

A mortise–tenon joint inspired interface structure design for synergistically enhancing the mechanical properties and thermal shock resistance of Cf/(HfNbTaTiZr)C–SiC composites

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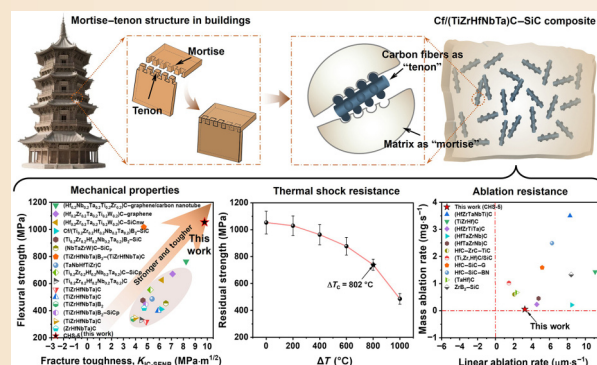
ABSTRACT: Carbon fiber-reinforced high-entropy carbide ceramics (Cf/HECs) are considered promising candidates for ultrahigh-temperature structural applications. The fiber–matrix interface plays a crucial role in determining the overall performance of these materials. This study proposes a novel interface design strategy inspired by the traditional Chinese mortise–tenon joint. In this design, microscale carbon spheres are deposited on the surface of carbon fibers to function as the “tenon”, while the matrix serves as the corresponding “mortise”. Furthermore, a TiC interfacial layer is introduced to improve the interfacial bonding through atomic diffusion. Owing to this distinctive interface structure, the resulting Cf/(TiZrHfNbTa)C–SiC composite exhibits excellent mechanical properties, with a flexural strength of 1053.33 MPa and a fracture toughness of 9.77 MPa·m^{1/2}. Additionally, the composite demonstrates remarkable thermal shock resistance, with a critical thermal shock temperature difference (ΔT_c) of 802 °C. It also displays superior ablation resistance, characterized by a linear ablation rate of 3.27 $\mu\text{m}\cdot\text{s}^{-1}$ and a mass ablation rate of 0.05 $\text{mg}\cdot\text{s}^{-1}$.

KEYWORDS: high-entropy carbide; carbon fiber; interface design; mortise–tenon joint; mechanical properties

1 Introduction

High-entropy carbide ceramics (HECs), represented by (TiZrHfNbTa)C, are important components of the new generation of ultrahigh-temperature ceramics (UHTCs) and have shown broad application prospects in extreme service environments, such as spacecraft thermal protection systems, gas turbine hot-end components, and nuclear reactor cladding materials [1–3], owing to their excellent oxidation resistance and ablation resistance [4,5], extremely high melting points, low thermal conductivity [6], and good hardness and strength matching [7,8]. However, compared with that of traditional binary carbides, the fracture toughness of HECs has not been significantly improved (2–4.5 MPa·m^{1/2}) [9,10]. Given that almost all extreme conditions impose high demands on the damage tolerance of materials [7,11], the inherent brittleness of HECs has become a key factor restricting their further application in ultrahigh-temperature structural components.

Carbon fibers possess remarkable characteristics, such as low density, near-zero thermal expansion coefficient, ultrahigh specific strength and modulus, and excellent thermal conductivity [12]. The UHTC-based composites constructed by introducing carbon fibers into the UHTC matrix not only achieve a significant increase in fracture toughness, which is difficult for a single ceramic material to reach, but also have comprehensive performance advantages, such as low density, high mechanical strength, and excellent thermal shock resistance [13–15]. However, the mechanical properties of UHTC-based composites highly depend on an efficient load transfer mechanism between the fibers and the matrix [16,17]. The insufficient interfacial bonding strength has become a key bottleneck restricting the performance improvement. This issue stems mainly from the poor intrinsic wettability between carbon fibers and ceramic matrices, which leads to weak interfacial bonding and causes load transfer failure, resulting in the actual reinforcing efficiency of the fibers being far lower than the theoretical prediction [18,19].



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Therefore, the precise control of the fiber–matrix interface structure has become the core issue for fully exploiting the reinforcing potential of carbon fibers.

To optimize the fiber–matrix interface performance, researchers have developed various fiber surface modification techniques [20,21]. Among them, coatings such as pyrolytic carbon (PyC), silicon carbide (SiC), and titanium carbide (TiC) have been proven to effectively improve wettability and enable controlled adjustment of the interface structure between fibers and ceramic matrices [22–25]. For example, Cai *et al.* [26] fabricated Cf/(HfNbTaTiZr)C–SiC composites via a precursor infiltration–pyrolysis process and introduced a PyC/SiC interfacial layer to achieve a weak interface design, significantly enhancing the fracture toughness of the composites. However, the weak van der Waals interactions between the coating and carbon fibers can easily lead to fiber pull-out and interfacial debonding [26]. Additionally, the ceramic coatings themselves are brittle, which can cause stress concentration and premature failure of the carbon fibers [27,28]. In recent years, many studies have focused on introducing nanofillers such as carbon nanotubes and graphene into interfaces, which can form mechanical interlocking structures with the matrix, thereby effectively enhancing the interfacial strength [17,20,29]. For example, in carbon fiber-reinforced polymer composites, owing to the good fluidity of the resin, it can penetrate into the interior or gaps of the nanofillers, forming interlocking structures, which is conducive to improving the interface performance [17,30]. However, when such interlocking structures are introduced into UHTC-based composites, many challenges are faced. First, compared with resins, ceramic particles have poor fluidity and have difficulty fully filling the gaps between nanofillers, thus failing to achieve effective mechanical interlocking. Second, at extremely high preparation temperatures (> 2000 °C), nanofillers are prone to chemical reactions with the ceramic matrix, thereby destroying their structural integrity [31]. Therefore, in such extreme processing environments, developing interface structures with mechanical interlocking with stronger and more stable bonding capabilities is particularly crucial.

The mortise–tenon joint found in traditional Chinese wooden buildings achieves a high-strength and high-durability dynamic

connection between convex components (tenon) and concave components (mortise) through their ingenious physical interlocking mechanism, endowing the buildings with excellent seismic performance and structural stability [32,33]. This mechanical interlocking mode, which does not rely on chemical bonds, provides an inspiring new idea for the interface design of composites.

Inspired by this, this study proposes an interface structure design strategy based on mortise–tenon joints. This strategy uses carbon fibers with microscale carbon spheres deposited on their surfaces as the “tenon” and a (TiZrHfNbTa)C–SiC ceramic matrix as the “mortise” and realizes effective mechanical interlocking between the carbon fibers and ceramic matrix through spark plasma sintering (SPS) technology (Fig. 1(a)). On this basis, by introducing TiC coating modification technology, a chemical–physical bridging synergistic effect is achieved. The influence of this mortise–tenon structure on the mechanical properties and thermal shock resistance of carbon fiber-reinforced (TiZrHfNbTa)C–SiC (Cf/HEC–SiC) composites was systematically studied, and the strengthening and toughening mechanisms were analyzed. In addition, through oxygen–acetylene flame ablation tests, the thermal shock resistance and ablation resistance of the composites under extremely high temperatures (> 2000 °C) and aerodynamic load conditions were further evaluated, verifying their service reliability in extreme environments.

2 Experimental

2.1 Raw materials

Hafnium tetrachloride (HfCl_4), titanium tetrachloride (TiCl_4), zirconium tetrachloride (ZrCl_4), niobium pentachloride (NbCl_5), tantalum pentachloride (TaCl_5) (all with purities $\geq 99.5\%$), and furfural (purity $\geq 95\%$) were purchased from Shanghai Aladdin Reagent Co., Ltd. (China). Silicon carbide powder (SiC; particle size: 500 nm, purity $\geq 99.9\%$) was obtained from Kaihua Silicon Carbide Co., Ltd. (China). Titanium powder (particle size: 100 nm, purity $\geq 99.9\%$), glucose ($\text{C}_6\text{H}_{12}\text{O}_6$), hydrochloric acid (HCl),

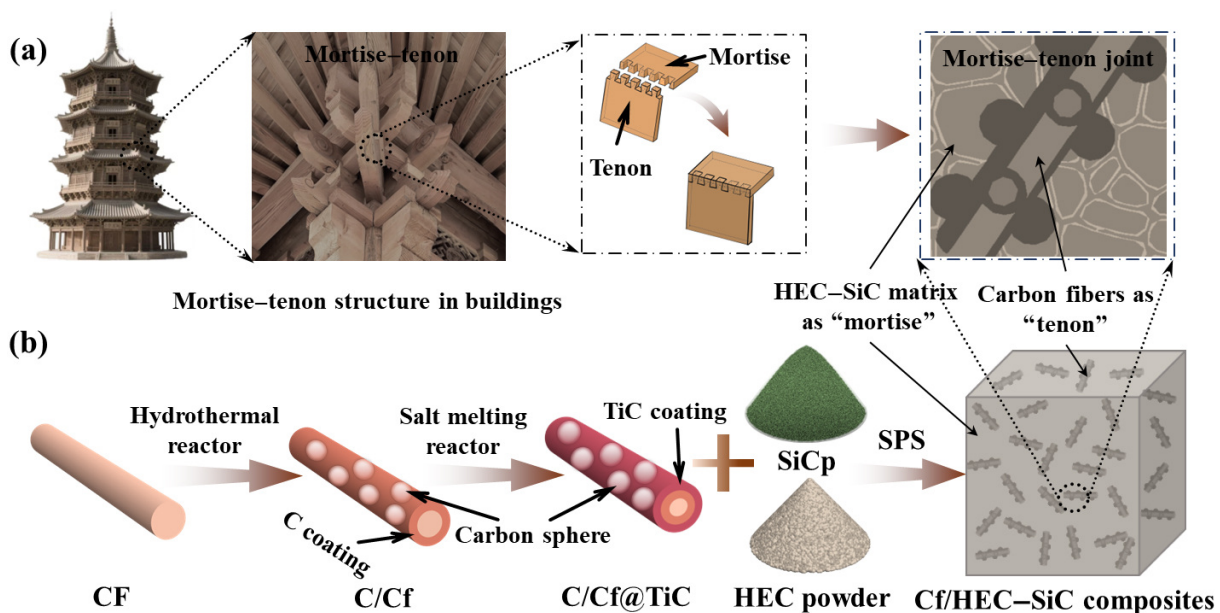


Fig. 1 (a) Schematic diagram of mortise–tenon joint design at interface between fibers and matrix in Cf/HEC–SiC composites. (b) Preparation process of Cf/HEC–SiC composites.

sodium chloride (NaCl), and potassium chloride (KCl) were produced by Sinopharm Chemical Reagent Co., Ltd. (China). T300-type chopped carbon fibers (diameter: 7 μm , length: 1 mm) were manufactured by Toray Industries, Inc. (Japan).

