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Characterization on microstructure of interface and failure analysis of SiC fiber reinforced Ti-17 composites under tension load

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Abstract. In this study, the ultimate tensile strength of unidirectional SiC-fiber/Ti-17 composites was measured in the as-produced condition at room temperature. Fracture and interfacial reaction zone was characterized by using laser confocal microscopy, field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy. Elemental distribution maps of the interfacial reaction layer and titanium matrix were quantitatively examined by electron probe micro-analyzer (EPMA). Micromechanical properties of SiC fiber and titanium matrix was inspected by Nano-indentation. The Fracture failure mechanisms was show that the key microstructural parameters which dominate damage initiation, damage growth and fracture behavior of the composites were explained in detail.

1. Introduction

With the continuous improvement of people's pursuit of high-performance engine thrust-to-weight ratio, people have put forward higher and higher requirements for materials and design. Traditional design concepts and material systems have been difficult to meet the requirement of future high thrust-to-weight ratio engines^[1,2]. Therefore, it is necessary to introduce new structures and new materials into the key components of the engine to realize the integration and weight reduction of the engine. The new generation of disc's materials includes powder superalloys and titanium matrix composites. The powder superalloys are mainly used for rotating parts of turbine, the internal defects of its have influence on fatigue properties ^[3,4]. The continuous SiC fiber-reinforced titanium matrix composites (TMCs) has higher specific strength, specific modulus, and working temperature than traditional titanium alloys, is being consistently identified as one of the enabling technologies for many applications in both military and commercial arena to meet the design requirements for materials displaying high strength and stiffness-to-density ratios at moderate to high temperatures (400 to 800°C)^[5, 6].

The longitudinal tensile properties is one of the most important mechanical properties of continue fiber reinforced metal matrix composites. The ultimate tensile strength (UTS) for titanium matrix reinforced with silicon carbide fibers is typically 60% higher than that for homogeneous titanium without any reinforcement^[5]. The UTS is closely related to the microstructure of TMCs, especially the interface between SiC fiber and titanium matrix, besides, microstructure of titanium matrix and SiC fiber have a huge impact on the mechanical behavior of TMCs, crack initiation, propagation and



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fracture is complicated process, there are many influencing factors, including properties of matrix and fiber, fiber volume fraction and test strain rate et al. A lot of progress has been made in the investigation on mechanical behavior of SiC_f / Ti composites ^[6-12] (e.g, with lower fiber volume ~35% SCS-6/Ti-6Al-4V), and research on microstructure evolution^[13,14] and interface properties^[15-18] degradation of SiC/Ti-17 alloy during hot exposure and different fabrication process ^[19-21], however, there are only a few investigation on tensile behavior and failure mechanism of SiC fiber reinforced Ti-17 alloy (Ti - 5Al - 2Sn - 4Mo - 2Zr - 4Cr) with higher fiber's volume (~50%).

In the present study, the damage initiation, propagation and fracture process under tension load at room temperature are investigated in order to obtain insight into the failure mechanism.

2. Experiment

First of all, the continuous SiC fibers with W core were produced by using CVD method, and then which was coated $2\sim3\mu$ m thickness carbon. Secondly, The Ti-17 alloy was deposited onto continuous SiC fibers by magnetron sputtering to prepare Ti-17 precursor wire. Finally, The Ti-17 alloy was used as canning material and HIP processing was employed to accomplish the consolidation of the precursor wires and canning at the temperature of 920 °C under an gas pressure of 120 MPa for a holding time of 3 hours^[14,15], then furnace cooling to room temperature to obtain as-receive composites. The specimen for tensile properties test was cut to dog bone (length 52 mm and diameter 3 mm), the tensile test was carried out on the INSTRON electronic universal testing machine. sample 1, UTS = 1024 Mpa and sample 2, UTS = 2494 Mpa were picked for investigation.

Fractographic analysis was performed using a Laser confocal microscopy(Keyence VK-X200, USA) and scanning electron microscopy with backscatter electron (SEM, Quanta 650, USA), the fractured specimen was further grinded and polished along fiber direction(Longitudinal direction) and vertical fiber direction(Transverse cross section). In order to study the microstructure of samples, focused-ion beam (FIB, FEI/Thermo Fisher Scientific Quanta 3D FEG Dual Beam, Hillsboro, USA) was used to prepare specimen of SiC fiber for transmission electron microscopy (TEM, FEI/Thermo Fisher Scientific Titan G2 ChemiSTEM, Hillsboro, USA) investigation. Nano-indenter (HYSITRON 950, Thermo-fisher-science, USA) was used to examine fiber and matrix micromechanical property.

