PROCESSING & PROPERTIES OF CONTINUOUS FIBRE REINFORCED TITANIUM MATRIX AXI-SYMMETRIC COMPOSITES

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SUMMARY: This paper describes a unique vacuum plasma spray technique for the manufacture of axi-symmetric titanium matrix composite structures. The process involves the simultaneous winding of a reinforcing fibre and vacuum plasma spraying the matrix onto a rotating and axially translating mandrel followed by hot isostatic pressing. The attributes of Ti-6Al-4V rings reinforced with 142 µm diameter silicon carbide fibre produced using this technique are described, including: the response to hot isostatic pressing and heat treatment, interfacial reactions, and interface shear strength. The interface shear strength compares favourably to Ti-6Al-4V/SiC structures produced by other processes. By adjusting the rotational speed, and/or axial mandrel motion, the fibre distribution and volume fraction can be controlled. The results indicate that this process represents a viable technique for the manufacture of fibre reinforced titanium matrix composites for axi-symmetric geometries.

KEYWORDS: titanium, MMC, fibre, SiC, axi-symmetric, plasma spray, gas turbine, fibre push-out, nanomechanical probe

INTRODUCTION

Several proposed innovative designs of both axial and radial compressor disk structures for advanced gas turbine engines incorporate continuous fibre reinforced titanium matrix composites (TMC) [1,2]. These applications require axi-symmetrically shaped components of continuous fibre reinforced TMCs with the fibre oriented in the circumferential direction. For TMCs to be successful in these applications, a manufacturing process that is both technically and economically feasible is required. The development of a mechanically simple continuous manufacturing process would improve the economic feasibility, while at the same time minimise the technical difficulty.
The thermal deposition of titanium alloy powders via vacuum plasma spraying (VPS) has been explored previously for the manufacture of fibre reinforced TMCs [3-5]. However, most prior work has involved producing a monotape by VPS deposition of the matrix onto a single layer of fibre and subsequent consolidation of multiple monotapes. Such multi-step processes may not be economically favourable or suited for the fabrication of axi-symmetric geometries. Given the foregoing, the next section describes a single step VPS process for the manufacture of continuous fibre reinforced TMCs for axi-symmetric geometries, compatible with subsequent hot isostatic pressing (HIPing) and heat treatment [6]. Subsequent sections describe the attributes of Ti-6Al-4V rings reinforced with SiC fibre produced using this technique.

**PROCESS DESCRIPTION**

The process consists of concurrently plasma spraying the matrix material and winding the reinforcing fibre onto a rotating and translating mandrel, as shown schematically in Fig. 1, with the whole process occurring within a vacuum chamber. The matrix powder is heated in the plasma to a molten or semi-molten state, before spray deposition onto the substrate of fibre and previously sprayed matrix. On impinging this substrate, the heated matrix powder will deform around the reinforcing fibre and rapidly cool, thereby minimising detrimental matrix-fibre reactions. Adequate fibre tension is maintained by the fibre guide and back tension applied by the fibre unwind spool. In this manner an axi-symmetric part with multiple layers of fibre aligned in the circumferential direction is produced directly in one step. By adjusting the rotational speed, the mandrel axial movement, or the feed rate of the matrix powder to the plasma torch, the fibre distribution and volume fraction can be controlled. A wide variety of compatible fibre-matrix systems can be utilised. However, due to the short contact time of the fibre with the semi-molten matrix, the process is particularly advantageous for reactive fibre-matrix systems. This advantage is demonstrated in the following sections through application of the process to manufacture Ti-6Al-4V/SiC axi-symmetric composite rings.

**MATERIALS & EXPERIMENTAL PROCEDURE**

The matrix powder is Ti-6Al-4V with a total interstitial content of <0.2 weight per cent. The powder particles have an irregular shape with a maximum dimension of 70 to 100 µm. The reinforcing fibre is SCS-6 SiC fibre manufactured by Textron Specialty Metals. This fibre is...
manufactured by chemical vapour deposition of SiC onto a 40 µm carbon core and has a 3 µm outer carbon coating for an overall diameter of 142 µm.