2.2 Material preparation

2.2.1 Surface morphology and component modification of carbon fibers

Carbon fibers with microscale carbon spheres deposited on their surfaces, which serve as “tenon” components, were prepared via the hydrothermal carbonization method [34]. The specific synthesis process is shown in Fig. 1(b). First, a 30 wt% glucose solution was prepared, and the pH of the solution was adjusted to 1, 3, and 5, respectively, with HCl. Then, short-cut carbon fibers were fully dispersed in the above glucose solution and transferred to a high-pressure reactor for a 6-h solvothermal reaction at 200 °C. After the reaction, the obtained products were subjected to a 2-h heat treatment at 800 °C under an argon protective atmosphere to obtain the target structure, which was denoted as C/Cf. To improve the wettability between the carbon fibers and the HEC–SiC matrix, TiC coatings were coated on the surfaces of the carbon fibers via the molten salt method. In the experiments, NaCl and KCl (molar ratio of 1 : 1) were used as the molten salt medium, C/Cf was used as the carbon source, and titanium powder was used as the titanium source. The molar ratio of C to Ti in the molten salt system was C : Ti = 1 : 0.2. After the abovementioned raw materials were thoroughly mixed, they were placed in an alumina crucible and subjected to a salt bath at 1100 °C for 2 h under a protective atmosphere of flowing argon. During this process, Ti and C/Cf reacted *in situ* to form a TiC coating. After the reaction, the system was cooled to room temperature, and the resulting product was then dissolved in boiling water to remove NaCl and KCl. The solution was filtered through a 300-mesh sieve to remove impurities, residual salts, and unreacted titanium powder. Finally, C/Cf coated with a TiC layer was obtained and denoted as C/Cf@TiC.

2.2.2 Fabrication of Cf/HEC–SiC composites

HEC powders were synthesized using NbCl_5 , TaCl_5 , HfCl_4 , TiCl_4 , and ZrCl_4 as metal precursors and furfuryl alcohol as the carbon source. The specific synthesis steps are detailed in our previous research [35]. Three types of C/Cf@TiC fibers with a proportion of 20 vol% each were subsequently mixed with 80 vol% HEC

powder or 20 wt% SiC powder. To fully disperse the fibers, a solvent-assisted dispersion method was adopted. Anhydrous ethanol was used as the dispersion medium, and the fibers, HEC powder, and SiC powder were subjected to prolonged magnetic stirring (6 h) in ethanol, which ensured a macroscopically uniform mixture. Additionally, intermittent ultrasonic treatment was applied during the stirring process. The high-frequency vibration generated by the ultrasonic waves could effectively break up the fiber bundles at the microscale, achieving good separation of individual fibers. After the mixing was complete, a slow evaporation drying method was employed, which helped maintain the uniform distribution achieved during the solvent removal process and minimized the reagglomeration and migration of the fibers. The Cf/HEC–SiC composite material was prepared via the SPS process under a high vacuum (vacuum degree $< 10^{-3}$ Pa) with a heating rate of 100 °C/min and a pressure of 40 MPa, heating to 2000 °C and holding for 10 min. The preparation process is shown in Fig. 1(b). During the sintering process, the HEC powder can fully fill the gaps between the microscale carbon spheres on the surface of the carbon fibers and eventually form an interface structure with mortise–tenon joint characteristics in the composites. The prepared composites were named CHS-1 (pH = 1), CHS-3 (pH = 3), and CHS-5 (pH = 5) according to the initial pH of the glucose solution.

2.3 Mechanical property test

The flexural strength of the composites was determined by a three-point bending method. The test parameters were set as follows: span of 20 mm, head movement rate of 0.5 mm/min, and sample size of 3 mm $\times$ 4 mm $\times$ 25 mm. To reduce the influence of surface defects and edge stress concentrations on the test results, all the samples were chamfered and finely polished before testing. Additionally, the fracture toughness ($K_{\text{IC-SENB}}$) of the composites was evaluated under the same three-point bending conditions based on the single-edge notched beam method (SENB). The sample size was 3 mm $\times$ 4 mm $\times$ 20 mm, the crossbeam rate was set at 0.5 mm/min, and the support span was 16 mm. The pre-made notches were cut using a diamond blade and finely polished by hand with a diamond polishing agent to ensure notch geometry accuracy. The notch depth and width were 0.5 and 0.3 mm, respectively. The $K_{\text{IC-SENB}}$ value was calculated based on Eqs. (1) and (2) [36]:

$$K_{\text{IC-SENB}} = ((10^{-6} F_{\text{max}} L) / (WB^{3/2})) f(a/B) \quad (1)$$

$$f(a/B) = \frac{3(a/B)^{1/2} \{1.99 - (a/B) (1 - a/B) [2.15 - 3.93a/B + 2.7(a/B)^2]\}}{2(1 + 2a/B)(1 - a/B)^{3/2}} \quad (2)$$

where F_{max} represents the maximum load (N) recorded for the composites in the SENB test; L is the support span (m); W is the specimen thickness (m); B is the specimen width (m); a is the notch depth (m).

The work of fracture (γ_{work}) is calculated based on Eq. (3):

$$\gamma_{\text{work}} = \int F(\delta) Fd\delta / (2A) \quad (3)$$

where $\int F(\delta) Fd\delta$ is the integral of the area under the load–displacement curve, representing the total energy absorbed during fracture in the SENB test; A is the projected area of the fracture surface. All the aforementioned mechanical property data were obtained by conducting five repeated tests for each material and taking the average to enhance the reliability of the results. Additionally, the interaction between the crack and the fibers was

observed using the indentation method. During the test, a load of 2 kg was applied to the sample surface and maintained for 15 s. To ensure the accuracy of the statistical results, more than 50 indentation tests were conducted, and the obtained crack lengths were statistically analyzed.

2.4 Thermal shock resistance test and oxyacetylene torch ablation tests

The thermal shock behavior was measured by the water quenching method. The three-point bending samples were heated to the set temperature in a muffle furnace and held for 10 min. The samples were then rapidly immersed in a water bath maintained at a constant temperature of 25 °C to generate thermal shock. The thermal shock temperature difference (ΔT) range was set from 200 to 1000 °C. After quenching treatment, the residual

flexural strength of the samples was determined by a three-point bending test. The critical thermal shock temperature difference (ΔT_c) of the composites was defined as the temperature difference corresponding to the residual strength remaining at 70% of the room temperature strength, which was obtained by linear interpolation of the residual strength data [37]. The flexural strength attenuation rate (σ^*) was calculated using Eq. (4):

$$\sigma^* = (\sigma_0 - \sigma_t) / \sigma_0 \times 100\% \quad (4)$$

where σ_0 represents the flexural strength of the sample before the thermal shock test; σ_t represents the flexural strength of the sample after being treated with a temperature difference (ΔT).

The thermal shock resistance and ablation resistance of the composites were evaluated using an oxyacetylene flame to verify their service reliability in extreme environments. Cylindrical samples with dimensions of $\phi 30 \text{ mm} \times 3 \text{ mm}$ (diameter $\times$ height) were exposed perpendicularly to the oxyacetylene flame for 60 s, and this process was repeated three times. During the test, the distance between the oxyacetylene flame nozzle (inner diameter of 2.0 mm) and the sample was maintained at 10 mm, and the heat flux density was $2.48 \text{ MW} \cdot \text{m}^{-2}$. The flow rates of O_2 and C_2H_2 were 0.244 and 0.167 L/s, respectively, and the pressures of O_2 and C_2H_2 were 0.4 and 0.095 MPa, respectively. The surface temperature changes of the samples were monitored in real time using an infrared thermometer (Raytek Mrscsf, USA). The mass ablation rate (MAR) and linear ablation rate (LAR) were calculated on the basis of the changes in mass and thickness of the samples before and after ablation, respectively. The specific calculation formulas are presented in Eqs. (5) and (6):

$$\text{MAR} = (m_0 - m_1) / t \quad (5)$$

$$\text{LAR} = (l_0 - l_1) / t \quad (6)$$

where t represents the ablation time; m_0 and m_1 are the masses before and after ablation, respectively; l_0 and l_1 are the thicknesses of the ablation center region before and after ablation, respectively.

2.5 Microstructural characterizations

The sample's apparent density was determined using Archimedes' method, whereas the theoretical density was calculated on the basis of the lattice parameters. The relative density is the ratio of the apparent density to the theoretical density. The phase composition was analyzed using an X-ray diffractometer (XRD; D/max-2550VB, Rigaku Co., Japan) with a Cu K α radiation source. In addition, the microstructures and elemental distributions of the samples were jointly analyzed by a field emission scanning electron microscope (FE-SEM; Hitachi S-4800, Japan) and an energy dispersive X-ray spectrometer (EDS; X-MaxN, Oxford Instruments, UK). A transmission electron microscope (TEM; Talos F200x, Thermo Fisher Scientific, USA) was further used for high-resolution microstructure observation. A focused ion beam (FIB; Helios Nanolab 600i, FEI, USA) technology was employed to extract slices for interface structure observation. The modulus and adhesion force in the interface region were investigated using an atomic force microscope (AFM; Dimension Icon, Bruker, USA).

3 Results and discussion

3.1 Surface morphology and chemical composition of C/Cf and C/Cf@TiC fibers

After treatment with glucose solutions of different pH values, the

surface morphology of the fibers significantly changed, as illustrated in Fig. 2. At pH = 1, a C coating, approximately 1.3 μm thick, was formed on the surface of the carbon fibers, which was smooth and dense (Figs. 2(a1)–2(a3)). At pH = 3, the thickness of the C coating slightly decreased to approximately 1.2 μm , and a few carbon spheres began to appear on the surface (Figs. 2(b1)–2(b3)). At pH = 5, the C coating further thinned to approximately 1.0 μm , and numerous microscale carbon spheres formed on the surface, with some spheres adhering to each other (Figs. 2(c1)–2(c3)). This is an important signal indicating that, under these conditions, the generation rate and migration rate of microscale carbon spheres are already quite high. It can be inferred that if the pH is further increased, the adhesion and agglomeration phenomena will intensify. To verify this hypothesis, samples prepared at pH = 7 were supplemented, as shown in Figs. 2(d1)–2(d3). The microscale carbon spheres clearly underwent excessive agglomeration and formed large, irregular clusters, with voids existing within the clusters. The size, density, and thickness of the C coating of the microscale carbon spheres were statistically analyzed, and the results are shown in Figs. 2(e)–2(h). When the pH increased from 3 to 5, the diameter of the microscale carbon spheres slightly decreased from 7.26 to 6.07 μm . The density of the microscale carbon spheres increased from 1.49×10^{-2} to $3.53 \times 10^{-2} \mu\text{m}^{-2}$, meaning that the number of microscale carbon spheres per $100 \mu\text{m} \times 100 \mu\text{m}$ on the carbon fiber surface increased from approximately 1 to approximately 3. When the pH increased to 7, the diameter of the microscale carbon spheres decreased to 5.23 μm , whereas their density sharply increased to $7.64 \times 10^{-2} \mu\text{m}^{-2}$, which is twice that of the microscale carbon spheres at pH = 5. It can be predicted that during the subsequent sintering process, the voids within the clusters will have difficulty being fully filled by the ceramic matrix, ultimately resulting in defects such as pores at the interface of the composite material. These defects undoubtedly have a negative effect on the final mechanical properties and ablation resistance of the composite material. Therefore, only the performance of the composite materials prepared at pH = 1, 3, and 5 will be studied in subsequent research.

