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TITANIUM MATRIX COMPOSITES

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ABSTRACT

Titanium matrix composites reinforced by continuous carbon fibres were prepared, using powder metallurgy and for part, techniques derived from ceramic and polymer matrix composites processing methods. Major difficulties encountered during fibres/matrix coupling and composites consolidation, were overcome through specific studies related to preparation of fine titanium powder, impregnation of carbon fibre yarns by a slurry containing matrix powder in a viscous transfer fluid made of polymeric binder in solvent, thermal elimination of binder, as well as mechanisms of consolidation and densification of titanium matrix under pressure and moderately high temperature.

KEYWORDS

A: Metal matrix composites, A: Carbon fibres, D: Scanning Electron Microscopy, E: Powder processing.

1. INTRODUCTION

The need of satisfying more and more specific functions during design of high performance structures leads to conceive multimaterials such as selectively reinforced composite parts. For instance, the need for missile applications of thin wall structures combining particularly high specific buckling strength, that requires excellent specific bending stiffness, and noticeable failure energy, leads to design titanium plates with a reinforcement only located in the vicinity of their upper and lower surfaces. Indeed, reinforcing only 50% of a plate thickness by stiff fibres placed in cross section area where bending strains are maximal,

enables 90% of the bending stiffness related to a completely reinforced plate, to be reached.

According to another presentation, it is possible to compare the bending stiffness of partly reinforced plates (E^*) and that of completely reinforced plates (E_c) by keeping constant the total volume of fibres in the plates. The effect of concentrating the fibres near the surface of the plate is illustrated in figure 1. For instance, if the fibres homogeneously distributed in a plate whose V_f is 30%, are concentrated in 40% of the plate thickness near its surfaces ($k=0.6$), the ratio E^*/E_c reaches 1.9, that to say a 90% increase in bending stiffness (point A in fig.1).

In the case of structures made of titanium, which already offers high specific mechanical performance, rigidity has to be improved, particularly at high temperature, to satisfy the need of the previously evocated space application. Stiffening of 1mm thick plates requires very thin high modulus fibres. The use of reinforcements such as SiC filaments obtained by chemical vapour deposition (CVD) is inadequate, since the related diameters are too large (100-140 μm) for concentrating stiffening effect in some tenth millimetres of titanium plates thickness. Besides, high modulus thin fibres, exhibiting a sufficiently good chemical compatibility with titanium, are not yet available [1]. Thus, high modulus carbon fibres can be considered as presently remaining good candidates for selective reinforcement of small titanium thickness, provided to control the vigorous titanium/carbon reactivity and to set up a suitable processing method.

Aiming at this objective, attention had been focused on showing the feasibility of titanium matrix composites (TMC) reinforced by continuous thin carbon fibres. Vapour, liquid and solid routes of processing were tested by various authors according to different methods for manufacturing titanium alloy matrix composites, but only few of them are adaptable to thin reactive fibres. As a matter of fact, very small size of carbon fibres obliges a thin subdivision of matrix for interpenetrating fibres while reaching a noticeable volume fraction. Thus, FFF (foil/fibres/foil) solid route processing method which requires both matrix foils thickness close to fibres diameter and distances between fibres allowing solid titanium flow, can be

used with big SiC_{CVD} filaments. However, it is unsuitable for incorporating 7 µm diameter carbon fibres. Similarly, liquid matrix droplet sizes related to plasma spray are too large for infiltrating fibres bundles [2, 3]. Furthermore, the process is not able to insure a sufficiently rapid extraction of heat to prevent fibres degradation [4]. This difficulty is even more accurate for other processing techniques based on liquid route. A sufficiently rapid cooling is the more difficult to be achieved that titanium thermal conductivity is relatively poor [5-9]. Otherwise, physical vapour deposition (PVD) offers simultaneously an excellent subdivision of matrix and a possibility of fibres/matrix coupling at low temperatures which prevents any fibres degradation [10-13]. However, process kinetics of most PVD techniques are not rapid enough to forget the cost parameter which is also affected by necessity of spreading reinforcement in case of fibre bundles [14]. Besides, powder metallurgy which has been mainly used for ceramic and intermetallic matrix composites as well as reinforcement of titanium alloys by particles, whiskers or short fibres, combines many advantages provided to overcome some major difficulties [15-26]. When on the one hand, adaptation of PM to continuous fibre reinforced composites under the so called powder cloth method, is suitable for refractory materials, that is for decreasing processing temperatures which is quite interesting for Ti/C coupling; on the other hand, powder fineness required for incorporating matrix within fibres bundles is difficult to obtain if the material is ductile and very reactive as it is the case for titanium.

