

## Chapter 2

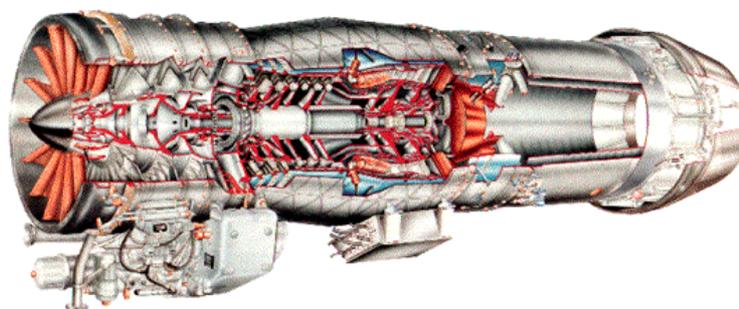
### The Ti6Al4V/SiC<sub>f</sub> composite

#### 2.1 Introduction

A great effort has been made in the last 20 years to develop new composites with Ti-alloy matrix reinforced by long ceramic fibres [1-7], which result equipped unidirectional with high-performance. About that the research and development about specific components are still active in many countries and few technical details are published in this regard.

In a conventional aircraft engine, most of the weight depends by mechanical components as rotors or anchorages, that a lot of parts is in the rotation during work condition. So it would be desirable to decrease the weight of the external elements to reduce the radial contribution. An high savings about this weight it is possible to have by the composites, that reduce the need for heavy structures through their high strength along the circle by optimal radial property.

.The Ti6Al4V-SiC<sub>f</sub> composite has proved a promising material for aeronautical applications including turbine components and structural high stressed parts (Fig.1), and however that applications including high thermic-mechanical stresses.



*Fig.1 Application for the composite Ti6Al4V-SiC<sub>f</sub>*

To value the real suitability of the material, in this section several studies have been carried out especially about the fiber /matrix interface. In fact mechanical behaviour is governed especially by the fibre-matrix interface where diffusion of atomic species and

chemical reactions may occur leading to structural instability. So the main target of this work was to assess how mechanical and micro-chemical properties are affected by long-term heat treatments.

Moreover an important part of the experimental study has been the comparison between two type of the aforesaid composite, different about their specific fabrication process. This situation has been developed about not only the structural aspects, but also a cost analysis.

## 2.2 State of the art

Since the real and the potential Ti6Al4V-SiC applications are attractive for both industrial and scientific aim, especially in recent years various studies about the chemical, thermal mechanical characterization of this material has been developed.

The studies have been directed mainly to microstructural analysis of the fiber-matrix interface and to the diffusion kinetics about the same area, trying to predict and to monitor any changes in the mechanical properties of the material.

In most cases the situation has been studied by SEM and TEM micrograph analysis, or XPS and AES technical, adopted on the perpendicular plane to the major axis of fiber. In particular firstly there the interaction between C and Ti at moderate temperature (between 400 and 750 ° C) has been reported. At these temperatures during short heat exposure time, there is the formation of a titanium carbide compound TiC, controlled by the diffusion of Ti in C [8]. The study element diffusion evaluated through the XPS spectrum, the analysis of the thickness of the carbide layer and in particular the comparison with the more general diffusion theories, have reported discrepancies in the results [8].

These discrepancies appear to be due to difficulties about the assessing the real value of the carbide thickness and the activation energy for the diffusion of C in TiC, higher in this case than for the diffusion C-Ti [8].

The complexity of the diffusion phenomena increases logically on the interface zone between the fiber and the matrix of a composite material. First, by the literature this situation seems to be confirmed, i.e. the formation of titanium carbide increase with the temperature. The presence of SiC fibers coated with a carbon layer also includes a complex diffusion that involves more elements and especially the aforesaid carbon layer degradation. A situation, can cause the contact Ti-SiC and the formation of elements such as Ti<sub>5</sub>Si<sub>3</sub>. In fact, while with the presence of the carbon layer it is possible to note the

presence of only TiC in the form of thin particle, with the following contact SiC-matrix layer, adjacent to the matrix and composed of a mixture of TiC particle and  $Ti_5Si_3$ , forms near the already constituted TiC layer. This fact means that both the C that the Si take part in the diffusion reactions [9].