Previous studies have shown that the hydrothermal carbonization process of carbohydrates, which are mainly composed of glucose, proceeds according to the growth mechanism of hydrophobic maturation [38]. During hydrothermal treatment, carbohydrates are first converted into 5-hydroxymethylfurfural (HMF) molecular clusters through intramolecular dehydration, and then, through continuous aldol condensation reactions, the molecular clusters gradually become hydrophobic and eventually precipitate from the solution in solid form, generating primary particles. The research results reported by Song *et al.* [39] indicate that reducing the pH value of a solution can increase the electrolyte concentration of the solution, which will reduce the repulsive force between these primary particles and promote their adhesion to the surface of carbon fibers [40]. Owing to the electrical neutrality of carbon fibers, particles are more likely to adhere to the surface of carbon fibers. However, when the electrolyte concentration is low (i.e., when the pH increases), the surface of the carbon fibers becomes charged after adhering to a certain number of particles. This limited the uniform adhesion of the particles on the surface of the carbon fibers, leading to the formation of microscale carbon spheres. Therefore, through hydrothermal carbonization and adjustment of the pH of the solution, carbon fibers with microscale carbon spheres deposited on their surfaces were successfully prepared as the “tenon” structural units. Additionally, a C coating of

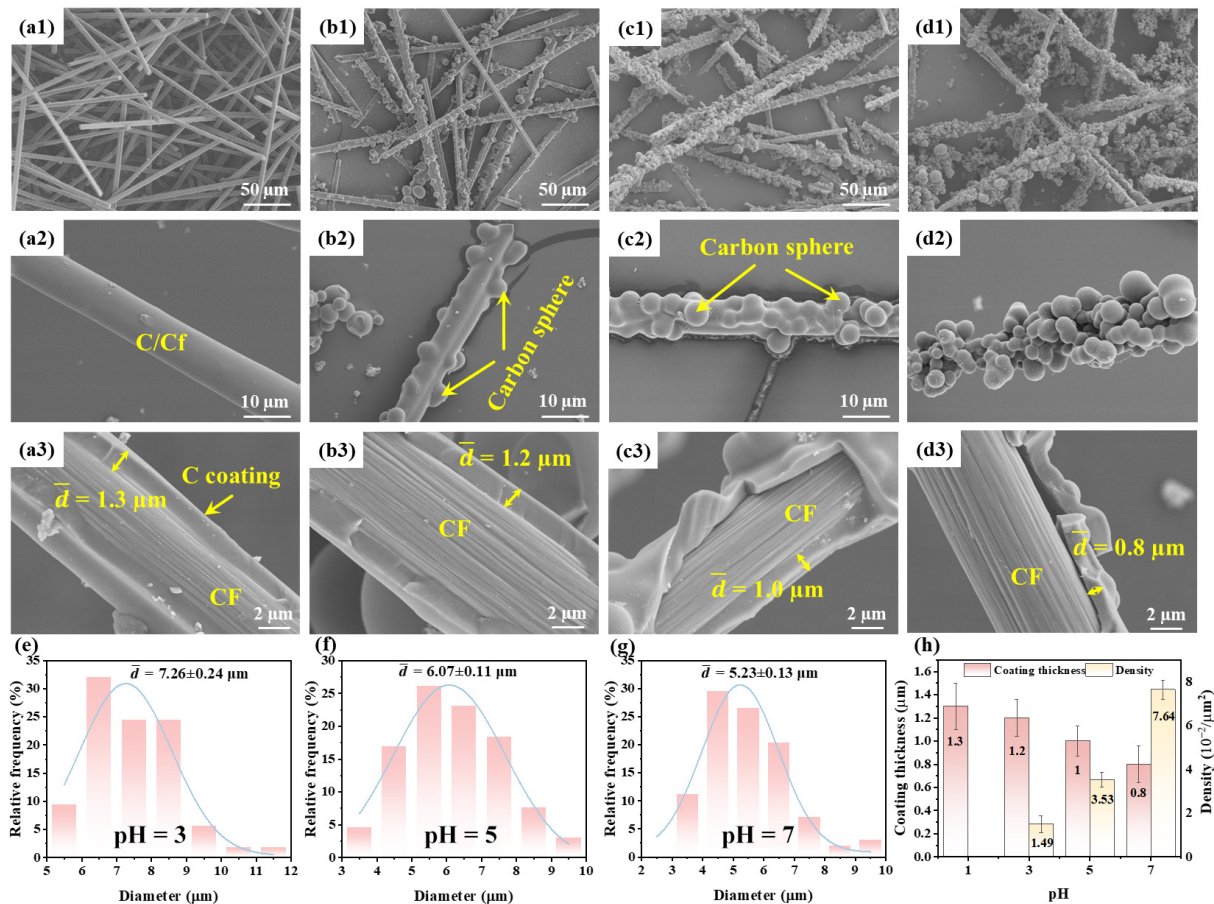


Fig. 2 Morphological changes of C/Cf surface prepared by glucose solution with different pH values: (a1–a3) pH = 1, (b1–b3) pH = 3, (c1–c3) pH = 5, and (d1–d3) pH = 7; (e–g) histograms of size distribution of microscale carbon spheres at pH 3, 5, and 7; (h) changes in density of microscale carbon spheres and thickness of C coating.

appropriate thickness not only serves as a carbon source for the subsequent formation of TiC coatings but also effectively protects the internal carbon fibers from chemical reactions and thermal damage during subsequent processes [41].

Figure 3 shows the surface and fracture morphology of the C/Cf@TiC fibers coated with TiC by the molten salt method. For both the smooth surface of C/Cf (Fig. 2(a2)) and the surface with carbon sphere structures (Fig. 2(c2)), a dense and uniform TiC coating was successfully formed on the surface (Figs. 3(a)–3(c)), with a coating thickness of approximately 0.8 μm (Figs. 3(d)–3(f)). The elemental distribution map (Fig. 3(g)) and XRD pattern (Fig. 3(h)) further confirm that the TiC coating structure was successfully prepared on the surface of the C/Cf. Among them, TiC shows good crystallinity, whereas the C coating exists in the form of amorphous carbon.

3.2 Microstructure and phase composition of Cf/HEC–SiC composites

To observe the complete dispersion of short carbon fibers, SEM images of the polished surfaces of the three composites at low magnification are obtained. As shown in Figs. 4(a1)–4(c1), these images clearly demonstrate that the carbon fibers are randomly and uniformly embedded in the HEC–SiC ceramic matrix. High-magnification SEM images of the three composites are shown in Figs. 4(a2)–4(c2). The carbon fibers are closely embedded in the HEC–SiC matrix, and the C coating effectively isolates the direct contact between the matrix and the carbon fibers, playing a good protective role. Furthermore, in the CHS-3 and CHS-5 samples, the ceramic particles fully filled the pores between the carbon

spheres on the surface of the carbon fibers, effectively enhancing the mechanical interlocking between the matrix and the fibers. The structural feature is similar to that of the mortise–tenon joint found in traditional wooden architecture, especially in the CHS-5 sample, where this phenomenon is more pronounced (Fig. 4(c2)). The mechanical interlocking effect is a typical strengthening mechanism in composites [42], and it is expected to have an important influence on the mechanical properties of Cf/HEC–SiC composites. The relevant content is discussed in detail in the subsequent mechanical properties section. The element distributions corresponding to Figs. 4(a2)–4(c2) are shown in Figs. 4(a3)–4(c3). The results indicate that the gray areas in the composites are the SiC phase, and the white areas are the HEC phase. The constituent phases are uniformly distributed in the material. The XRD patterns of the composites are shown in Fig. 4(d). Owing to the addition amount of carbon fibers being much lower than those of SiC and HEC, the diffraction peak intensity of C is relatively low, and the signal is weak. The relative densities of the three composites are all close to 99% (Fig. 4(e)), which is significantly higher than that of the HEC–SiC composites [35]. This phenomenon can be attributed to the introduction of high-thermal-conductivity carbon fibers, which improve the internal heat conduction of the composites during the SPS process and promote the densification sintering process between particles.

The characterization results of the interface structure between the fibers and the matrix in the composites are shown in Fig. 5. An SEM image of the interface morphology is presented in Fig. 5(a). The results indicate that no TiC coating was observed between the C coating and the matrix; instead, an interface

