Interface characterization of Al/SiC\textsubscript{p} compacts

Mariana Ciurdas, Daniela Alina Necsulescu, Roxana Marina Solea
Faculty of Materials Science and Engineering, University Politehnica of Bucharest, Spl. Independentei 313, Bucharest, Romania
mariana.ciurdas@upb.ro

Abstract. In the last decade, several authors and scientists are interested more and more to understand the phenomena which are take place at aluminum matrix and silicon-carbide interface. This type of composite material is especially used in aerospace and automotive industries which involve high temperature applications. Aluminum metal matrix composites containing 5,15,25 weight % SiC\textsubscript{p} were processed by powder metallurgy route. The interaction between metallic matrix (aluminum) and particulate ceramic reinforcement (silicon-carbide) was studied. At the beginning the solid-state reaction behavior of the Al/SiC powder mixture compacts was investigated. The density, porosity, compressibility of Al-SiC\textsubscript{p} composites were calculated. Maximum densification of the composite material reinforced with 5% SiC is obtained at a compactation pressure of 420 MPa (94.5%). For 15% SiC content densification increase with higher compactation pressure given the density values of 95.66% and 96.46% for the 25% SiC composite. Conventional scanning electron microscopy revealed a faceted interface between SiC particulate and Al matrix. This chemical reaction at the interface is the result of dissolution of silicon carbide particulates in aluminum matrix during the liquid sintering process at high temperature:1100,1300,1450 and 1750°C in different sintering atmospheres. For intermetallic compounds identification was used an energy dispersive X-ray spectroscopy (EDX) investigation. The presence of compounds and phases such as: Al\textsubscript{2}C, Al\textsubscript{2}O\textsubscript{3}, Al\textsubscript{2}SiC\textsubscript{4}, SiO\textsubscript{2}, Al\textsubscript{2}O\textsubscript{3}, confirmed by X-ray diffraction analysis is given by different sintering temperature. It is important to know the structure and chemical composition of the interface and near to the interface zone for study the mechanical properties of the reinforced metal matrix composites.

1. Introduction
The continuous development of aircraft and automotive industries impose new solutions for new advanced materials. Metal matrix composites represent a promising class of such materials, especially aluminum composites [1].

The composites properties depend on the metal matrix properties and on the reinforced particles form, distribution and properties. Also, the composition and physicochemical properties of the interface between metal matrix and reinforced particles must be known. From many techniques suitable to obtain the metal matrix composites the powder metallurgy is used broaden [2], [3].

The processing of composite materials with Al matrix represents an important and distinct field in the powder metallurgy. It allows us to obtain materials with complex properties, almost impossible to obtain through classical technological processes. At the industrial level, there are several conventional techniques for obtaining aluminum sintering products and aluminum based composites using powder metallurgy, but the most commonly used are: [4], [5]
a. elementary state powder mixing (Al, Cu, Mg, etc) or binding matter - powder mixing with the ceramic particles, followed by (one direction, two direction or isostatical) warm pressing and thermal treatment; by powder mixing, cold isostatic press and warm extrusion/lamination with degasification and specific thermal treatment.

b. these techniques require specialized equipments and specific endowment. Under laboratory conditions there can be obtained composite materials by mixing the elementary state powders or by mixing the binding matter used in the pressing process with State the objectives of the work and provide an adequate background, avoiding a detailed literature survey or a summary of the results. the powder, followed by cold pressing process, controlled atmosphere sintering process and thermal treatment. The obtained composite materials can be acquired at a lower price and can have specific characteristics according to the applications used for.

