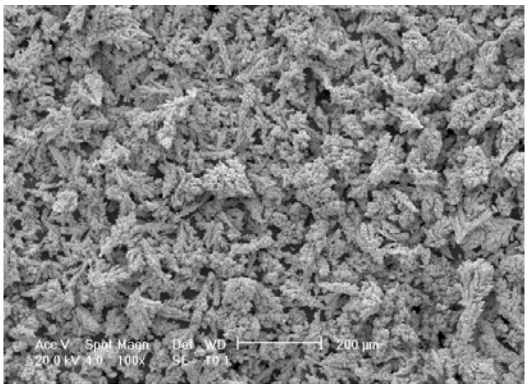
3 Problem Solution

3.1. Characterization of raw and mixture powders

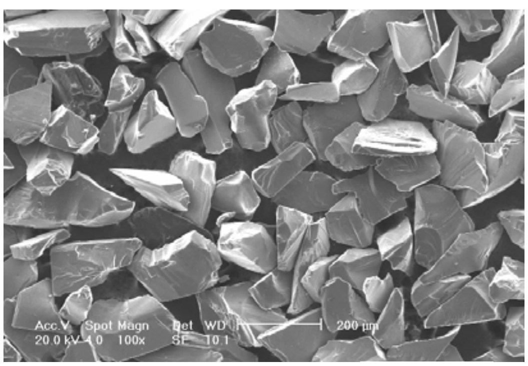
In the experimental work have been intended to obtain homogeneous powder mixtures and highly pressed compacts which involve obtaining of sintered composites with high physical, mechanical characteristics.
The results of (macro) chemical analysis of aluminum powder show the existence of maximum content of 0.02%O_2; 0.14% Fe; 0.18% Si; 0.02% Zn and aluminum active balance. The chemical analysis of reduced electrolytic copper powder has resulted a content of 0.1% O_2, and copper balance, and the chemical analysis of silicon carbide show us a content of 0.14%Fe_2O_3, 2.96%C in excess and trace of Si and SiO_2.
The morphology of elemental powders (Al, Cu, SiCp) used as raw powders, using ESEM are presented in Figure 1.



a) 200 µm



b) 200 µm

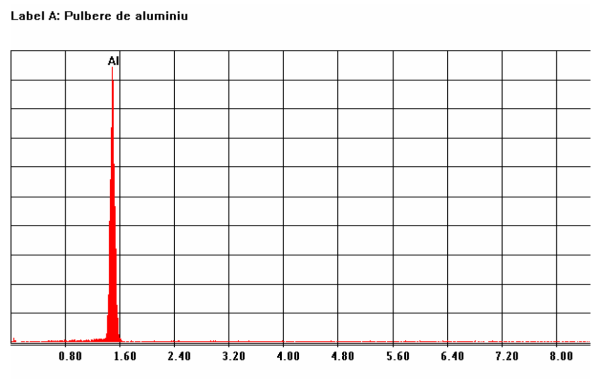


c) 200 µm

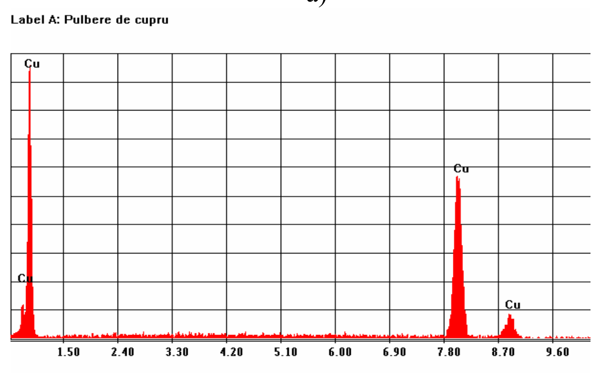
Fig. 1 Scanning electron micrographs and of (a) aluminum powders; (b) electrolyte copper powder; (c) Silicon carbide particles

We observed that the aluminium particles are droplet-like shape, the size and the shape of copper particles are dendritic and the ceramic particles shape are poliedric. The Quantitative X-ray Microanalysis (Figure 2) show as that the copper powder was reduced in the proportion of about one hundred percent, and SiC powder has been dried properly (no oxygen present in the composition). The amount of silicon in the composition of SiC particles is 57,15% weight and the carbon weight of 42.85%.

