Chapter 5

Mechanical characterization

5.1 FIMEC tests

The study of mechanical characterization for the Ti6Al4V-SiC_f composite has been developed firstly by the FIMEC indentation. Fig. 1 shows the FIMEC curve of composite tested at room temperature. For $p>p_Y$ the curve is characterised by a sudden slope decrease and the material starts to protrude around the imprint.

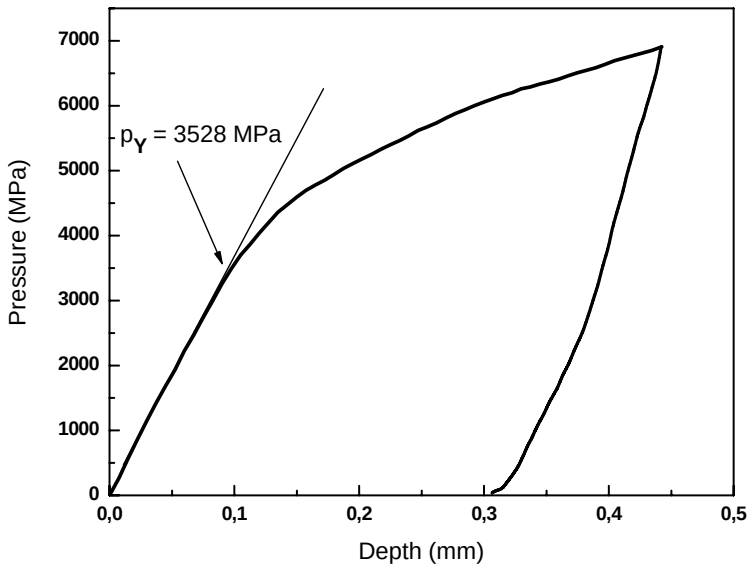

Fig.1 FIMEC curve of the as-fabricated composite recorded at room temperature.

In this case the slope change occurs for a pressure p_Y = 3528 MPa thus $\sigma_Y = p_Y / 3$ = 1176 MPa. The corresponding value obtained by tensile tests is 1154 MPa thus $\Delta = (\sigma_Y - p_Y/3) / \sigma_Y$ is ~ 2%.

FIMEC tests have been carried out not only on composite but , for comparison, on the monolithic Ti6Al4V alloy too, at increasing temperature up to 500 °C. About that, FIMEC curves of as-fabricated composite and Ti6Al4V alloy at increasing temperature up to 500 °C are reported in Fig. 2 (a-b). Tab.1 reports the yield stress values ($\sigma_Y = p_Y /3$) obtained from the tests.