In the reaction zone presence of elements apparently “unrelated” were detected, too. In fact on the fiber oxygen high concentrations has been identified especially by Auger spectroscopy, but also the existence of high-temperature empty ( $\sim 950$  °C) in the same zone, due to a vacancy saturation [10]. To this point, about the interesting zone quantity of solid precipitates and solutions is a detectable, involving an hardness increase and ductility reduction of the areas on the matrix immediately adjacent to with the fiber, with the consequent influence about the ability to transfer load from matrix to fiber [10].

After the measures of the elements present at the interface and their concentration, the diffusion kinetics evaluation is fundamental. Generally, a study of this kind begins by observing the thickness of the reaction zone by optical microscopy [11]. In this case the difficulties, however, are caused by the irregular nature of this area especially in the part devoted to the matrix. That's why you choose an “average” value thickness is placed.

It is known primarily as the formation rate of the reaction zone grows very rapidly with the temperature increasing of a heat treatment [11]. This growth can be characterized, as shown in [4], by a not constant value of the diffusion coefficients during the heat exposure time . This situation first is due to the interaction between the outer layer of carbon fiber and titanium (with formation of titanium carbide), then to the interaction with the SiC (with the formation of more complex reaction ) that produces a second zone that may be related to different diffusion phenomena, as soon as the aforesaid layer is consumed [11].

About aluminium and vanadium, alloy elements in the matrix, they seem to be detected in the interface only in very little amount (especially vanadium can be found at the  $Ti_5Si_3$ ). However it results demonstrated that the rates of growth are lower in this case than that in the fiber- pure Ti diffusion, reflecting the influence of these factors about the kinetics of reactions.

### 2.3 Property and production

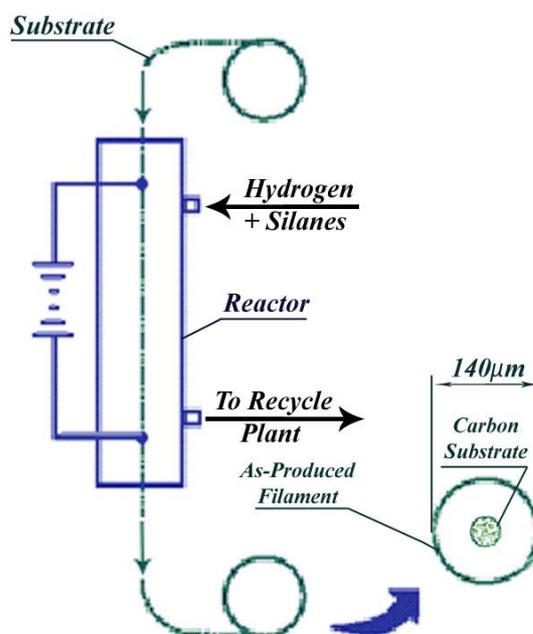
The studied material is a composite constituted by a titanium matrix in Ti6Al4V alloy, used in the 90% of the aerospace and aeronautical applications. About the micro-

structural aspect, this alloy is a mixed phase  $\alpha+\beta$  (with few percent of  $\beta$  phase), where the  $\alpha$  phase is a {hcp} phase,  $\beta$  a {fcc} phase. About the additional elements in the alloy (Al and V), they act especially as stabilizing for the two phase into the titanium alloy: Al is a substitutional element in the  $\alpha$  phase, V is an interstitial element in  $\beta$  phase.

