

Oxidation behaviors and shear properties of z-pinned joint made of two-dimensional carbon fiber reinforced silicon carbide composite

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ABSTRACT

Chemical vapor infiltration has been introduced for preparing z-pinned joint, which is made of two-dimensional carbon fiber reinforced silicon carbide composite. The effects of oxidation on the shear properties of the joint were investigated. The results showed that the joint strength increases with the increase of oxidation temperature, which is consistent with the oxidation consumption of the carbon phases. An exponential relationship is presented between the weight loss and the joint strength. In contrast, linear relationships are presented between the weight loss and the mechanical properties of the composite. The exponential relationship results from the coupled shear and bending stress states of the pin, according to the failure mechanisms of the joint. It is observed that in-plane and intra-layer cracks are formed under the shear stress. And these cracks are bridged by the fibers under the bending stress. Accordingly, the fiber bridging mechanism contributes to the joint strength before and after oxidation. For the conditions of this study, the joint strength can be roughly estimated as the plus of the in-plane shear strength and the tensile matrix cracking stress.

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1. Introduction

Carbon fiber reinforced silicon carbide composites (C/SiCs) are increasingly applied in aerospace components such as thermal protection systems (TPS) and hot structures since C/SiCs retain high specific mechanical properties under high temperature oxidizing environments [1–4]. In design of such complex components, C/SiC joints with C/SiC fasteners are widely used to assemble the parts of these components [5,6]. And the joints suffer from oxidation damages and external loads under service conditions [7]. Hence, the C/SiC joint becomes the weakest link of the components [8]. Under this circumstance, several kinds of C/SiC joints have been developed, such as bolted joints [9,10], Miller joints [8,11] and screw joints [12]. However, they are susceptible to brittle rupture with unexpected low joint strengths because of oxidation damages in the region of stress concentrations (e.g. threads or notches) [9]. Thus, developing an oxidation-resistant C/SiC joint with high mechanical performances has become one of the key technologies for C/SiC applications [3].

Many researches have focused on oxidation mechanisms of C/SiCs [13,14], as well as oxidation effects on their mechanical properties [15–17]. The results demonstrated that the decreased mechanical properties of C/SiCs are caused by oxidation

consumption of carbon phases (pyrolytic carbon (PyC) interphase and carbon fibers). In addition, the large-scale fiber bridging mechanism dominates the mechanical properties of C/SiCs even after oxidation damages [17]. However, the fiber bridging mechanism was rarely discussed in developing oxidation-resistant C/SiC joints. On the other hand, many studies have revealed relationships between constituent (matrix, fiber, and interface) properties and mechanical properties of C/SiCs [18–22]. These relationships could benefit the design of C/SiC joints. However, they are seldom involved due to the complex three-dimensional stress states within the joints [8,12,23]. In conclusion, current C/SiC joints could be improved based on in-depth understanding of oxidation mechanisms and failure mechanisms of C/SiCs.

In this paper, chemical vapor infiltration (CVI) was introduced for preparing z-pinned joints made of two-dimensional carbon fiber reinforced silicon carbide composite (2D C/SiC). Oxidation effects on the shear properties of z-pinned joint were investigated in order to demonstrate the joint oxidation resistance. Finally, the shear properties of the joint were analyzed based on the failure mechanisms.

2. Experimental procedures

2.1. Preparation and microstructures of 2D C/SiC z-pinned joint

Z-pinned joint was prepared via two main steps, preparation of

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raw plates and preparation of z-pinned joint (Fig. 1). The raw plates of 2D C/SiC were prepared by CVI [6]. Firstly, plain weave cloths were stacked to prepare the plates. Then, the PyC interphase was deposited on the cloths, using C_3H_6 precursor at 900 °C. The thickness of the interphase was about 200 nm. After that, the plates were annealed in argon at 1800 °C. At last, SiC matrix was infiltrated into the plates at 1000 °C using methyltrichlorosilane (MTS, CH_3SiCl_3) as SiC precursor.

Using the raw plates, the substrate plates and the pins were cut off and assembled with an interference fit. After removing the protrusions, the assembled joint was fully densified by the infiltration of SiC matrix. Finally, z-pinned joint was prepared with the density of 2.0 g/cm³. The geometrical dimensions of z-pinned joint are illustrated in Fig. 2.

Fig. 3 shows the microstructural features of z-pinned joint, which are inspected by micro-computed tomography (micro-CT) (Y. Cheeta, YXLON, German). Because the SiC matrix is progressively infiltrated into the joint, a region of density gradient was formed around the surfaces. Therefore, a uniform SiC coating was formed outside of the joint. Meanwhile, the pin and the plates were firmly bonded. In contrast, the two plates were scarcely bonded due to the large clearance (about 0.2 mm). Note that few residual pores are closed due to the bottle-net effect of CVI.