In order to explore this last route, the present contribution has focused on the adaptation of both a processing method based on powder metallurgy and several features related to polymer matrix composites processing techniques.

2. COMPOSITES PROCESSING METHOD

The method which has been studied for processing continuous carbon fibre reinforced titanium matrix composites (CFRTMC) can be schematically defined by the following steps:

- preparing a slurry made of a mixture of titanium powder and dissolved polymer used as binder and as viscous fluid allowing the powder to be transferred within fibrous preforms,

- impregnating fibre bundles, fabrics or more generally fibre preforms by the slurry,
- shaping specimens or parts from formable fibrous elements placed for instance in moulds,
- drying preforms for solvent elimination and pre-composite handling,
- eliminating the polymeric binder at moderate temperature under vacuum,
- consolidating preforms at high temperature under compression in order to facilitate composite matrix densification by plastic and creep deformation of powder grains.

This processing method can be adapted to either unidirectional compression or hot isostatic pressing (HIP), but requires in any case the availability of non contaminated titanium powders sufficiently fine to be incorporated within fibre bundles and to prevent any fibre mechanical degradation during compression steps.

3. POWDER/FIBRES COUPLING

When using the powder cloth method for performing brittle matrix composites, association between fibres and matrix is not a major problem, when matrix brittleness enables elaboration of apparently viscous slurry exhibiting a very fine distribution of powder. That is particularly the case of most ceramic and intermetallic matrices for which it is possible to obtain, without too much difficulty, very fine powders by chemical route or by grinding. In contrast, the chemical reactivity of titanium and its great ductility are important handicaps with respect to the cloth powder method.

3.1. Conditions of matrix powder incorporation within fibre bundles

Some methods based on dry route were used for processing polymer and aluminium matrix composites [27-30]. However the related methods require both fibre bundles spreading and the use of electrostatic effects, without systematically resulting in homogeneous distributions of fibres. Furthermore, if powder exhibits a great reactivity, the cost of methods based on dry route becomes exorbitant because of security equipment requirements.

Besides, using a liquid vector can be helpful for incorporating powder within fibre bundles or preforms, provided:

- (1) the liquid vector physical properties are able to make fibres yarns spreading,

(2) the stable and quasi homogeneous powder/liquid suspension can be elaborated and
(3) the slurry's apparent viscosity can be adjusted to facilitate fibre bundles impregnation [31,32]. In addition, liquid vector must not contaminate powder and must be capable to be completely eliminated without using excessive temperatures. If these conditions can be satisfied, fibre bundles have only to be run into slurry or fibrous preforms has to be impregnated by slurry, to obtain powder incorporation [33]. It is worthy of note that in the second case, the ratio between liquid vector viscosity and apparent slurry viscosity is an important parameter of impregnation. The higher this ratio, the lower the filtering effect of powder by the fibrous architecture is.

Otherwise, it is obvious that for a given architecture, powder incorporation is only possible if powder grain size is smaller than inter-filament distances authorised by fibrous preform architecture. This requirement is less strict in the case of fibre bundles which are able to spread, but powder grain size also controls inter-filament distances that is to say composite fibre volume fraction. This correlation is so important that influence of powder grain size on quality of impregnation has been studied elsewhere [34]. Indeed, L. Tang and al. have proposed a model which enables composite fibre volume fractions to be determined as a function of powder grain size for given fibre diameters. For instance, according to this model and as shown in figure 2, for carbon fibres whose diameter is about 7 μm , obtaining a fibre volume fraction of 30% would require a powder grain size of about 15 μm , whereas using a 35 μm grain size leads to hope a reinforcement of only 12%. These considerations show how crucial is the availability of non contaminated fine titanium powder. However, before tackling this difficulty, preparation conditions of slurry, whose liquid vector and binder could be completely eliminated, had to be investigated.