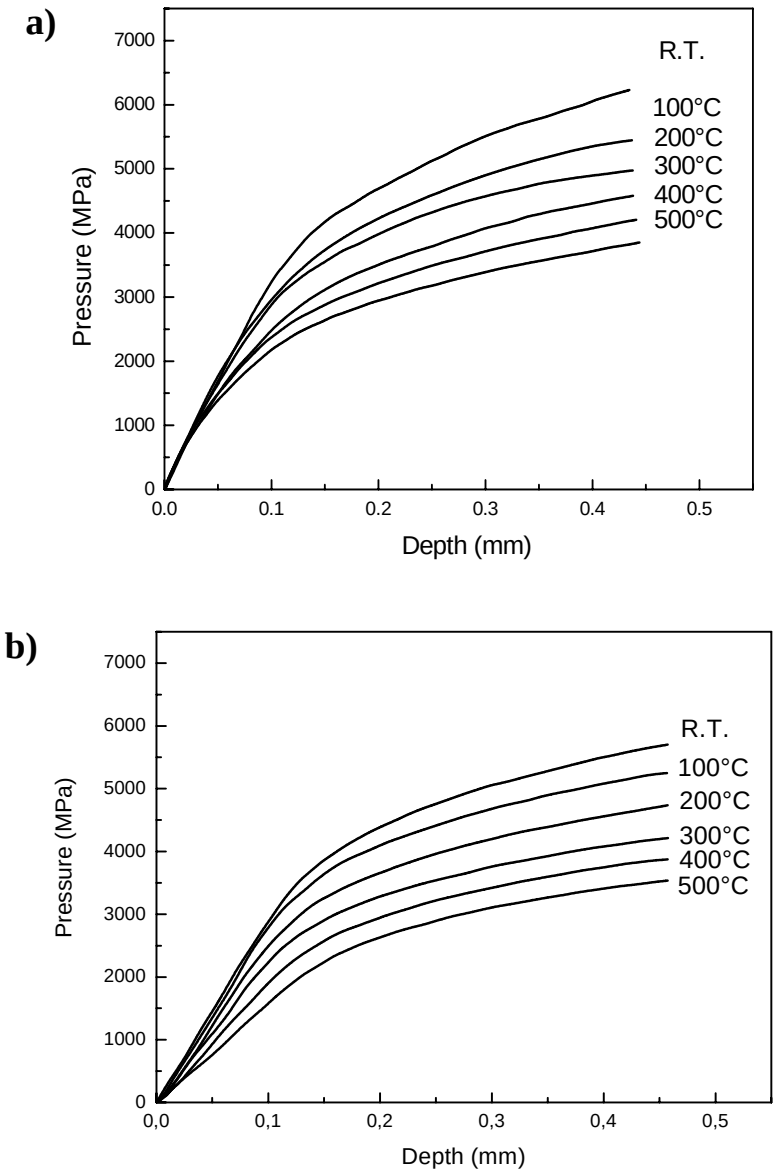

Fig.2 (a-b) FIMEC curves of as-fabricated composite (a) and monolithic Ti6Al4V alloy (b).

The results show that the yield stress of the composite is always higher than that of the matrix alloy but the difference becomes progressively smaller as temperature increases.

T [°C]	25	100	200	300	400	500
Composite	1176	849	807	722	595	550
Ti6Al4V alloy	904	807	747	671	569	548

Tab.1 Yield stress σ_Y in [MPa] of as-fabricated composite and monolithic Ti6Al4V alloy from FIMEC tests at increasing temperature

5.2 Tensile tests

Fig.3 shows the results of tensile tests carried out at room temperature and 600 °C on probes in as-fabricated condition and after heat treatments in vacuum (P = 5 x 10^{-6} mBar) at 400 and 600 °C with exposure times of 100, 500 and 1,000 hours. Yield stress and ultimate tensile strength are not affected by heat treatments also in the most severe conditions.

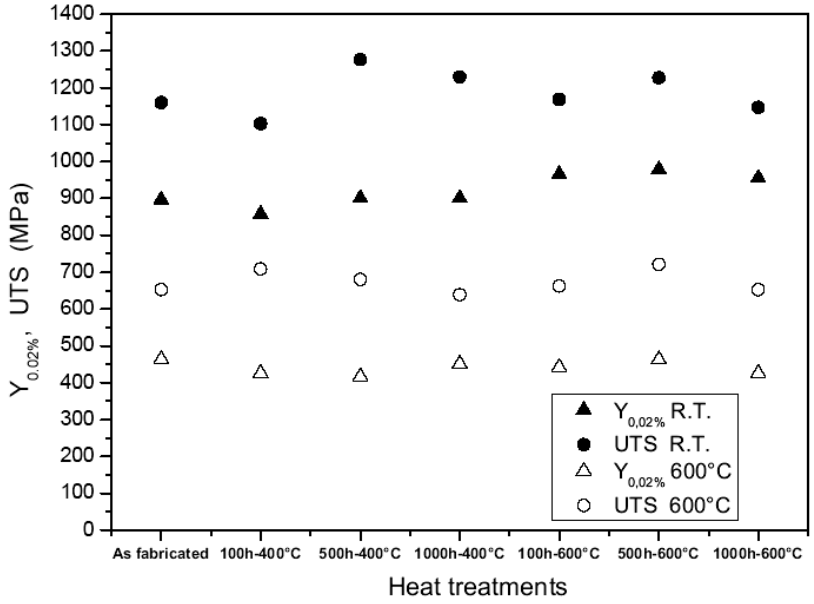

Fig. 3 Yield stress ($Y_{0.02\%}$) and ultimate tensile strength (UTS) at room temperature and 600°C of the composite in as-fabricated condition and after the heat treatments.