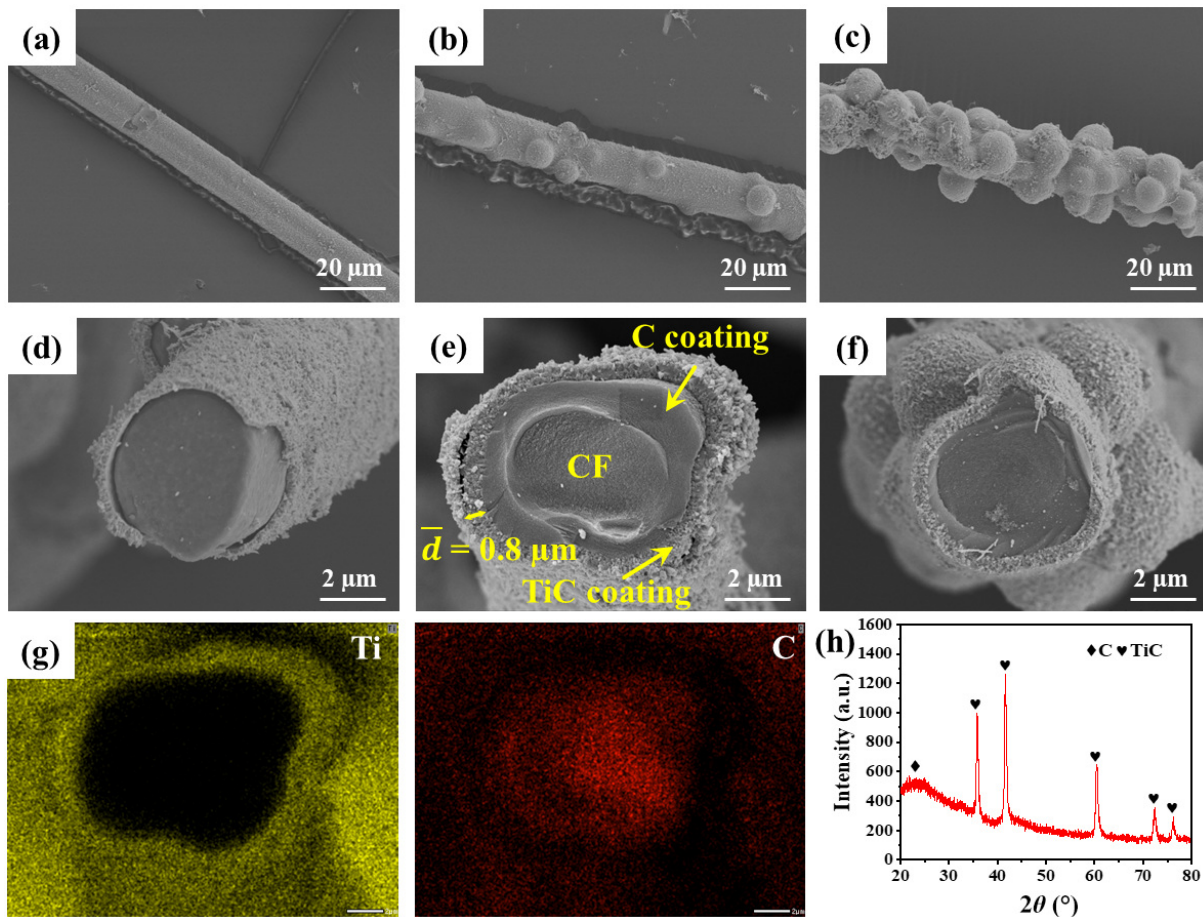


Fig. 3 Surface and cross-sectional morphologies of C/Cf@TiC fibers with TiC coatings prepared by molten salt method: (a, d) pH = 1, (b, e) pH = 3, and (c, f) pH = 5. (g) EDS mapping corresponding to (e). (h) XRD patterns.

reaction layer with a thickness of approximately 500 nm was found. To further analyze the microstructure and composition characteristics of this interface reaction layer, a thin slice sample was prepared in the interface area using FIB technology and used for TEM analysis. The extraction position of the FIB slice is indicated by the white box in Fig. 5(a). Figure 5(b) shows a TEM image of the interface reaction layer, revealing many dense and intertwined ribbon-like network structures. The elemental mapping results in Fig. 5(c) indicate that this ribbon-like network structure is mainly composed of C and contains a small amount of doped metal. The high-resolution TEM (HRTEM) images in Figs. 5(d1)–5(d3) and their corresponding selected area electron diffraction (SAED) analysis further confirm that this ribbon-like network structure is a multilayer graphene (MLG) network. In addition, the HRTEM images of the interfaces between the MLG and the matrix (Fig. 5(e1)) as well as between the MLG and the C coating (Fig. 5(f)) indicate that a tight bond has formed between them. This phenomenon is of great significance for regulating the stress distribution between the fibers and the matrix and achieving effective load transfer [43]. Notably, many embedded nanoparticles can be observed inside the MLG connected to the C coating (Fig. 5(f)). The HRTEM image (Fig. 5(g)) and elemental mapping results (Fig. 5(h)) indicate that these nanoparticles are HEC particles. In addition, an enrichment zone of metal elements was detected in the interface region between the MLG and the C coating. This phenomenon further confirms that the elements in the TiC coating and the substrate diffused into the C coating during the sintering process and induced the graphitization of the C coating. According to the line distribution characteristics of the

Ti element in Fig. 5(i) and the point distribution results of the Ti element in Fig. 5(j), the Ti atoms in the TiC coating clearly diffuse not only into the C coating but also into the HEC–SiC substrate, forming a Ti-rich HEC solid solution. This bidirectional diffusion behavior helps to enhance the chemical bonding and mechanical interlocking between the MLG and the C coating, as well as between the MLG and the substrate.

A comparison of the diffusion coefficients of each single component reveals that the diffusion coefficients of HfC ($1.16 \times 10^{-16} \text{ m}^2 \cdot \text{s}^{-1}$), ZrC ($4.56 \times 10^{-15} \text{ m}^2 \cdot \text{s}^{-1}$), TaC ($4.47 \times 10^{-15} \text{ m}^2 \cdot \text{s}^{-1}$), and NbC ($2.46 \times 10^{-14} \text{ m}^2 \cdot \text{s}^{-1}$) are significantly lower than that of TiC ($6.16 \times 10^{-14} \text{ m}^2 \cdot \text{s}^{-1}$) [44]. This result further validates the intense diffusion behavior of TiC during the SPS process. In fact, owing to the lattice distortion in HECs causing a lag effect [45,46], the diffusion rates of each component in the HEC matrix are much lower than their diffusion coefficients as single components, making it difficult for them to diffuse into the C coating. Therefore, the formation of the MLG reaction layer is mainly controlled by the diffusion behavior of the Ti in the TiC coating. Previous studies have shown that Ti atoms have excellent catalytic graphitization ability [47,48]. During the SPS process, Ti atoms diffuse into the amorphous C coating and promote the transformation of amorphous C to a graphite structure through the formation and decomposition mechanism of TiC under high-temperature and high-pressure conditions [47].

The interfacial bonding characteristics between the MLG reaction layer and the HEC–SiC matrix as well as the C coating were further evaluated by PeakForce QNM AFM technology. The measurement schematic is shown in Fig. 6(a), and the

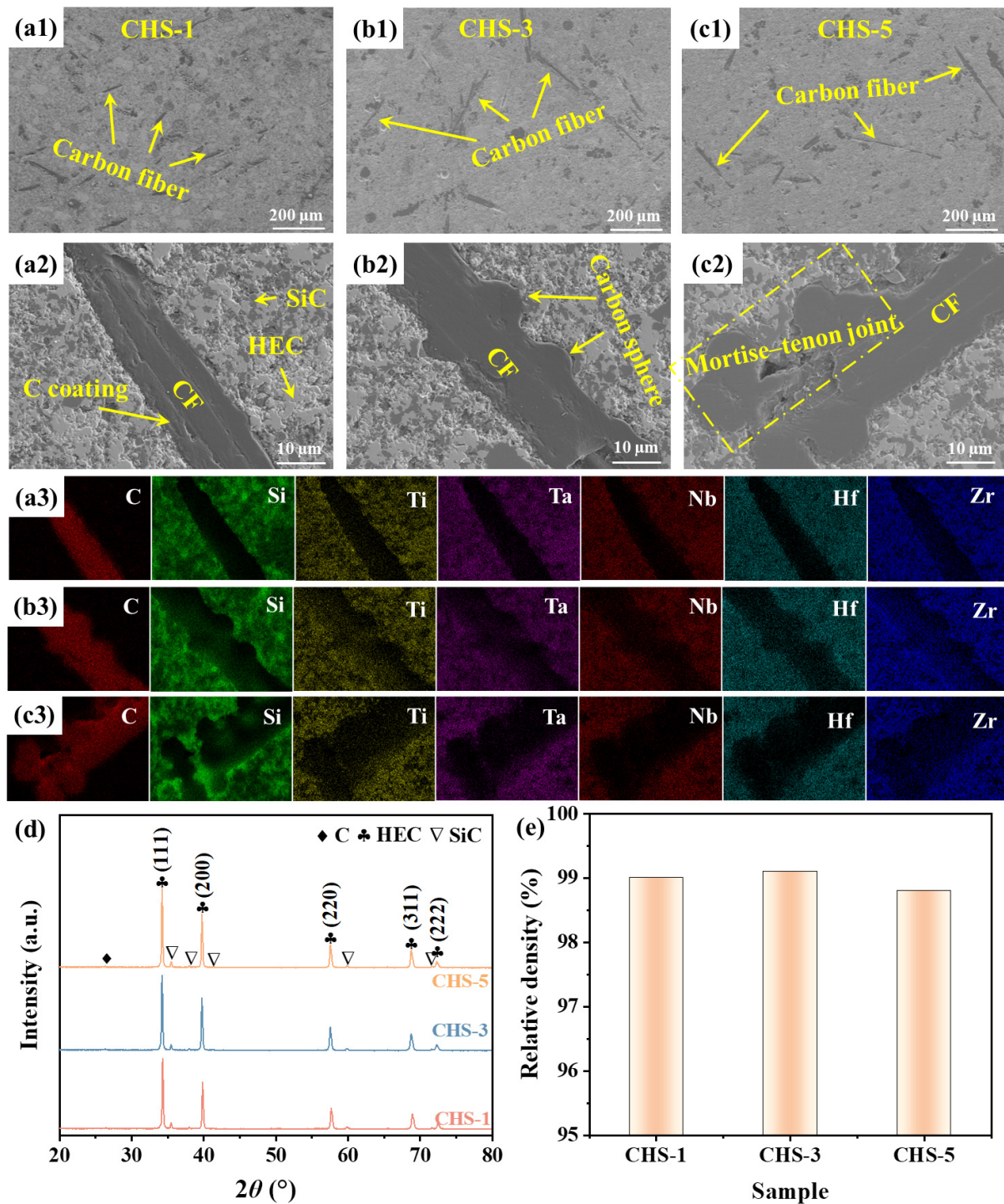


Fig. 4 SEM and EDS images of surface of composites: (a1–a3) CHS-1, (b1–b3) CHS-3, and (c1–c3) CHS-5. (d) XRD patterns. (e) Densification of composites.

measurement results are presented in Figs. 6(b)–6(g). The displacement module (Fig. 6(b)) shows the displacement changes in each phase during interaction with the probe. MLG exhibits a high displacement mapping value, indicating its good flexibility, which is more fully reflected in its displacement distribution diagram (Fig. 6(e)). The modulus module (Fig. 6(c)) reflects the modulus changes during the measurement process. The results show that the modulus of the MLG reaction layer is similar to that of the C coating but significantly lower than that of the HEC–SiC matrix. Notably, no sudden drop in modulus was observed between the MLG and the C coating, whereas a gradient

transition trend in modulus was observed between the MLG and the matrix (Fig. 6(f)). The adhesion force module was used to evaluate the adhesion performance between each phase and the probe (Fig. 6(d)), and it can indirectly reflect the interfacial bonding strength [18]. The adhesion force distribution diagram (Fig. 6(g)) shows that there are obvious adhesion force peaks between the MLG and the HEC–SiC matrix as well as between the MLG and the C coating, indicating that each interface has a strong bonding ability, which is highly important for improving the stress transfer efficiency between the fiber and the matrix and enhancing the overall mechanical properties of the composites [18].