The studies accomplished so far regarding the obtaining process of the composite materials trough the powder metallurgy based on the nature of the component materials and technologically parameters reveal the fact that there are still many unsolved problems with direct implications upon the properties of these materials. One of the problems encountered is the difficult pressing and sintering of aluminum powders and especially of Al/SiC powders comparing to pressing and sintering of other ferrous or copper powders. The process of cold one-direction pressing of the mixtures of metal-ceramics powders gives rise to densification problems because it requires pressing forces much higher than those used in the case of metal-metal mixtures. Aluminum sintering is difficult because each particle of Al is covered with a thin layer of a very stable aluminium oxide which cannot be reduced during the sintering process and because of that it locks the diffusion process which is necessary for the particles binding process to take place [3]. In order to solve all these problems, we have to be able to fully characterize the composite materials at the microstructural level. Structural information from X-ray powder diffraction can be obtained using the Rietveld method. Rietveld refinement of X-ray powder diffraction data can yield considerable amount of crystallographic information, in addition to quantitative phase estimation. The method itself is a whole pattern fitting least squares technique that uses the entire pattern rather than a limited number of reflections to extract the information required. In this method, the observed pattern for each phase is compared with the calculated one, and any differences between them are minimized by refining structural as well as profile related parameters. Since the method uses all lines, severely overlapping reflections are not a problem. Finally, we will give informations about weight fraction of the present phases, atoms positions in the unit cell, mean crystallite size and mean square microstrain etc.

2. Experimental details
Because of limited information concerning the interaction study taking place at the interface of the Al/SiC system, we have selected Al as a metallic matrix component and SiC powder as reinforced material. The route for processing Al/SiC_p composites were powder metallurgy based on results in using the diffusion process in order to study the chemical reactions between metallic matrix and ceramic reinforcement.

The powder metallurgy processing advantages are: the obtaining of isotropic materials with complex configuration at sintering temperatures that inhibits the interaction of the matrix with ceramic particles. This process implies the restriction of forming specific breakable chemical compounds. The absence of metallic melt or the existence of metallic melt in low quantity excludes or diminish the unreactibility problems [6].

For their good characteristics of wear resistance, roughness, high temperature resistance was made the selection of aluminum matrix-based composites with discontinuous reinforced with rough particles of silicon carbide. We have also considered the possibility to process these materials trough conventional and economic technologies, using the existing equipment and for the low price of the raw material.

The starting materials were: green air atomized, 99.5 % purity AA300 grade aluminum metallic powder of 100-300 µm mean size, delivered by SC Alba Aluminium Zlana, Romania was used as metal matrix and
fine ball milled α SiC green powder with 80-100 μm mean size, delivered by Casirom Turda, Romania. Before mixing, the SiC powder was cleaned and then washed in cold water and dried.

In order to distinguish the physical and chemical processes taking place in the processing of selected systems and to optimize the technological parameters, the ratio of SiC in the composite mixture was 5,15,25 weight % of SiC were homogenized in a TURBULA T2F mixer.

The combination of two materials is mixed in a removable container preventing cross-contamination between batches and dusting when mixing powders. The mixing time for homogenisation was 30 minute for 5 % SiC mixtures and 2 hours for 25 % SiC mixtures with rotational speed of 20 rpm. In order to avoid the agglomeration of the powder and deposition were used rotational speed of 20 rpm. In order to avoid the agglomeration of the powder and deposition were used uniaxial pressing with 200, 240 and 420 MPa. The dimensions of the cylindrical die used for pressing with 200 MPa was h=84,5 mm and d=32 mm, and for pressing with 240 and 420 MPa the dimensions was: h=84,5 mm and d=10 mm. Four sets of green compacts were sintered at different temperature and atmospheres as it shown in table 1.

| Sample      | Sintering temperature (°C) | Sintering atmosphere          |
|-------------|----------------------------|-------------------------------|
| Al-SiC-green| 1100°C                     | Reducing CO                  |
| Al-SiC-green| 1300°C                     | protective gas (Ar)          |
| Al-SiC-green| 1450°C                     | protective gas (Ar)          |
| Al-SiC-green| 1750°C                     | air                          |

The heat treatment of sintering was performed in CO reducing atmosphere at 1100°C, protective gas (Ar) atmosphere at 1300-1450°C and air atmosphere at 1750°C in Siemens-Plania and Balzers IOV 16 type furnaces.

Almost all the researches upon composite materials are focused on the effects associate to the interface because these effects are crucial to the optimization of composites performances and to their processing technology.