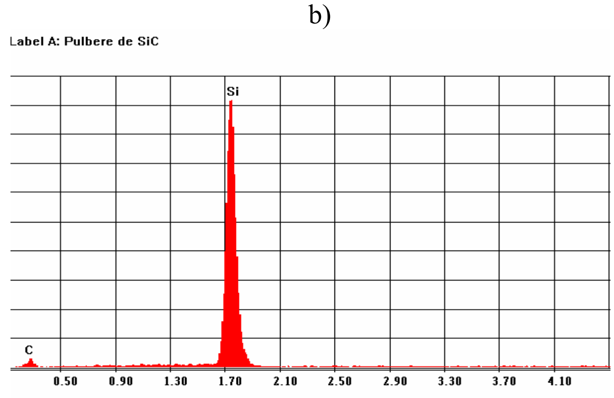
Fig. 2 The Quantitative X-ray Microanalysis of (a)



a)



b)



c)

aluminum powders; (b) electrolyte copper powder; (c) Silicon carbide particles.

The physical and technological characteristics of raw powders are presented in Table 1 and 2.

Table 1

Powder type	Particle size distribution , %				
	>100 µm	100 – 63µm	63 –50 µm	50 –40 µm	<40 µm
Al	0.7	32.3	22.4	43.5	1.1
Cu	-	12.8	31.7	14.3	41.2
βSiC	-	-	1.6	4.5	93.9

Table 2

Powder type	Technological and physical characteristics			
	Apparent density, g/cm^3	Tap density, g/cm^3	Flow rate s/50g	Area surface m^2/cm^3
Al	1.29	1.374	12.8	0.12
Cu	2.35	2.84	38	0.41
βSiC	1.37	1.648	16	1.36

The graphical representation of evolution of technological and physical characteristics of Al-Cu/ SiCp mixtures depending on SiC p addition are presented in Figures 3 (apparent and tap densities) and in Figures 4 and 5 (Flow rates and area surface).

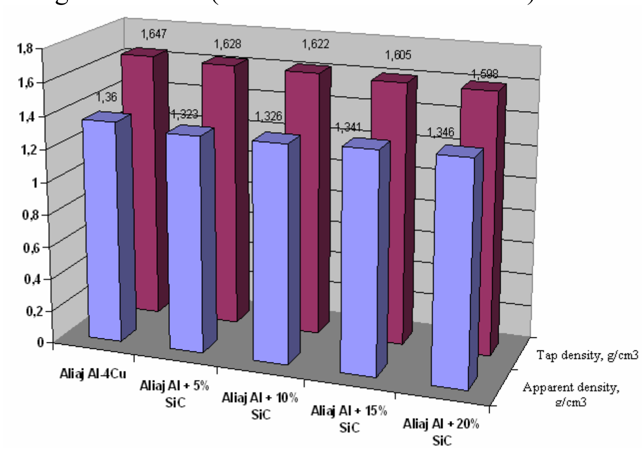


Fig. 3 Apparent and tap density evolution depending on SiCp addition in Al-Cu matrix

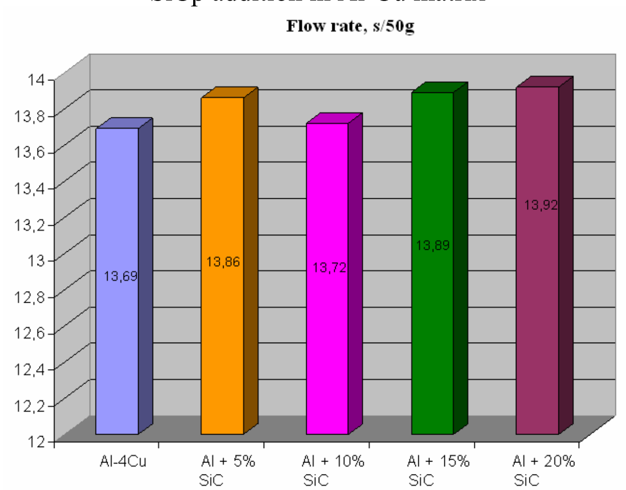


Fig. 4 Flow rate evolution depending on addition of SiCp

3.2 Densification of mixture powders and characterization of compact composites

The mixed homogenous powders were compacted at room temperature in a double action hardened steel die (13,4 mm in diameter and 20 mm height) with a hydraulic press of 30 tone force, Meyer Type.