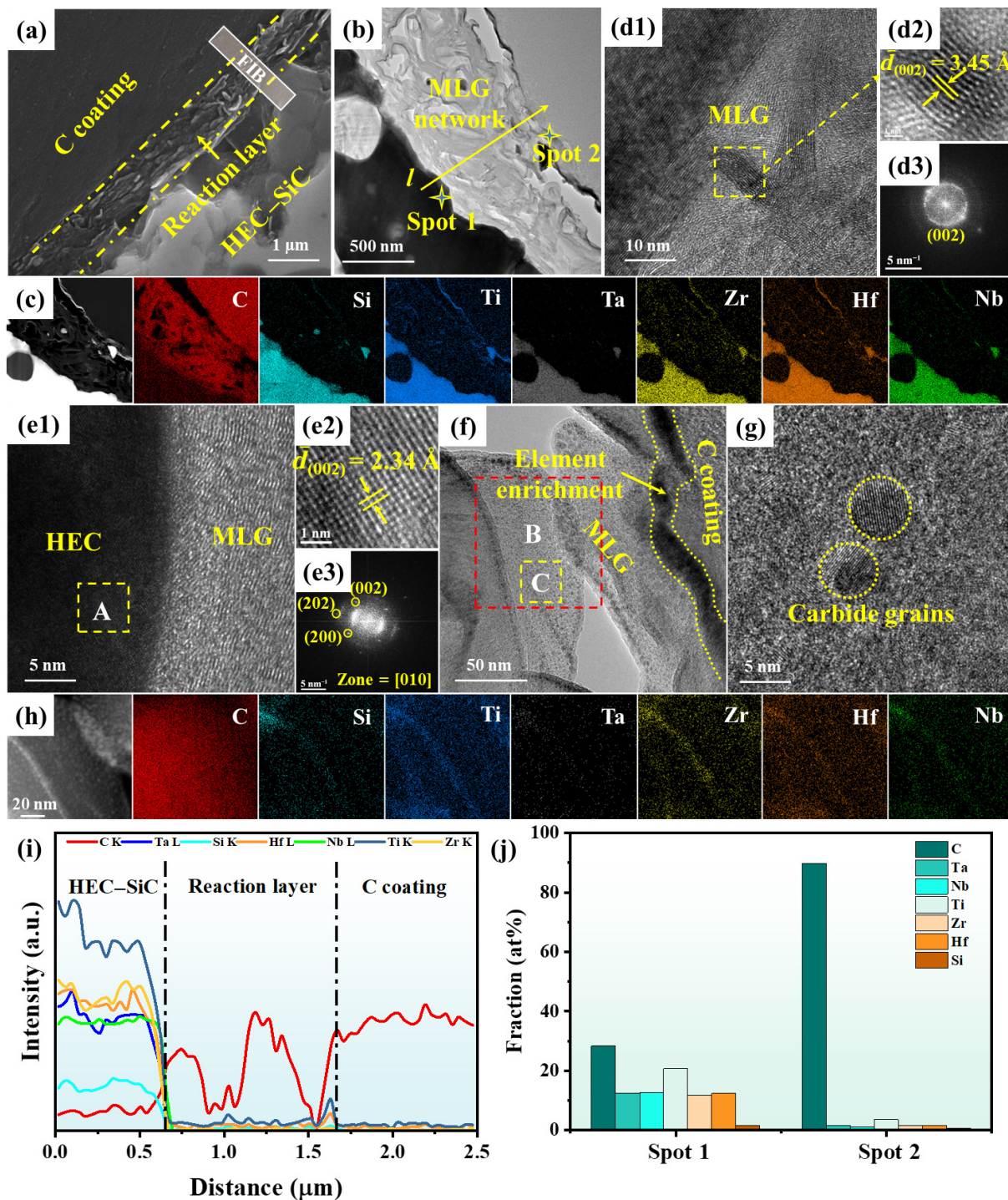


Fig. 5 Morphology and structural analysis of interfacial layer between fibers and matrix: (a) SEM image; (b) TEM image; (c) EDS mapping corresponding to (b); (d1–d3) HRTEM images of reaction layer, interplanar spacing measurement, and SAED pattern; (e1–e3) HRTEM images of interface between matrix and reaction layer, interplanar spacing, and SAED pattern of HEC; (f) TEM image of interface between reaction layer and C coating; (g) HRTEM image in (f); (h) EDS mapping corresponding to red-framed area in (f); (i) line and (j) point distributions of elements in (b).

3.3 Mechanical behavior of Cf/HEC–SiC composites

The results of the mechanical performance evaluation of the three composite materials are shown in Fig. 7. The load–displacement curves obtained from the tests are presented in Fig. 7(a). Although all three composites exhibited brittle fracture characteristics, the CHS-5 sample demonstrated the highest flexural load, reaching 1264 N, compared with the CHS-1 sample. More importantly, its displacement also reached a maximum value of 0.18 mm. These

results indicate that the introduction of the mortise–tenon structure can significantly enhance the deformation tolerance of the composites [7]. The fracture energy calculated through SENB testing is shown in Fig. 7(b). The work of fracture increased from $65.71 \text{ J}\cdot\text{m}^{-2}$ for the CHS-1 sample to $102.47 \text{ J}\cdot\text{m}^{-2}$ for the CHS-5 sample, representing an increase of 56%. The work of fracture reflects how much energy is consumed during the formation of a new fracture surface as the damage process occurs [49]. Therefore, the mortise–tenon structure can effectively consume more

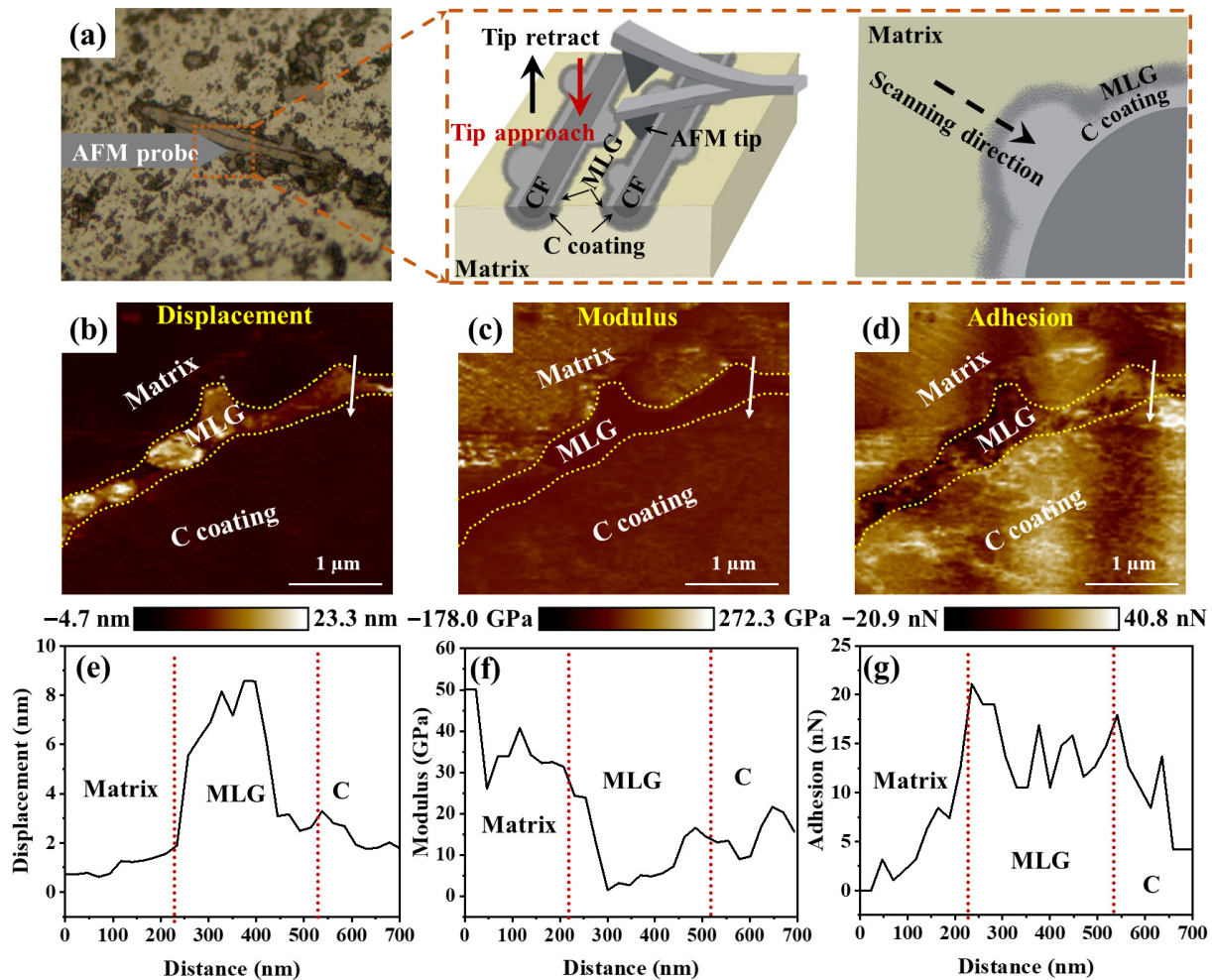


Fig. 6 (a) Schematic diagram of interfacial bonding strength test; (b–d) displacement, modulus, and adhesion force mapping; (e–g) displacement, modulus, and adhesion force distribution diagrams corresponding to positions indicated by solid lines in (b–d).

fracture energy. The CHS-5 sample exhibited the highest flexural strength and fracture toughness (Fig. 7(c)), reaching 1053.33 MPa and $9.77 \text{ MPa}\cdot\text{m}^{1/2}$, respectively, which were 58% and 30% higher than those of the CHS-1 sample (663.51 MPa and $7.50 \text{ MPa}\cdot\text{m}^{1/2}$), fully demonstrating that the fiber–matrix interface with the mortise–tenon structure has a more significant reinforcing effect than the straight interface does. In our previous research [35], we prepared HEC–SiC ceramics via the same SPS process. Its reported flexural strength was approximately 530 MPa, and its fracture toughness was approximately $5.48 \text{ MPa}\cdot\text{m}^{1/2}$. A comparison of the performance of the CHS-5 sample with that of the HEC–SiC ceramics revealed that the flexural strength and fracture toughness increased by approximately 98% and 78%, respectively. This significant improvement undoubtedly proves the excellent reinforcing and toughening effects of carbon fibers as “tenons”. Figure 7(d) shows a comparison of the flexural strength and fracture toughness of the CHS-5 sample prepared in this study with those of representative high-entropy ceramics and their composites reported in the literature [11,26,36,50–58]. The results indicate that the mechanical properties of Cf/HEC–SiC composites with a mortise–tenon structure (CHS-5) are particularly outstanding, even surpassing those of composites reinforced with advanced toughening mechanisms such as graphene or carbon nanotubes [36].