Microstructural analysis of the composite powders was studied by scanning electron microscopy and by X-ray diffraction, a FEI XL-30-ESEM Philips electron microscope with EDS analyzer and a DRON UM1 diffractometer in Bragg-Brentano parafocusing geometry with horizontal goniometry. A flat graphite monochromator in diffracted beam improved the pattern resolution and eliminate the Kβ line. The experimental conditions for data acquisition were: radiation type CuKα (λ=1.547 Å), 35 kW, 30 mA, 2θinitial = 20°, 2θfinal = 100°, Δθ step = 0.05°, time of acquisition per step = 8 s. For data acquisition/analysis was used specialized data acquisition software and preliminary pattern analysis software. The presence of reaction’s products and component’s heterogeneity leads to local variations of the charge transfer. Indifferently to the phases that bind together, the interface microstructure presents interesting particularities, having major influences upon mechanical properties of the composite [7].

### 3. Results and discussion

#### 3.1. Morphological changes

The morphological characteristics of the powders it can be observing a uniform distribution of SiC particles in the Al matrix which is an important factor to be monitored in order to obtain better physical and mechanical properties of the final composites.
In order to determine some physical and technological features of the samples, the Reichart metallographic optical microscope with qualitative and quantitative imagine analysis was used. By optical microscopy the following parameters can be determined: particle’s shape, distribution of particles, specific surface and apparent density. Chemical microanalysis and samples morphology were determined using ESEM FEI XL-30 (Environmental Scanning Electron Microscopy) with incorporated EDS analyzer. Figure 1 shows a relatively homogeneous distribution of polyhedral particles and porosity and also some of SiC particles are cracked.

![Figure 1](image1.png)

**Figure 1.** Optical micrographs of Al-25%SiC composite sintered at 1300°C in protective atmosphere (Ar), x200

In the case of aluminum matrix composite material reinforced with silicon carbide particles sintered in protective atmosphere (figure 1) we can observe a non-homogeneous distribution of the SiC particles with formation of an intermediate layer at the interface. According to figure 1 are parts where SiC particles are cracked and some particles are damaged.

![Figure 2](image2.png)

**Figure 2.** Optical micrographs of Al-25% SiC composite sintered at 1450°C in protective atmosphere (Ar), x200

From the optical microstructures of the composite materials sintered at temperature of 1450°C reinforced with silicon carbide particles can be observed changes in the appearance of the area of interaction (reaction) for the purpose of better adhesion at the interface with formation of a layer with relatively constant thickness (Figure 2) which confirms the existence of chemical reactions, the reaction precipitates or compounds.

3.2. Structural evolution
The characterization of Al/SiC green compacts composites is been reveals by density and compressibility changes and dimensional modification in the powder morphology. Material density is an important physical parameter being interrelated with the main mechanical parameters of materials (solids) [8-10]. To determine the effect of reinforcement content the compacts density was measured and compared with the theoretical density of the powder mixtures which represent the maximum
density of material attained in the final stage, and is calculated by the rule of mixtures, after the following relationship:

\[ \rho = \frac{\sum_{i} x_i \rho_i}{\sum_{i} x_i} \]  

(1)

where: \(x_i\) is the fraction component \(i\) in mixture (\(i\) being Al, SiC, APV and Mg stearate) and \(\rho_i\) represent the density of component \(i\) (g/cm\(^3\)).