Fig.4 shows the fracture surface of a probe heated 1,000 hours at 600 °C. The fracture surface is not planar with several pull-out of fibres. The external mantle of the fibres shows a clear reaction with the matrix indicating a correct load transfer from the matrix to the fibres.

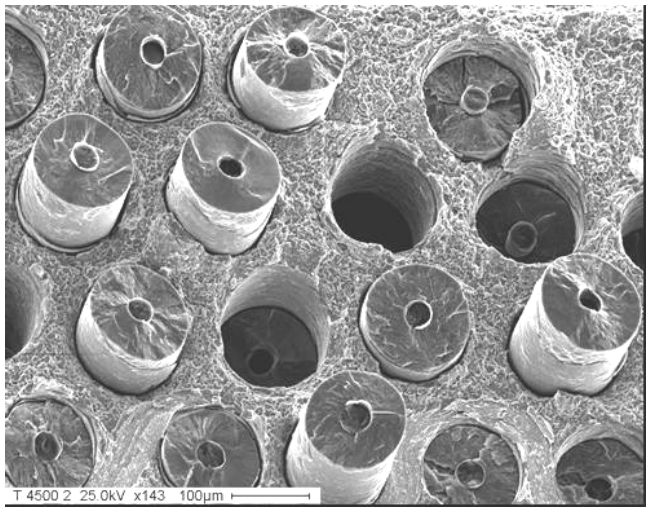

Fig. 4 Fracture surface of a probe exposed at 600°C for 1,000 hours.

The main reason of such mechanical performances is the stability of fibre-matrix interface after long-term heat treatments. To have explanation of this mechanical stability, the thickness of the reaction zone, i.e. the zone between carbon coating and matrix affected by chemical reactions, has been measured by means of SEM observations (see Fig. 5).

Since local irregularities are present at fibre boundary a statistical approach (at least 10 measurement in 8 different fibre-matrix positions rotated of 45°) has been used. Tab.2 reports the results which demonstrate that the growth of the reaction zone is quite slow. For example, its thickness, which is 0.85 μm in the as-fabricated material, becomes 0.98 μm after 1,000 hours at 600 °C.

Another factor contributing to preserve the mechanical properties after long-term heat treatments is grain size which substantially does not change.