To reveal the mechanism of mechanical property enhancement in Cf/HEC–SiC composites, the crack propagation paths and fracture morphologies of the CHS-1 and CHS-5 samples were

compared and analyzed. The crack propagation morphologies are shown in Fig. 8. As shown in Figs. 8(a) and 8(b), when the cracks propagated from the matrix to the carbon fibers, significantly large-angle deflections occurred. However, owing to the presence of the mortise–tenon structure, the degree of crack deflection in the CHS-5 sample was significantly greater than that in the CHS-1 sample. Additionally, the crack length in the CHS-5 sample reached $40.32 \text{ }\mu\text{m}$ (Fig. 8(b)), whereas that in the CHS-1 sample was only $30.77 \text{ }\mu\text{m}$ (Fig. 8(a)). Moreover, the crack lengths were statistically analyzed, and the results are shown in Figs. 8(d) and 8(e). The average crack length of the CHS-5 sample was $40.41 \text{ }\mu\text{m}$, which was 36% longer than that of the CHS-1 sample ($29.82 \text{ }\mu\text{m}$). A longer crack propagation path helps dissipate more fracture energy, thereby enhancing the fracture toughness of a material. When observing the interface between the fibers and the matrix after crack propagation, many MLG pull-outs and bridging phenomena were observed (Fig. 8(c)), which are typical toughening mechanisms of graphene [59].

The fracture morphologies of the CHS-1 and CHS-5 samples are shown in Figs. 8(f1)–8(g3). In both samples, toughening mechanisms such as fiber pull-out and interfacial debonding were observed (Figs. 8(f1), 8(f2), 8(g1), and 8(g2)), and the fiber surfaces were relatively rough, with a large amount of pulled-out MLG adhering to them (Figs. 8(f3) and 8(g3)). Therefore, on the basis of the morphological features observed in Figs. 8(c), 8(f3), and 8(g3), the cracks passing through the substrate preferentially propagate within the MLG layer rather than along the

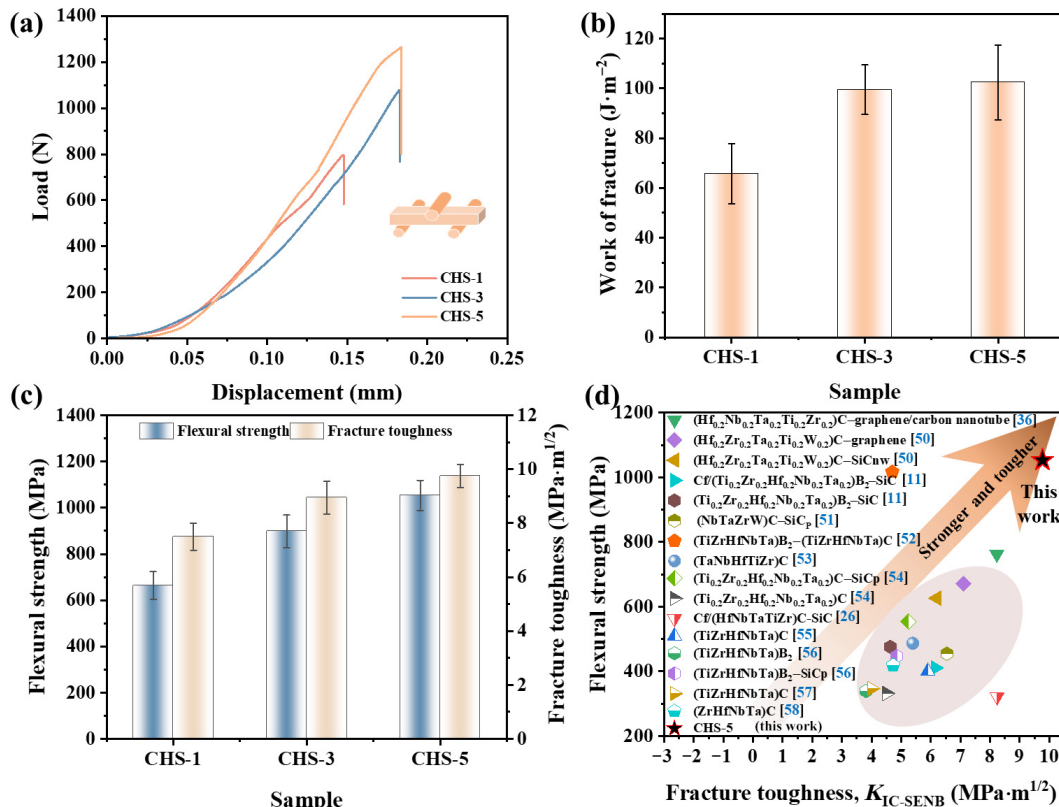


Fig. 7 (a) Load-displacement curve; (b) work of fracture; (c) flexural strength and fracture toughness; (d) comparison of mechanical properties.

MLG/substrate interface or the MLG/C interface. This propagation path is determined by the structure of the MLG layer and its interfacial characteristics. First, the HRTEM results (Fig. 5(b)) show that the *in situ*-formed MLG layer is approximately 500 nm thick and is composed of many interwoven and curved MLG nanoribbons. This considerable thickness and unique interlocking network structure make the MLG layer itself no longer a fragile two-dimensional interface but rather a three-dimensional nanostructured region with significant deformation capacity (Fig. 6(b)). Additionally, there is a modulus transition zone between the MLG and the HEC-SiC matrix (Figs. 6(c) and 6(f)), and significant adhesion force peaks are detected at both the MLG/matrix and MLG/C interfaces (Figs. 6(d) and 6(g)). This is directly related to the composition gradient formed by the diffusion of elements such as Ti, as observed in Figs. 5(c) and 5(i). Therefore, this “composition gradient” structure can effectively alleviate the stress concentration caused by the modulus mismatch between the substrate and the fiber, prevent the initiation of microcracks at the rigid interface, and enhance the interface bonding at both the MLG/matrix and MLG/C interfaces. When the crack propagates from the substrate to the fiber-matrix interface, the MLG layer with significant deformation capacity captures the crack and absorbs and dissipates energy through the bending, sliding, and reorientation of the graphene sheets within the MLG layer. This is an extremely ideal failure mode because it fully utilizes the nanoscale toughening mechanism of graphene and achieves noncatastrophic cracking at the interface. These results prove that MLG plays an important role in enhancing the interfacial bonding between the matrix and the fibers. Therefore, the significant improvement in the interface performance enables the CHS-1 sample to exhibit superior mechanical properties compared with those of traditional carbon fiber-reinforced ceramic matrix composites [11,60]. Compared with the fracture morphology of the CHS-1 sample, in

the CHS-5 sample, in addition to the MLG sample, the surfaces of the pulled-out fibers also retained the original carbon sphere structure (Figs. 8(g1) and 8(g2)), which indicates that the carbon sphere structure has a strong bonding force with the carbon fibers and does not fall off during the fiber pull-out process but is pulled out together with the fibers. This also confirms that the carbon spheres play a key role in constructing the mortise-tenon joint between the fibers and the matrix. Through the firm mortise-tenon joint between the fibers and the matrix, effective interface load transfer from the matrix to the fibers can be achieved [61]. This unique reinforcement mechanism significantly enhances the mechanical properties of the composites. Notably, in Figs. 8(f2) and 8(g2), a toughening mechanism in which carbon fibers are pulled out from the C coating can be observed. This is because the interfacial resistance when the fibers are pulled out from the matrix exceeds the bonding strength between the carbon fibers and the C coating, thus resulting in the phenomenon of the carbon fibers being pulled out from the C coating. The observation of the surface morphology of the carbon fibers before and after sintering further confirms that the carbon fibers are well protected. The surface morphology of the carbon fibers before sintering is shown in Figs. 2(a3)–2(d3), revealing a typical groove structure. The surface morphology of the carbon fibers after sintering can be observed through SEM images of the fibers pulled out at the fracture surface, as shown in the inset of Fig. 8(f2). This groove structure can still be clearly observed. This finding indicates that even under extreme sintering conditions (2000 °C), the structural integrity of the carbon fibers was well preserved under the protection of the C coating.

3.4 Thermal shock resistance and ablation resistance of Cf/HEC-SiC composites

The enhancement of toughness contributes to improving the

impact resistance and service reliability of composites. The residual strength after water quenching with different thermal shock temperature differences is a recognized parameter for

evaluating the thermal shock resistance of composites [62]. Figures 9(a) and 9(b) summarize the residual strength and strength attenuation rates of the above three composites after

