Processing by P/M route and characterization of new ecological Aluminum Matrix Composites (AMC)

Ileana Nicoleta Popescu, Simona Zamfir, Violeta Florina Anghelina, and Carmen Otilia Rusanescu

Abstract—The continuous development of technology in automotive manufacturing process has required new solutions adapted to the growing requirements of lightweight, non-pollution for the environment materials with a low cost production. According with these required characteristics of materials, the aim of this paper was to manufacturing Al-Cu/SiCp composites by powder metallurgy (P/M) processing route and characterization of the powders and compacted/sintered mixture powders. Was developed a complex experimental program consisted in variation of silicon carbide proportion (5-20%wt.) in the composites and also was established the effect of proportion of SiC’s addition on: a) characteristics of powders mixtures Al-Cu/ SiCp; b) press densification of materials at different applied pressure (50-450 MPa); c) the porosity evolution (sintering densification) respectively dimensional variation, the homogeneity and hardness of sintered materials, in the solid state (at 520-548°C) and in the liquid phase (548-620°C). To characterize powders, mixtures of powders and obtained composite materials from physic-mechanical, chemical and technological point of view were used for investigation, classical and also modern techniques, such as: Environmental Scanning Electron Microscopy, Electron Probe Micro Analyzer and X-ray diffraction. Hardness was correlated with physical and microstructural characteristics, thereby determining the sintering temperature and optimum proportion of carbide particles that assure the best densification of materials and the best mechanical characteristics. After investigations was resulted that the best characteristics of composite materials was obtained at a proportion of 10 and 15% wt. SiCp, cold pressed at 450 MPa, dewaxed / presintered between 300 and 4000C, for 30 min (maintenance temperature/ time) and sintered at 6200C/60 min in protective atmosphere.

Keywords—Characterization of powders, Densification, Ecological AMC obtained by P/M, Electron Probe Micro Analyzer, X-ray diffraction

I. INTRODUCTION

The increased demand for new high-performance materials, ecological, for the automobile industry, in the last decades, led to the intensification of researches in this field. The materials which accomplish in many parts these characteristics are the light weight composites discontinuous reinforced with ceramic particles, like aluminum based composites [1], [2]. These composites combine the characteristics of aluminum and aluminum alloys matrix (low density in comparison with ferrous materials, good corrosion resistance and machinability) with the characteristics of ceramic particles (e.g. SiC, TiC, B4C, Al2O3, SiO2, etc.) which improve in special mechanical, tribological and thermal expansion characteristics [1]- [3].

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 presintering - sintering the powder mixtures to the near shape [1]-[6]. Taking into account that dewaxing - sintering is a very complex process, the experimental processing and results of similar Al-based composites reinforced with ceramic particles, especially with SiCp, were analyzed. For understand these physical-chemical processes requires a comprehensive study of the composites fabrication at different parameters of manufacturing. In the scientific papers of O. Lapshin and others [7] is specified that in the case of aluminum or aluminum alloy reinforced with SiCp composite materials, dewaxing-degassing operation took place at 400°C-420°C temperature for 30-60 minute and for the unreinforced aluminum used like matrix took place at 300°C for 3 hours. Sintering parameters (sintering temperatures, isothermal sintering time and the sintering atmosphere) varies in very wide limits, owing to the diversity of materials used as matrix and the shape, size and distribution of silicon carbide [6-21]. Sintering temperatures used in aluminum and its alloys is between 520 and 640°C, while the isothermal maintaining time at sintering temperatures is between one and 16 hours [8]-[24]. The sintering atmosphere could be hydrogen, nitrogen, and dissociated ammonia with dew point of very low – 40°C or vacuum [8], [11], [13]-[24].