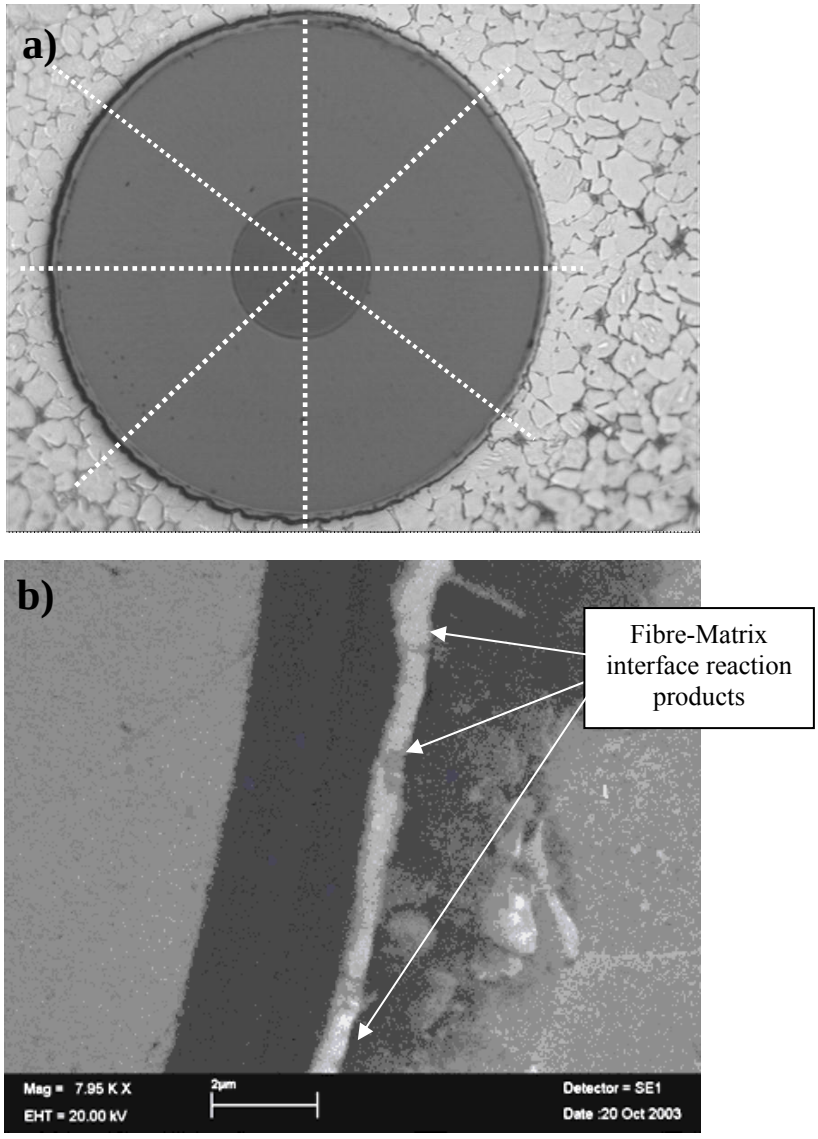

Fig.5 (a-b) Directions where the thickness of reaction zone has been measurement (a). Irregularities at fibre boundary (b).

Heat treatment	Mean thickness [μm]
As-fabricated	0,85
400 °C, 100 hours	0,86
400 °C, 500 hours	0,95
400 °C, 1000 hours	0,98
600 °C, 100 hours	0,94
600 °C, 500 hours	0,97
600 °C, 1000 hours	0,98

Tab.2 Thickness of the reaction zone in samples after different heat treatments.

5.3 Fatigue tests

The results of fatigue tests are displayed in Fig. 7; they show that the heat treatments at 600 °C produce a progressive reduction of the number of cycles to failure N_f as the exposure time increases. However, for the treatment times considered here, such reduction is very small.

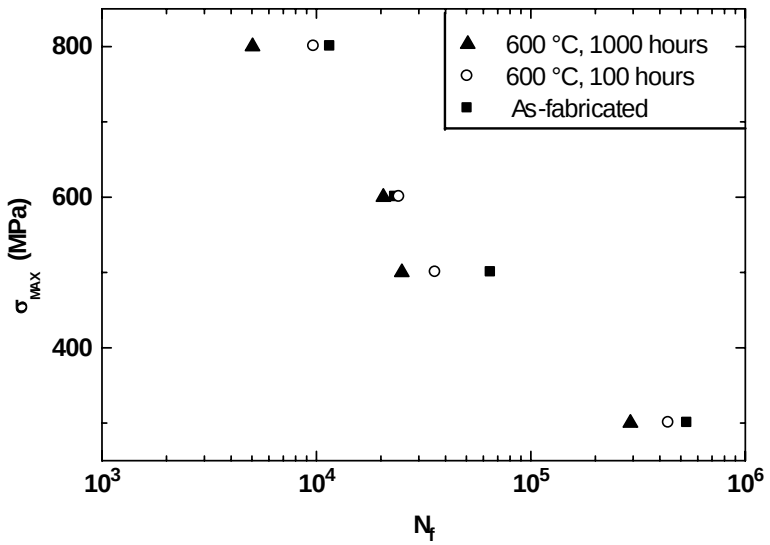

Fig. 6 Data of fatigue tests on the composite in as-fabricated condition and heat treated for 100 and 1,000 hours at 600 °C.