In such a way, slurries were prepared by using acetone as fluid vector whose viscosity could be controlled by dissolving a small amount of pure polymethacrylate of methyl (PMMA). This polymer also plays the role of binder for coupling powder grains and fibres, in order to

make possible composite handling as a prepreg, before binder elimination immediately followed by the high temperature treatment of consolidation.

3.2. Elimination of organic binder

Pure PMMA was chosen among various polymers which were investigated as binders. Indeed, polystyrene (PS), polyoxyethylene (POE), polydimethylsiloxane (PDMS) and styrene-butadiene-styrene (SBS) were also considered, but, thermogravimetric analyses have shown that most of these polymers give rise to slight amounts of residual carbon which are apparently completely eliminated at 380°C in the case of PMMA (fig. 3). However, despite these positive results obtained for PMMA by thermogravimetry after short times of thermal exposure, embrittlement effects could be pointed out by tensile tests on consolidated titanium powder about 25 μm in size. It has been observed that the longer the binder elimination treatment, the higher the consolidated material ductility, which is of about 5% after 2 h of heat treatment under vacuum and can exceed 6% after 5h. Since a much more significant embrittlement effect may be expected from the incorporation of carbon fibres, PMMA binder was used for a first approach of this type of titanium matrix composites. As a matter of fact, prior to focusing on improvement of matrix ductility, it was necessary to assess the concept validity of carbon fibres reinforced titanium matrix composites.

4. CONDITIONS OF CONSOLIDATION AND DENSIFICATION OF COMPOSITES

Since the main advantages of processing methods based on powder metallurgy (PM) deal with moderate temperatures and duration of composite densification and consolidation, processing condition studying was oriented toward the use of high compaction pressures.

4.1. High pressure processing

In order to manufacture quasi net shape titanium parts, hot isostatic pressing of titanium alloys was studied during the 80th and has demonstrated possibilities of decreasing significantly processing temperatures thanks to the application of high pressures. For instance, when conditions generally used for Ti6Al4V densification are 870-925°C during 2 h under

105 MPa, it is possible to lower temperatures of about 250°C and to limit densification times at 15 min., by multiplying pressures by 8 [35-41].

These results previously reported in literature have shown that processing of titanium matrix composites by powder metallurgy could be performed at temperatures as low as 600-650°C during half an hour with pressures of about 300 MPa. A first confirmation of the related feasibility was achieved with the help of rough model and numerical simulation of densification mechanisms which combine successively quasi instantaneous plastic deformation of powder grains, followed by their creep and sintering. The model is based on plastic and then creep deformations of compact stacking (fcc) of identical powder grains and on a rather short contribution of metal transfer by diffusion. Despite important simplifications of the model described elsewhere [42], the numerical simulation of powder compaction at elevated temperature as illustrated in figure 4, has shown that the main contribution to densification is related to initial plastic deformation. The creep contribution is rather moderate and quasi achieved after 10 minutes for 200 mesh powder 20 minutes for 400 mesh powder. Finally, the level of compaction under high pressure at elevated temperature is so high that sintering contribution during one hour is negligible. As an example, for powder of 200 mesh (average size of about 60 μm) the two predominant mechanisms lead to predict a theoretical density of 99.7% after half an hour of compression under 600 MPa at 600°C and 98.5% after one hour under 100 MPa at 700°C (figure 4 and table 1). It is worthy of note that when on the one hand, pressure and temperature are not predominant parameters in the range of respectively 100-600 MPa and 600-700°C, on the other hand, the finer the powder, the more difficult the densification by powder grain deformation is.

Besides the previous considerations, experimental validation of the processing method feasibility with titanium specimens and the perspective of manufacturing CFRTMC specimens required the set up of specific hot compression equipment [42]. The unidirectional feature of the apparatus compared to an isostatic system often used in industry, is able to

induce beneficial shearing and allows in the present case pressures up to 1000 MPa at 1000°C to be reached, provided dies are able to undergo these conditions.

Conditions of matrix densification and consolidation were studied with disks of 12 mm in diameter and 2 mm thick. Specimens were hot pressed by a tungsten carbide piston in a cylindrical tungsten carbide die. Applied pressures range from 20 to 600 MPa, and temperatures from 550 to 750°C. Various powder grain size distributions corresponding to 0-10 µm, 10-30 µm, 30-45 µm, 50-75 µm and 100-150 µm, were tested in first series of experiments to point out the influence of this parameter for composite processing.