For example, in the scientific research of D. Sâbăduș [17], for processing of Al/SiCp from elemental powder mixtures by P/M, different sizes of initial powder under the same conditions of compaction pressure, the sintering temperatures chosen for research were between 530 and 610°C and the optimal sintering condition was established for sintering temperature between 580 and 590°C, sintering time at 4 hours for a mixture of different grain size fractions. W.F. Calley and collaborators [9] studied the fabrication of Al-based composites reinforced with 14%wt. SiCp by cold isostatic pressure at 187 MPa. They use the follows processing temperatures: 400°C for dewaxing of prealloyed powders Al-0.5Mg-4Cu (AA2014) and for sintering temperatures ranged...
from 605-620°C, maintenance at sintering temperature for 1, 2 and 4 hours. They demonstrate that the optimum temperature of sintering is 620°C, at a sintering time of 4 hours. J. Zhou and J. Duszczyzk [16] obtained by powder metallurgy (151MPa, uniaxial cold pressing pressure, delubricating temperature of 420°C for 25 min, degassing between 420 and 590°C and sintering temperature at 590°C for 30, 60 respectively 90 minutes) a composite AA2014/10% vol. SiC. They decided that: the best properties (strength and elongation) is obtained at sintering temperature of 590°C, holding at sintering temperatures for 60 minutes and the heat-treated at T6 condition. For better consolidation these materials were subjected of extrusion [16].

II. 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 Al2O3 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 research 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/SiCp) and manufacturing process and were determined the optimal technological parameters of densification of composites.

A. 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. The raw powders selected must be ecological from compositional point of view and have required characteristics. Thus, a commercial air atomized aluminum and electrolytic copper powders, both with particles size less than 100 µm were selected 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.

B. Experimental Procedure

The determined characteristics of elemental powders and mixtures were following: particle size and shape, distribution and surface area (the physical characteristics), chemical macro-analysis 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 Environmental 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 permeameter, 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” type 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 automatic hydraulic press of 30 tone force, Meyer Type. The compaction pressures were varied from 100 to 450 MPa. In order to minimize the processing costs, the dewaxing, degassing and sintering were combined into a single operation. The compacts were dewaxed for 30 min, degassing during heating up from 420°C to 520°C with duration of 30 min and sintered at 520-620°C for 60 min in presintered-
sintered furnace (Siemens-Plania type) in a protective atmosphere (hydrogen) and then furnace cooled.

The existence of good bonding between metallic matrix and ceramic particles at interface and the morphology and distribution of pores and carbides in the matrix we can appreciate. The green density of the compacts was determined by physical measurements. The density of the sintered composites was measured using the Archimedes method (ASTM B 328-92). The sintered Al-Cu/βSiC composites were analyzed from physical, mechanical and microstructural point of view. The physical characterization consists in determine the density and porosity of composites and unreinforced matrix alloy and the mechanical characterization was Brinell hardness measurements. The Brinell hardness test was done on a device FRANKOSCAP universal type by pressing on the surface material with a ball of 2.5 mm and load of 62.5 Kgf for 30 seconds.

For investigate the dimensional changes and the physical characteristics exhibited by Al-Cu/ βSiC compacts we made dimensional measurements - the Volume of compacted samples (Vc) and sintered samples (Vs) - before and after sinterization of samples at all 6 temperatures ranges between 520-620°C.

The sintered density of specimens was measured using the Archimedes techniques. The microstructural analysis was made with Electron Probe Micro Analyser JXA-5A JEOL type. With this apparatus we could investigate the morphology and distribution of particles, the homogeneity of material, etc. The X-ray diffraction was made in order to put in evidence the presence of phases α solid solution, θ (CuAl2) and SiCp hard particles.

III. PROBLEM SOLUTION

A. 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%O2; 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% O2, and copper balance, and the chemical analysis of silicon carbide show us a content of 0.14% Fe2O3, 2.96% C in excess and trace of Si and SiO2.

The morphology of elemental powders (Al, Cu, SiCp) used as raw powders, using ESEM are presented in Figure 1. We observed that the aluminum particles are droplet-like shape, the size and the shape of copper particles are dendritic and the ceramic particles shape is 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).

Table 1

<table>
<thead>
<tr>
<th>Powder type</th>
<th>Particle size distribution, %</th>
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<tr>
<td></td>
<td>&gt;100 μm</td>
</tr>
<tr>
<td>Al</td>
<td>0.7</td>
</tr>
<tr>
<td>Cu</td>
<td>-</td>
</tr>
<tr>
<td>βSiC</td>
<td>-</td>
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</table>

The amount of silicon in the composition of SiC particles is 57.15% weight and the carbon weight of 42.85%. The physical and technological characteristics of raw powders are presented in Table I and II.

