ID 1036

MECHANICAL BEHAVIOUR OF SiC MONOFILAMENTS IN ORTHORHOMBIC TITANIUM ALUMINIDE COMPOSITES

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SUMMARY : Continuous fibre-reinforced titanium metal matrix composites (TMCs) are particularly attractive for use in aircraft gas turbine engines owing to their specific mechanical properties at high temperature. In the present paper, the compatibility of SiC monofilaments (SCS-6, SM 1140+) with an orthorhombic titanium aluminide matrix (Ti-22Al-27Nb) has been investigated. It has been shown that the triode sputtering method developed at ONERA enables to produce high quality SiC/Ti-22Al-27Nb composites. The thermal stability of the reinforcement is a property of critical importance during the manufacture of the composite. The fibre tensile properties were evaluated after each step of the composite processing using an adapted test technique. A slight decrease in the SM 1140+ fibre strength was observed that can be attributed to the beginning of the W-core/SiC reaction.

KEYWORDS : titanium alloy, silicon carbide fibre, matrix-coated fibre process, mechanical testing, aerospace applications.

INTRODUCTION

Metal matrix composites are one of several classes of advanced materials which are expected to play a significant role in the development of future aerospace applications. Due to the availability of high performance continuous fibres, these composites exhibit much improved properties, in terms of specific strengths and stiffness at room and elevated temperatures, compared to unreinforced structural materials. Among metal matrix composites, TMCs are of particular interest for aero-engines applications and mainly for compressor discs where the potential weight savings due to improved specific properties are the greatest [1].

Titanium alloys which are the most widely used as matrices are Ti-6Al-4V and Ti-6Al-2Sn-4Zr-2Mo. However, the increased operating temperature of advanced aero-engines requires matrices with improved high temperature properties. Orthorhombic titanium aluminides based on the Ti_2AlNb compound offer the best alternative to conventional titanium alloys in terms of oxidation behaviour and high temperature specific strengths (tensile, creep). The matrix compositions of interest range from Ti-(21 to 25)Al-(17 to 27)Nb (at.%). Among them, the Ti-22Al-27Nb alloy displays an attractive balance of mechanical characteristics [2-4].

Silicon carbide monofilaments are currently the only commercially available fibres for reinforcement in TMCs. Two types of SiC monofilaments are produced by chemical vapour deposition : the ones manufactured by Textron in USA (SCS-6, SCS-Ultra) have a carbon core (33 µm in diameter) whereas those fabricated by DERA-Sigma in United Kingdom (SM 1140+) have a tungsten core (13 µm in diameter). The advantage of using a SiC fibre with a tungsten core is that the tungsten wire is cheap and available in large spools, unlike carbon monofilament. As a consequence, the production cost of W-core/SiC fibre is lower than C-core/SiC. All these fibres possess an outer protective coating to limit the interaction with the matrix during the fabrication of the composite or service conditions. That coating consists mainly of pyrocarbon with a thickness, a composition and a morphology varying with the fibre.

The manufacture of TMCs is difficult because of the high melting point and the extreme chemical reactivity of titanium-based alloys. Therefore, processing of composites is limited to solid state routes. Various fabrication methods have been developed which differ in the way used to assemble fibres and matrix before consolidation. These different routes are termed as foil-fibre-foil, tape casting, plasma spray and physical vapour deposition (PVD). PVD processes offer the advantage to obtain a composite with a fairly uniform fibre distribution. These processes involve in a first step the coating of SiC fibres with a thick layer of the matrix. Then, bundles of matrix-coated fibres are consolidated into a bulk composite [5-8]. The matrix material is provided entirely by the coating, thus avoiding the expensive cost of foils or powders required for other routes.

The thermal stability of the reinforcement is a property of critical importance since the consolidation of TMCs is performed at high temperature (> 900°C). The thermal stability of SiC fibres with a carbon core is superior to the ones with a tungsten core : silicon carbide begins to react with tungsten at 950°C with the formation W_5Si_3 and W_2C [9] and this reaction induces a decrease in tensile properties of the monofilament [10]. The behaviour of the SCS-6 fibre (Ø = 140µm) and the SM 1140+ fibre (Ø = 108µm) in an orthorhombic matrix (Ti-22Al-27Nb) has been compared in the present study.

The objectives of the research work were (i) to demonstrate the feasability to manufacture SiC/orthorhombic titanium aluminide composites using the triode sputtering method developed at ONERA [7], (ii) to study the influence of process conditions on the properties of SCS-6 and SM 1140+ fibres.

PROCESSING OF COMPOSITE MATERIAL

Triode sputtering method

Among the different PVD techniques, triode sputtering was chosen for the various advantages that it provides :
- possibility to use any matrix alloy, metallic or intermetallic, with a precise control over the chemical composition of the coating,
- cleanness of the coating atmosphere,
- high metal utilisation efficiency (i.e. most of the metal sputtered is collected onto the fibres),
- good adhesion of the coating on the substrate.