4.2. Determination of matrix densification conditions

The processing efficiency was assessed through evaluations of specimen density, hardness and intergranular controlled ductility, in order to validate the feasibility of high pressures/moderate temperatures densification of titanium powder and to optimise the processing parameters. Specimen densities were measured by using Archimedes method with a precision of about 0.1% related to mass measurements. Hardness was determined by Vickers macro indentation and ductility was approached thanks to four points bending tests. Because of the small specimen size, a specific bending device was used with the exploitation method described elsewhere [43].

A first series of experiments with two types of powder size (200 and 400 mesh) of ex hydride commercial titanium (purity 99,9) has confirmed theoretical predictions (although powder grain shape was not spherical) and except in case of the highest pressure (600 MPa). For this high pressure, a Poisson's effect related to the piston generates a friction induced reduction of pressure effectively applied on powder. Despite this effect, table 1 has shown a rather good agreement between predicted and experimental results.

These previous results, as well as those related to other powder sizes and reported in table 2, confirm the negative influence of powder fineness on densification: the smaller the powder size, the smaller the densification rate.

Nevertheless, hardness values also reported in table 2, show that the influence of powder fineness could be attributed, not only to lower efficiency of plastic and creep deformations in the case of smaller spherical powder grains but, also to microstructure refinement or/and titanium contamination by interstitial impurities. As a matter of fact, the smaller the powder grain size, the finer the consolidated titanium microstructure results, the higher the titanium powder specific surface submitted to all types of contamination is, and the higher the corresponding increases in yield strength and brittleness are.

Concerning temperatures, pressures and hot compression times, table 1 was not able to depict their influence for further processing conditions optimisation. Thus, a designed experiment method had to be used with a larger domain of parameter values as defined in table 3.

Experimental results related to a 400 mesh powder (average grain size: 25 μm) are reported in table 3. They lead to the following remarks:

- the influences of hot compression time on density and hardness are not significant in the domain of study. It is not surprising since contributions of creep and sintering mechanisms are expected to be according to predictive numerical simulations,
- on the contrary, putting up pressure in the range of 20-200 MPa, gives rise to high increases in density and hardness. Also, the lower the temperature, the stronger the increase of density as pressure is raised. These remarks confirm the important role of grain deformations in densification mechanisms,
- similarly, the smaller the pressure, the more significant the density increase when raising temperature is, which confirms again the predominance of the initial plastic deformation mechanism in densification; indeed the higher the temperature, the lower the titanium yielding strength.

The whole theoretical and experimental results previously presented have led to consider that processing titanium matrix composites by powder metallurgy at moderate temperature (600-700°C), for a short time (30-60 min.) should be possible provided pressures higher than 100 MPa (200 MPa) are applied. However, these processing conditions must not generate any

substantial fibre/matrix unfavourable degradations: mechanical interactions due to high pressures or /and chemical interaction leading to an interfacial zone whose brittleness might induce premature composite failure.

5. COMPOSITES ELABORATION AND EVALUATION

5.1. Processing parameters

According to the general procedure briefly described in section 2, with the densification conditions approached in section 4.1 and using the binder and its conditions of elimination defined in section 3, CFRTMC were elaborated at 600, 650 and 700°C according to the temperature T and pressure P cycles illustrated in figure 5. All specimens were elaborated from a 400 mesh commercial titanium powder (purity: 99.9). The carbon fibrous reinforcement was chosen after trying Cu/W based diffusion barriers and studying carbon/titanium interaction at moderate temperatures [44, 45]. From these studies, it has been concluded that artificially interposing a very thin continuous TiC layer between Ti and C would be the best solution for impeding the natural uncontrolled formation of angular crystallites of TiC. In this direction and awaiting the availability of TiC coated C fibres yarns obtained by reactive CVD on a pyrolytic C pre-coating, FT500 carbon fibres coated by 40 nm of pyrolytic carbon were used (560 GPa of rigidity and 3610 MPa of strength). The aim of the C pre-coating was to protect the reinforcement from natural TiC formation which was expected to remain limited for the composites processing conditions [45].

