Titanium matrix reinforced composites produced by H.I.P. of plasma sprayed preforms

T. Valente, C. Testani, M. Tului

The present work has been focussed on the fabrication of long fibre reinforced titanium composites by hot isostatic pressing (HIP) of plasma sprayed preforms. Silicon carbide long fibers were used together with two different metallic matrices, the first being a Ti-6Al-2Sn-4Zr-2Mo alloy and the second a Ti-4.5Al-3V-2Mo-2Fe alloy, also named SP700. All the spraying trials were performed by using a Controlled Atmosphere Plasma Spraying equipment (CAPS), allowing deposition processes also in the high pressure range. After optimisation of the plasma spraying procedure, on the basis of results obtained with preliminary spraying trials carried out according to a DOE methodology, composite multilayers were fabricated by HIP with different combinations for the process parameters temperature/time/applied pressure, in the range 850-940°C, 30-45 min, 1200 bar. They were then qualified by microstructural investigations and tensile testing at room and high temperature (up to 600°C). Obtained results are reported and discussed with particular reference to differences evidenced for the two titanium matrices.

Parole chiave: titanio e leghe, materiali compositi

INTRODUCTION

Long fiber reinforced metal matrix composites are taking on a primary role in the field of advanced materials for aerospace structural applications, due to their specific properties guaranteed by the coupling of a ceramic reinforcement with a metallic matrix [1-5]. To date, the majority of these composites have been fabricated by way of diffusion bonding techniques by means of the foil-fiber-foil method or by using woven fiber mats in alternating layers with the matrix foil. In the first production route the use of an organic binder is required; the processing cycle must be controlled to remove the binder, as volatilisation occurs with temperature, and at the same time to ensure suitable matrix plastic flow to obtain the final component. With woven fiber mats in alternating layers with the matrix foil, fibers are held in place with a wire or ribbon interweave material and consolidation is achieved by vacuum hot pressing or by hot isostatic pressing, without the use of any binder. As alternative fabrication route to the above mentioned processes, composite monolayers can be fabricated by a controlled atmosphere plasma spraying and then consolidated by a secondary consolidation techniques as hot isostatic pressing (HIP) to obtain a composite multilayer. Main advantages are the elimination of possible contamination by any binder residue and of any technical problem related to interweave material composition; in addition plasma spraying enables the use of any pre-alloyed powder as matrix material and minimizes any fiber-matrix deleterious chemical reaction for the very short liquid/solid contact time (about 100 ms), so resulting in high quality composites monotapes.

In this paper plasma sprayed preforms were used to fabricate multilayered composites by using two different titanium alloy matrix materials, reinforced with long silicon carbide fibers. Microstructural and mechanical characterization tests were performed on the multilayers in order to qualify the obtained composites.

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MATERIALS AND METHODS

The plasma spraying manufacturing process of composite monolayers basically consists of deposition of successive metallic layers to form a compact metallic matrix on previously arranged ceramic long fibers wound onto a cylindrical substrate (Fig. 1). Two types of gas atomised powders have been used: a Ti-4.5Al-3V-2Mo-2Fe alloy and a Ti-6Al-2Sn-4Zr-2Mo alloy, both with average size in the range of 40-80μm (Figs. 2-3).

SCS-6 fibers (Textron System Divison) have been used as reinforcements, consisting of silicon carbide deposited by CVD of β-SiC onto a graphite core, already used for other experiences with plasma sprayed Ti6Al4V titanium matrices [6-7]. Average fiber diameter was 142μm. Details on fibers set-up and spraying procedures adopted are fully described in Ref. [8]. All spraying trials were performed by using a CAPS [9] (Controlled atmosphere plasma spraying) equipment in an inert environment (Ar atmosphere), by following the experimental matrix reported in Table1.

