
# Fabrication and characterization of $Ti_3SiC_2$ composites by Spark Plasma Sintering

Miguel Lagos[1*], Charlene Pellegrini[2], Iñigo Agote[1], Maria Parco[1], Laura Silvestroni[3], Diletta Sciti[3]

*1 TECNALIA, Industry and Transport Division, Mikeletegi pasealekua 2, E20009 Parque Científico y Tecnológico de Gipuzkoa, San Sebastián, Spain; 2 Ecole Nationale Supérieure de Céramique Industrielle (ENSCI) 87100 European Ceramics Centre, Limoges, France, 3 CNR-ISTEC, Institute of Science and Technology for Ceramics, Via Granarolo 64, I-48018 Faenza, Italy*

**Corresponding author: miguel.lagos@tecnalia.com*

***Highlights***

- Densification of composites of $Ti_3SiC_2$ with carbon fibers.
- SPS parameters for full densification.
- Microstructure and mechanical properties of the obtained materials.
- Interaction between the fibers and the matrix

## 1. Introduction

MAX phases are layered carbides or nitrides with crystal structure of hexagonal symmetry. These materials combine ceramic and metallic characteristics, including high electrical and thermal conductivities, machinability, damage tolerance and good thermal shock resistance [1, 2]. Among MAX phases, Titanium silicon carbide ($Ti_3SiC_2$) has roughly half the density of the alloys used in today's jet engines and maintains its creep resistance even at higher temperatures [2]. In addition, its biocompatibility in combination with the previously mentioned mechanical properties makes $Ti_3SiC_2$ a promising candidate for bioceramic composites [3]. Moreover, $Ti_3SiC_2$ can be used as a ceramic binder in cutting tools [4].

In order to improve the properties of $Ti_3SiC_2$ different composites have been developed increasing the hardness and the fracture toughness of the material [3, 5]. As reinforcement different materials like SiC, TiC, SiC wiskers, etc have been used [6]. In this work, $Ti_3SiC_2$ is reinforced with carbon fibers in order to reduce the density and increase the thermal shock resistance at high temperature.

## 2. Methods

$Ti_3SiC_2$ powder (particle size >15 microns, KANTHAL) was dry mixed with carbon fibers (maximum length 500 microns, mean diameter 11 microns, Nippon Graphite Fiber Corporation) to obtain composites with a fiber content of 5, 20 and 40 vol%. The consolidation of the composites was performed by Spark Plasma sintering. The powder was loaded into a graphite die in between two graphite punches. During the tests, the chamber was maintained under vacuum ($10^{-2}$ Pa). Temperature was controlled by a pyrometer that measured the temperature in the interior of the graphite punch. Maximum temperature was 1390 ºC with a holding time of 5 min. The load (50 MPa) was applied before the heating cycle was started.

Density was measured by Archimedes method using a Mettler AE 240 weight balance and porosity was analyzed according to the standard UNE-EN ISO 4605:1978. Microstructure and semi-quantitative chemical composition were analyzed by optical and SEM microscopy (Zeiss Nvision 40 and Zeiss EVO50). XRD analysis was carried out using a BRUKER D8 ADVANCE apparatus (Cu_Kα radiation, $\lambda$ = 0.154 nm, 2$\theta$ =20–100°, 40 kV–30 mA, scan step 0.1°). 4-point bending tests were performed in an INSTRON machine according to the standard EN-843-1:2006.

## 3. Results and discussion

It was possible to obtain a complete densification of the composite with the 5 and 20 vol % of carbon fibres. Density values close to the 99 % of the theoretical value were achieved. With the 40 % of carbon fibers the density was around the 94 %. Figure 1 presents the microstructure of one composite with the 40 vol% of carbon fibres. Fibers are distributed within the matrix but some agglomerations can be observed. The porosity is linked to the agglomeration of the fibers.

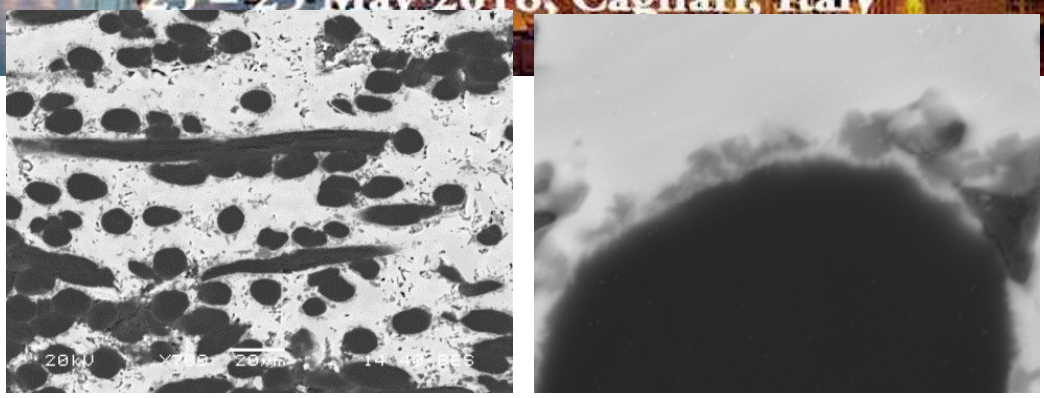

**Figure 1.** Microstructure of the Ti3SiC2 composite with 40 vol% of carbon fibres. Magnification X500 (left), Magnification X5000 (right)

In addition, it is possible to observe a reaction zone between the matrix and the fibers. The EDX analysis of the reaction zone confirms a Si rich phase with an important amount of carbon. It is known from the ternary Ti-Si-C system equilibrium diagram at 1300 ºC [7] that $Ti_3SiC_2$ can lose Si in a carbon rich environment forming Si (g) and TiC. This Si (g) can react with C forming SiC at the surface of the fibers [7]. In SPS the reaction time is very short and it helps to the relatively low reaction of the fibers with the matrix. XRD analysis of the composites was also performed to confirm the obtained results. Regarding the mechanical properties of the obtained composites, bending tests were performed at room temperature. A reduction of the strength was observed with increasing the amount of reinforcement. However, with 20 % of fibres, bending strength was around 400 MPa, similar to conventional CMCs. In the material with 40 % of fibres, the strength was lower, this was caused by the porosity. A detailed explanation about the microstructure, crystallographic phases and mechanical properties will be presented.

## 4. Conclusions

This work presented the densification of $Ti_3SiC_2$-$C_f$ composites by Spark Plasma Sintering. It is possible to obtain fully dense composites up to 20 vol% of carbon fibers. There is a reaction between the fibers and the matrix, but the damage of the fibers is not very significant due to the short processing time of SPS. Mechanical properties were also explained according to the amount of carbon fibers. In the future, other interesting properties like thermal shock resistance will be measured.

## Acknowledgements

This work has received funding from the European Union's Horizon 2020 "Research and innovation programme" under grant agreement N°685594 (C3HARME).

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The reference format is provided below [1 – 3]. [Times New Roman 10].

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***Keywords***

Insert a maximum of 4 keywords separated by ";". [Times New Roman 10].