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Fabrication of Ti/SiC_f metal matrix composites by vacuum plasma spraying

Y.Y. ZHAO, P.S. GRANT and B. CANTOR

Oxford Centre for Advanced Materials and Composites, Department of Materials, University of Oxford, Parks road, Oxford OX1 3PH, England

ABSTRACT

Ti/SiC_f MMC monotapes have been successfully fabricated by vacuum plasma spraying. The as-sprayed MMC monotapes have a uniform fibre distribution, dense matrix and no fibre-matrix interfacial reaction. The effects of processing parameters such as H₂ flow rate, plasma current, chamber pressure and spray distance on the monotape temperature, porosity and fibre infiltration have been evaluated quantitatively. The mechanism of fibre infiltration is discussed in detail.

1. INTRODUCTION

Titanium metal matrix composites (MMCs) are potential materials for application in the aerospace industry, which requires low density materials with good mechanical properties at elevated temperatures. Ti by itself has high specific strength at room and moderately elevated temperatures, and excellent general corrosion resistance. By combining Ti with continuous silicon carbide fibres (SiC_f) increased stiffness and decreased weight can be achieved^[1]. Ti MMCs can be used in virtually every sector of the aerospace industry: aeroengines, airframes, spacecraft and guided weapons^[2-5].

The manufacture of bulk Ti MMCs is difficult because of the high melting point and high reactivity of Ti. Vacuum pressing, diffusion bonding and hot isostatic pressing of foil/fibre/foil layups are currently the most widely used techniques to manufacture Ti MMCs^[1,6,7], although a number of other consolidation techniques have also been investigated over the past three decades^[8]. However, processes such as diffusion bonding require thermal cycles which often produce significant interfacial reaction between Ti and SiC_f^[1,7,9]. Moreover, Ti foils are expensive and time-consuming to produce.

Vacuum plasma spraying (VPS) is a promising primary manufacturing route for the fabrication of Ti MMCs. In VPS, a potential difference of 10 - 100 V is applied across two fixed electrodes in a plasma gun, with an inert gas flowing between the two electrodes to create and maintain a hot gas plasma jet. Metallic powders of 20 - 100 μm are fed continuously into the plasma where they are melted and propelled at high velocity onto a prepared workpiece supporting a fibre preform as shown in Fig. 1. The molten droplets deposit and infiltrate into the fibre preform to produce an MMC monotape. The as-sprayed Ti MMC monotapes are subsequently cut, stacked and hot pressed to form a fully dense bulk Ti MMC component.

The advantages of VPS for the manufacture of Ti MMCs are as follows^[10-13]:

Fig. 1 Schematic of the plasma spraying process

- (1) High droplet cooling rates on impact with the fibre preform prevent excessive fibre-matrix reaction, and promote rapid solidification benefits such as small matrix grain size, uniform composition and extended solid solubility;
- (2) Inert, low chamber pressure atmosphere allows reactive metals such as Ti to be sprayed without excessive oxidation;
- (3) Consolidated bulk MMCs exhibit a uniform fibre distribution, because the densely deposited matrix holds the fibres rigidly in place during secondary hot pressing.

This paper describes an investigation into the effect of processing conditions on the microstructural characteristics and thermal histories of Ti/SiC_f MMC monotapes fabricated by VPS.

2. EXPERIMENTAL

Ti MMC monotapes containing SiC_f reinforcement were manufactured by spraying commercially pure Ti powder onto prepared SiC_f preforms. The composition of the powder was: Ti 99.65%, Fe 0.24%, Al 0.11%, with particle diameters of 0 - 90 μ m. The SiC_f preforms were made by winding 15 revolutions each of 100- μ m-diameter uncoated and TiB₂/C-coated continuous SIGMA SiC_f with a spacing of 200 μ m on a flat mild steel substrate mounted on a holder, as shown schematically in Fig. 2. The substrate was grit blasted before winding to improve adhesion between the deposit and the substrate.

The VPS experiments were performed in a Plasma Technik A2000 system shown schematically in Fig. 3. The chamber was first evacuated to below 10^{-1} mbar, and then back filled with Ar to 50 - 250 mbar. The plasma gas consisted of a mixture of primary Ar and secondary H₂, and the Ti powder was fed continuously into the plasma using an Ar carrier gas. The plasma gun was preprogrammed to weave over the substrate 25 times, with each weave containing 13 parallel passes at 5 mm spacing, with a gun traverse speed of 300 mm/s. The temperature of each monotape during spraying and cooling was measured by a thermocouple projecting out of the substrate surface between the two fibre preforms as shown in Fig. 2, with temperatures recorded every 2 s on a Schlumberger 3531 datalogger. Typical spraying conditions were: primary Ar flow rate 35 l/min, secondary H₂ flow rate 8 l/min, chamber pressure 150 mbar, plasma voltage 62 V, plasma current 700 A, powder feed rate 0.4 g/s, carrier Ar flow rate 1.6 l/min and spray distance 300 mm. The different spraying parameters were varied independently to investigate their effect on the resulting MMC monotapes.