5.2 Composites properties

With 400 mesh titanium powders, that is to say average grain sizes of about 25 μm , only small volume fractions ($V_f \text{ max}=12\%$) of 10 μm diameter C fibres could be obtained as predicted by the model illustrated in figure 2. Consequently, strong increases in performance could not be expected with V_f of about 8-10%, since the theory of mixture for a ductile titanium matrix reinforced by brittle high modulus carbon fibres, leads to estimate that no strength improvement cannot be obtained for V_f lower than a critical value of about 8%. However, the experimental values obtained through tensile tests and reported in table 4 are

disappointing since only 12% of rigidity increase was obtained with a fibres volume fraction of 11% whereas the rule of mixture leads to expect a rigidity increase of 45%. As regards to ductility, the presence of brittle carbon fibres within the ductile Ti matrix (30% of elongation) was obviously a source of embrittlement, although the matrix remains ductile as illustrated in figure 6. Any way, the matrix ductility drop is significant and generates some decrease in strength whereas a slight increase in yielding strength (8.5%) can be already related to a V_f of 11%. Although, a deeper analysis of the experimental results is required to explain the rather disappointing performance of the elaborated CFRTMC, several remarks can be made and will be developed elsewhere:

- (1) the fibre matrix interaction is limited to the pyrolytic C pre-coating as illustrated in figure 7. The small pyrolytic carbon layer is transformed into TiC crystallites forming a thin discontinuous TiC layer of about 100 nm in average thickness whose corresponding notch effect on fibres should not be dramatic,
- (2) the noticeable discrepancy between expected and measured rigidities may be related to (i) fibres breakage induced by high pressure or/and high speed of pressure application, (ii) fibre disorientations enhanced by the strong graphite anisotropy,
- (3) the disappointing performance of the elaborated CFRTMC particularly its small strength has mainly to be related to insufficient fibres volumes fractions which are determined by the used powder grain size.

To increase fibres volume fractions, very fine titanium powders (1-3 μ m in size) prepared by grinding brittle commercial Ti hydride powders have been used and allowed the elaboration of CFRTMC containing about 30% fibre volume fractions. The related composites illustrated in figure 8 exhibit rather good reinforcement distributions but also an excessive brittleness. Fractographic analyses of this 30% CFRTMC have shown similar limited fibre/matrix interaction as already illustrated in figure 7. Furthermore, the absence of dimple on the matrix rupture surface, leads to consider the composite brittleness is mainly related to interstitial contamination of the fine titanium powder either by residual hydrogen or by oxygen.

6. CONCLUSION

Carbon fibres reinforced titanium matrix composites could be manufactured through the adaptation of the “powder cloth” method while using specific conditions to take into account the great chemical reactivity between titanium and carbon. Matrix consolidation and densification temperatures could be decreased until 650-700°C for 1h, thanks to the application of high pressures of about 200 MPa. The feasibility of the proposed processing method is strongly dependant upon the ability to obtain very fine and non contaminated titanium powders whose morphology and size monitor achievable reinforcement volume fractions. Such a method induces very moderate fibre/matrix interaction and consequently limits reinforcement degradation. However, tensile tests performed on specimens with low and higher fibre volume fractions ($V_f=7-11\%$ and $V_f=30\%$) showed that rigidity and strength could not be completely transferred into composite material behaviour. This might be attributed to various causes such as matrix embrittlement and non negligible fibre degradation or/and disorientation, which have to be more accurately investigated.

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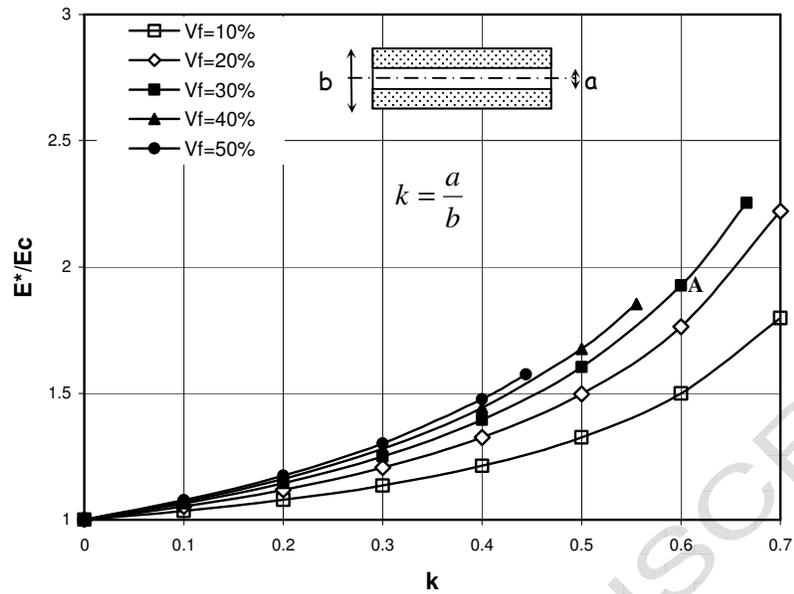


