

# MATRIX CONSOLIDATION MECHANISM ON THE Ti/SiC/C COMPOSITES PRODUCED BY CONTINUOUS BINDER-POWDER COATING

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**Abstract.** Matrix consolidation mechanism has been investigated in the Ti/SiC/C composites produced by Continuous Binder-Powder Coating – CBPC. Titanium metal matrix composites reinforced with continuous SiC/C filaments (SM 1145<sup>+</sup>) were analysed in different densification conditions. The results showed that during processing, densification occurs by several mechanisms including a complex elasto-viscoplastic flow and diffusion bonding. The matrix consolidation depends on many processing conditions such as pressure and temperature, mainly. Using correct conditions of pressure and temperature, the titanium matrix composites produced by this process present a good matrix consolidation, without porosity and a weak interaction between matrix and fiber. These good agreements between matrix consolidation and weak chemical interaction between matrix and fibre are obtained when pressures up to 150 MPa and temperatures below 1600°C are applied. In these conditions, supplementary heat treatments can be performed either in alpha or beta domains.

**Keywords:** Metal-matrix composites; Consolidation mechanisms; Binder-powder processing.

## 1. Introduction

The matrix consolidation mechanisms have been investigated in different manufacturing techniques for titanium metal matrix composites (Kim *et al* 2004; Schüler *et al*, 2000; Choo *et al.*, 2002; Even *et al*. 2001; Nicolaou *et al*. 1995). During the last decade, performance improvement of these materials has been carried on by aeronautic and aerospace industries. Based on the part shapes and the method of metal/matrix coupling, various fabrication routes have been developed in industrial field (Ward-Close *et al*. 2000; Osborne *et al*. 2001; Warrier *et al*. 1995; Jin *et al*. 2004; Xun *et al*. 2000). Independently of the manufacturing technique, in all pressing conditions, the carbon protective coating of these continuous reinforcements (SiC/C) tends to react with matrix during consolidation temperatures above 800°C, leading to the deterioration of mechanical and physical properties of the resulting composites, as reported e.g. in Ward-Close *et al*. (2000). In order to optimize the matrix consolidation without damage the SiC/C reinforcements, it is necessary that the thermomechanical behavior of each processing is well understood. Different theoretical models have been proposed (Kim *et al* 2004; Schüler *et al*, 2000) to understand and justify the matrix consolidation. Kim *et al*. (2004) showed that the matrix consolidation depends on many processing conditions such as pressure, temperature, and geometrical factors as fiber arrangements. According to Schüler *et al*. (2000) the influence of fiber volume fraction is slight in the range of 10-50% by volume. The only effect appears to be a shift in the maximum consolidation rate to higher levels of density. The thermal effects were analogically compared by Choo *et al*. (2002) as a mechanical loading (hydrostatic tension), because the lattice expansion of matrix and fiber during heating. In order to avoid the fiber degradation produced either by pressure (bended and broken) or temperature (chemical reactions), the mechanisms of matrix consolidations is analyzed and discussed in this article based on these thermomechanical models. Particularly, this article reports the effects of the pressure and temperature during matrix consolidation of Ti/SiC/C produced by CBPC or Continuous Binder-Powder Coating. As previously reported by Sanguinetti Ferreira *et al*. (2005), CBPC process is an alternative fabrication route, which the fibers are coated by impregnation of a mixing binder/powder producing a concentric binder-powder layer. After coating, the fibers are laid-up and hot pressed in a tungsten carbide mould, for matrix consolidation.

## 2. Experimental methods

Titanium Matrix Composites (TMC) were processed by using the CBPC method according which, silicon carbide fibers (SM 1140<sup>+</sup> in the present contribution) is continuously coated by a mixture of polymer binder and titanium powder. The binder is a polymeric solution consisting in poly-methyl methacrylate (PMMA) dissolved in acetone, at 0,20g/ml composition ratio. In this process, a commercial Ti-powder (99,5 % at), having a sponge aspect and large distribution of grain size with an average value of 100  $\mu\text{m}$ , was used in this process. After acetone cleaning, the fibers are dipped into the binder solution and pulled out vertically, passing through a coating diameter limiting the binder deposited layer to about 150  $\mu\text{m}$ . This layer regulator was specially developed for the CBPC apparatus. During these operations, both the acetone during cleaning and the polymeric solution (binder) during coating are maintained at ambient temperature. Then, the fiber is run through a container of titanium powder for gluing titanium grains on the polymeric coating (fig.1).