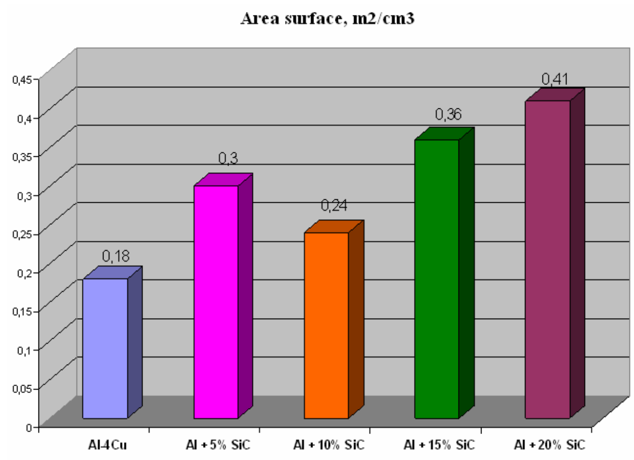


Fig. 5 Area surface evolution depending on % SiCp

The densification curves for all five types of mixtures are presented in Figure 6. According with densification curves (fig.6) we note that for achieving maximum densification of un-reinforced aluminum alloys (96.77 relative density) it is sufficient an applied pressure of 250 MPa, for composites reinforced with 5% SiC the good densification it is obtained at 350MPa (94.96%) and for an applied pressure of 450MPa for composites reinforced with 10-20% SiC the relative density achieved was 92-89%. The theoretical and calculated green density, respectively the relativ density of the resulted cold compacted materials, at pressure which we obtained the maximum /very good densification are presented in Table no. 3.

Table 3

Specimens	Theoretical density, g/cm^3	Relativ density, %	Porozity of green parts, %
Al-4Cu	2.69	96.77	3.23
Al-4Cu- 5%SiC	2.72	94..92	5.08
Al-4Cu-10%SiC	2.74	92.15	7.85
Al-4Cu-15%SiC	2.76	91.63	8.37
Al-4Cu-20%SiC	2.78	89.49	10.51

Theoretical density of powder mixtures represent the maximum density of material attained in the final stage, where, ideally is considered zero porosity and is calculated by the rule of mixtures, after the following relationship:

$$\rho_{mixture} = \frac{100}{\sum_{i=1}^{n} \frac{x_i}{\rho_i}},$$

where xi is the fraction component i in mixture (i being Al, Cu, zinc stearate or/and SiC); ρi is the component density i (g/cm3).

The measured porozity (the pore volume fraction) P of compacted respectively sintered parts was made can be determied by the equation:

$P = \left(1 - \frac{\rho_u}{\rho_{mixture}}\right)$ 100, %, where ρ_u - the measured density of green compact or sinterd part ; and $\rho_{mixture}$ is the theoretical density, g/cm^3.

The Al-Cu and Al-Cu-SiCp mixtures exhibits uniform die filling and provides good reproduction of part configuration.

In order to understand the mechanisms that occur during the densification through cold compaction of composite mixtures and to highlight the stages that take place during the compaction process, we correlated the physical characteristics of the green compact (relative density) with their microstructural characteristics (microstructural analysis using the Electron Probe Micro Analyzer JXA-5A JEOL (Fig. 7). In Fig. 7 we can see the morphology and distribution of particles: Al and Cu particles have oval shape and SiC particles have polyedric ones.