Fig. 1- Evolution of a the ratio between partially reinforced plate bending stiffness E^* and completely reinforced plate bending stiffness E_c as function of the fraction k of unreinforced plate thickness for various average volume fraction V_f of reinforcement

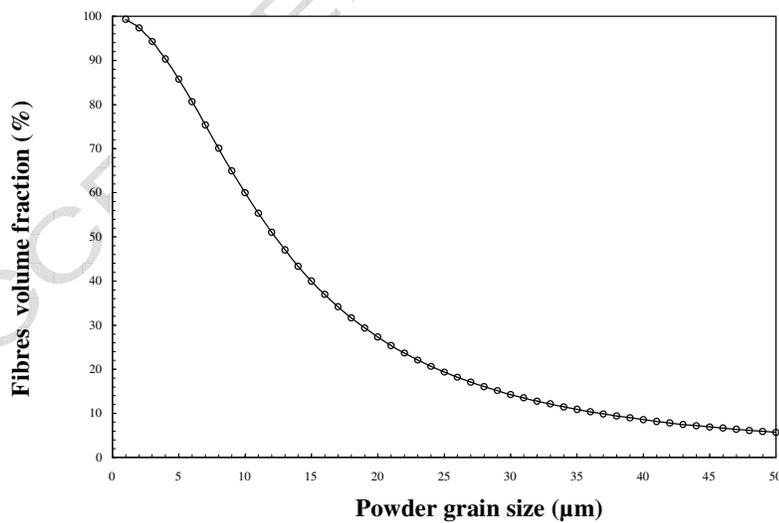


Fig. 2- Correlation between the powder grain size and the achievable volume fraction of 10 μm diameter fibres.

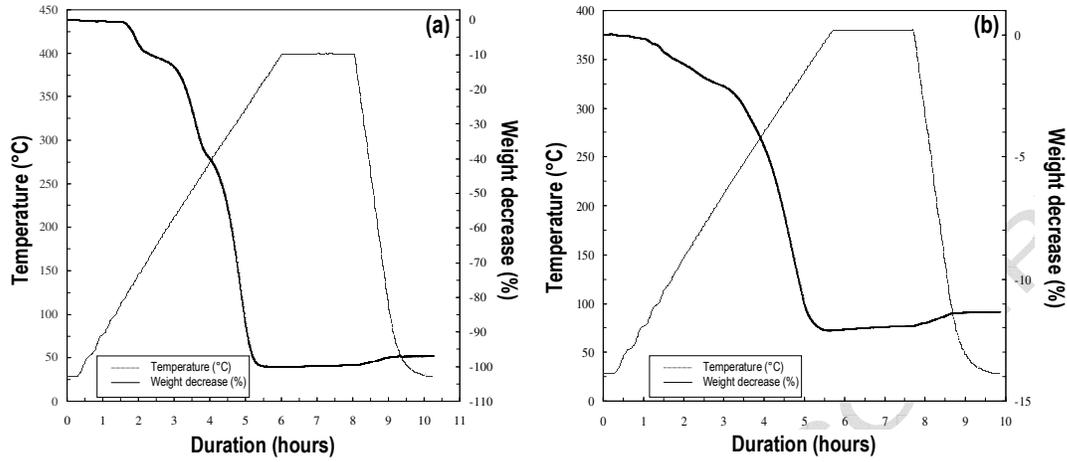


Fig. 3- Thermo gravimetric analyses of (a) pure PMMA and (b) a resulting prepreg.