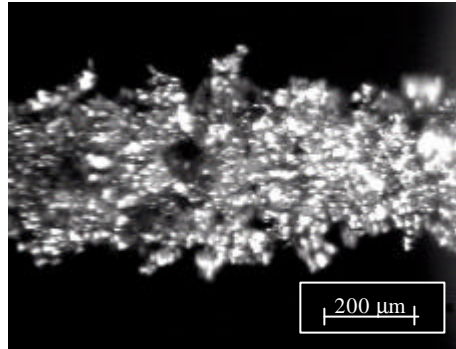


Figure 1. Binder-powder coated fiber.

During all the coating process, the fiber running speed is maintained closed to 70-75 cm/min. Finally, the coated fibers are dried. After this first step of TMC processing related to the preparation of a semi product, the coated fibers were cut into short lengths (around 42 mm) and aligned in a tungsten carbide (WC) mould for composite consolidation. Thin Ti-sheets about 40  $\mu\text{m}$  thick, were placed above and below of the coated fibers, allowing initial arrangement control. The composites were processed under vacuum hot pressing (VHP) in a specific apparatus where the tungsten carbide tooling was placed inside a HF furnace. Under secondary vacuum, better than  $10^{-5}$  Torr, heat and pressure were applied following the thermomechanical cycle depicted in figure 3.

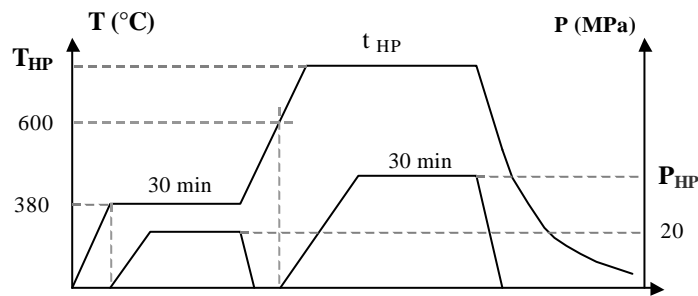


Figure 2. Termomechanical cycle used to produce TMC by CBCP process.

For eliminating the binder, the composite was initially heated up to 380°C at a rate of 20°C/min and maintained at this temperature during 30 minutes for degassing. Once the binder was eliminated at 380°C, a pressure of 20 MPa was applied for at least 20 minutes and then relaxed. This weak pressure was progressively applied at a rate of 5 MPa/min. Then, the rate of heating was maintained at 20°C/min until the temperatures of hot pressing (650, 700, 750 and 800°C). During heating, when the temperature reached 600°C, the pressure was again applied at a rate of 5 MPa/min up to 100 or 200 MPa; allowing the control of the fiber arrangement and avoiding damage or breakage of fiber. This pressure was maintained for 30 minutes in T<sub>HP</sub> condition for matrix grains flowing, creeping and consolidation. After hot-pressing, specimens were slowly cooled inside the furnace.

Samples for microstructural characterization were prepared following classical metallographic techniques. Different diamond liquids were used during polishing. The microstructure was observed using optic (OM) and scanning electron microscopes (SEM).

### 3. Results and Discussion

Using the coating conditions leading to a binder/powder layer of 400-420  $\mu\text{m}$ , gives rise after hot-pressing to TMC presenting a fiber volume fraction of 14-16%, depending on the consolidation pressure. An increase in volume fraction can be obtained with a smaller diameter of binder layer regulator.

Particular care has to be taken during processing when the maximum pressure is applied. As a matter of fact, the fibers arrangement can be seriously disturbed because of irregular coated surfaces. During hot-pressing, at pressure the SiC-fibers were bended and broken when the pressure was rapidly applied. This damaging effect is not only occurring for the highest consolidation pressures but was also commonly observed for rather small pressures. For instance, fiber breakage was systematically induced by pressure application at rates higher than 5 MPa/min, even though the final pressure is smaller than 180 MPa. This effect can be explained by considering that for high rates of pressure increase, the isostatic compressive component of the stress field is insufficient, compared to the stresses induced by sharp points of Ti-powder (Sanguinetti Ferreira *et al.* 2005). Tacking into account these preliminary considerations, rates of pressure increase were limited at 5 MPa/min and the maximum pressure was lowered from 200 MPa to 150 MPa. Following the conditions previously defined (Sanguinetti Ferreira *et al.* 2005), allowed an acceptable distribution of reinforcements to be obtained.

Results showed that depending on the pressure and temperature conditions, significant porosity in matrix can be produced, particularly, in lower temperatures even if an elevated pressure is applied. For temperature of around 650°C, grains in vicinity of the fiber show that the matrix consolidation is not completely achieved (fig. 3). After 30 minutes in this hot pressing condition, the plastic flow transforms the sharp-pointed grains into equiaxed grains.