3. Results and Discussion

3.1. Fracture Characterization

Fracture morphologies of SiC_f /Ti-17 under tensile load. The fracture surface is flat, 3D image using confocal microscope show that the difference between the highest point and the lowest point of the sample 1 fracture surface is about 950 μ m, and the flat area is large, accounting for about 2/3 of the entire fracture surface, only a few fibers were pulled out, as shown in Figure 1a. The roughness of fracture surface of Sample 2 is more than sample 1 obviously, gap between the highest point and the lowest point is about 1150 μ m. The flat area occupies a small area, about 20% of the entire fracture area. Fiber pullout is main fracture feature, as shown in Figure 1b. The microscopic morphology of the fracture surface showed that only a small number of fibers were pulled out in sample 1, and there was no sign of de-bonding at the interface between the pulled-out fibers and the matrix. The TiCx reaction layer was observed in the interface area, which was intergranular and cleavage mixed fracture are shown in Figure 2. On the other hand, on the fracture surface of Sample 2, there are obvious signs of fiber being pulled out, and the number is large. The interface between the pulled out fiber and the matrix has all been de-bonded, multiple fracture cracks were found on the interface layer and carbon coating, the matrix fracture characteristic is dimples, as shown in Figure 3.

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Fig. 1 the macro-morphology of tensile fracture with laser confocal microscopy (a) sample 1 and (b) sample 2



Fig. 2 Fracture morphologies of the fracture surface of the sample 1 at RT (The secondary electron images)



Fig. 3 Fracture morphologies of the fracture surface of the sample 2 at RT (The secondary electron images)

3.2. Microstructure and chemical element distribution of $SiC_f/Ti-17$.

The transverse microstructure of Sample 1 shows that the fiber arrangement is good, the matrix is equiaxed α phase and β phase, the thickness of the interface reaction layer is about 2.3~2.5 um, and the size of α phase is about 10µm, as shown in Figure 4. The longitudinal section of sample 1 (near the fracture area) can clearly see the crack, and when the crack passes through the fiber, there is no deflection or cutting mechanism, as shown in Figure 4(d)~(f). Two different organization compositions were found in sample 2, one is equiaxed α and a small amount of β , and the other is Widmanstatten with columnar crystal, as shown in Figure 5(a)-(c). Under such a mixed structure, the static tensile test results showed the highest result (UTS = 2494Mpa). The internal microstructure of the fracture longitudinal sample showed that the crack was deflected during the propagation process after initiation and expanded along the fiber direction, see Figure 5(d)~(f), where Figure 5(f) shows that the interface reaction layer and carbon coating of sample 2 change the direction of crack propagation, and the matrix with good plasticity captures the direction from the interface reaction layer to the substrate. The cracks that propagate in the body prevent the cracks from propagating to the matrix and cause catastrophic fracture. The interface of sample 1 is characterized by intergranular fracture. At that point, the average thickness of the interface reaction layer of sample 2 is about 1.8µm. Because the cracks stay in the sample for a short time, the cracks are penetrating, that is, crack initiation Later, under the external load, the cracks rapidly propagated around and remained on a plane without deflection or expansion along the fiber, indicating that the interface did not play a good role during the load transfer. The electron probe microanalysis results show that sample 2 presents a mixed structure in which Widmanstatten structure and equiaxed structure coexist. For titanium alloys, Widmanstatten structure has the highest UTS and has poor plasticity, while equiaxed structure has better overall performance.



Fig. 4 Microstructure of sample 1 cross section (a)~(c) transverse cross section; (d)~(e) longitudinal cross section



Fig. 5 Microstructure of sample 2 cross section (a)~(c) transverse cross section; (d)~(e)longitudinal cross section

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Fig. 6 Chemical element distribution mapping analysis by EPMA *3.3. Microstructure and micromechanical behavior of SiC fiber.*

The ring pattern was found on fiber of sample 1 as shown in Fig. 7a. The microstructure of SiC fiber was used TEM to examine with SADP mode shown that a colony small size grain have different growth direction with mainly growth direction of columnar grain. The micromechanical behavior results show that the ring pattern has a great influence on performance stability of SiC fiber, see Fig.8.



Fig. 7 The microstructure of SiC fiber



3.4. The failure mechanisms of SiCf/Ti-17 under tensile load.

Based on the above experimental observations and analysis, the mechanisms of initiation and propagation of damage in the un-notched unidirectional continue SiC fiber reinforced Ti-17 matrix composites under tensile loading is classified into two types. In one case, the crack initiated in the interfacial reaction layer is captured by matrix with better ductile, the mixed microstructure (Widmanstatten and equiaxed) with strong fiber(microstructure uniformity) and low interfacial bonding strength (the lower thickness of reaction layer) can cause interfacial de-bonding along the reaction layer and the fiber, for case of composites can occur multiple fracture in interfacial reaction layer caused by load transfer between the fiber and matrix. In another case, strong interfacial reaction with weak fiber(defects on the surface or microstructure non-uniformity), even if matrix is ductile the crack initiated in the interfacial reaction layer cannot be arrested by matrix or stop propagating by fiber. In the case of composite, the crack can easily extend to the fiber and tough matrix lead to immediate fracture and lower mechanical properties.

4. Conclusions

(1) Interfacial debonding, a lot of fibers was pull-out and crack initiated in interfacial reaction layer can arrested by tough matrix and strong fiber under tensile load, which lead to better mechanical properties $_{\circ}$

(2) Interface play more important role during load transfer, however, microstructure of matrix and fiber also can cause the key influence of mechanical behavior of titanium matrix composites, the failure mechanisms under tensile load show that different type of matrix microstructure with crystals orientation can lead to different mechanical behavior, fiber's properties and microstructure uniformity is more and more important in titanium matrix composites with higher volume fraction of fiber(\sim 50%).

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