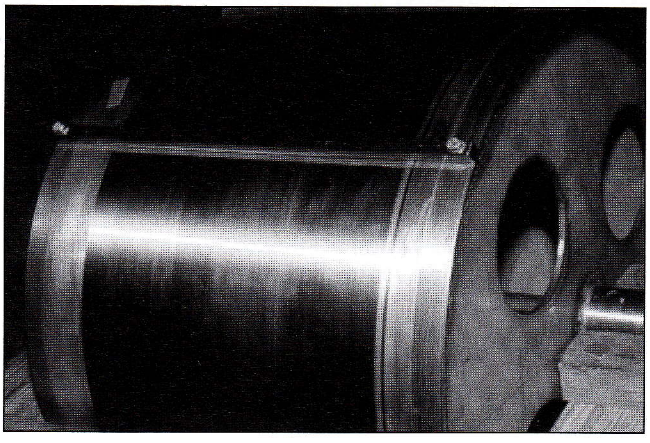

Fig. 1 Fiber arrangement onto a cylindrical substrate

Fig.1 Disposizione delle fibre su un substrato cilindrico

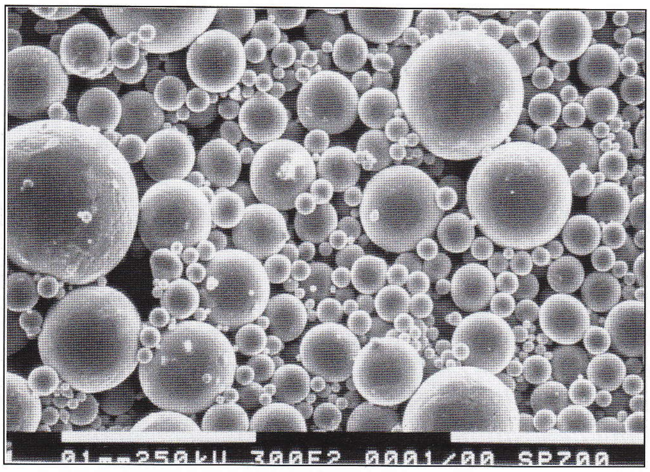

Fig. 2 Ti-4.5Al-3V-2Mo-2Fe alloy powder

Fig.2 Polvere Ti-4.5Al-3V-2Mo-2Fe

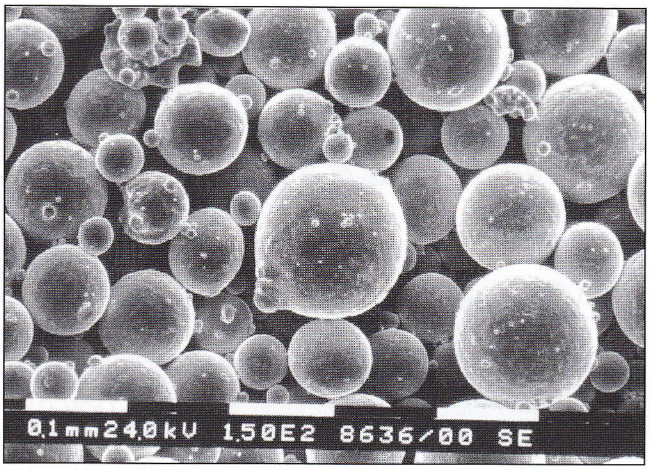

Fig. 3 Ti-6Al-2Sn-4Zr-2Mo alloy powder

Fig. 3 Polvere Ti-6Al-2Sn-4Zr-2Mo

Run	Pressure (mbar)	Spraying distance (mm)	Ar plasma gas (SLPM)	Current intensity (A)
1	1500	100	20	810
2	1500	120	10	810
3	2500	100	10	810
4	2500	120	10	700
5	2500	100	20	700
6	2500	120	20	700
7	1500	100	10	810
8	1500	120	20	700
9	2000	variable	15	750
10	2000	variable	15	750

Table 1 Experimental matrix for spraying runs (monolayers fabrication)

Tab. 1 Matrice delle prove sperimentali (fabbricazione dei monostrati)

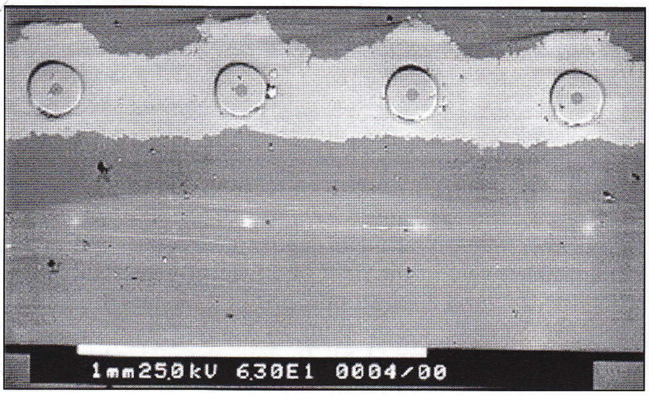

Fig. 4 Cross section of Ti-4.5Al-3V-2Mo-2Fe matrix monotape (SEM image)