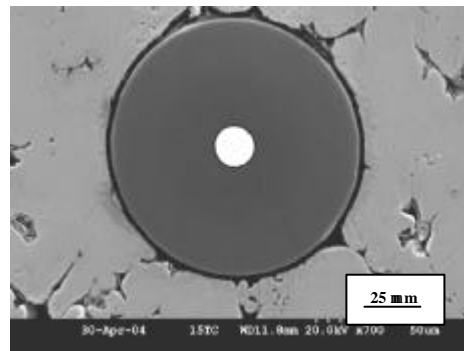


Figure 3. Microstructure of composites processed up to 200 MPa for 30 min at 650°C.

On the other hand, in zones nearby the upper surface, fiber damage is always produced by elevated pressure. Fissures can be produced both in carbon protective coating and fiber core, as has been previously shown and discussed (Sanguinetti Ferreira *et al.* 2005). If a pressure of 100 MPa is applied during densification, the fibers are not blended or broken.

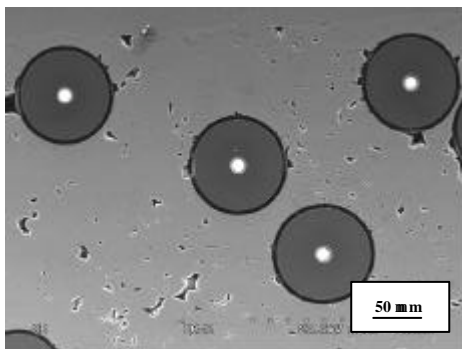


Figure 4. Microstructure of composites processed up to 100 MPa for 30 min at 700°C

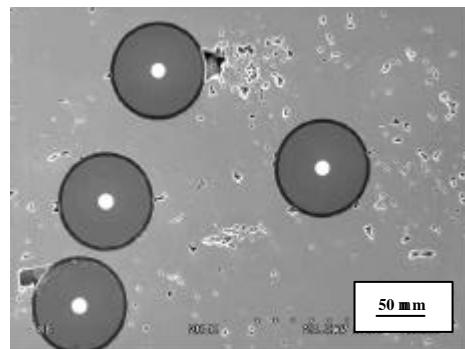


Figure 5. Microstructure of composites processed up to 100 MPa for 30 min at 800°C.

Nevertheless, the matrix is not well consolidated, even though the temperatures increase from 650 up to 800°C. As illustrated (fig. 4 and 8), poor ameliorations were produced when the temperature increased from 700 to 800°C. Porosity is always observed in the matrix. A good compromise between matrix consolidation and weak chemical reaction is produced when a pressure of 150 MPa and temperature of 800°C are applied during 30 minutes. Under this condition, the diffusion bonding increases producing a good matrix consolidation (Fig.6), while the chemical reactions at interface are limited a few nanometers; as previously explained by analysis (Sanguinetti Ferreira *et al.* 2005) performed in Auger electron spectroscopy (AES).

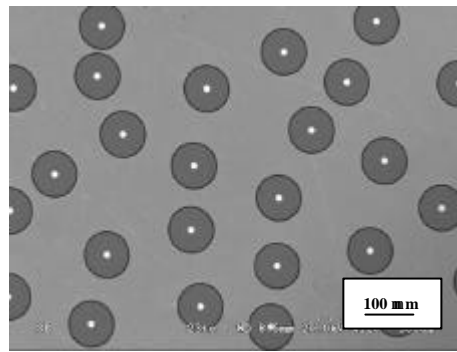


Figure 6. Microstructure of composite processed up to 150 MPa during 30 min at 800°C.

Generally, the matrix consolidation has been described in TMC studies (Kim *et al.* 2004; Schüler *et al.*, 2000; Choo *et al.*, 2002; Even *et al.* 2001; Nicolaou *et al.* 1995) by using of unified mechanical models, despite different mechanisms concerned; above all at the beginning of the densification cycle. For TMC produced by Continuous Binder-Powder Coating, special attention must be made, particularly when heat and pressure are applied in vacuum hot pressing (VHP) for porosity elimination. Results showed that SiC/C fibers are broken if a high pressure (greater than 200 MPa) is applied. In the other hand, in low pressure (100 MPa), a poor matrix consolidation is always produced, even if the processing occurs at elevated temperature.

In CBPC process, results showed that both pressure and temperature are important parameters for matrix consolidation, contrarily to fiber-foil-fiber process in which the temperature has a less important role, as compared with the pressure (Kim *et al.* 2004; Schüler *et al.*, 2000). Despite of the differences between binder-powder coating and fiber-foil-fiber manufacturing conditions, the final results concerning the matrix consolidation are in agreements (Kim *et al.* 2004; Schüler *et al.*, 2000). For all temperatures between 700 and 800°C performed in CBPC process, both the plastic deformation and the diffusion bonding (as consequence) are weak and insufficient for porosity elimination, if a pressure of 100 MPa is applied.