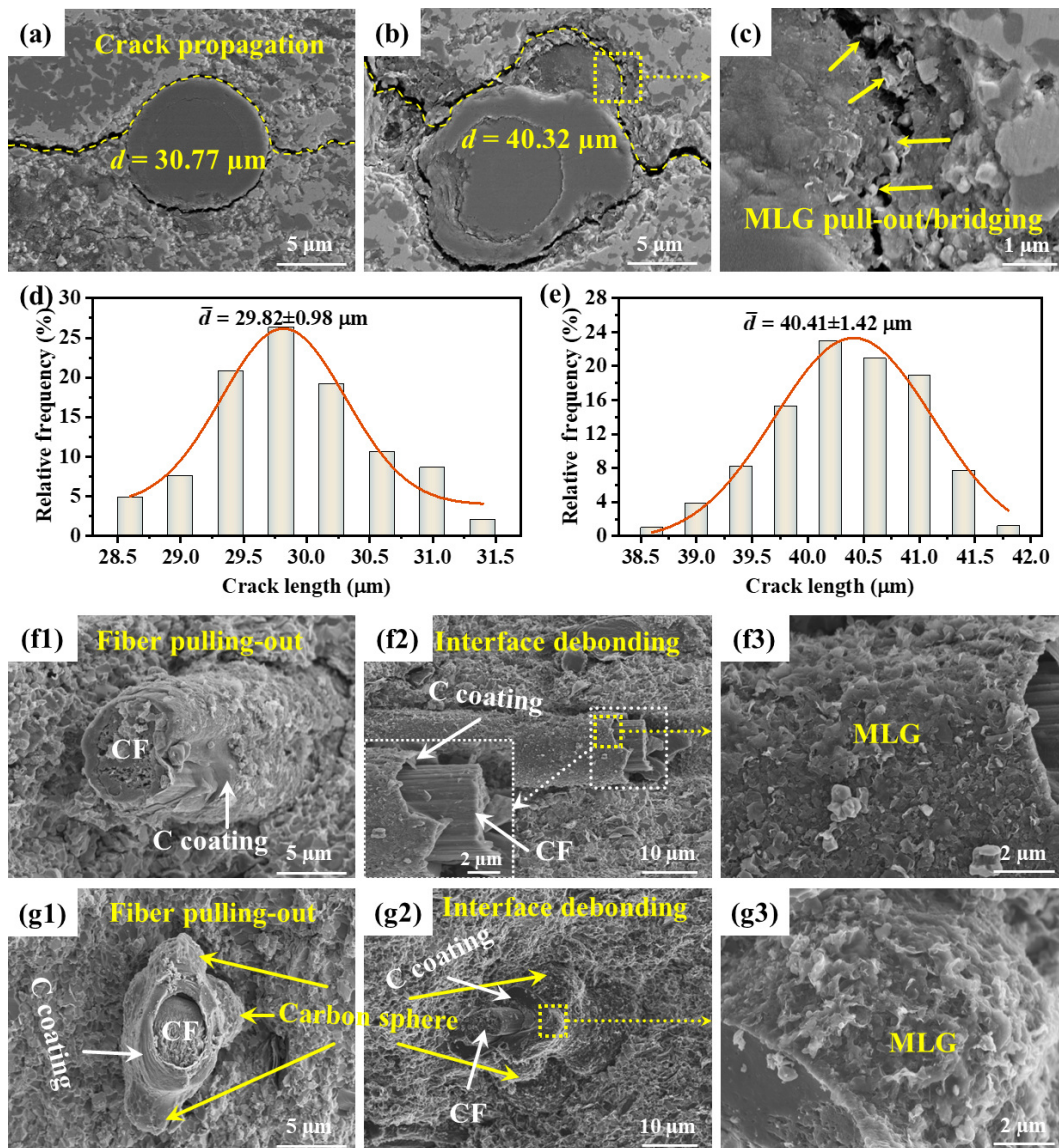


Fig. 8 Crack propagation morphology of (a) CHS-1 and (b) CHS-5 samples. (c) Interfacial morphology after crack propagation. Crack length statistics of (d) CHS-1 and (e) CHS-5 samples. Fracture surface morphology of (f1–f3) CHS-1 and (g1–g3) CHS-5 samples.

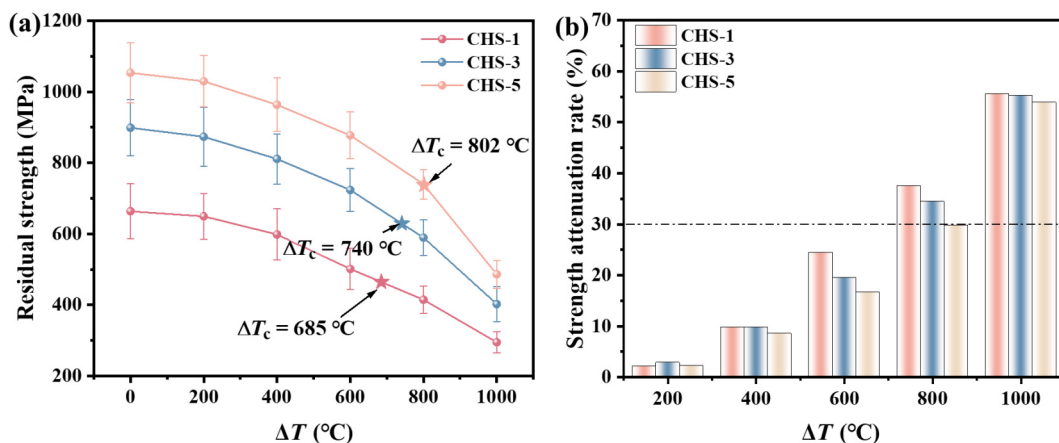


Fig. 9 Comparison of thermal shock resistance of composites: (a) residual strength variation and (b) strength attenuation rate variation.

water quenching with thermal shock temperature differences of $\Delta T_c = 200\text{--}1000\text{ }^\circ\text{C}$. The ΔT_c of each material was determined by linear interpolation of the residual strength values (Fig. 9(a)). The results show that ΔT_c increased from $685\text{ }^\circ\text{C}$ for the CHS-1 sample to $802\text{ }^\circ\text{C}$ for the CHS-5 sample. Additionally, the ΔT_c of the CHS-5 sample is approximately twice that of traditional UHTC materials ($\Delta T_c < 400\text{ }^\circ\text{C}$) [37], demonstrating excellent thermal shock resistance. The strength attenuation rate in Fig. 9(b) further indicates that after thermal shock at $800\text{ }^\circ\text{C}$, the strength attenuation rates of the CHS-1 and CHS-3 samples exceeded 30%, whereas that of the CHS-5 sample remained below 30%. Furthermore, under all ΔT_c conditions, the increase in the

strength attenuation rate of the CHS-5 sample remained at the lowest level, indicating that the strong interface bonding formed by the mortise–tenon joint played a crucial role in the dissipation of thermal stress.

Three types of composites were subjected to three cycles of oxyacetylene flame ablation tests to further investigate their thermal shock resistance and ablation resistance at high temperatures ($> 2000\text{ }^\circ\text{C}$) and under aerodynamic loads, verifying their service reliability in extreme environments. The results are shown in Fig. 10. Figure 10(a) presents the macroscopic morphological characteristics of the three samples after three cycles of ablation. The CHS-1 sample exhibited through-cracks on

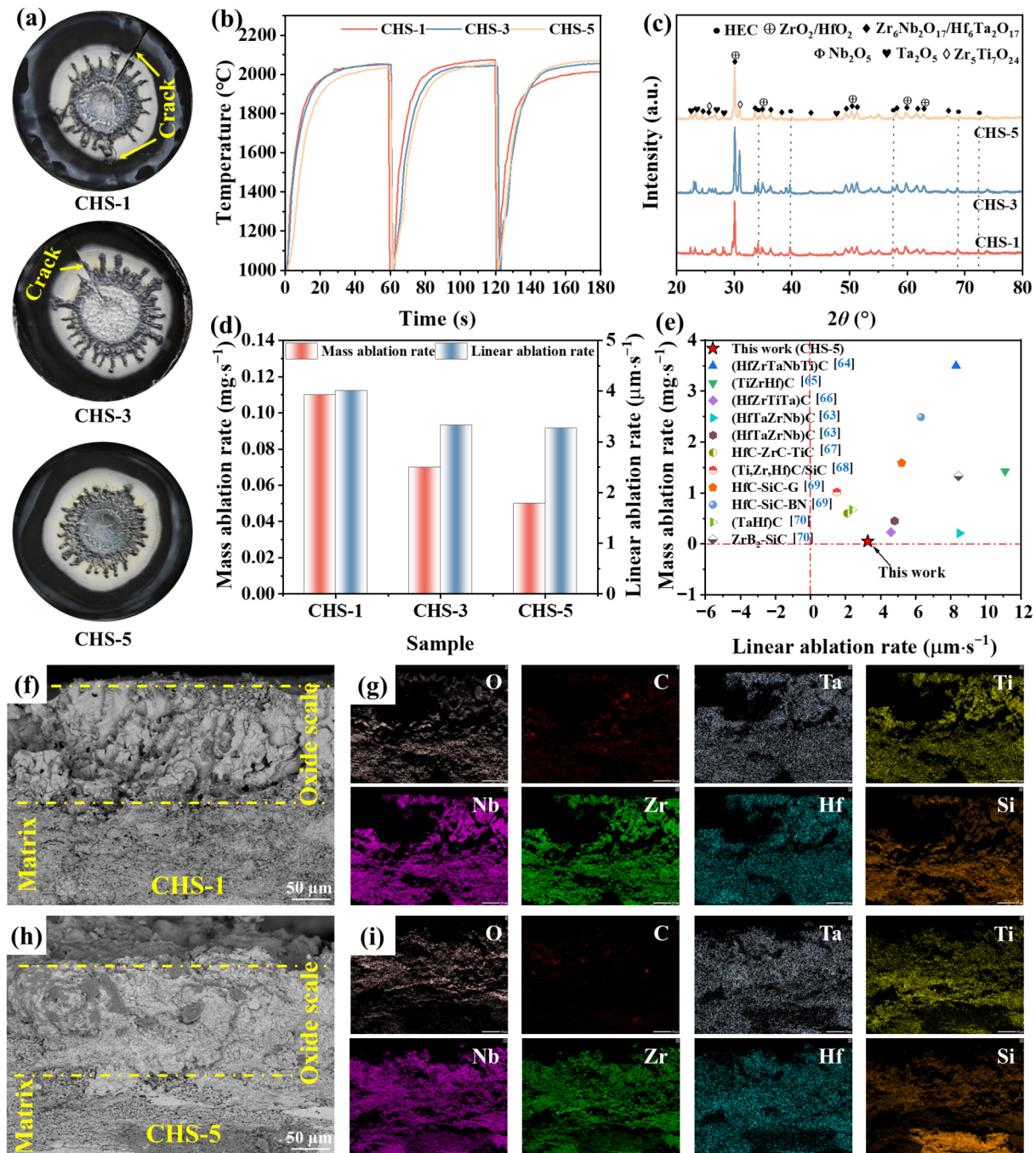


Fig. 10 Comparison of ablation resistance of composites: (a) surface morphology variation; (b) surface temperature variation; (c) phase distribution after ablation; (d) mass ablation rate and linear ablation rate; (e) comparison of ablation resistance; (f–i) cross-sectional morphology and elemental distribution of oxide scale of CHS-1 and CHS-5 samples.

its surface, characteristic of catastrophic cracking and failure, demonstrating poor thermal shock resistance. In contrast, the CHS-3 sample did not show through-cracks but only a few microcracks. In sharp contrast, the CHS-5 sample maintained good structural integrity without any signs of cracking, once again confirming its excellent thermal shock resistance. Therefore, the mortise–tenon structure design of the fiber–matrix interface significantly enhances the thermal shock resistance of the Cf/HEC–SiC composites, which still performs well even under extreme test conditions.