Fig. 2 Design of preform holder for fibre winding and VPS

Fig. 3 Schematic diagram of the VPS system

Fig. 4 Schematic diagram of Ti/SiC_f MMC monotape microstructure

As-sprayed samples were cut, mounted, ground, polished with 30 and then 6 μm diamond, final polished with OP-S, and then etched in a modified Keller's solution for subsequent observation in a Quantimet 900 image analyzer and a Hitachi S530 scanning electron microscope. The image analyzer was used for quantitative measurements of porosity ϵ and fibre infiltration ϕ defined as the ratio of matrix density ρ in MMC and monolithic regions of the deposit (A and B as shown in Fig. 4).

$$\phi = \frac{\rho_A}{\rho_B} = \frac{1-\epsilon_A}{1-\epsilon_B}$$

3. RESULTS AND DISCUSSION

3.1 Size distribution of Ti powder

The morphology and size distribution of the Ti powder feed are shown in Figs. 5 and 6 respectively. The Ti powder was highly angular as shown in Fig. 5, with 90 wt% in the range 32 - 90 μm . Selection of suitable powder size and morphology is needed to prevent powder agglomeration and poor flowability. Small particles may evaporate in the plasma before reaching the substrate, and large particles may not melt fully in flight. The Ti powder shown in Figs. 5 and 6 had good flowability for plasma spraying although, under some processing conditions, the larger particles were not fully melted.

3.2 MMC microstructure

Figs. 7(a)-(c) show typical low magnification micrographs of the as-sprayed Ti/SiC_f microstructure in the MMC monotapes. The SiC fibres were distributed uniformly in a near dense Ti matrix. The Ti matrix had a fine-scale splat microstructure, although unmelted particles were occasionally present.

Fig. 8 shows high magnification micrographs of the Ti/SiC_f interface. There was no observable interfacial reaction between the Ti matrix and either uncoated or TiB₂/C-coated SiC fibres. In electric arc spray formed Ti MMC monotapes^[14], 2 - 10 μm long TiB needles are formed during spraying at the Ti-6Al-4V matrix/TiB₂/C-coated SiC_f interfaces, because of a high deposition rate (~ 0.9 g/s) and corresponding low cooling rates in large molten droplets (~ 200 μm). In VPS, lower deposition rates (~ 0.2 - 0.4 g/s) and smaller droplets (< 90 μm) lead to much higher cooling rates of 10^3 - 10^6 °C/s^[15], which inhibit fibre-matrix interfacial reaction.

3.3 MMC thermal history

Figs. 9(a)-(d) show the effect of H₂ flow rate (F), plasma current (I), chamber pressure (P) and spray distance (Z) respectively on the MMC monotape thermal history during spraying. In all cases, the monotape temperature increased during spraying and then decreased gradually after the end of spraying. The monotape temperature increased with increasing H₂ flow rate and plasma current, and with decreasing chamber pressure and spray distance.

Fig. 9(a) shows that the monotape temperature increased with increasing H₂ flow rate, reaching 460 °C after about 5 mins of spraying with F = 2 l/min compared with 620 °C with F = 8 l/min. Increasing the H₂ flow rate in the plasma gas increases the plasma enthalpy because of the heat of H₂ dissociation^[11], leading to higher droplet temperatures at the point of deposition and a hotter plasma striking the growing deposit surface. Similarly, Fig. 9(b) shows that the monotape temperature increased with increasing plasma current, again because of increased plasma enthalpy, from 510 °C after about 5 mins of spraying with I = 500 A to 680 °C with I = 850 A. Fig. 9(c) shows that the monotape temperature increased with decreasing chamber pressure, from 560 °C with P = 250 mbar to 790 °C with P = 50 mbar; and Fig. 9(d) shows that the monotape temperature increased with decreasing spray distance, from 430 °C at Z = 400 mm to 660 °C at Z = 200 mm. Decreasing chamber pressure lengthens the plasma flame, so both effects are again caused by hotter droplet temperatures at the point of deposition and a hotter gas plasma striking the growing deposit surface.