The experimental arrangement used to coat SiC fibres is illustrated in Figure 1. A tungsten filament heated by Joule effect emits electrons which are accelerated by an anode. These electrons interact with the low pressure argon atmosphere to give Ar^+ ions. A columnar plasma is created by means of magnetic coils. The target which consists of the matrix alloy is polarised to a negative potential. This target is sputtered under the bombardment of Ar^+ ions and metallic atoms are collected on fibres mounted on a rotating holder.

Fibre coating

The rotating fibre holder is cylindrical and made of titanium alloy Ti-6Al-4V. Sixty fibres 140mm in length can be mounted on it. The coating parameters should be adjusted so that (i) the chemical composition of the coating is close to that of the sputtering target, (ii) the deposition temperature should not exceed 900°C to limit the interaction between titanium and the carbon protective layer of the fibre, (iii) the deposition rate should be maximum. The sputtering time is calculated as a function of the fibre volume fraction expected in the bulk composite.

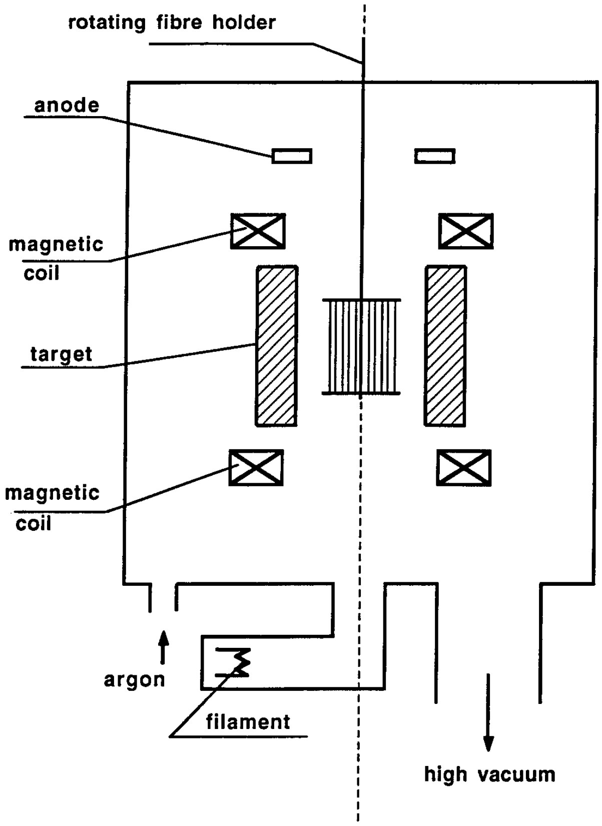

Fig. 1 Schematic of the triode sputtering unit.

A key parameter in PVD techniques is to obtain a coating with a chemical composition as close as possible to that of the target material. Using triode sputtering, we have shown previously the possibility to deposit various titanium alloys (Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo, TIMETAL 834) on SCS-6 fibre with a coating composition very similar to the one of the target [7]. The results of the present study are reported in Table 1. Metallic elements were analysed by ICP spectroscopy whereas oxygen and nitrogen contents were determined by inert gas fusion extraction. Results which appear in Table 1 were obtained after optimisation of deposition parameters. From a general point of view, the chemical compositions of the coatings and the target are very similar. Only a slight decrease in the aluminium content in the coatings was measured. Furthermore, it is well recognised that the pollution by interstitial elements severely affects mechanical properties of titanium alloys and the triode sputtering unit was designed to avoid any gas contamination in the coating. Table 1 shows that the oxygen content is lower in the coatings than in the target.

Table 1 Chemical composition (wt.%) of the sputtering target and fibre coatings.

	Al (%)	Nb (%)	O (ppm)	N (ppm)
Target	9.7	44.6	880	75
Coating on SCS-6 fibre	9.45	43	440	-
Coating on SM 1140+ fibre	9.3	43.6	400	75

Consolidation of the composites

Matrix-coated fibres are put into a tube of an inner diameter of 2.8mm and an outer diameter of 10mm made of the same orthorhombic alloy. Once the tube has been filled, it is inserted in a low carbon steel capsule which is outgassed and sealed by welding under high vacuum. The composite is consolidated by hot isostatic pressing (HIP) and optimised HIP conditions have been determined. At first, the temperature is raised up to 1000°C, then a pressure of 800 bars is applied for one hour. After consolidation, the temperature is decreased gradually (5°C/min) and the pressure is released when the temperature is about 700°C, in order to minimise thermal residual stresses in the composite [11].

