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Microstructure evolution of C/Mo double-coated SiC fiber reinforced Ti6Al4V composites

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ABSTRACT

C/Mo double-coated SiC fiber reinforced Ti6Al4V (SiC_f/C/Mo/Ti6Al4V) composites fabricated by foil-fiber-foil (FFF) were thermally exposed for the purpose of investigating thermal stability of the C/Mo duplex coating at high temperatures of 700–900 °C. It is revealed in the study that the SiC_f/C/Mo/Ti6Al4V composites have an excellent thermal stability at temperature below 700 °C compared to the poor thermal stability at 800 °C. When the composites were thermally exposed at 900 °C for 100 or 200 h, a large number of TiC, Ti_2AlC or Ti_3AlC particles were identified as reaction products in the matrix by means of energy-dispersive spectrometer analysis, X-ray diffraction analysis and transmission electron microscopy. The formation mechanism of these particular phases was analyzed in terms of thermodynamics, composition condition and element diffusion path. In addition, the longitudinal tensile performance of the composites thermally exposed under 900 °C/200 h was tested at room temperature, and the corresponding failure behavior was analyzed.

1. Introduction

Continuous SiC fiber-reinforced Ti-based composites are very promising in aerospace field because of their high specific strength and stiffness [1,2]. However, because of the severe fiber/matrix interfacial reaction, the service life of the composites is limited. Therefore, effective measures should be taken to retard or even prevent the interfacial reaction. Many investigations have confirmed that fiber protective coating is the most efficacious method to control the interfacial reaction [3–6].

Fiber/matrix interface modified by coating is generally evaluated by studying the interfacial reaction and coating consumption conditions after thermal exposure treatment. Yang et al. have studied the interfacial processes in SCS-6 SiC/TiB_2/super α_2 composites via vacuum thermal exposure treatment [7]. Their results shows that the interfacial reaction became more severe along with the increase of the duration, and the interfacial phase sequence of the composites thermally exposed under 900 °C/400 h is SiC/C/fine TiC/Ti_5Si_3/large TiC/Ti_3Si+TiB/Ti_3AlC+TiB/matrix. They concluded that TiB_2 can effectively slow down the diffusion of Ti prior to the formation of TiB.

However, due to the increase in the duration of thermal exposure and the depletion of the coating, the matrix adjacent to the coating is inevitably affected by the composition variation resulted from elements diffusion. In our previous work, Cu/Mo duplex coating was used to modify the SiC_f/Ti6Al4V composite, and it was found that Widmänstaten structure containing Ti_2Cu and α-Ti was formed as the diffusion of Cu into the matrix [8]. So we studied the effect of single Mo coating on SiC_f/Ti6Al4V composites [9]. After the composites were thermally exposed at 800 °C, the $\alpha+\beta$ matrix adjacent to the coating was β-stabilized by sufficient diffusion of Mo element. But no further investigation has been conducted in relation to the evolution of the global matrix and the corresponding effect on mechanical properties of the composites. And only a few studies emphasizing the matrix of the composites can be found out [10–12]. For instance, Martina et al. have studied the effect of isothermal exposure on the failure behavior of 1D-SCS6/super-α_2 composites [13]. The decreased performance of the composites was ascribed to the embrittlement of the super α_2 matrix resulting from the coarsening of the grains during processing, but no change in the phase composition of the matrix was found along with the exposure duration.

Wang et al. [14] utilized C/Mo duplex coating as interfacial modification layer of SiC_f/Ti6Al4V composites. The effect of C/Mo coating on the interfacial element diffusion and interfacial reaction was focused on in the study. According to the study, the C/Mo coating in the composites can be well retained under the thermal exposure below 700 °C, but the interfacial microstructure changes as more severe interfacial reaction took place in condition of thermal exposure at 800 °C. Therefore, a further study of thermal stability of the C/Mo coating and the microstructure evolution of the composites is necessary. In this work, thermal stability of the

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composites was investigated to evaluate the C/Mo duplex coating, and microstructure evolution of the matrix and the fractography of the composites after thermal exposure were also studied.

2. Experimental

The SiC$_f$/C/Mo/Ti6Al4V composites were fabricated by the foil-fiber-foil method with hot pressing parameters of 900 °C + 60 MPa + 60 min. The detailed fabrication process can be seen in our earlier work [14]. Fig. 1 shows a schematic of the C/Mo coated SiC fiber before hot pressing. After the preparation of the SiC$_f$/C/Mo/Ti6Al4V composites, a part of them was thermally exposed in vacuum in order to study the microstructure evolution of the composites. The thermal exposure conditions are 700 °C/100 h, 700 °C/200 h, 800 °C/100 h, 800 °C/180 h, 900 °C/200 h. Then metallographic specimens were prepared by conventional preparation methods of metallographic samples. After that the interface morphology and matrix microstructure of the samples were analyzed by a Zeiss SUPRA 55 field-emission scanning electron microscope (SEM) equipped with an Oxford INCA X-ray energy-dispersive spectrometer (EDS). A PANalytical X′Pert Pro MPD X-ray diffractometer (XRD) with a Cu target and an FEI Tecnai F30 transmission electron microscope (TEM) were also utilized to identify the phases in the matrix.

In order to study the influence of thermal exposure on the failure of the composite, a tensile test of the composite thermally exposed under 900 °C/200 h was carried out by an Instron-3382 tensile test machine with a tensile velocity of 1 mm/min. After that the fractography was carried out by SEM.