Fig. 4 Impianto CAPS

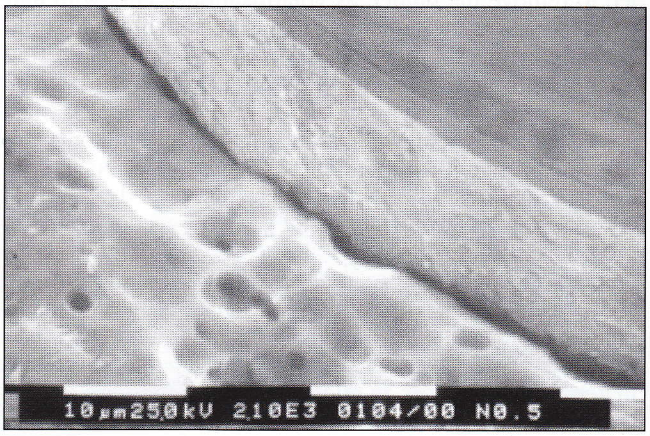

Fig. 5 Fiber-matrix interface after chemical etching

Fig. 5 Sezione del mononastro a matrice Ti-4.5Al-3V-2Mo-2Fe (immagine SEM)

All monotapes were qualified by microstrucural investigations and mechanical testing [8] and obtained results, analysed on the basis of a DOE response method, were used to define optimum spraying parameters, finally adopted to fabricate monolayers for hot isostatic pressing. By using different process parameters combinations - 850°C/1200bar/45min; 910°C/1200bar/45min; 940°C/1200bar/30 min - three different sets of composites plates (200mm x 200mm x 2mm) were fabricated, by joining and pressing five composite monolayers. Specimens for microstructural investigations and tensile testing at different temperatures (RT, 300°C, 400°C, 600°C) were cut from the mentioned plates in order to evaluate mechanical performances in fiber direction. Average fiber volumetric fraction calculated for the hot pressed composites was 21 %.

RESULTS AND DISCUSSION

In Fig. 4 a cross sections of optimised Ti-4.5Al-3V-2Mo-2Fe matrix monotape, fabricated by high pressure spraying at 2500 mbar, is reported as an example. In Fig. 5 a detail of fiber-matrix interface after chemical etching (2 min; 48 cc H_2O – 1 cc HF, 1 cc HNO_3) is also reported. No evidence of interactions due to possible deleterious chemical reactions was observed as well as no titanium matrix oxidation after spraying, as also confirmed by XRD analyses which did not revealed detectable amounts of new phases. No modifications of starting powder composition were detected by EDS analyses carried out on the sprayed matrix.

As an example cross sections of the SP700_940 and Ti6242_940 specimens at different magnifications, are reported in Fig. 6-7. Neither signs of imperfect bonding among various layers or fiber-matrix interaction could be observed. Samples of Ti-4.5Al-3V-2Mo-2Fe/SiC were identified as SP700_850, SP700_910, SP700_940 on the basis of the three pressing cycles adopted; samples of Ti-6Al-2Sn-

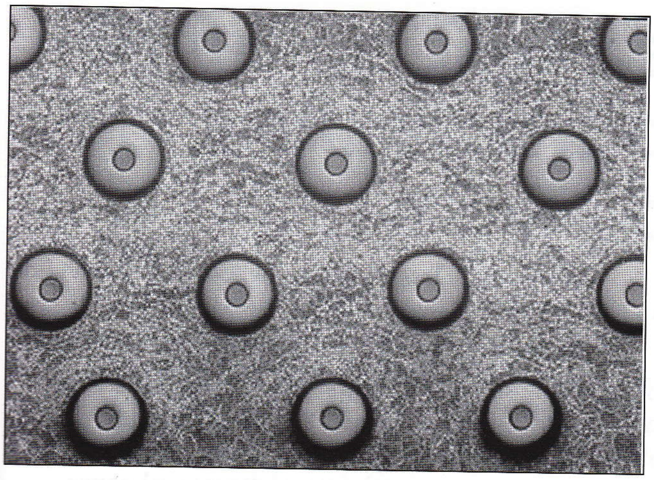

Fig. 6 Cross section of SP700_940 sample (optical microscope)

