



A study of interface reaction zone in a SiC fibre/Ti-17 composite

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ABSTRACT

The interface reaction zone (RZ) in a unidirectional continuous carbon-coated SiC fibre reinforced Ti-17 titanium alloy composite is investigated. Micro-computed tomography (CT), scanning and transmission electron microscopy are employed to characterize the fibre/matrix interface. It is revealed that the interface RZ is a 400 nm thick titanium carbide (TiC) layer which is composed of two sublayers, a 60 nm thick fine-grained sublayer and an approximate 340 nm thick coarse-grained sublayer. The RZ is formed through chemical reaction between carbon coating on the SiC fibre surface and Ti, Zr and Sn in the alloy matrix. The reaction is controlled by atom diffusion occurring at the fibre/matrix interface. However, in the reaction process, Al, Cr and Mo in the matrix are rejected and piled up in front of the RZ on the matrix side. A structure model is proposed to describe the formation mechanism of the interface RZ.

1. Introduction

Unidirectional continuous fibre reinforced titanium alloy matrix composites (TMCs) have gained significant attractiveness for material researchers and engineers since they were initially proposed in 1960s due to their high specific strength, stiffness and fatigue and creep resistance, consequently, the possibility of marked weight reductions of 30–70% compared to titanium alloys, steels or nickel-based superalloys. The TMCs give rise to interests of the potential applications in the compressor stage of aero engines (Campbell, 2012; Chou et al., 1985; Lissenden et al., 1995; Saito, 2004; Singerman and Jackson, 1996; Takahashi et al., 2012; Vassel, 1997).

In determining the processing/manufacturing parameters and macroscopic performance of the TMCs, fibre/matrix interface plays a critical role since the fibre and matrix are interconnected and interactive at such interface. Therefore, investigations have been largely focused on the interface in order to improve TMC processing/manufacturing technology and, consequently, its performance. For example, by investigating the interfacial reaction phenomena, the stability and compatibility of the relevant materials can be evaluated. The outcome of such evaluation provides the necessary knowledge for interface design in TMCs for optimising the processing/manufacturing technology and improving product performance (Fromentin et al., 1996; Lacoste et al., 2015; Li et al., 2011; Ngai et al., 2011; Yang et al., 1998b).

Multi-layered reaction zone at the fibre/matrix interface has been

observed in various titanium alloy matrix composites reinforced by SiC fibre with protective carbon-rich coating in previous investigations. The RZ commonly contains titanium carbides due to the chemical reaction between carbon and titanium (Dudek et al., 1997; Hall and Ni, 1995; Lacoste et al., 2015; Yang and Dudek, 1997; Yang et al., 1998b). Other phases, such as titanium silicides, were also identified in the RZ due to the reaction between titanium and silicon in the carbon-rich coating (Dudek et al., 1997; Hall and Ni, 1995; Yang and Dudek, 1997; Yang et al., 1998b). In addition, titanium aluminium carbide may also be present in the RZ owing to the reaction between carbon and titanium/aluminum in the alloy matrix, for example, Ti–25Al–10Nb–3V–1Mo matrix (Hall and Ni, 1995; Yang and Dudek, 1997; Yang et al., 1998b). The structure of the interface RZ varies significantly depending on the elemental components of both protective carbon coating on SiC fibre surface and matrix alloy. The other critical factor that influences the RZ structure is the fabrication process of the composites (Dudek et al., 1997; Fan et al., 1997; Guo et al., 1998; Lacoste et al., 2015; Zhang et al., 2014, 2013; Zhao et al., 2013). Thus, in the present study, the interface RZ in a SiC fibre reinforced Ti-17 matrix composite newly developed by Aero Engine Corporation of China (AECC) is investigated in order that its characteristic structure can be fully understood, which is essential for optimising the fabrication process and the property of the composite.

Further, chemical reaction at the fibre/matrix interface takes place through atom diffusion which is driven by the difference in chemical

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potential formed by the element concentration gradient at the interface. Therefore, investigation on the atom diffusion across the interface is also the focus in the field (Guo et al., 1998; Guo et al., 1993; Hall and Lirn, 1992; Jeng et al., 1989; Martineau et al., 1984; Metcalfe, 1974; Naka et al., 1997; Xun et al., 2000; Zhang et al., 2014). But very few studies investigate the behaviour of non-reactive atoms in the regions adjacent to the RZ. To the best of the authors' knowledge, only the rejection of aluminium by the RZ in the C-coated or B₄C /boron-coated SiC fibre reinforced titanium composites has been reported (Hall and Lirn, 1992; Jeng et al., 1989; Martineau et al., 1984; Metcalfe, 1974). In order to further understand the interfacial diffusivity of the alloying elements in the Ti-17 alloy, and their contribution to the interface reaction and stability, the present study also investigates the diffusion phenomena of all the alloying elements at the fibre/matrix interfacial region in the composite. A structural model is proposed to describe the formation mechanism of the reaction zone at fibre/matrix interface and the associated atom diffusion.

2. Experimental

The composite investigated in this study is a unidirectional continuous SiC fibre reinforced titanium alloy Ti-17 (Ti-5Al-2Sn-2Zr-4Cr-4Mo) composite, which is fabricated using matrix coated fibre (MCF) route followed by hot isostatic pressing (HIP) at 860 °C/180 MPa for 30 min. The HIP temperature is the typical forging temperature to achieve equiaxed α structures in the matrix alloy. The diameter of the MCF before HIP is 150 μm . The final dimension of the composite bar is 15 mm (diameter). Ti-17 is selected as the matrix alloy due to its good tensile properties, creep resistance and fracture toughness over other titanium alloys such as Ti-6Al-4V, which is mainly used for the manufacturing of fan blades and compressor disks of aero engine. The SiC fibre of 100 μm diameter has a tungsten core of 14 μm diameter and 1 μm thick pure carbon coating. A high fibre volume fraction is employed for improvement of thermal and mechanical responses of the composite and more weight saving.