Optical micrographs of transverse cross sections through SCS-6/Ti-22Al-27Nb and SM 1140+/Ti-22Al-27Nb composites in the as-consolidated condition are shown in Figures 2 and 3. The composites are fully dense and no structural defect is visible. The fibre coating thickness was adjusted so that to obtain composites with a fibre volume fraction of 35%. The micrographs reveal the very uniform fibre distribution that can be achieved using the triode sputtering process.

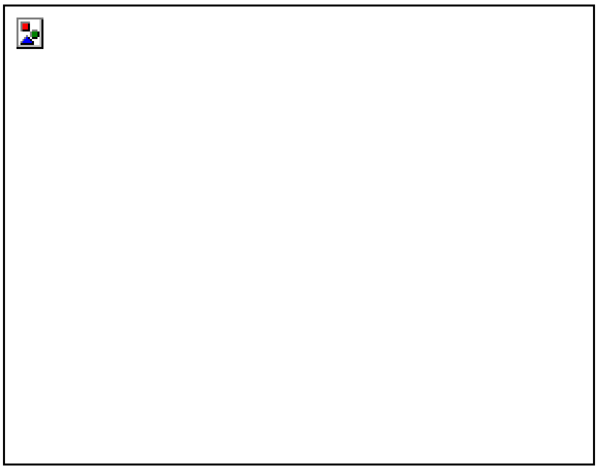

Fig. 2 Transverse optical micrograph of the SCS-6/Ti-22Al-27Nb composite.

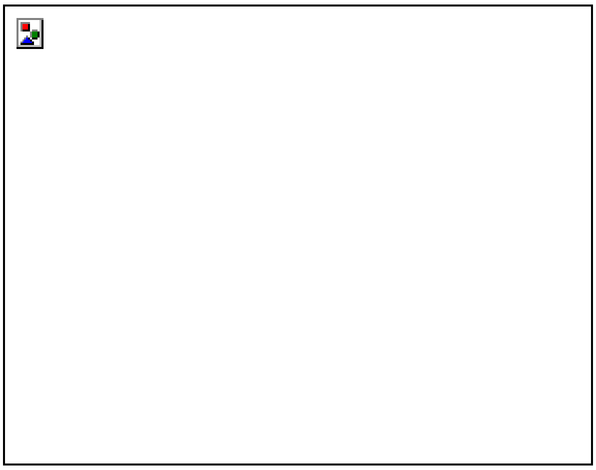

Fig. 3 Transverse optical micrograph of the SM 1140+/Ti-22Al-27Nb composite.

Fibre/matrix interaction

Figure 4 shows a SEM micrograph of the fibre/matrix interaction zone in the SCS-6/Ti-22Al-27Nb composite. The interaction zone consists of two layers of precipitates : the inner one close to the fibre and the outer one adjacent to the matrix are about 0.5µm and 0.2µm thick respectively. A qualitative EDS analysis reveals that the inner zone contains titanium, niobium and carbon mainly plus a very small amount of silicon. It is very likely that the chemical composition of the reaction product is of the type $(Ti,Nb)_xC_{1-x}$. The outer zone contains titanium, niobium and carbon like the inner zone but also silicon and aluminium. That latter zone is a mixture of carbide and silicide. The quantitative analysis could not be performed owing to the too small size of the precipitates. There is a rather good agreement between our observations and a TEM investigation carried out on a SCS-6/Ti-22Al-23Nb composite manufactured by the foil/fibre/foil route [4]. Three zones were identified at the fibre/matrix interface : the two ones adjacent to the fibre are composed of (Ti,Nb)C plus a small amount of $(Ti,Nb)_5Si_3$ whereas the outer zone close the matrix is a $(Ti,Nb)_5(Si,Al)_3$ compound.

The fibre/matrix interaction zone does not look the same in the SM 1140+/Ti-22Al-27Nb composite (Figure 5). Here, the reaction product consists of rather nodular precipitates which delineate the outer surface of the carbon coating. Their size lies in the range 0.6-0.9µm. The qualitative EDS analysis reveals that the reaction product is $(Ti,Nb)_xC_{1-x}$. Unlike SCS-6/Ti-22Al-27Nb composite, no silicon was detected in the fibre/matrix interaction zone. The explanation is that the outer shell of the carbon coating of the SCS-6 fibre contains silicon whereas the coating of the SM 1140+ fibre is made of pyrocarbon only.

Fig. 4 SEM micrograph of the reaction zone in the SCS-6/Ti-22Al-27Nb composite.

Fig. 5 SEM micrograph of the reaction zone in the SM 1140+/Ti-22Al-27Nb composite.

THERMAL STABILITY OF THE REINFORCEMENT

The mechanical degradation of fibres resulting from composite processing at high temperature may arise either from an internal chemical reaction within the fibre or from the brittle reaction products formed at the fibre/matrix interface.