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

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

**Table II**

<table>
<thead>
<tr>
<th>Powder type</th>
<th>Technological and physical characteristics</th>
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<tr>
<td></td>
<td>Apparent density, (g/cm³)</td>
</tr>
<tr>
<td>Al</td>
<td>1.29</td>
</tr>
<tr>
<td>Cu</td>
<td>2.35</td>
</tr>
<tr>
<td>βSiC</td>
<td>1.37</td>
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**B. 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. 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% SiCp the good densification it is obtained at 350MPa (94.96%) and for an applied pressure of 450 MPa for composites reinforced with 10-20% SiC the relative density achieved was 92-89%. The theoretical and calculated green density and respectively the relative density of cold compacted materials, at pressure with maximum/ very good densification are presented in Table III.

Table III

<table>
<thead>
<tr>
<th>Specimens</th>
<th>Theoretical density, (g/cm³)</th>
<th>Relative density, (%)</th>
<th>Porosity of green parts, (%)</th>
</tr>
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<tbody>
<tr>
<td>Al-4Cu</td>
<td>2.69</td>
<td>96.77</td>
<td>3.23</td>
</tr>
<tr>
<td>Al-4Cu-5%SiC</td>
<td>2.72</td>
<td>94.92</td>
<td>5.08</td>
</tr>
<tr>
<td>Al-4Cu-10%SiC</td>
<td>2.74</td>
<td>92.15</td>
<td>7.85</td>
</tr>
<tr>
<td>Al-4Cu-15%SiC</td>
<td>2.76</td>
<td>91.63</td>
<td>8.37</td>
</tr>
<tr>
<td>Al-4Cu-20%SiC</td>
<td>2.78</td>
<td>89.49</td>
<td>10.51</td>
</tr>
</tbody>
</table>

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

\[
P = \left(1 - \frac{\rho_u}{\rho_{mixture}}\right) \times 100\% 
\]

where \( \rho_u \) - the measured density of green compact or sintered part ; and \( \rho_{mixture} \) is the theoretical density, g/cm³.

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 Figure 7 we can see the morphology and distribution of particles: Al and Cu particles have oval shape and SiC particles have polyedric ones.

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 0.02-0.16 mm.

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.
C. Dimensional and porosity changes during sintering of composite samples

Dimensional changes, in volume of sintered composites and his un-reinforced matrix alloys at different sintering temperature for Al-4Cu aluminum alloys and Al-4Cu/SiCp composites (5-20wt.%SiC as reinforcement in matrix) are presented in Figure 8.

To observe the evolution of porosity depending on addition of ceramic particle tough material, and sintering temperature was drawn graph in Figure 9 presented below.

Fig. 8 Dimensional changes, in volume of sintered composites and alloys.
Brinell hardness values obtained at different sintering temperatures on aluminum alloy sintered composite materials that are presented in Table IV.

Table IV

<table>
<thead>
<tr>
<th>Sintering T, oC</th>
<th>Brinell Hardness</th>
<th>Al-4Cu 0%SiC</th>
<th>Al-4Cu 5%SiC</th>
<th>Al-4Cu 10%SiC</th>
<th>Al-4Cu 15%SiC</th>
<th>Al-4Cu 20%SiC</th>
</tr>
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<tbody>
<tr>
<td>520</td>
<td>30</td>
<td>28</td>
<td>28</td>
<td>30</td>
<td>27.5</td>
<td></td>
</tr>
<tr>
<td>540</td>
<td>36</td>
<td>29</td>
<td>27.5</td>
<td>28</td>
<td>29</td>
<td></td>
</tr>
<tr>
<td>560</td>
<td>40</td>
<td>45</td>
<td>38</td>
<td>36</td>
<td>35</td>
<td></td>
</tr>
<tr>
<td>580</td>
<td>33</td>
<td>29.5</td>
<td>26</td>
<td>28</td>
<td>28.5</td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>50.5</td>
<td>52</td>
<td>51</td>
<td>55</td>
<td>52</td>
<td></td>
</tr>
<tr>
<td>620</td>
<td>58</td>
<td>57</td>
<td>59</td>
<td>62</td>
<td>59</td>
<td></td>
</tr>
</tbody>
</table>

After analyzing of the porosity evolution respectively dimensional variation of sintered materials we observed:

The appearance of progressive dilatation of all materials at temperatures between 520 and 580°C, and at temperatures above 580°C, the differentiate contraction of materials took place, according to the SiC particles proportion in the matrix.