2.2. Oxidation treatments, testing and characterization

A total of 12 z-pinned joints were prepared and they were divided equally into four groups. Three groups were heat treated at 700 °C, 1000 °C and 1300 °C for 10 h in static air. The weight loss was measured by an analytical balance (Mettler Toledo, AG 204, Switzerland). According to the single pin configuration, these joints were named as SPJ-RT, SPJ-7, SPJ-10, and SPJ-13. These oxidation environments were chosen to compare with the previous studies on oxidation mechanisms of C/SiCs [13–17]. For the same purpose, the tensile and the shear strengths of 2D C/SiC were measured before and after oxidation.

As shown in Fig. 2, z-pinned joint was tested on a universal testing machine (Instron-5567). A displacement control mode was performed with a loading rate of 0.2 mm/min. The joint strength is calculated by F/S , where S is the cross-section area of the pin, and F is the maximal load. The joint stiffness is the initial slope of the load–displacement curves.

After tests, the diameters of the pin were measured by optical microscope (Stemi2000-C, Carl Zeiss). The failure morphologies of the joints were observed by scanning electron microscope (SEM) (Hitachi S-2700, Tokyo, Japan).

3. Results and discussion

3.1. Oxidation effects on shear properties of z-pinned joint

Fig. 4(a) shows the typical load–displacement curves of z-pinned joint before and after oxidation. The average joint strengths are 66.10 MPa of SPJ-7, 90.28 MPa of SPJ-10, and 101.27 MPa of SPJ-13, which are lower than the virgin strength of 176.79 MPa of SPJ-RT (Fig. 4(b)). Before the maximum load, all curves have a similar nonlinear profile. It indicates that similar failure mechanisms should occur before and after oxidation although the load capacity of the joint is degraded. After the maximum load, softening stages are observed after oxidation, which is different from the abrupt fracture of SPJ-RT. Hence, oxidation could not lead to catastrophic failure of z-pinned joint.

3.2. Oxidation damages to the carbon phases in z-pinned joint

Fig. 5 shows the failure morphologies of z-pinned joint before and after oxidation. Overview of the ruptured pin indicates that it is progressively oxidized from the periphery to the interior (the yellow rectangles). It is observed that the oxidized depths are different from each other (Fig. 5(a)–(d)), which are the distances between the oxidized zones and the periphery of the pin. The depth of SPJ-7 is the largest while the depth of SPJ-13 is the smallest, and the depth of SPJ-10 is in between. In spite of the oxidized depths, similar oxidized morphologies are observed within the fiber tows (Fig. 5(e)–(i)). That is, the carbon fibers within a oxidized fiber tow can be divided into the superficial oxidation zone, the transitional oxidation zone, and internal non-oxidation zone (i.e., surviving fibers) [9]. The carbon fibers are locally consumed within the superficial oxidation zone, which would lead to notch-like degradations [15]. In the tests, these fibers can be easily sheared off with a sharp end (Fig. 5(j)–(m)). According to the oxidized depths and morphologies, the amount of the oxidized carbon fibers can be qualitatively determined (Fig. 4(b)). Clearly, it is largest in SPJ-7, which is consistent with the weight loss.

As shown in Fig. 4(b), the weight loss decreases as the increase of oxidation temperature, which can be elucidated by the oxidation mechanisms [16,17]. During the cooling process of CVI, multiple matrix microcracks initiate due to thermal misfit stress between SiC matrix and carbon fibers. Along these micro-cracks, the carbon phases react with oxygen. In addition, the reaction is governed by the crack opening displacement (δ). Under 700 °C, δ cannot be eliminated so that oxygen can easily diffuse into the composite to react with the carbon phases. And the speed of oxygen diffusion is much greater than the oxidation rate. Then, the oxidation rate dominates the weight loss. Under 1000 °C, δ is narrowed because thermal misfit stress disappears (the infiltration

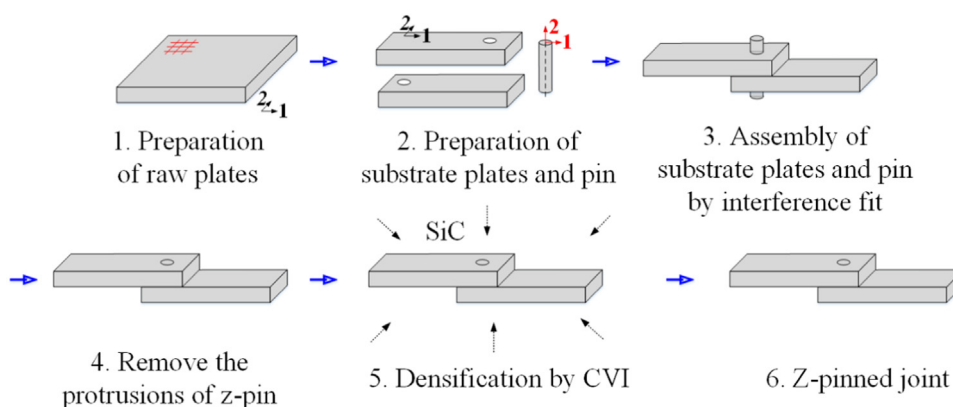


Fig. 1. Preparation procedures of 2D C/SiC z-pinned joint by CVI.

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