The theoretical density, calculated with relation (1) obtained for the studied samples were: \(\rho_{\text{Al+5\%SiC}}=2,725\text{g/cm}^3\), \(\rho_{\text{Al+15\%SiC}}=2,775\text{g/cm}^3\), \(\rho_{\text{Al+25\%SiC}}=2,825\text{g/cm}^3\). The physical characteristics of the compacts were determined by measuring and weighting. Densities of the samples obtained after each processing step are given in table 2. The maximum densification it can be observed at 420 MPa pressure in the 25% SiC reinforced samples. Best values of density after compaction are recorded on sample Al-25 % SiC composites. During the compaction process the particles overlap much better; the spaces between the larger particles are filled with smaller particles, thus the volume of pressed sample is reduced. With increasing the compacting pressure was resulted a decrease on the porosity of compacts and also, the shape of particles became much more uniform, hindering the compaction process; the empty spaces among the particles of matrix and reinforcements remain unfilled properly. The processes observed during the cold unidirectional pressing operation confirm the existing data in the literature following the three stages namely: in the first stage of the pressing the particles is rearrange, allowing a better packaging of powders, then the decreasing of densification at intermediate pressures at values up to 80-85% of compaction, actually corresponds to stage II of the pressing, when occurs the contact surfaces increasing between particles by elastic-plastic deformation of the metallic particles, respectively fragmentation of ceramic particles, this increasing in density reveals from compressibility curves of our mixtures (figure 3).

### Table 2. Densities, porosity and compressibility measured after compaction

| Sample       | Pressing pressure MPa | \(\rho_t\) g/cm\(^3\) | Apparent density \(\rho\) g/cm\(^3\) | Compressibility % | Porosity % |
|--------------|------------------------|------------------------|----------------------------------------|-------------------|------------|
| Al - 5%SiC\(_g\) | 240                    | 2,72                   | 2,44                                   | 89,70             | 10,3       |
|              | 420                    | 2,72                   | 2,57                                   | 94,48             | 5,5        |
| Al-15%SiC\(_g\) | 240                    | 2,77                   | 2,47                                   | 89,16             | 10,3       |
|              | 420                    | 2,77                   | 2,65                                   | 95,66             | 4,3        |
| Al-25%SiC\(_g\) | 240                    | 2,83                   | 2,49                                   | 86,70             | 12         |
|              | 420                    | 2,83                   | 2,73                                   | 96,46             | 3,5        |

Processes corresponding to the third stage of pressing were presented in table 2, it is observed that with porosity decreasing and increasing of pressure occurs a corresponding maximum densification of massive plastic deformation and an increased number of contacts between the particles. The porosity for each composition increases with raising of SiC content reaching values of 3.5% (for samples with 25% SiC) at a pressure of 420 MPa and values of 10, 3% (for samples with 5% SiC).
It is known the form filling shaped unidirectional cold pressing of metal-ceramics powder mixtures it create densification problems compared with metal-metal mixtures for these reasons was studied the influence of silicon carbide proportion on the pressing behaviour of the materials, the main results of research can be summarized as follows:

- Analyzing the plotted compressibility curves (Figure 3) we can conclude by the addition in the matrix of the hard and brittle SiC particles, the compressibility decreases. It’s difficult to press the mixtures (higher pressing forces) with increasing of SiC amount and is explained by the fact that hard SiC particles, delay the densification by taking the load for pressing, until their broke, maximum densification taking place by repackaging SiC fragments in aluminum matrix.
- Maximum densification of the composite material reinforced with 5% SiC is obtained at a pressure of 420 MPa (94.5%). For 15% SiC content densification increase with higher pressing pressure given the density values of 95.66% and 96.46% for the 25% SiC composite.

![Figure 3. Compressibility curves for Al matrix reinforced with 5, 15, 25 % SiC compacts](image)

![Figure 4. SEM images of the Al-25%SiC composites after sintering at a) 1100°C b) 1300°C c) 1450°C](image)
From the three experimental mixtures, and based on the results one of the representative contents (Al-25% SiC) was chosen to sintered and to study microstructural and compositional point of view. The structure of sintered sample was investigated by SEM, XRD and illustrated in figure 4 and 5.

![Figure 4](image1.png)  ![Figure 5](image2.png)

**Figure 4.** X-ray diffraction patterns of Al-25 % SiC composites sintered at a) 1100 °C b) 1300 °C c) 1450 °C d) 1750 °C

In table 3 are presented the Rietveld refinement results for the analyzed XRD patterns which demonstrate the presence of reaction compounds and phases identified at the Al/SiC composites interface.