3. Results and discussion

3.1. Thermal stability of the C/Mo coating

Fig. 2 shows a cross-sectional SEM-backscattered electron (SEM-BSE) image of the interfacial zone of the as-prepared SiC$_f$/C/Mo/Ti6Al4V composites. According to our previous study [14], the interfacial phase sequence of the as-prepared composites from SiC side to the matrix is SiC|C/Mo|TiC|Mo$_{residual}$ + β-Ti|β-Ti + α-Ti$_{primary}$|Ti6Al4V.

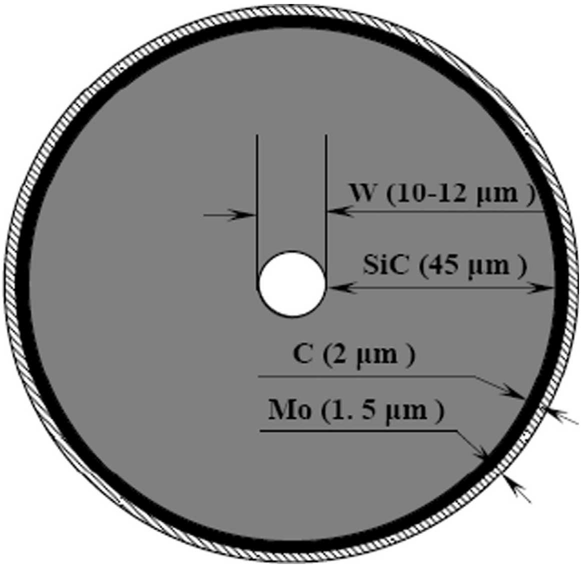

Fig. 1. Schematic of the C/Mo double-coated SiC fiber before hot pressing.

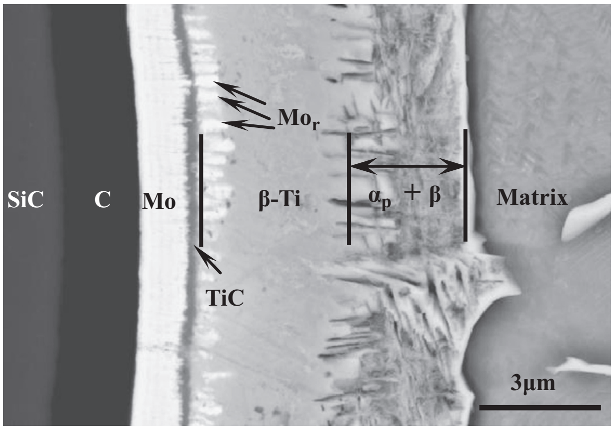

Fig. 2. SEM-BSE micrograph of the as-prepared SiC$_f$/C/Mo/Ti6Al4V composite.

Thermal stability of the C/Mo duplex coating on the interface was evaluated by vacuum thermal exposure experiments. Fig. 3 shows the interfacial zone SEM-BSE morphologies of the SiC$_f$/C/Mo/Ti6Al4V composites after being thermally exposed at 700 °C and 800 °C, respectively. From Fig. 3a, it is seen that the interfacial constitution has no obvious evolution after the composite was thermally exposed at 700 °C for 200 h. From Fig. 3b, it can be seen that, after 800 °C/180 h heat treatment, the C/Mo coating thinned obviously because of its diffusion to the matrix, and the primary α phase cannot be observed. Moreover, black particles which may be TiC or Ti$_3$AlC phase precipitated from the thickened β-Ti layer according to EDS analysis [14].

The thicknesses of C and Mo coatings were carefully measured in the relevant micrographs, and their average thicknesses were calculated. According to the average thickness data, the "thickness versus square root of time" curves were plotted in Fig. 4. It can be seen that the depletion of C and Mo coatings is controlled by diffusion. Therefore, the C/Mo coating depletion obeys parabolic law, i.e. $H = kt^{1/2} + b$ (where k is the rate constant, b and H are the original and final coating thicknesses, respectively). The fitted depletion formulas of the C and Mo coatings are listed in Table 1.

From the fitted formulas, it can be seen that the depletion of C coating is faster than that of Mo coating at 700 °C because of the larger absolute value of the rate constant k. This phenomenon can be understood in terms of diffusion mechanism. The radii of C and Mo are 0.086 nm and 0.140 nm, respectively, so C atoms are interstitial diffusion while Mo atoms are substitutional diffusion. When the composites were thermally exposed at 700 °C, C atom diffused across the TiC layer by vacancies while Mo cannot easily diffuse across the TiC layer, which is also supported by the view that TiC can be good reaction barrier coating [15,16]. Therefore, the Mo coating located between C coating and TiC layer cannot easily diffuse into the matrix, which is the main reason why the depletion of Mo coating is slower than that of C coating.

The depletion rate constants of C and Mo coatings increase when the composites were thermally exposed at higher temperature of 800 °C, which indicates the C/Mo coating is not very stable at 800 °C. In addition, the depletion rate of Mo coating becomes a little faster than that of C coating. The poor thermal stability of C/Mo coating is caused by two reasons: one is that both C and Mo have high diffusion coefficients at 800 °C, the other is that the formed β-Ti layer can accelerate element diffusion.

According to the aforementioned analysis, it can be concluded that the SiC$_f$/C/Mo/Ti6Al4V composites have an excellent thermal stability at temperatures of 700 °C and below. Furthermore, when the composites were thermally exposed at 800 °C, the C/Mo coating depleted quickly. However, in comparison with that of