

A user-friendly heat-resistant modified polymer-based adhesive for joining and repair of carbon/carbon composites



Mingchao Wang, Xiaoxia Hu, Xiqing Xu, Ziqi Yun, Jiachen Liu *, Haiyan Du, Anran Guo

School of Materials Science and Engineering, Key Lab of Advanced Ceramics and Machining Technology of Ministry of Education, Tianjin University, Tianjin 300072, PR China

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ABSTRACT

A user-friendly heat-resistant modified polymer-based adhesive was developed to join C/C composites. After calcination at 1300 °C, the bonding effect of the adhesive reached the highest as more heat-resistant ceramics and high-temperature melting glass were generated in the adhesive. Its bonding strength was kept above 15 MPa during test from RT to 500 °C and the corresponding joints ruptured at C/C substrates. Besides, after repeated thermal-cycling at 1300 °C, the bonding strength at this temperature was maintained at about 12 MPa. For cured adhesive without calcination, its bonding strength could be maintained above 5 MPa during the whole heating process, which made it to have direct application in practice after curing.

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1. Introduction

Carbon/carbon (C/C) composites are eminent heat-resistant structural materials for applications in harsh ultra-temperature environments due to their excellent properties, such as low coefficient of thermal expansion, good thermal shock resistance and advantageous high-temperature mechanical properties [1]. With the rapid development of high-temperature technology fields, the requirement for C/C components with large sizes and complex shapes becomes more and more urgent and wide. However, their direct preparation is always limited by certain disadvantages involving long production time, high cost and their brittle characteristic [2,3]. Besides, in order to make them serve long time and reduce maintenance cost, it is significant to seek practical approach for repairing damaged C/C parts after being employed for many times. One alternative way to manufacture complex components or repair damaged parts is the joining technology.

Compared to several mature joining technologies like diffusion bonding, active metal brazing and glass or glass ceramic bonding, the heat-resistant adhesive bonding is the most promising and practical method as it can realize effective joining under a relatively low temperature and guarantee the joint in good condition after the heating process, which makes the fabrication, repair and assembly processes more feasible, especially on the occasion that these high-temperature joining methods are hardly operated [4]. For example, it is crucial to guarantee the function of reinforced C/C composite thermal protection

systems of the space shuttle during the re-entry conditions, as even a single small exterior crack or pore could lead to the hot plasma and other gaseous species getting through the thermal protection system. In that case, a heat-resisting and low-temperature-curing repair adhesive (e.g., GRABER) can facilitate the reparative process and effectively prevent the high temperature gas, while other methods are usually hard to be carried out [5]. In addition, due to the adhesive's viscous flow in the polymeric state, it can distribute the load well and stress concentration in the joining layer is minimized early [6].

Two kinds of heat-resistant adhesives have been commonly used in practice, including inorganic adhesive and polymer-based adhesive. According to our previous research on phosphate-based inorganic adhesives [7], although they possess outstanding heat resistance, their lower bonding strength restricts their application. Generally speaking, the polymer adhesive can provide high strong joining after the polymer's ceramic conversion reaction [8,9]. However, since the polymer's strength was very low during the conversion process [10], as-received joints are always needed to be calcined at temperature above conversion temperature before being applied in practice, which reduces the polymer-based adhesive's practicability. Fortunately, this disadvantage can be improved by adding several modified fillers. NASA Glenn Research Center once developed an excellent heat-resistant adhesive called GRABER prepared from phenolic resin as matrix [5]. In this work, we developed a modified polymer adhesive by using silicon resin as matrix and Al, SiC, B₄C and glass powders as additives. It could be directly applied in practice after curing and provide high bonding performance during the heating process. The high-temperature bonding effect of both cured adhesive and calcined adhesive during the heating process was investigated and compared.

* Corresponding author.

E-mail address: jcliutju@gmail.com (J. Liu).

2. Experimental

C/C specimens ($24 \times 6 \times 3$ mm and $10 \times 6 \times 3$ mm) were cut from bulk 3-D C/C composites fabricated by chemical vapor deposition (CVD) with a density of 1.75 ± 0.02 g/cm³. After being polished, they were ultrasonically cleaned in anhydrous ethanol and dried at 80 °C for 4 h.

Silicone resin adhesive was prepared from MK resin (polymethylsilsesquioxane) and isopropanol as the matrix and Al, SiC, B₄C and low-temperature melting glass powders (SSP) as the modified fillers. The solid MK powder was firstly dissolved in isopropanol and magnetically stirred for 1 h, and then successively added with various active additives and finally magnetically stirred in high vacuum chamber for 2 h. The adhesive was universally brushed on each bonding surfaces in uniform thickness of 200 μ m by using the adjustable coating machine. Then the specimens were overlapped to establish bonding joints according to the configuration shown in Fig. 1A. The obtained joints were then cured at 200 °C under pressure of 2.5 MPa for 1 h. A part of these cured joints were calcined in vacuum at different temperatures from 300 °C to 1500 °C for 0.5 h.