The deformation mechanism is controlled by matrix plasticization (see Fig.8). This is confirmed by the "Universal-Slope" model [47], which describes fatigue results. Eq. (1) describes the composite behaviour when controlled by the fibre fractures, eq.(2) when controlled by the matrix plastic deformation.

$$\varepsilon_{\max} = A_f (N_f)^{\alpha_f} \tag{1}$$

$$\varepsilon_{\max} = A_m \frac{\sigma_{ult}}{E}(N_f)^{\alpha_m} + B_m(N_f)^{\beta_m} \quad (2)$$

being ε_{max} the maximum deformation imposed to the probe, σ_{ult} and E the ultimate tensile strength and the Young's modulus of the matrix, N_f the number of cycles to failure, α_f, α_m, β_m, A_f, A_m and B_m parameters to determine by the statistical regression analysis.

The fatigue results have been interpolated by the equation (4) reported below (Rd^2= 0.96):

$$\varepsilon_{\max} = -0.46584 \cdot N_f^{-0.80498} + 0.507653 \cdot N_f^{-0.84546} \quad (3)$$

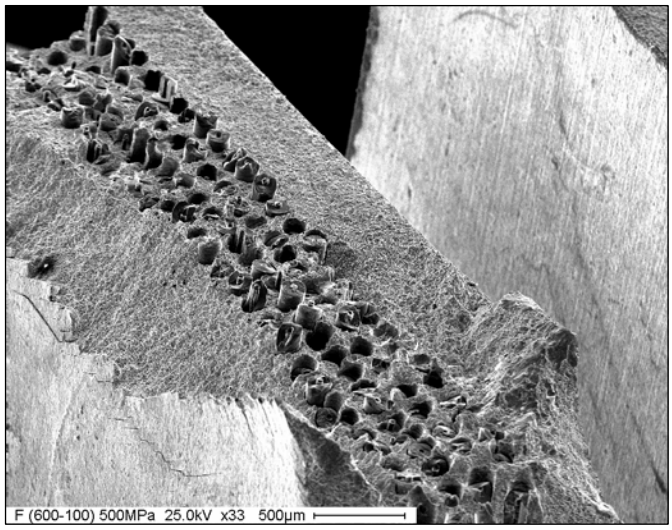

Fig. 7 Fatigue fracture surface with heavy deformed matrix

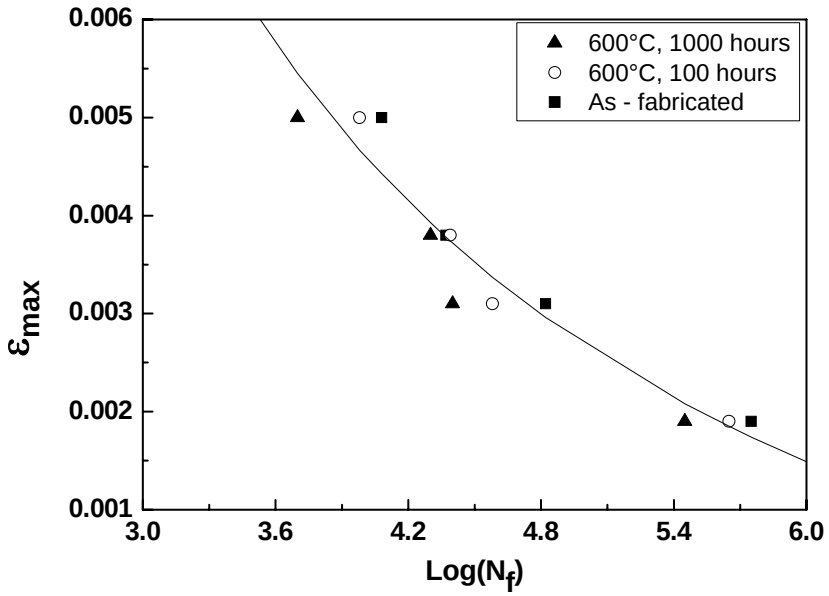

Fig.8 Fatigue data and interpolation curve

5.4 Dynamic modulus

Dynamic modulus trends (measured as function of the resonance frequency by the equation (11) showed in Cap.3) described of composite and monolithic alloy vs. temperature are displayed in Fig. 9.