After three cycles of ablation, all three samples showed varying degrees of ablation oxidation on their surfaces. The surface temperature changes of the samples during the ablation process are shown in Fig. 10(b). The surface temperatures of all the samples exceeded 2000 °C. Notably, the surface temperature of the CHS-1 sample decreased significantly during the third ablation compared with those of the previous two samples, indicating that it cracked. This occurred because the heat from the oxyacetylene flame could escape through the crack gaps, resulting in a lower surface temperature. The phase composition after ablation is shown in Fig. 10(c). The main diffraction peaks were highly consistent with the crystal structures of $\text{Nb}_2\text{Zr}_6\text{O}_{17}$, $\text{Ta}_2\text{Hf}_6\text{O}_{17}$, ZrO_2 , and HfO_2 . Additionally, some weaker diffraction peaks were identified as $\text{Zr}_5\text{Ti}_7\text{O}_{24}$, Nb_2O_5 , and Ta_2O_5 . Owing to the high volatility of SiO_2 at high temperatures, its diffraction peak was not detected in the XRD patterns. Importantly, in high-temperature environments, the diffusion of metals and oxygen is highly active, promoting the formation of many complex multicomponent solid solutions. This inevitably leads to the overlap or shift of XRD peaks, making phase analysis difficult [63]. Therefore, XRD analysis of the ablated surface can provide only possible phase combinations. The ablation rate calculation results for the composites are shown in Fig. 10(d). The mass ablation rate of the CHS-5 sample was $0.05 \text{ mg}\cdot\text{s}^{-1}$, and the linear ablation rate was $3.27 \mu\text{m}\cdot\text{s}^{-1}$, which were 54.54% and 18.45% lower than those of the CHS-1 sample at $0.11 \text{ mg}\cdot\text{s}^{-1}$ and $4.01 \mu\text{m}\cdot\text{s}^{-1}$, respectively. Moreover, Fig. 10(e) shows a comparison of the ablation rate of the CHS-5 sample in this study with those of other ceramic materials reported in the literature [63–70]. After three cycles of 60-s ablation tests, the CHS-5 sample still exhibited excellent ablation resistance. Therefore, by introducing carbon fiber reinforcements with carbon spheres on the surface and forming a mortise–tenon structure with the HEC–SiC ceramic matrix, the thermal shock resistance and ablation resistance of the Cf/HEC–SiC composites can be effectively enhanced.

Owing to its poor thermal shock resistance, the CHS-1 sample suffered fatal cracking after three cycles of ablation (Fig. 10(a)). At such macroscopic crack defects, the rates of flame erosion and oxygen diffusion accelerate. This further deteriorates the integrity of the oxide scale and reduces the ablation resistance of the composite material. It can be clearly seen from the cross-sectional morphology of the oxide scale at the cracked area of the CHS-1 sample (Fig. 10(f)) that the oxide scale has suffered severe damage, and even the substrate is partially exposed. The distribution of oxygen (Fig. 10(g)) confirms that oxidation occurs inside the substrate. In sharp contrast, the CHS-5 sample did not crack after three cycles of ablation (Fig. 10(a)), which is attributed to the mortise–tenon structure and the MLG layer significantly enhancing the thermal shock resistance of the composite material. This means that a continuous oxide scale can be formed on the surface of the CHS-5 sample. The cross-sectional morphology of the oxide scale of the CHS-5 sample (Fig. 10(h)) shows that the oxide scale is dense and well-bonded to the substrate, and the

distribution of oxygen (Fig. 10(i)) also proves that the oxide scale provides good protection to the substrate. Therefore, by introducing the mortise–tenon structure and MLG interlayer, the thermal shock resistance and ablation resistance of the Cf/HEC–SiC composite material can be effectively improved.

3.5 Toughening and strengthening mechanisms of Cf/HEC–SiC composites

On the basis of the above observations and analyses, the toughening and strengthening mechanisms of Cf/HEC–SiC composites with different morphologies of carbon fibers in terms of their mechanical properties and thermal shock resistance can be summarized, as shown in Fig. 11. First, to address the weak adhesion and poor wettability between the ceramic matrix and carbon fibers, an efficient and stable MLG bonding layer was formed *in situ* between the fibers and the matrix through the diffusion effect of the TiC coating and the catalytic graphitization effect of Ti (Figs. 5 and 6), significantly enhancing the interfacial bonding strength between the matrix and carbon fibers. During the fracture process, when the crack propagates from the matrix to the fiber–matrix interface, the MLG layer with significant deformation capacity captures the crack and makes it propagate within the MLG layer, absorbing and dissipating energy through the pull-out and bridging of the graphene sheets within the MLG layer (Fig. 8(c)). The carbon-coated fibers were subsequently pulled out from the matrix, which carried many graphene sheets (Fig. 8(f1)). However, the pull-out and bridging of the graphene sheets and the rough surface increase the resistance between the interfaces during the pull-out process (Fig. 8(f3)). When the resistance exceeds the bonding strength between the C coating and the carbon fibers, the C coating peels off, and the carbon fibers are pulled out from the C coating (the inset of Fig. 8(f2)). Therefore, compared with traditional carbon fiber-reinforced ceramic matrix composites, the CHS-1 sample still has superior mechanical properties. However, despite the multiple toughening mechanisms mentioned above, the relatively straight surface of ordinary carbon fibers still easily leads to rapid fiber pull-out and debonding, as well as rapid crack propagation, as shown in Fig. 11(a). In contrast, the mechanical interlocking interface with a mortise–tenon connection structure effectively solves the above problems. As shown in Fig. 11(b), on the basis of the above multiple toughening mechanisms, the further improvement in the mechanical properties of the composite material can be attributed to the following three mechanisms: First, the carbon fibers with carbon spheres attached to the surface can form a good mortise–tenon joint structure with the matrix, thereby achieving effective stress transfer from the matrix to the fibers. Under this mechanism, the relative slip between the fibers and the matrix is restricted, significantly delaying the fiber pull-out and debonding process and thereby enhancing the material's strength. Second, the carbon spheres embedded in the matrix produce a pinning effect, which can hinder crack propagation and induce continuous large-angle deflection. Third, the presence of carbon spheres causes the fiber surface to present an irregular morphology, significantly extending the crack propagation path and thereby dissipating more fracture energy and improving the fracture toughness of the material. In summary, by introducing the interfacial bonding method imitating the mortise–tenon joint, effective mechanical interlocking between the fibers and the matrix is achieved, significantly enhancing the interface performance. Under this structural design, the carbon fiber-reinforced Cf/HEC–SiC composite achieves synergistic enhancement of strength and toughness.

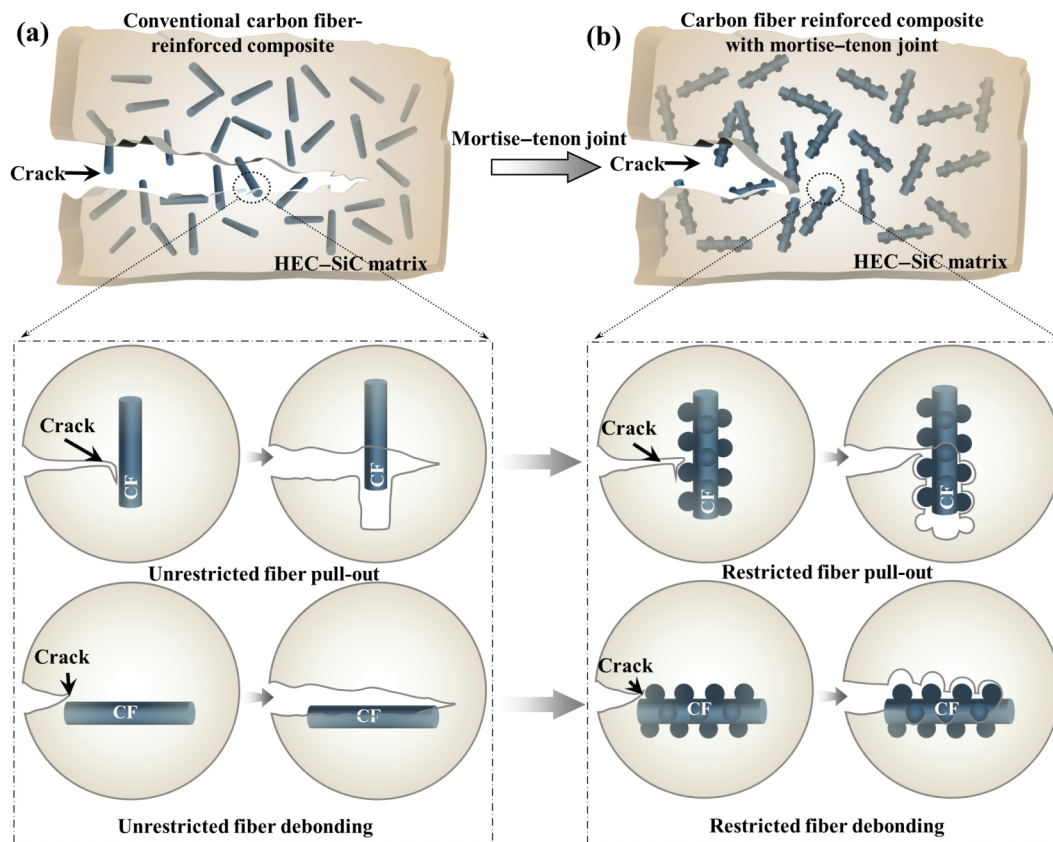


Fig. 11 Toughening and strengthening mechanisms of Cf/HEC-SiC composites under different fiber-matrix interface reinforcement methods: (a) straight interface and (b) interface with a mortise-tenon joint.

4 Conclusions

(1) A novel “mortise-tenon” joint interface has been successfully developed in Cf/(TiZrHfNbTa)C-SiC composites. In this design, microscale carbon spheres deposited on carbon fibers act as the “tenon”, whereas the matrix serves as the corresponding “mortise”. Additionally, a TiC interfacial layer was introduced to enhance interfacial bonding strength. The TiC coating promotes the catalytic graphitization of the C layer, resulting in the formation of an MLG interlayer at the interface. This interface structure enables synergistic reinforcement through both chemical bonding and mechanical interlocking.

(2) The composite featuring the “mortise-tenon” joint interface shows exceptional mechanical performance, achieving a flexural strength of 1053.33 MPa, a fracture toughness of 9.77 MPa·m^{1/2}, and a fracture energy of 102.47 J·m⁻². Furthermore, this material exhibits outstanding thermal shock resistance, with a critical thermal shock temperature difference (ΔT_c) of 802 °C, along with excellent ablation resistance, characterized by a linear ablation rate of 3.27 $\mu\text{m}\cdot\text{s}^{-1}$ and a mass ablation rate of 0.05 mg·s⁻¹. These properties highlight its promising potential for applications in high-temperature and extreme environments.

(3) The improvements in strength and toughness can be attributed primarily to the robust “mortise-tenon” joint interface. This interface configuration provides effective physical anchoring points for stress transfer, significantly enhancing the load-bearing efficiency while delaying fiber debonding and pull-out, ultimately improving the material's strength. Additionally, this architecture effectively hinders crack propagation by inducing large-angle crack deflection, which substantially lengthens the crack propagation path and dissipates greater amounts of fracture energy. Therefore, the “mortise-tenon” joint design strategy offers

a practical and feasible approach for developing high-performance UHTCs.

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Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

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