Ti	Al	V	Fe	C	O <sub>2</sub>	N <sub>2</sub>	H <sub>2</sub>
Balance	5,5÷6,76	3,5÷4,5	<0,25	<0,08	<0,2	<0,05	<0,015

*Tab.1 Ti6Al4V chemical composition*

About the SiC fibers, these are as long fibers reinforcement. The fibers, usually denominated by the mark SCS-6, have a diameter of 140  $\mu\text{m}$  and they are produced by PVD deposition, where the SiC accretion occurs on a carbon precursory (Fig.1).



*Fig.1 PVD technology production to the SiC fiber (type SCS-6) fabrication*

The final microstructure consists in a fiber constituted by three concentric parts: a first part is the internal carbon core of the fiber; the second is a silicon carbide (by two different phases) part; the third is a carbon coating by a thickness of 3  $\mu\text{m}$  ( this part has used to increase the strength of the fiber by a curing of surface defects). The fiber structure is showed in Fig.2.

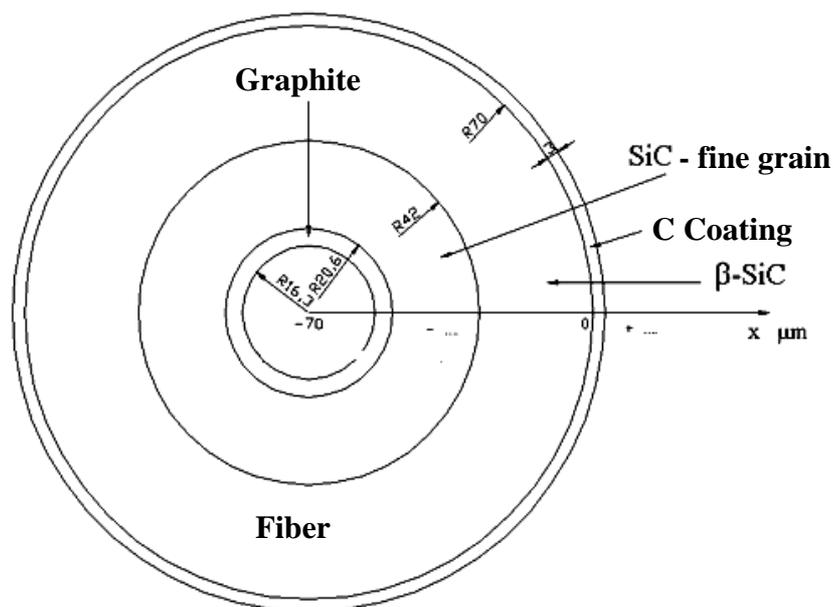


Fig.2 SCS-6 fiber microstructure on the transversal section

The fiber property are summarized about the following points:

- Not too expensive costs
- High strenght
- High resistance to high temperatures (up 1200÷1400 °C)
- Low density
- Excellent chemical and corrosion resistance
- Good wettability by metals

As summarizing, in the Tab.2 is possible to see the principal mechanical and thermic property for the two components of the studied composite.

Matrix Ti6Al4V		Fiber SiC-C (SCS-6 type)	
Density	4,5 g/cm <sup>3</sup>	Density	3 g/cm <sup>3</sup>
Tensile Strength, Yield	830 MPa	Diameter	140 μm
Modulus of elasticity E	114 GPa	Modulus of elasticity E	400 GPa
Tensile Strength, Ultimate	980 MPa	Tensile Strength, Ultimate	4 GPa
C.T.E.	9 ppm/K	C.T.E.	4 ppm/K

Tab.2 Mechanical and thermic property for the components of the Ti6Al4V-SiC<sub>f</sub> composite

The composite has produced at C.S.M. laboratories in Castel Romano (Rome). It has been fabricated by hot isostatic pressure (HIP) in a multi-step process. Preforms (450 x 200 mm<sup>2</sup>) made of four layers of unidirectional SCS-6 fibres alternated with five Ti6Al4V

sheets (thickness of 0,6mm for the external, 0,1 for the internal sheets) have been prepared. They were then put inside a AISI 304 steel die, which was then evacuated ( $3 \times 10^{-6}$  mbar) and sealed. HIP cycle (showed in Fig.3) has been realized by a hot isostatic press ASEA-QH21, which permits to control independently temperature and pressure. The material (Fig.4) was kept at 1163 K for  $1,8 \times 10^3$  s under a pressure of 1200 bar; after cooling to room temperature the panels were extracted from the die. Finally by that for the experimental tests samples (with dimension of about 10 mm x 20 mm) have been obtained by spark erosion wire cutting (Fig.5).