A series of preliminary experiments were undertaken to determine the appropriate VPS process parameters. For the results presented in this paper a plasma gas mixture of nearly equal volumes of argon-hydrogen-helium was used and the matrix powder feed rate was 23 g/min. The rotation and translation of the mandrel shown in Fig. 1 were set between 45 to 60 rpm and 1.5 to 2 mm/s respectively. The composite rings produced have an inside diameter of about 50 mm, corresponding to a fibre feed rate of about 10 m/min. As indicated previously, the matrix powder feed rate and mandrel motions control the fibre volume fraction and arrangement.

After VPS fabrication, the rings were cut into sections for metallographic analysis or further processing. The metallographic analysis consists of optical and electron microscopy and X-ray diffraction analysis using standard techniques. Some sections were vacuum encapsulated and HIPed for 2 hours at 210 MPa at temperatures between 870°C and 1040°C. As well, some sections were heat treated, according to various thermal profiles, by encapsulating in quartz tubes backfilled with a partial pressure of argon.

Mechanical properties were evaluated by microhardness measurements and fibre push out tests using a nanomechanical probe. For the latter tests, fibres were pushed out from 0.2 µm slices of the composite using a 100 µm diameter flat diamond indenter. Using an optical microscope, the indenter was positioned at the centre of the fibre, and the fibre pushed out using a motorised stage with a constant displacement rate of 1.47 µm/s, while acquiring data for load and displacement. To minimise bending, the composite sample was clamped between two stainless steel plates. For each of the conditions reported, at least 20 fibres were pushed out. Complete details of the nanomechanical probe and fibre push out tests are presented elsewhere [7,8].

RESULTS & DISCUSSION

A portion of a Ti-6Al-4V/SiC ring produced via the technique of Fig. 1 is illustrated in Fig. 2. The fibre was wound and the matrix sprayed onto a copper mandrel of 50 mm diameter. This ring has a radial thickness of ≈2 mm, an original axial length of 8 cm and consists of 4 layers of fibre. The total plasma spraying time to produce this ring was about 6 minutes. Fig. 3 illustrates a section through a Ti-6Al-4V/SiC ring consisting of 37 layers of circumferential SiC fibre arranged in an orderly array. The section of Fig. 3 is cut from a composite ring of about 65 mm diameter and 50 mm axial length, which required 35 minutes of plasma spraying to produce. An important feature of Fig. 3 is the orderly array of fibre, indicating good control of fibre placement during VPS processing. Therefore, the fibre volume fraction could be increased substantially from that of Fig. 3 without problems associated with fibre touching.
A typical microstructure of the composite in the as-sprayed condition is illustrated in Fig. 4. Importantly, no damage to the fibre coating is evident as a result of impingement by high velocity titanium powder spray. However, image analysis indicates that the matrix contains about 16% porosity. Furthermore, a higher density of porosity exists on the ‘inner’ side of the fibre – the side of the fibre in the ‘shadow’ of the plasma spray. A similar porosity distribution has been noted in other VPS produced Ti/SiC composites [9,10]. It is expected that utilising two plasma torches and/or moving the plasma torch during matrix powder spraying would improve the volume fraction and distribution of porosity.

The microhardness of the matrix in the radial direction is shown in Fig. 5. The as-sprayed condition shows large fluctuations in hardness. This is likely a result of individual plasma sprayed layers within the matrix. As the matrix powder traverses through the plasma to the composite substrate, individual powder particles become partially or fully molten. Therefore, on impinging the composite substrate individual particles bond together and deform around the fibres. The matrix rapidly cools, since the mandrel on which the composite substrate accumulates, is water-cooled, Fig. 1. Thus, subsequent layers of matrix are sprayed onto the previous cooler layer, reducing the interlayer bonding. This disadvantage is mitigated by the advantage of minimal fibre-matrix contact at high temperature, ensuring that the fibre coating remains intact, Fig. 4.