![Figure 5](image3.png)  ![Figure 6](image4.png)

**Figure 5.** X-ray diffraction patterns of Al-25 % SiC composites sintered at a) 1100 °C b) 1300 °C c) 1450 °C d) 1750 °C

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12 ectropositive metal) this liquid, chemically
of the composite material processing. Thus, in the manufacture of composite materials
SiC melt, chemical reac
existing chemical reactions between Al matrix and silicon carbide. According to (3.3)
process, which depends on temperature and time. This time
deceases since aluminum is very reactive (el
concentration; the risk of cracks is very high. In generally some areas is as compact and without
3. As a result of chemical reactions between the matrix and the reinforcement material
form compounds which affect the quality of the composite, due to the emergence of an area of stress
fraction, which obtaining is not a simple process, which depends on temperature and time. This time-temperature dependence is given by the
existing chemical reactions between Al matrix and silicon carbide. According to (2) relationship in Al-
SiC melt, chemical reaction occurs:

\[ 4Al + 3SiC \rightarrow Al_4C_3 + 3Si \] (2)

\[ 4Al + 4SiC \rightarrow Al_4SiC_4 + 3Si \] (3)

Some authors affirm [12], [13] the processing of pure Al – SiC that obtaining is not a simple
process, which depends on temperature and time. This time-temperature dependence is given by the
existing chemical reactions between Al matrix and silicon carbide. According to (2) relationship in Al-
SiC melt, chemical reaction occurs:

\[ Al + SiC \rightarrow [Al-Si] + Al_4C_3 + SiC \] (4)

where liquid aluminum matrix will become the type Al-Si matrix, which depends on the temperature
of the composite material processing. Thus, in the manufacture of composite materials Al/SiC
interfacial reaction between Al and SiC depends on production parameters: temperature, atmosphere
and chemical composition of the matrix and reinforcement material.
The reactions at the interface can be: the dissolution of the SiC in Al; precipitation of a phase III; segregation of the third phase at the interface. The reaction products formed at the interface are fragile and under certain conditions may affect in negative way the properties of the composite. For our experimental conditions we can observe the following reactions produced at 1300°C and 1450°C in the same sintering atmosphere (protective):

\[
\begin{align*}
4\text{Al} + 3 \text{SiC} & \rightarrow \text{Al}_4\text{C}_3 + 3 \text{[Si]} \text{ reaction at the Al} - \text{SiC interface} \quad (5) \\
4\text{Al} + 4\text{SiC} & \rightarrow \text{Al}_4\text{SiC}_4 + 3 \text{[Si]} \text{ reaction at the Al} - \text{SiC interface} \quad (6) \\
\text{SiC} + \text{Al}_4\text{C}_3 & \rightarrow \text{Al}_4\text{SiC}_4 \quad (7) \\
\text{Si} + \text{Al} & \rightarrow (\text{Al}, \text{Si}) \text{ with Si diffusion in matrix and adsorption in aluminum matrix} \quad (8) \\
4\text{Al} + 3\text{O}_2 & \rightarrow 2\text{Al}_2\text{O}_3 \text{ reaction at Al-O interface (pores)} \quad (9) \\
\text{Si} + \text{Al} & \rightarrow \text{Al}_2\text{Si} \text{ with Si diffusion in matrix without adsorption in aluminum matrix} \quad (10)
\end{align*}
\]

Therefore, it is recommended to conduct partial reactions at the interface by controlling the thickness of the reaction zone. Increasing the reaction layer is limited by diffusion processes of the elements, the silicon carbide particles and the molten aluminum above its melting temperature (up to 1100°C).

Areas subject to EDS chemical microanalysis investigation to spot focused areas: matrix, interface, and silicon carbide particles. The aluminum content (table 4) from aluminum matrix is less than 100%.