The apparent shear strength test was applied to both calcined joints and cured joints under two different conditions: at room temperature (RT) and at high temperature (HT). The apparent shear strength tested at RT for all these joints was named as RTSS-X (X: treatment temperature). It should be noted that RTSS-1300 °C was the largest, which would be discussed in Section 3.2. In order to detect the properties of 1300 °C-calcined adhesive in high-temperature environment once again, the apparent shear strength for 1300 °C-calcined joints was tested at different temperatures from RT to 1300 °C and named as ATSS-Y (Y: test temperature). Moreover, ATSS-1300 °C after the thermocycling from RT to 1300 °C was measured and the thermo cycling was performed several times for better estimating the property of adhesive being repeatedly used. To investigate the direct effect of cured adhesive without calcination during heating process, the apparent shear strength for cured joints was tested at different temperatures from RT to 1300 °C and named as HTSS-Y. RTSS was tested by CSS-44001 Universal Testing Machine at a loading speed of 0.5 mm/min and the same speed was applied to ATSS and HTSS checked by RDL-15 High Temperature Tensile Testing Machine. The high temperature test was performed after maintaining the test temperature for 0.5 h in vacuum. Each shear strength value was obtained as the average of five specimens. In addition, for

comparison purposes, the interlaminar shear strength (ILSS) of the substrate C/C was also determined under the same testing conditions as the above apparent shear strength tests. Fig. 1B shows the ILSS of C/C at different temperatures. With the temperature increasing, ILSS increased from 15 MPa at RT to a maximum of 20.5 MPa at 900 °C, and then decreased to about 19.8 MPa at 1500 °C, which conformed to the regular pattern in Ref. [9].

The compositions of adhesive under different conditions were identified with D/Max-2500 X-ray diffraction (XRD). The structure morphology of both the cross-section and fracture surface of ruptured joints was analyzed by S-4800 field emission scanning electron microscopy (SEM) equipped with X-ray energy dispersive spectrometers (EDS). Moreover, the analysis for stress distribution was carried out using a finite element program ANSYS. The following parameters were used for the initial model: three-dimensional plane, linear elastic, linear elements and isotropic material properties [11]. Three-dimensional eight node solid elements integrated in ANSYS were used to model the behavior of the rupture element in this three-dimensional problem, and the solid elements (SOLID185) were used for substrate and the solid-shell elements (SOLSH190) were for the adhesive.

3. Results and discussion

3.1. Compositional variation analysis

As seen from Fig. 2A, with the temperature increasing from RT to 500 °C, only the area of broad amorphous diffraction peak of resin reduced and nothing happened to crystalline peaks of Al and SiC, indicating that the network structure of resin began to decompose and that Al and SiC did not react at 500 °C, respectively. According to the spectrum at 700 °C, silicon resin had decomposed completely with the formation of SiC because the peak intensity of SiC obviously enhanced and Al had also been oxidized into γ -Al₂O₃. Besides, the crystallization of SSP glass occurred, forming Sn, SiO₂ and hexagonal AlPO₄ in the adhesive. From 700 °C to 900 °C, SSP glass had definitely disappeared and much more SiO₂ and hexagonal AlPO₄ generated. The identified crystalline phases of the adhesive after treatment at 1300 °C included mullite, orthorhombic AlPO₄ and α -Al₂O₃. Mullite formed from the reaction of SiO₂ and Al₂O₃, while orthorhombic AlPO₄ and α -Al₂O₃ came from the transition of hexagonal AlPO₄ and γ -Al₂O₃, respectively. In addition, there was a

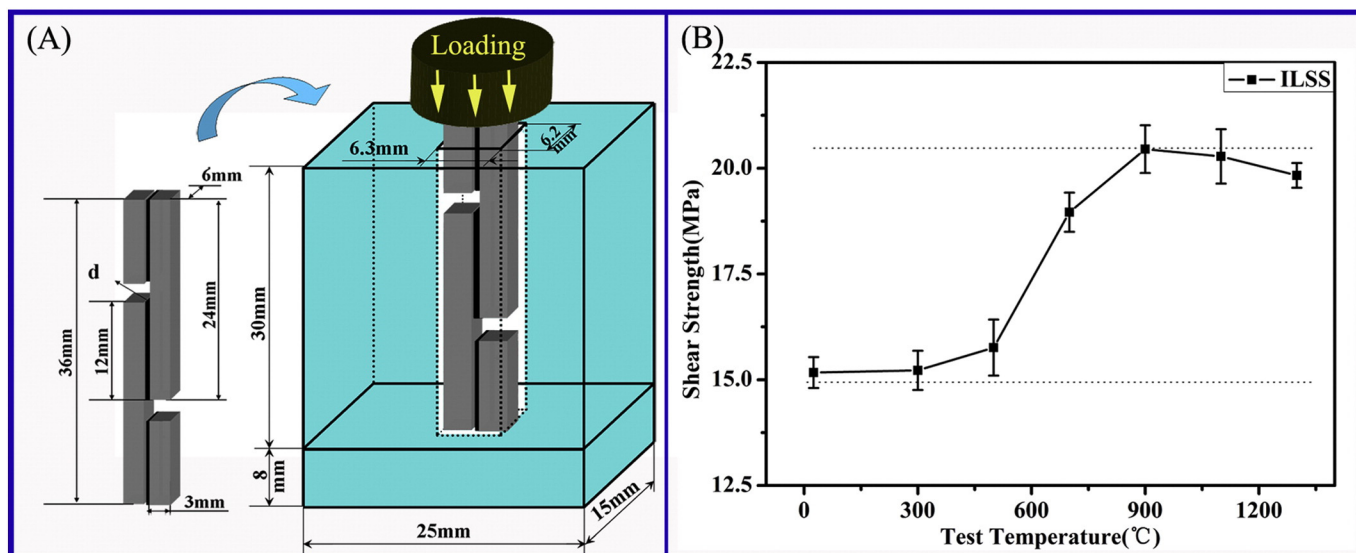


Fig. 1. Sample configuration and the procedure of shear test (A) and ILSS of C/C at different temperatures (B).

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