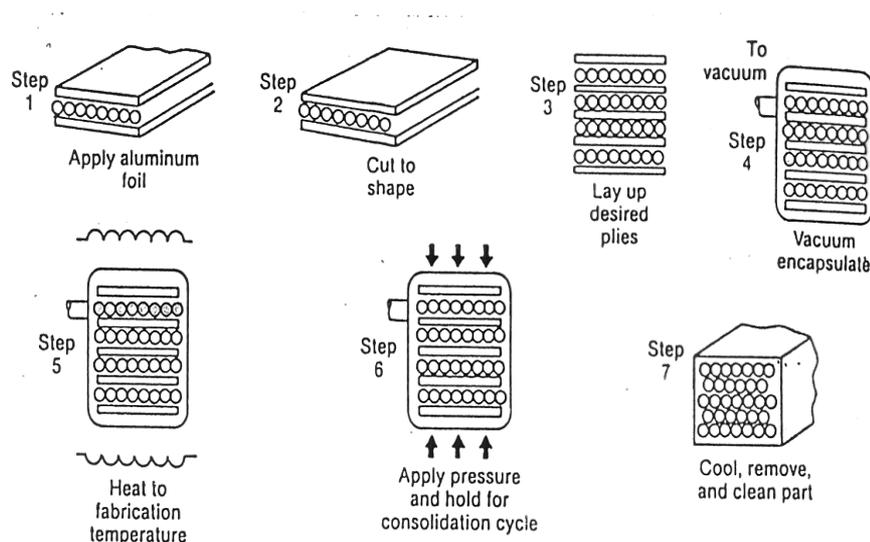


Fig.3 H.I.P. (Hot Isostatic Pressure) process to the fabrication of the composite

$T_{\max}$ (°C)	$P_{\max}$ (bar)	Time stop die (min)
890	1200	60-90

Tab.3 Process parameters for the HIP production

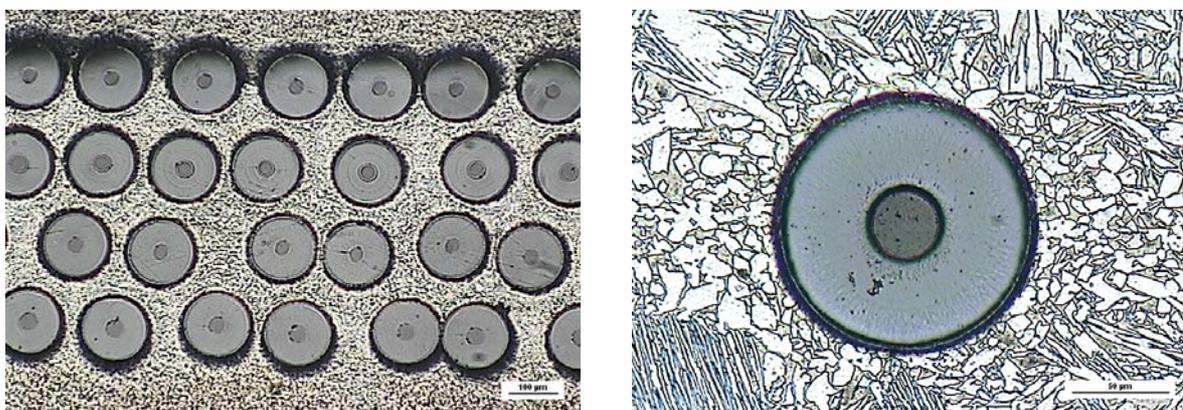


Fig.4 Ti6Al4V-SiC<sub>f</sub>: result of the HIP process and particular micrograph