The SM 1140+ possesses a tungsten core and it has been mentioned previously that an internal chemical reaction occurs with SiC at temperatures higher than 950°C. As the SM 1140+/Ti-22Al-27Nb composite has to be consolidated at 1000°C, it is important to evaluate the effect of the consolidation cycle on fibre characteristics.

The fibre strength degradation during the different steps of the composite processing was assessed using a fibre tensile test technique that has been described previously [12]. Batches of SCS-6 and SM 1140+ fibres corresponding to the four following conditions were tested at room temperature : as-received, after triode sputtering coating, after consolidation of the composite and after heat treatment (1000°C, 2 hours) under vacuum in a quartz tube simulating the thermal cycle of the consolidation process. Thirty fibres at least were tested for each condition. In the cases of matrix-coated fibres and bulk composite, fibres were extracted from the matrix by etching in a HF-HNO_3-H_2O solution at 50°C. It was checked that this acid solution has no harmful effect on fibre strength.

Tensile tests results are listed in Table 2. The mean ultimate tensile strength and the standard deviation have been reported for each condition. It has been found that the fibre tensile strength after the triode sputtering process does not differ significantly from the one in the as-received condition for both fibres. The absence of degradation can be explained by the rather low deposition temperature (800°C) and the very thin reaction zone layer (0.3µm) in SCS-6/Ti-22Al-27Nb and SM 1140+/Ti-22Al-27Nb systems.

An apparent slight decrease in the mean tensile strengths of SCS-6 and SM 1140+ fibres has been measured after HIP consolidation. Then, a significance test (Darmois model) has been used to decide whether there is a difference between the mean tensile strengths in the as-received condition and after consolidation. The Darmois model reveals, with a confidence limit higher than 95%, that the difference of fibre strength is not significant in the case of the SCS-6 fibre whereas a slight degradation occurs in the case of the SM 1140+ fibre.

Table 2 Tensile properties of batches of SCS-6 and SM 1140+ fibres under different conditions.

	SCS-6		**SM 1140+**	
	Mean UTS (MPa)	St. dev. (MPa)	Mean UTS (MPa)	St. dev. (MPa)
As-received	4330	350	3170	110
After triode sputtering coating	4280	520	3110	120
After HIP consolidation	4100	430	2970	320
1000°C - 2 hours	4360	410	2970	80

The moderate decrease in SM 1140+ fibre strength after consolidation may originate either from the interaction with the matrix or from the W-core/SiC reaction. As the mean tensile strengths of fibres extracted from the composite or heat treated for 2 hours at 1000°C are the same, it can be concluded that the slight degradation is due to the very beginning of the W-core/SiC reaction. Furthermore, considering the 5µm thick protective carbon coating on that fibre, it is unlikely that the thin fibre/matrix reaction zone (< 1µm) may have a detrimental effect on fibre characteristics. Besides, the very good thermal stability of SiC monofilaments with a carbon core has been confirmed.

CONCLUSION

It was shown that the triode sputtering technique can produce high quality Ti_2AlNb-based alloy coatings on SiC monofilaments with a chemical composition very close to the one of the target material. Optimised HIP consolidation parameters were determined to manufacture composites from matrix-coated fibres.

The fibre/matrix interaction zone is thin in SCS-6/Ti-22Al-27Nb and SM 1140+/Ti-22Al-27Nb composites in the as-consolidated condition (< 1µm). A reaction zone of two layers was identified in the SCS-6/Ti-22Al-27Nb composite : an inner layer of $(Ti,Nb)_xC_{1-x}$ and an outer layer composed of a mixture of carbide and silicide. The reaction zone in the SM 1140+/Ti-22Al-27Nb composite consists of a single layer of $(Ti,Nb)_xC_{1-x}$ compound.

Tensile tests on batches of SCS-6 and SM 1140+ fibres were performed to assess the strength degradation that the different steps of the composite processing may induce. Thus, it was shown that the coating operation has no detrimental effect. Fibres extracted from SCS-6/Ti-22Al-27Nb and SM 1140+/Ti-22Al-27Nb bulk composites revealed a very slight decrease in the mean tensile strength of the SM 1140+ fibre. A statistical analysis of tensile tests results enabled to establish that the moderate loss of strength was due to the beginning of the W-core/SiC reaction in that fibre.

It can be concluded from that study that both SCS-6 and SM 1140+ monofilaments are compatible with orthorhombic matrices using the triode sputtering manufacturing route.

ACKNOWLEDGEMENTS

The authors are very grateful to S. Landais and E. Candale for their advice and help in conducting triode sputtering coatings and to M. Raffestin for her expertise in metallography. They also would like to thank DGA for financial support.

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