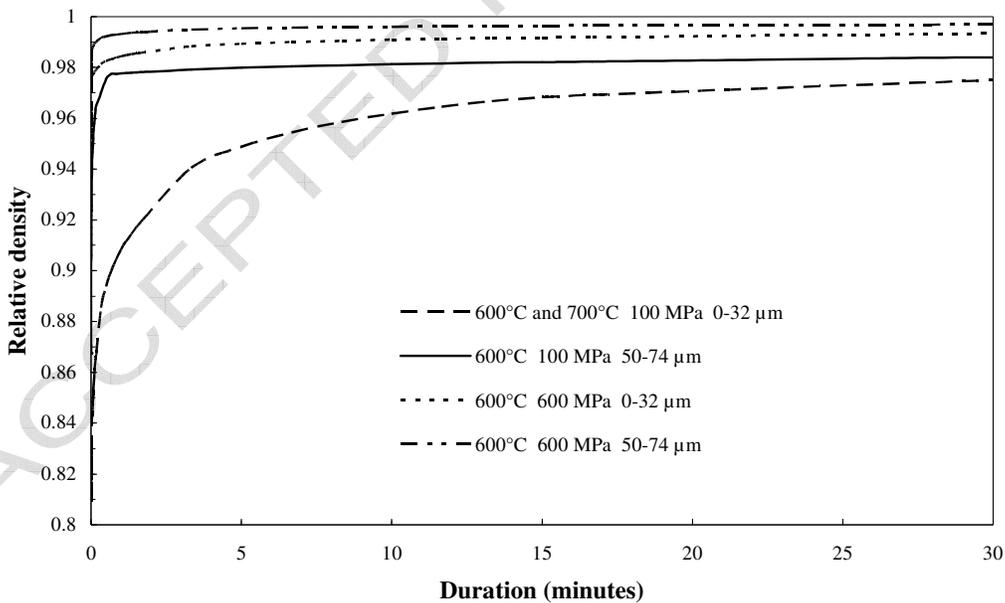


Fig.4- Computed compacted powder density as function of hot compression time

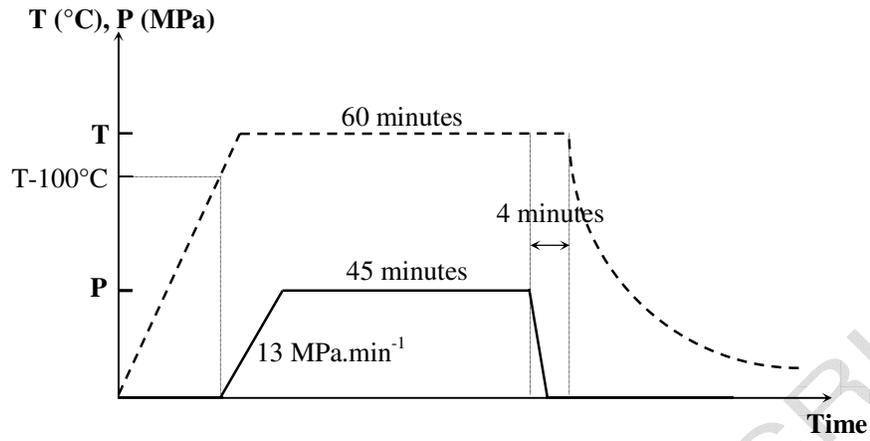


Fig.5- Temperature and pressure cycles of CFRTMC processing procedure

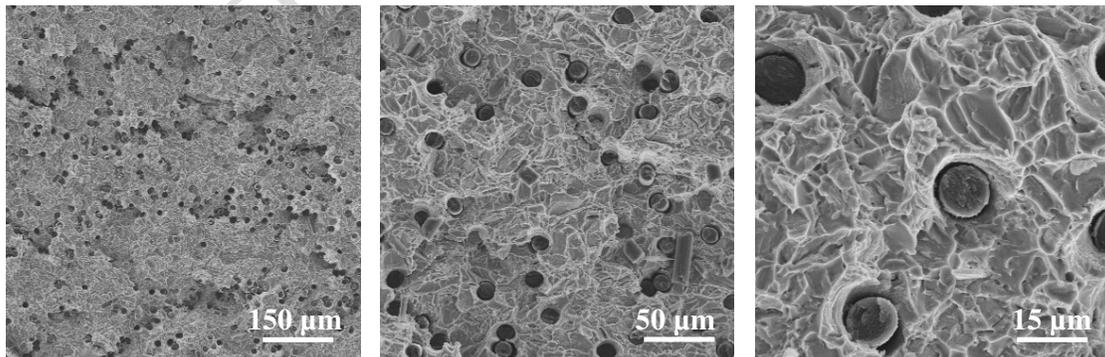


Fig.6 - Fractography of CRFTMC containing 8% vol. of C fibres (failure strain 13%)