Fig. 6 Interfaccia fibra matrice dopo attacco chimico

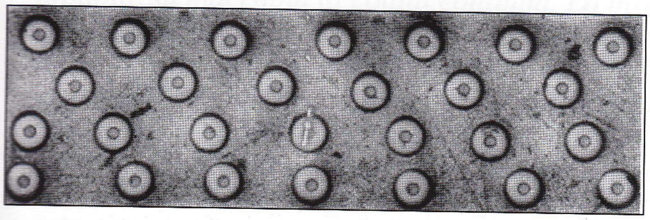

Fig. 7 Cross section of Ti6242_940 sample (optical microscope)

Fig. 7 Sezione metallografica del provino SP700_940 (microscopio ottico)

4Zr-2Mo/SiC were identified as Ti6242_850, Ti6242_910, Ti6242_940. Tensile tests were performed at room temperature, 300°C, 400°C and 600°C. Results in terms of ultimate tensile strength and yield stress are reported in Figs. 8-9 as a function of testing temperature.

In all cases, for both matrices, a relevant dependence of experimental values by temperature was observed. The following considerations can be pointed out:

- at room temperature the highest tensile strength was obtained for the Ti-4.5Al-3V-2Mo-2Fe matrix; the measured average value was 1230 MPa for samples obtained at 850°C/1200mbar/45m (SP700_850). This value can be compared with that of the bulk materials [10] which is, after annealing, 1025 MPa. Highest value for the Ti-6Al-2Sn-4Zr-2Mo matrix was 1168 MPa (sample obtained at 910°C/1200mbar/45m - Ti6242_910) against 963 MPa for the bulk material in the duplex annealed state;
- at high temperatures for the Ti-4.5Al-3V-2Mo-2Fe matrix, the following sequence for the tensile strengths was obtained: 1039 MPa at 300°C, 813 MPa at 450°C and 457 MPa at 600°C for the samples SP700_850. Literature data [10] referred to sheet materials report ultimate tensile strength values of about 750 MPa at 300°C, 600 MPa at 450°C and 280 MPa at 600°C;
- at high temperatures for the Ti-6Al-2Sn-4Zr-2Mo matrix, the following sequence for the tensile strengths was obtained: 1068 MPa at 300°C and 865 MPa at 450°C for samples Ti6242_910. Literature data [10] referred to sheet materials report ultimate tensile strength values of about 840 MPa at 300°C and 740 MPa at 450°C;

All measured values for the fabricated composites were higher than those of unreinforced corresponding titanium alloys after usual thermal treatments. Average values of Young modulus in fiber direction at room temperature were in the range 160-170 GPa, values comparable with those predictable by the role of mixture; elongation at rupture in