**Table 4.** EDS analysis of Al-SiC composite sintered at temperatures of 1100 °C, 1300 °C, 1450 °C and 1750 °C

| Temperature | Zone       | Sample | Al, % weight | Si, % weight | C, % weight | O, % weight |
|-------------|------------|--------|--------------|--------------|-------------|-------------|
| 1100°C      | Matrix     | Al-25 %SiC | 85.23        | 1.14         | 6.33        | 7.29        |
|             | Interface  | Al-25 %SiC | 67.61        | 2.92         | 0           | 29.48       |
|             | Particle   | 1.01     | 75.89        | 22.56        | 0.54        |
| 1300°C      | Matrix     | Al-25 %SiC | 83.05        | 8.93         | -           | 8.02        |
|             | Interface  | 71.11    | 5.69         | -            | 23.20       |
|             | Particle   | 0.87     | 75.02        | 23           | 1.12        |
| 1450°C      | Matrix     | Al-25 %SiC | 88.32        | 7.36         | 2.96        | 1.37        |
|             | Interface  | 40.74    | 33.86        | 6.33         | 19.07       |
|             | Particle   | 0.60     | 73.97        | 25.43        | -           |
| 1750°C      | Matrix     | Al-25 %SiC | 90.21        | 5.94         | -           | 3.85        |
|             | Interface  | 74.26    | 0.67         | -            | 25.07       |
|             | Particle   | 55.52    | 2.50         | 7.46         | 34.52       |

This content can be explained by the diffusion phenomena, dissolution and / or reaction of silicon and carbon in the matrix. The amount of oxygen in the aluminum matrix can explained due to the reaction of aluminum with air provide from the pores of the material. The presence of silicon and carbon in the metal matrix cannot be explained only by the decomposition of the silicon carbide particle.

Estimated reaction products and intermetallic compounds are: Al, Al$_2$O$_3$, (Al, Si), Si, Al, Al$_4$O$_4$C, 3Al$_2$O$_3$3SiO$_2$, SiO$_2$, α Al$_4$SiC$_4$, SiC$_{14}$SiC$_{11}$, Al$_4$C$_3$ which are indentified by XRD analysis (figure 5). The detailed EDS / SEM analysis (figure 6-8) has revealed the interface morphology between SiC particles and aluminum matrix. As shown above, with the increasing of temperature, the composites phase structure becomes more complex, with the apparition of new interphases and a good interface zone.
In the paper were investigated the influence of the reinforcement content, the sintering parameters and conditions on interface between Al/SiC composites obtained by powder metallurgy route can be concluded:

- the technical applications and benefits have been studied for first time the behavior of composite materials of Al-SiC at high temperatures (1100, 1300, 1450 and 1750°C) by powder metallurgy (sintering in the liquid phase). Composites were made in different environments, namely: reducing (CO), inert (Ar) and oxidant (air). Following the characterization of the three types of composites with different contents of one polytypic state (α SiC polytype I, II) silicon carbide (5, 15, 25%) and obtained was found that the strongest interaction takes place in the composites material with composition of 75% Al and 25% SiC. By optical and electron microscopy analysis have showed the composites structure where the SiC particles are dispersed in the Al matrix.
the EDS analysis effectuated on a concentration line has showed the soft shape the interface layer at the interface of the Al/SiC composite. The XRD analyses have established the composition of the interface layers. From the phase analysis we were able to identify the following phases: at $1100^\circ$ C: Al, Al$_2$O$_3$, SiC (polytype II); at $1300^\circ$ C: Al, SiC (polytype II), Si; at $1450^\circ$ C: Al, SiC (polytype II), Si; at $1750^\circ$ C: Al, Al$_2$O$_3$, Al$_2$O$_4$C, Al$_2$C$_3$Si, SiC (polytype II), SiC (polytype I). Sintered powders in reducing atmosphere have led to the apparition at the interface of an Al$_2$O$_3$ layer which acted as a diffusion barrier [14], [15].

Acknowledgment(s)
The article is financed by the University Politehnica of Bucharest, through the project “Engineer in Europe”, online system, registered at the Ministry of Education under no. 140 / GP / 19.04.2021, by using the fund to finance special situations that cannot be integrated in the form of financing institutions state higher education.

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