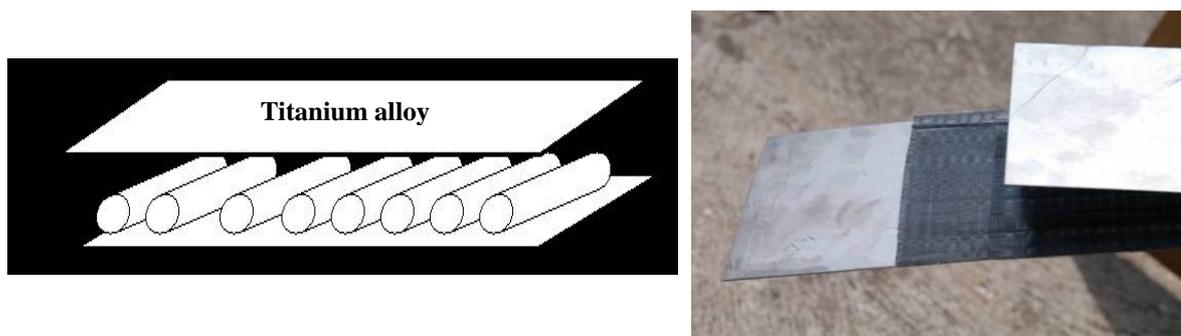
*Fig.5 Samples of the composite obtained to the experimental tests*

About the internal friction probes, samples constituted by only one layer of fibers have been tested, cause to the necessary vibrational characterization of the sample during the starting test.

The experimental development of this work has regarded the analysis and the studies also about the Ti6Al4V-SiC<sub>f</sub> composite produced by Roll Diffusion Bonding, thanks to a innovative pilot project realized at the C.S.M. laboratories. Details are present in [12].

The process consists in the manufacturing of composite samples reinforced by the unidirectional SCS-6 fibres, by the mean of a semi-continuous/continuous approach. The matrix is constituted by Ti6Al4V sheet.

The materials are assembled as a lay-up of two metallic sheets between that one layer of fibers is inserted, as shown in Fig.6.



*Fig.6 Composite lay-up and preparation of a precursor for the studied composite*

During the Roll-Diffusion Bonding phase, the just-assembled sample (precursor) has been put under a backward and forward strip-tension under inert gas. As well as the pressure also the rolling speed and mill-stand temperature is computer controlled to improve the bonding reaction and this is the reason for the process called: Roll-Diffusion-Bonding (RDB). The metallic strips play the role of the composite matrix and under the combined effects of temperature and pressure flows from the surfaces through the fibre

inter-spaces inside the fabric resulting in a complete metallic and ceramic-to-metal bonding.

The process phase is summarised as follows:

- 1- heating-up of a portion of the lay-up in the rolling-inert gas chamber;
- 2- plastic deformation of the metal matrix that starts the flow between the fibres;
- 3- first chemical reactions (inter-diffusion) at the fibre-matrix interfaces;
- 4- facing and contact between the two metal flows in the spaces between the fibres;
- 5- matrix recrystallisation in the joining surfaces;

The bonding process is stabilised and is completed in about 10 seconds. A further diffusion bonding phase is improved by mean of post-heat treatment just controlling the composite strip temperature to improve the diffusion strength.