Using the foil-fiber-foil manufacturing technique, Osborne *et al.* (2001) explained the porosity of the matrix, hot pressed in similar condition, considering the residual stress which arises during cooling. Because of the difference in coefficients of thermal expansions between SiC fiber and Ti matrix, porosity can be formed (Osborne *et al.* 2001). Even though the interface reaction is weak, these differences in thermal expansion cause an inherent residual stress in matrix, in both the normal and radial directions, which contributes to porosity in matrix. Choo *et al.* (2002) showed that in transverse direction, particularly, the matrix and the fiber expand independently over the whole temperature range, indicating that the thermal load is not shared. Dudek *et al.* (2002) explained the voids formation (porosity in reaction zone) based only on the volume contraction produced by chemical reaction on the interfacial zones. In our manufacturing conditions, the porosity cannot be attributed to volume contraction (chemical reaction) or differences in thermal expansion. In absence of chemical reactions (see fig. 4, 5 and 6), this weak consolidation condition can only be explained by changes in the stress field during hot-pressing. When the pressure is slowly applied and reaches to 100 MPa, the sharp points of the grains contribute to plastic deformation. The local stress in these sharp points is elevated, holding the plastic flow. After a number of minutes at 100 MPa, the sharp points are fully deformed and the grains became approximately equiaxed, as shown in figure 3. In this deformation condition, the distortion component (deviatoric) of the stress field tends towards zero, so the isostatic stress field becomes only the hydrostatic stress field, stopping the plastic deformation.

C. Even *et al.* (2001) explained the behavior of matrix consolidation in similar conditions, based on model which considers the elasto-viscoplastic deformations of the matrix. It has been shown that for a same pressure and a fixed grain size, no changes in matrix densification are meaningfully produced when the temperature increases from 600 up to 700°C. The model proposed by C. Even *et al.* (2001) may explain how after 20 minutes of hot-pressing at 100 MPa, no changes in matrix densifications were produced. Using CBPC manufacturing technique, it was necessary an increasing of the pressure to achieve the matrix consolidation. A good compromise between pressure (150 MPa) and

temperature (800°C) was obtained during matrix consolidation, without fiber degradation. In this condition, neither fiber breakages produced by pressure nor excessive chemical reaction produced by temperature were observed as previously shown (Sanguinetti Ferreira *et al.*, 2005). These results concerning the matrix consolidation are in agreement with Schüller *et al.* (2000). Through the use of finite element model (FEM), these authors showed that for pressure of 100 MPa and temperature of 800°C, 30 minutes is a critical value of time for matrix consolidation. Contrarily, using a pressure of 150 MPa at the same temperature, 30 minutes is sufficient to achieve full density (fig.6). Because of this weak interaction fiber/matrix during CBPC process, supplementary heat treatments can be performed both in alpha and beta domains, considering the microstructure ameliorations, if a titanium alloys is used.

Experimental and theoretical results show that differences of fiber volume fractions have a little influence on the matrix consolidation, particularly in fiber-foil-fiber process. Similar results concerning fiber volume fraction can be attempted in CBPC process. Nevertheless, it has already shown (Nicolaou *et al.*, 1995) that the fiber redistribution, resulting from a swimming process during matrix consolidation, may cause significant degradation in mechanical properties of TMC. So, further investigations are necessary to verify the influence of the fiber distribution in Ti/SiC/C composites produced by Continuous Binder-Powder Coating (CBPC).

## 5. Conclusion

Titanium Metal Matrix Composites can be produced by CBPC- Continuous Binder-Powder Coating. Pressure and temperature are the most important parameters in CBPC process during matrix consolidation. SiC/C fibers are broken if a high pressure (greater than 200MPa) is applied. In the other hand, if a low pressure (100 MPa) is applied, poor matrix consolidation is always produced, even though the processing occurs at elevated temperature. A good compromise between pressure and temperature (150 MPa at 800°C) can be obtained during matrix consolidation, without fiber degradation produced by pressure or excessive chemical reaction produced by temperature. Considering the weak interactions matrix/fiber produced during hot-pressing, supplementary heat treatments can be performed either in alpha or beta domains without damage meaningfully the reinforcements.

## Acknowledgements

Authors thankfully acknowledge Brazilian research agency **CNPq** – Conselho Nacional de Desenvolvimento Científico e Tecnológico for financial support for this work.

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