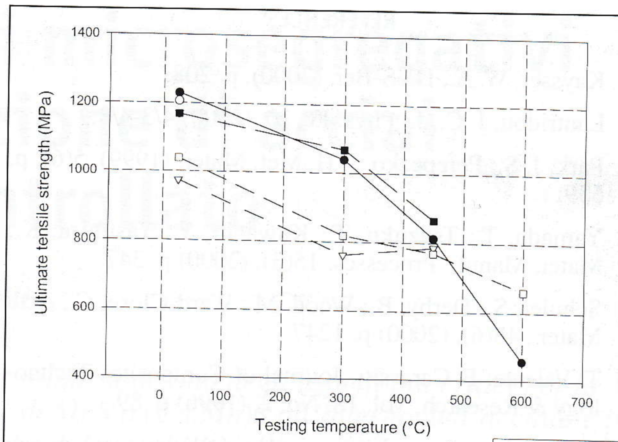

Fig. 8 Ultimate tensile strength - composite miltilayer

Fig. 8 Tensione di rottura per il composito multistrato

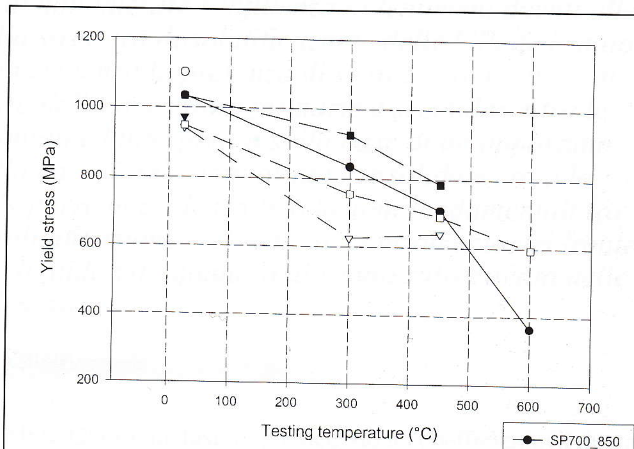

Fig. 9 Yield stress - composite multilayerf

Fig. 9 Tensione di snervamento per il composito multistrato

all tests were higher than 2.5% (room temperature) up to 10% for samples SP700_850 at 600°C.

CONCLUSIONS

Composite SiC fiber reinforced titanium alloys (Ti-4.5Al-3V-2Mo-2Fe; Ti-6Al-2Sn-4Zr-2Mo) preforms fabricated by high pressure plasma spraying in inert environment were hipped to obtain 2 mm thick composite multilayers, with 21% as volumetric fiber reinforcement. Despite the not very high value of fiber reinforcement fraction, significant improvements in mechanical properties, especially at high temperatures, in terms of tensile strength, as compared to values of the corresponding bulk titanium alloys were achieved, thus justifying the possibility to use the adopted technologies for the fabrication of structural components with potential application in the aerospace field.

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ABSTRACT

FABBRICAZIONE DI MATERIALI COMPOSTI A MATRICE DI TITANIO TRAMITE HIP DI PREFORME OTTENUTE CON DEPOSIZIONE AL PLASMA

Il presente lavoro è centrato sulla fabbricazione di materiali compositi multistrato a matrice di titanio, ottenuti mediante pressatura isostatica a caldo (HIP) di preforme realizzate con un processo di deposizione al plasma. Sono state utilizzate due diverse tipologie di matrici metalliche, la prima consistente in una lega Ti-4.5Al-3V-2Mo-2Fe, denominata anche SP700, la seconda in una lega Ti-6Al-2Sn-4Zr-2Mo. In entrambi i casi il rinforzo è stato ottenuto impiegando fibre continue di carburo di silicio del tipo SCS-6. La fabbricazione dei mononastri è stata condotta tramite l'uso di un impianto di tipo CAPS (Controlled Atmosphere Plasma Spray) realizzando deposizioni a pressioni in camera anche superiori al valore di pressione atmosferica. Dopo ottimizzazione dei parametri del processo di deposizione, sulla base di una campagna sperimentale di prove progettata in accordo con tecniche di Design of Experiment (DOE), i mononastri così realizzati sono stati utilizzati per la fabbricazione di compositi multistrato. Il processo di pressatura isostatica a caldo è stato condotto con diverse combinazioni dei valori dei principali parametri di processo variando la temperatura nell'intervallo 850-940°C, il tempo di stasi nell'intervallo 30-45 minuti e mantenendo fissa la pressione al valore di 1200 bar. I multistrati ottenuti sono stati sottoposti a prove meccaniche di trazione a temperatura ambiente e ad alta temperatura (fino a 600°C). Per le prove a temperatura ambiente il valore più alto di tensione di rottura, pari a 1230 MPa, è stato ottenuto per il multistrato a matrice Ti-4.5Al-3V-2Mo-2Fe realizzato con la combinazione dei parametri di pressatura a caldo 850°C, 1200 bar, 45 minuti. Ad alta temperatura sono stati ottenuti i valori di 1039 MPa, 813 MPa e 457 MPa rispettivamente a 300°C, 450°C e 600°C per il multistrato a matrice Ti-4.5Al-3V-2Mo-2Fe. Per il composito a matrice Ti-6Al-2Sn-4Zr-2Mo la sequenza ottenuta è stata di 1068 MPa e 865 MPa rispettivamente a 300°C e 450°C. I valori medi misurati del modulo di Young sono risultati compresi nell'intervallo 160-170 GPa.