Analyzing the porosity in function of the sintering temperatures and the proportion of SiCp was found that for sintered alloy matrix Al-4Cu (0% SiCp) and for the composites Al-4Cu / 5% SiCp at a 580°C sintering temperature, the maximum porosity were 5.94% respectively 6.80% and for composites with the same matrix (Al-4Cu) and 10, 15 respectively 20% SiCp at 560°C sintering temperature, the porosity were recorded between 9.13% and 12.63%.

It was found that the best densification was obtained at temperatures of 600 and 620°C in the presence of a sufficient quantity of eutectic liquid phase when the lowest values of porosity was 4.39% and 3.53% for composites with 5% SiCp. The values of porosity increase with the proportion of SiCp at 8.94%, at 600°C respectively 8.38% at 620°C for composites with 20% SiCp.

High porosity values for composites with high content of SiCp in the first stages of sinterization it is explained, on the one hand, by existing of a large initial porosity of compacts due to a lower pressure in the packaging of mixtures containing large SiC particles (Fig. 9 and 11) and on the other hand by reducing of self-diffusion between solid particles of Al-Al and reducing of diffusion between Al-Cu by hard particles of SiC.

The minimum values obtained at 600°C respectively at 620°C temperature are explained by the presence of the liquid phase in sufficient quantity (6% at 600°C and 20% at 620°C) to allow a maximum densification of the materials.

D. Microstructural analysis of sintered composite samples with Electron Probe Micro analyzer and X-ray investigations

The microstructural analysis with the Electron Probe Analyzer of pressed and sintered materials that confirm that the presence of SiC particles prevents proper densification of composites (Fig. 10).
It appears that the SiC particles are located around pores; the size and quantity of these are proportional to the amount of SiC and sintering temperature.

In Figure 11 is given the diffractometry of composite material with a high content of SiC (20%) sintered at 620°C.

We observed in Figure 12 the presence of α-Al phase, θ (Al₂Cu) and β-SiC carbide, which confirms that copper was completely dissolved in the material.

IV. 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.

It was found that the best densification was obtained at temperatures of 600 and 620°C in the presence of a sufficient quantity of eutectic liquid phase when the lowest values of porosity was 4.39% and 3.53% for composites with 5% SiCp, (Fig 8 and 9) in correlation with Brinell hardness. The microstructural analysis with the Electron Probe Analyzer of sintered materials confirms that the presence of SiC particles prevents a proper densification of composites (Fig. 10). The diffractometry of composite material with 20% SiC sintered at 620°C shows the presence of α-Al phase, θ (Al₂Cu) and β-SiC carbide, which confirms that copper was completely dissolved in the material. The best characteristics of composite was obtained at 10 and 15% wt. SiCp, pressed at 450 MPa, dewaxed / presintered between 300 and 400°C, for 30 min and sintered at 620°C/60 min in protective atmosphere.
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REFERENCES


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Physicist, Ph.D. Student Florina Violeta Anghelina has published in Proceedings of Conferences and Journals about 23 scientific research papers and presented 13 Poster and Oral Communications and has 6 research projects (as collaborator of project). She is Member of Chemistry Society from Romania, SChR (2008), Member of Romanian Association of Fracture Mechanics, ARMR (2008) and Member of the Romanian Society for Biomaterials SRB (2010).

C. O. Rusanescu was born in Gaesti, (Dambovita County, Romania) April 4th, 1970. She graduated in Materials Science and Engineering, Faculty of Material Science and Engineering, Speciality Plastic Deformations and Heat Treatment of the Polytechnic University of Bucharest.

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Lecturer Dr. Eng. Carmen Otilia Rusanescu published three books: 1) Elements of dynamic pollution, CO Rusanescu, I. Paunescu, M. Rusanescu, Student Publishing House, Bucharest 2007, , 2) Acquisition techniques and environmental monitoring, CO Rusanescu, Student Publishing House, Bucharest 2010; and 3) Dynamics and control of pollution, CO Rusanescu, Student Publishing House, Bucharest 2010. , accredited by the NURC Publishing. She published 11 papers in journals, 12 papers presented in national and international conferences and published in proceedings of conferences, nine posters presented at conferences. She is a member of the Romanian Society for Metallurgy (SRM) and the Association of Fracture Mechanics (ARMR) since 2000.