Modulus of composite is always higher (~ 20%) than that of the matrix alloy up to 600°C. In particular, the modulus value of composite at 600°C (112 GPa) is very close to that of the matrix at room temperature: this datum is very important especially by the point of view of solidity for the composite at middle-high temperature and at high vibrational work.

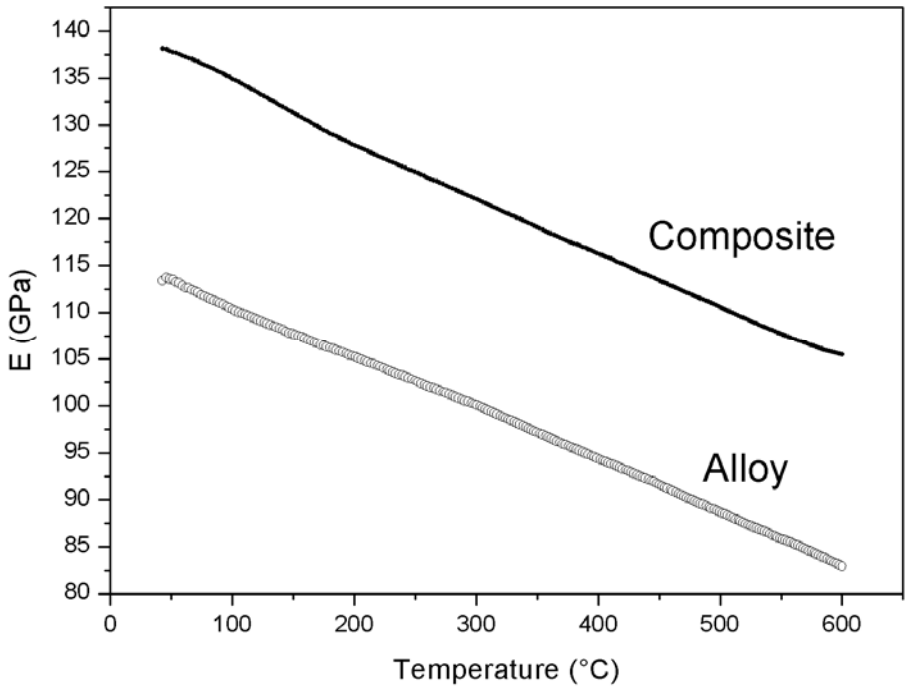

Fig.9 Dynamic modulus of Ti6Al4V monolithic alloy and composite at increasing temperature up to 600 °C.

5.5 Discussion

The main results can be summarized as follows.

First the FIMEC tests show that yield stress of the composite is always higher than that of the matrix alloy, even if the difference becomes progressively smaller as temperature increases.

Tensile and fatigue tests indicate that mechanical properties substantially do not change after long-term exposure to temperatures of potential service for Ti-MMC materials. It's possible to verify it analyzing the fracture surfaces, that show plastic deformation of the matrix and pull-out of fibres, i.e. a correct load transfer from the matrix to the fibres.

Then SEM observations showed that the growth of reaction zone between carbon coating and matrix following heat treatments is quite slow, keeping the stability of fibre-matrix interface: by this way (and through the preservation of the grain size), the composite is able to maintain its original properties after prolonged heat treatments.

Finally the dynamic measurements show that Young's modulus of composite is always higher (~ 20%) than that of the matrix alloy up to 600°C, confirming the composite solidity at high temperature during high vibrational work, too.