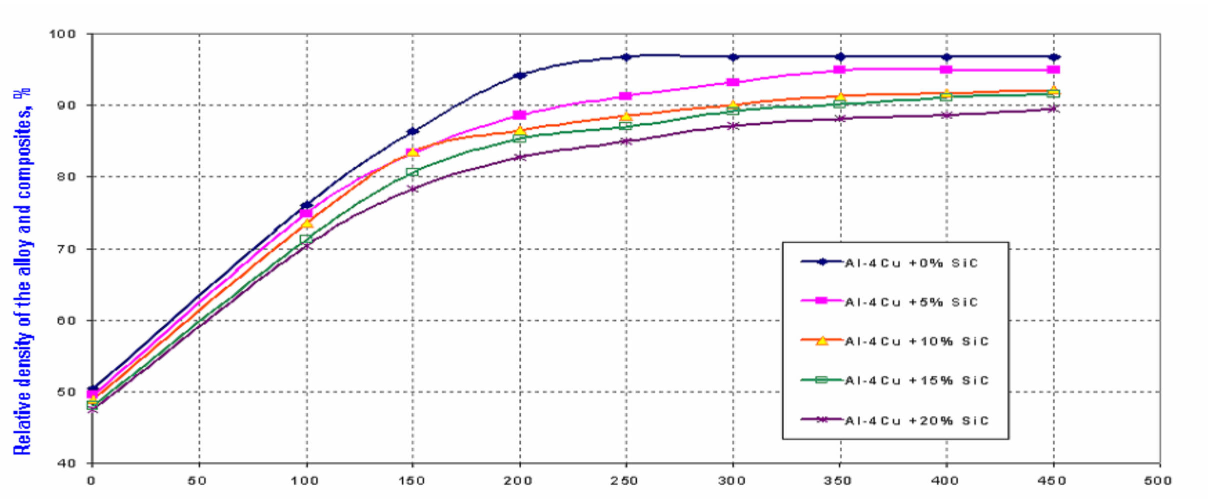


Fig. 6 The densification curves for Al-4wt.%Cu alloys and Al-4Cu/SiCp composites

The image composition shown in Figure 7 (a and c) it appears that after compression of the Al particles, were deformed ensuring better packing and deformed aluminum particle have sizes ranging between 0.02-0.16mm. In the same set of figures indicates that the presence of particles in composite materials SiC prevent good densification of the material. It appears that the SiC particles are located around the pores, their size and quantity is proportional to the amount of SiC material.

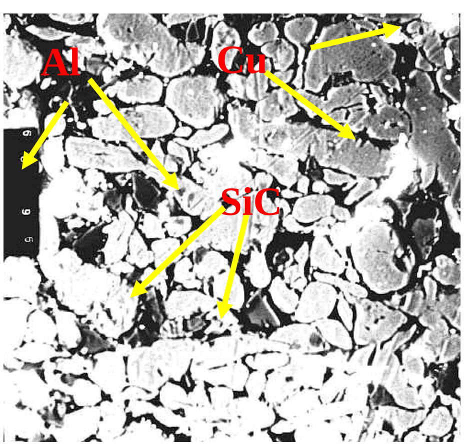


Fig.7 a)

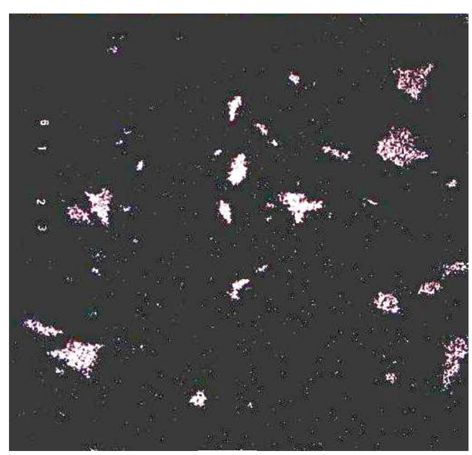


Fig.7 b)

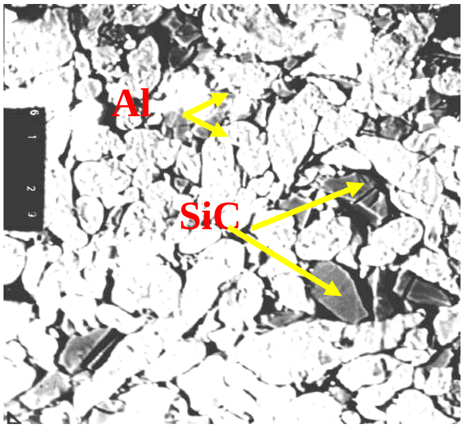


Fig.7 c)

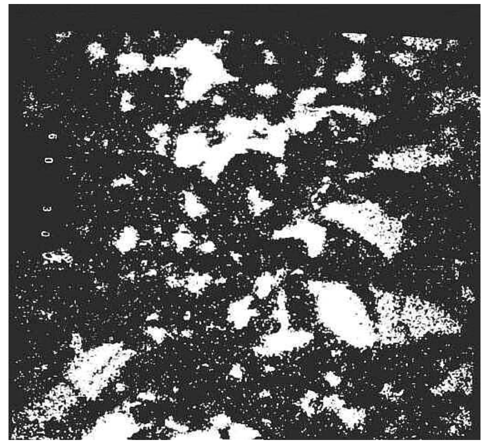


Fig.7 d)

Fig. 7 Electron MicroProbe Analysis (a) Compo Image of Al-4Cu/5%SiCp green compact samples; (b) X-ray distribution map of Cu in Al-4Cu/5%SiCp ; (c) Compo Image of Al-4Cu/20%SiCp; (b) X-ray distribution mapSi in Al-4Cu/20%SiCp composite sample

Conclusion

After analyzing densities tests we observed that the apparent and tap densities decrease with increasing SiC content in the mixture, the surface area and flow velocity mixtures have a weak increasing trend according to the proportion of mixed carbide inserted. The densification curves obtained for all mixtures show us that by adding the hard and fragile SiC particles in the metallic Al alloy mixtures, the compressibility decreases. The increasing difficulty to compact the mixtures (higher compaction forces) with the increase of the SiC content is explained by the fact that the hard SiC particles delay the densification by taking over the compaction charge until they break, the maximum densification occurring through the repacking of the SiC fragments in the Al alloy mass.

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