Fibre distribution in the composite was investigated using X-ray micro-computed tomography operated at 140 keV. Totally 1800 projections were acquired for 3D reconstruction. The morphology and chemical composition of interface RZ were characterised by scanning electron microscopy (SEM) with angle-selective backscattered (AsB) electron detector under 8 kV and energy dispersive X-ray spectroscopy (EDS) under 15 kV. Transmission electron microscopy (TEM) with EDS and selected area electron diffraction (SAED) were also employed under 300 kV to investigate the morphology, chemical composition and crystal structure of the RZ at the fibre/matrix interface.

Samples used for SEM examination were mechanically ground and polished with 3 μm finish. The polished sample surface was cleaned with plasma using a glow discharge optical emission spectroscopy (GDOES) instrument at an argon pressure of 600 Pa and applied voltage of 35 V with a power of 25 W for 10 s. The GDOES sputtering was employed to remove the contamination and artificial defects caused by deformation during mechanical grinding and polishing. Meanwhile the microstructure was revealed by the sputtering due to the sputtering rate variation at different microscopic sites with different elements concentrations and crystal structure. As for TEM specimens, 0.2 mm thick slices were taken from the block samples perpendicular to the fibre axis using low-speed diamond blades. The slices were then mechanically ground and polished on both sides with 0.25 μm finish until the thickness is smaller than 0.1 mm. The slices were finally punched into discs of 3 mm in diameter and milled in a precision ion polishing system (PIPS) at 4–6° tilt and 6 keV for 6 h until the disc is electron transparent in TEM under the accelerating voltage of 300 kV.

3. Results and discussion

The distribution of the carbon coated SiC fibres in the Ti-17 alloy

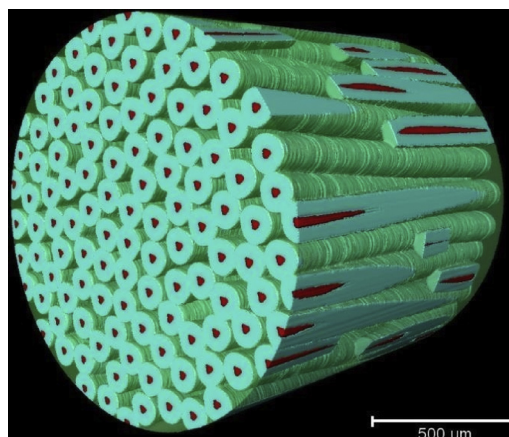


Fig. 1. X-ray 3D volumetric reconstruction showing the fibre distribution. Transparency is applied to the alloy matrix, and carbon coated SiC fibres are green with colour code R: 64, G: 227, B: 210 and tungsten cores are red with colour code R: 255, G: 0, B: 0 (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

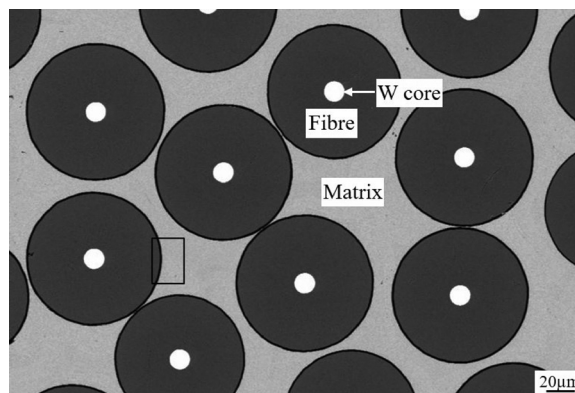


Fig. 2. Scanning electron micrograph of the cross section of the composite, showing the overview of the fibre distribution.

matrix is shown in Figs. 1 and 2. The fibres are statistically uniformly arranged in the composite although the fibre spacing varies at some local sites on microscopic scale (Fig. 2).

The reconstructed 3D X-ray micro-CT image (Fig. 1) demonstrates the configuration of the components in the composite. The Ti-17 matrix is transparent and the carbon coated SiC fibres are green with colour code R: 64, G: 227, B: 210 with red tungsten cores colour coded in R: 255, G: 0, B: 0. Fig. 1 also shows clearly that the fibres are not distorted or kinked in axis direction during fabrication. Direct impingement between the neighbouring fibres is not found on microscopic scales (Fig. 2).

The volume fraction of the fibres is determined to be 69.6% based on the 3D volumetric reconstruction. The calculated average fibre spacing is 113.2 μm using Eq. (1) (Hull and Clyne, 1996) based on the above measurement of fibre volume fraction.

$$V_f = \frac{\pi}{2\sqrt{3}} \left(\frac{R_f}{0.5S} \right)^2 \quad (1)$$

where V_f is the fibre volume fraction, R_f is the radius of the fibre and S is the fibre spacing which is described by the centre to centre distance between two neighbouring fibres in the idealised hexagonal arrangement of the fibres.

The typical structure of the fibre/matrix interface is shown in Fig. 3(a) which is obtained from the framed area in Fig. 2. Fig. 3(b) shows the framed area in Fig. 3(a) at an increased magnification, illustrating the interfacial region between the C coating on the fibre and

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