On these grounds it is possible to conclude that Ti6Al4V-SiC_f composite is suitable for aerospace applications where stable tensile and fatigue properties up to 600°C are requested.

The same aspects can be developed for the *RDB* composite. About this the studies are still at the beginning, but a first comparison can be observed by the following Tab.3, in which the tensile test results (also developed at high temperature) have been obtained about the same alloy matrix, same fiber volume fraction (about 10% vol), same fiber mono-orientation (0° fibres), and same perform.

Manufacturing process (fiber fraction: 10,6%)	**T (°C)**	**Yield Strength (MPa)**	**Ultimate Tensile Strength (MPa)**
Foil-Fibre-Foil process HIP	r.t.	893 $\pm$ 10	1298 $\pm$ 20
	400	710 $\pm$ 12	876 $\pm$ 35
	600	543 $\pm$ 13	659 $\pm$ 26
	800	285 $\pm$ 21	329 $\pm$ 24
Roll-Diffusion-Bonding process	r.t.	898 $\pm$ 35	1282 $\pm$ 11
	400	712 $\pm$ 34	942 $\pm$ 27
	600	553 $\pm$ 27	596 $\pm$ 23
	800	315 $\pm$ 29	337 $\pm$ 36

Tab.3 Tensile test result about HIP composite and RDB composite

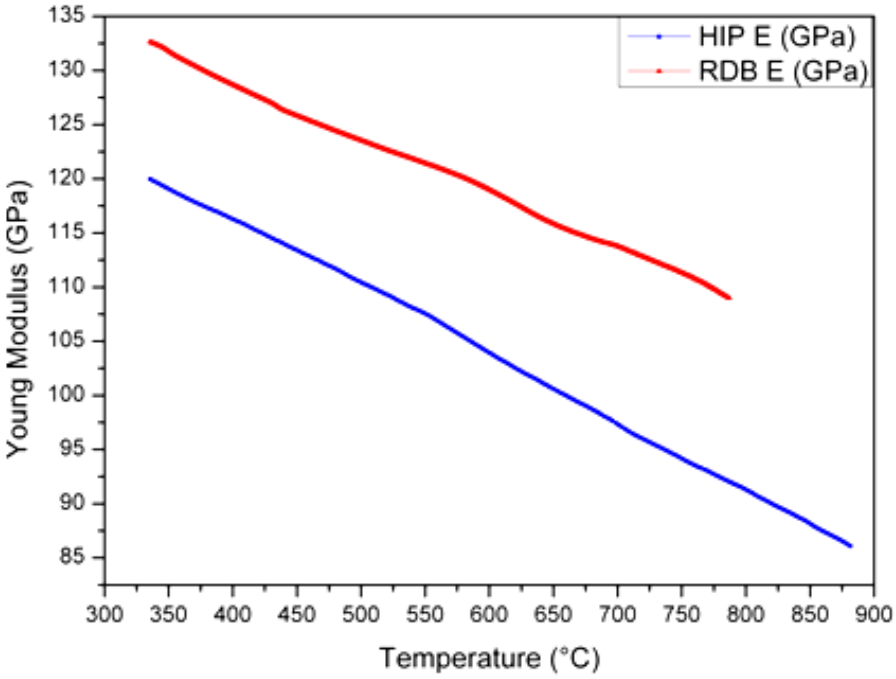

Fig.10 Dynamic modulus of Ti6Al4V-SiCf , produced by Hot Isostatic Pressure and Roll Diffusion Bonding

About the dynamic modulus studies for the *RDB* composite, these are yet at an initial state. However a first result is possible to note in Fig.10, remembering the eq.(11) in Cap.3 about the relation between temperature and dynamic elastic modulus.

In particular the comparison in the figure shows as the dynamic behaviour at high temperature remains fundamentally the same for both the composites, but for the *RDB* composite the modulus values result higher than that for the *HIP* composite.