Fig. 5 Morphology of Ti powder

Fig. 6 Size distribution of Ti powder

Fig. 7 Microstructure of the as-sprayed Ti/SiC_f MMC monotapes: a) cross section; b) longitudinal section; and c) etched cross section

Fig. 8 Fibre-matrix interfaces in VPS Ti/SiC_f monotapes: a) uncoated SiC fibre; and b) TiB₂/C-coated SiC fibre

Fig. 9 Thermal histories of Ti/SiC_f monotapes for different processing parameters:
a) H₂ flow rate; b) plasma current; c) chamber pressure; and d) spray distance

Fig.10 a) Good fibre alignment and infiltration and
b) poor fibre alignment and infiltration in VPS Ti/SiC_f monotapes

3.4 Fibre infiltration and monotape porosity

Figs. 10(a) and (b) show two examples of the Ti/SiC_f monotapes with different types of pores. The monotape in Fig. 10(a) had a well aligned fibre preform before spraying, and maintained good alignment during spraying. A few ~ 50 μm sized pores were formed behind the fibres and near the monotape-substrate interface, as shown at A in Fig. 10(a). Occasional ~ 80 μm sized pores were also present in the bulk deposit as shown at B in Fig. 10(a). The monotape in Fig. 10(b) had a loose fibre preform after winding, and the fibre became misaligned during spraying with much larger pores behind the fibres. Figs. 11 (a)-(d) show the variation of monotape porosity and fibre infiltration with H₂ flow rate, plasma current, chamber pressure and spray distance respectively. Also included in Figs. 11 (a)-(d) are the corresponding maximum temperatures, taken from Figs. 9(a)-(d).

Fig.11 Relationships between fibre infiltration, monotape porosity and precessing parameters:
a) H_2 flow rate; b) plasma current; c) chamber pressure; and d) spray distance

There are two factors which control the extent of fibre infiltration and therefore the final porosity in the MMC monotapes:

- (1) Droplet deposition from the gas plasma involves simultaneous spreading and solidification to form thin splats on the top surface of the growing deposit. With high droplet temperatures, spreading is complete before solidification impedes material flow, leading to good fibre infiltration. With low temperatures, however, the onset of solidification prevents efficient spreading, leading to poor fibre infiltration.
- (2) Some droplets arrive and solidify directly on the fibres themselves, and other droplets arrive between the fibres and solidify on the underlying substrate. With small interfibre spacings and large fibre-substrate separations, droplet solidification directly on the fibres restricts infiltration between the fibres, and creates large pores behind them at positions such as C in Fig. 4. With large interfibre spacings and small fibre-substrate separations, the droplets infiltrate readily and porosity is much reduced. Droplet solidification on the fibres can also sometimes lead to poor infilling and porosity in the bulk deposit at positions such as D in Fig. 4.

Fig. 10(a) shows relatively low porosity in the monotape, because the interfibre spacing is larger than the fibre diameter and the fibre-substrate separation is small. However, Fig. 10(b) shows much higher porosity because of a small interfibre spacing and large fibre-substrate separation. Good fibre infiltration was found to require a fibre-substrate separation below a critical value of about half of the fibre diameter, i.e. $50\ \mu\text{m}$. Fig. 10 (a) also shows a large pore above two adjacent fibres at the position marked B. A deep valley can form in this region where thickening Ti deposits on adjacent fibres meet, resulting in poor infilling and porosity.

Increasing H_2 flow rate and plasma current increase the plasma enthalpy. Decreasing chamber pressure increases the plasma length, and consequently increases droplet residence times in the hot plasma core. All these effects increase droplet temperatures, leading to better fibre infiltration and lower porosity as shown in Figs. 11(a)-(c). However, there is no improvement in fibre infiltration when the H_2 flow rate is above 4 l/min, because almost all of the droplets are fully molten. Fibre infiltration at $I = 850\ \text{A}$ is also not as high as expected. This is partly due to a loose fibre preform, and partly because of increased gas velocities and therefore reduced droplet

residence time^[6]. There is no apparent variation of fibre infiltration with chamber pressure from 50 to 150 mbar, because of a decreased plasma mean free path, leading to decreased heat transfer between the plasma and the droplets^[7]. Varying spray distance in the range of 300 - 500 mm has little effect on fibre infiltration, although monotape porosity is slightly increased with increasing spray distance as shown in Fig. 11(d). However, too short spray distances decrease droplet residence times, while too large distances result in droplet cooling, all leading to lower droplet temperatures.

4. CONCLUSIONS

- (1) Ti/SiC_f MMC monotapes have been fabricated successfully by vacuum plasma spraying. No interfacial reaction is observed between the Ti matrix and either uncoated or TiB₂/C-coated SiC fibres in the as-sprayed Ti/SiC_f monotapes.
- (2) Monotape temperature during spraying increases with increasing H₂ flow rate, increasing plasma current, decreasing chamber pressure and decreasing spray distance.
- (3) Deposit porosity decreases with increasing H₂ flow rate, increasing plasma current and decreasing spray distance. Fibre infiltration increases with increasing H₂ flow rate, decreasing chamber pressure and increasing plasma current, but is unaffected by varying spray distance.
- (4) Under optimum VPS processing conditions, high quality MMC monotapes can be produced, with porosity as low as 0.5% and fibre infiltration as high as 0.92.

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