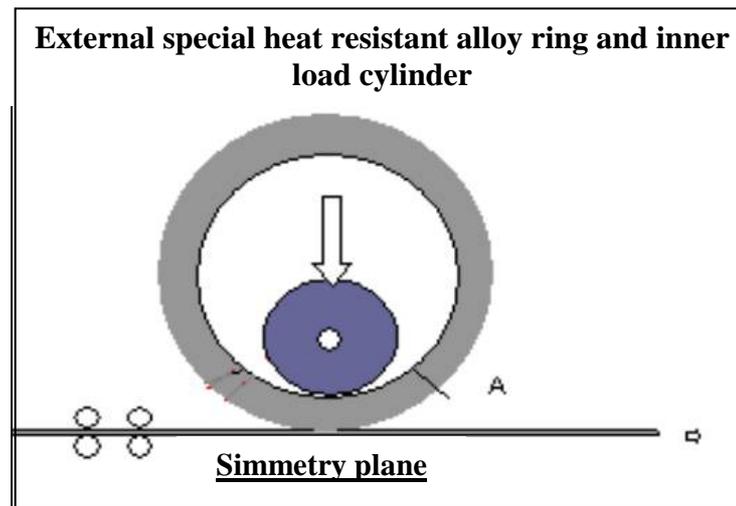
The design parameters adopted for the pilot equipment first dimensioning are reported in Tab.4

<b>Exercise temperature (°C)</b>	<b>Mean pressure in the contact arc (MPa)</b>	<b>Time (s)</b>
900	500	6

*Tab.4 Experimental reference parameters adopted for the pilot plant design*

About the composite production, the adopted solution consist in the heating up a sealed chamber with inside the work rolls by mean of electrical resistance, in fact all other devices, micro-waves, induction, joule effect have shown higher complexity and higher costs. By this way a compacted working region has been manufactured (electric resistance are distributed on a crown of about 240 spatial degrees on each work roll) where has been possible to control both composite strip and rolling tool temperature, with also the atmosphere. In particular the large thermal mass of the rolling tools, simulated as an infinite heat source, is able to heat up the Ti-MMC precursor reaching 900°C in about few tenths of seconds.

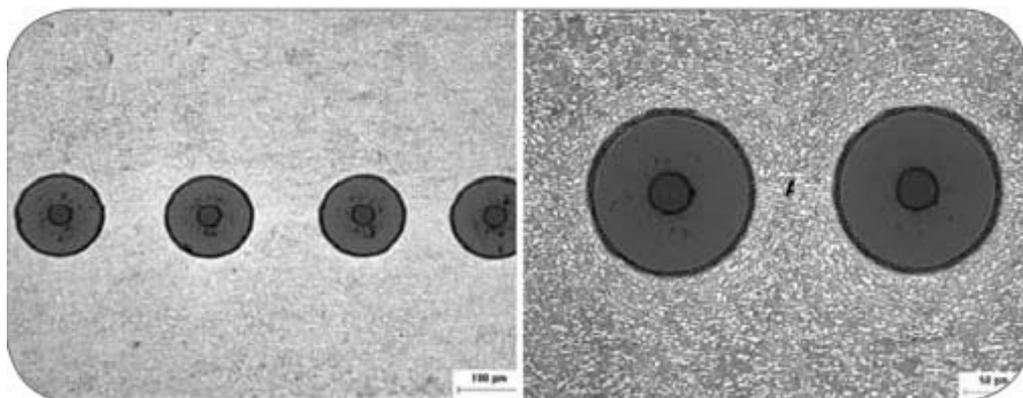
To warranty a safe working temperature of the work-rolls has been necessary a special cooling circuit. Moreover to avoid an excessive cooling effect, it was necessary to design special “concentric work-rolls”: an inner force-roll and an external high-temperature resistant alloy work-ring (Fig.7).



*Fig.7 Rolling Cylinders with the splitting of the thermal-tool (external ring) and the pressure-tool (inner cylinder)*

This solution has permitted two advantages: the heating up only of the external special alloy-rings, and the manufacturing of cheap standard tool-steel inner-cooled work-rolls. Obviously the space between inner and external tool has been insulated with special ceramic shields in order to reduce the thermal dispersions.

The work-rolls rotation starts when the external ring high temperature surface reaches a temperature that is sufficient to warranty at least 900°C in the contact-arc.



*Fig 8 Ti6Al4V-SiC<sub>f</sub>: result of the Roll Diffusion Bonding process and particular micrography*