Fig. 6 illustrates the microstructure following HIP’ing at 870°C. HIP’ing successfully reduces the volume fraction of porosity to <1%, with the fibre coating still remaining ostensibly intact, even during HIP’ing at 1040°C. As expected, the matrix microstructure varied with HIP temperature. HIP’ing at 1040°C results in matrix regions with a transformed β structure. HIP’ing improves the matrix uniformity and strength, as evidenced by the nearly constant hardness of the HIPed matrix, Fig. 5.
Heat treatment subsequent to HIPing can be used to modify the matrix microstructure. However, increasing thermal exposure of the composite, either during HIPing or heat treatment, increases the fibre-matrix interface reactions. While the interface shear strength depends on a well bonded fibre and matrix, excessive thermal exposure may cause the formation of deleterious interface phases. Even in the as-sprayed condition, XRD analysis indicates the presence of titanium carbides and silicides, Fig. 7. Since these are brittle phases they potentially may lead to premature failure at the fibre-matrix interface. After HIPing and heat treatment for 4 h at 950°C an almost continuous reaction layer exists at the fibre-matrix interface, Fig. 8.

A second ramification of thermal exposure during HIPing or heat treatment is diffusion of silicon from the fibre into the matrix. SEM-EDS measurements indicate that, relative to the as-sprayed condition, HIPing or heat treatment caused the matrix silicon content 10 µm from the fibre to increase from 0.09% to as high as 0.44%. Diffusion of silicon into the matrix has been observed previously for plasma sprayed Ti/SiC fibre composites [5], but the current results imply substantially larger diffusion distances than previously reported.

Fig. 9 illustrates typical load-displacement plots from fibre push out tests. The fibre-matrix interface shear strength, $\tau$, is estimated from the peak load, $P$, of Fig. 9 using the equation:

$$\tau = \frac{P}{2\pi Rh}$$

where $R$ is the fibre radius and $h$ is the thickness of the specimen. Accordingly, the interface shear strength of the as-sprayed and HIPed conditions is about 36 and 215.
MPa respectively. The HIPed shear strength compares favourably with fibre push-out results for TiSiC fibre composites produced by other fabrication techniques [3,11,12]. Indeed, specimen thickness influences the fibre push-out debonding strength, with specimens >0.6 mm required to achieve the maximum fibre push-out debond strengths [11]. Therefore, since the current tests used a sample thickness of only 0.2 mm, the fibre-matrix strength after HIPing is likely substantially higher than the measured strength of 215 MPa. Consistent with the increased interface strength of Fig. 9, a change in the interface failure mode also occurred. In the as-sprayed condition, interface failure occurs with minimal damage or deformation of the fibre or the matrix, Fig. 10(a). In contrast, after HIPing, SEM examination of the pushed-out fibre reveals substantial fibre cracking and matrix deformation, Fig. 10(b). The fibre push-out results and SEM examination clearly indicate that the interfacial reactions occurring during HIPing improve the fibre-matrix bond.

![Fig. 9. Fibre push-out load displacement plots for the composite of Fig. 3 in the (a) as-sprayed, and (b) HIP’ed conditions.](image)

![Fig. 10. SEM micrographs illustrating fibre-matrix interface after fibre-push-out testing for the (a) as-sprayed, and (b) HIP’ed conditions.](image)
SUMMARY & CONCLUSIONS

The results confirm that a fabrication process involving simultaneously winding the fibre and vacuum plasma spraying of the matrix onto a rotating mandrel, followed by HIPing is a viable technique for the manufacture of fibre reinforced titanium matrix composites for axi-symmetric geometries. The matrix microstructure can be modified by appropriate choice of the HIPing and heat treatment thermal profile, without causing deleterious fibre-matrix reactions. Indeed, post-VPS HIPing, and heat treatment, are essential to develop uniform microstructures and maximum fibre-matrix interface strength, which for the current technique compares favourably with Ti/SiC fibre composites produced by other methods.

The continuous nature, speed and simplicity of the process presented is a major advantage compared to the multi-step lay-up and consolidation procedures associated with many alternative fabrication technologies for fibre reinforced composites with axi-symmetric geometries. Future research will be aimed at increasing the volume fraction of reinforcing fibres in the axi-symmetric shapes.

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