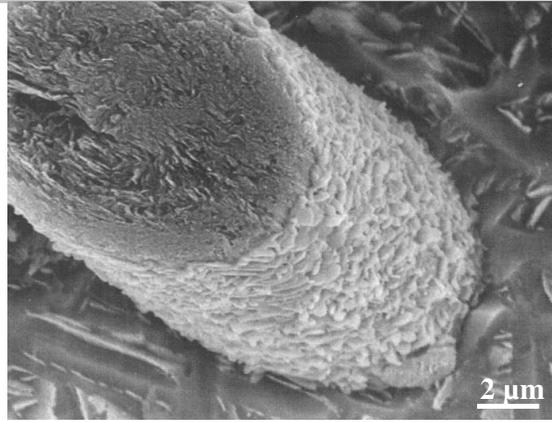


Fig.7 – SEM micrograph of TiC crystallites formation at C fibre/matrix interface after 1h of compression at 700°C

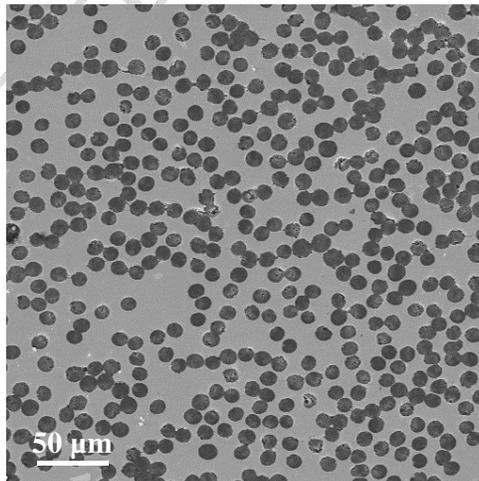


Fig. 8 - Micrograph of CRFTMC containing 35% vol. of C fibres

TABLES

Temperature (°C)	Pressure (MPa)	Time (min.)	Powder size (μm)	Measured density	Theoretical density	Deviation (%)
600	100	30	From 0 up to 32	97.4	97.50	-0.1
	600			97.4	99.34	-1.9
	100	60		97.3	97.71	-0.4
	600			97.4	99.46	-2.1
700	100	30		97.6	97.54	0.1
	600			97.2	99.32	-2.1
	100	60		97.4	97.71	-0.3
	600			97.2	99.44	-2.2
600	100	30	From 50 up to 74	98.9	98.40	0.5
	600			98.6	99.70	-1.1
	100	60		98.9	98.55	0.4
	600			98.5	99.75	-1.3
700	100	30		98.7	98.37	0.3
	600			98.6	99.70	-1.1
	100	60		98.9	98.53	0.4
	600			98.8	99.74	-0.9

Table 1- Theoretical and experimental densities obtained after hot compression

Powder size (μm)	Density (%)	Hardness (HV)
0-10	97.5	297
10-32	96.5	228
32-44	97.2	202
50-74	98.2	117

Table 2- Influence of the powder size for the following experimental conditions:

600°C; 200 MPa; 30 min.

	Temperature (°C)	Pressure (MPa)	Duration (min)
Upper value	750	200	120
Central value	642	63	86
Lower value	550	20	20

Table 3- Study domain of processing parameters

Temperature (°C)	Pressure (MPa)	Time (min)	Density (min)	Hardness (HV)	Bending yielding strength (MPa)
550	20	20	64.4	155	
750			87.3	121	
550	200	20	98.8	180	330
750				204	612
550	20	120	69.2	135	
750			92.3	123	
550	200	120	94.0	205	657
750			99.0	214	574
642	63	86			

Table 4- Density, hardness and yield strength related to specimens hot pressed from a 400 mesh powder