



Ultra-high-temperature tensile properties of mesophase pitch-based carbon fiber reinforced C/C composite

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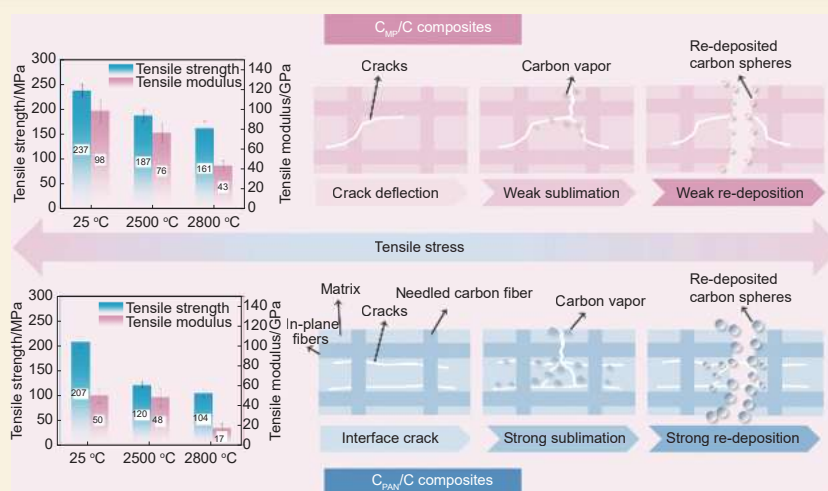
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Abstract: Integrated thermal protection and load-bearing structures for high-speed vehicles require carbon/carbon (C/C) composites capable of retaining structural integrity at ultra-high temperatures. However, their high-temperature mechanical behavior is governed by the combined effects of fiber structure, interfacial bonding, and the occurrence of thermomechanical mismatch-induced damage. A mesophase pitch-based carbon fiber reinforced C/C composite (C_{MP}/C) graphitized at 3000 °C was investigated, with a polyacrylonitrile-based carbon fiber reinforced C/C composite (C_{PAN}/C) as a comparison. The tensile behavior of both C/C composites was evaluated at room temperature, 2500 and 2800 °C. Combining experimental characterization with theoretical simulation, the correlations among fiber structure characteristics, interfacial residual stress distribution, crack propagation path, and high-temperature damage mechanisms were clarified. The results showed that the C_{MP}/C composite has a superior mechanical performance at ultra-high temperatures. Its tensile strengths at room temperature, 2500 and 2800 °C were 236.7, 186.6 and 160.9 MPa respectively, which are approximately 15%, 56% and 55% higher than those of the C_{PAN}/C composite (206.9, 119.6 and 103.7 MPa), the corresponding strength retentions at 2500 and 2800 °C were 79% and 68% for C_{MP}/C , markedly higher than the 58% and 50% for C_{PAN}/C . This exceptional ultra-high-temperature performance is attributed to the combined effects of reduced interfacial residual stress and enhanced crack-deflection capability, which collectively facilitate energy dissipation. These findings provide microstructural design guidelines for improving the ultra-high temperature reliability of C/C composites.

Key words: Mesophase pitch-based carbon fiber; C/C composites; Ultra-high-temperature mechanical performance; Tensile strength; Damage mechanism



1 Introduction

The development of high-speed vehicles toward higher Mach numbers, longer service durations, and greater heat flux densities imposes increasingly strin-

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gent requirements on load-bearing thermal protection components such as sharp leading edges, nose cones, nozzles, and their extensions. These components must withstand not only severe aerodynamic heating but also aerodynamic loads, vibration, and thermally induced stresses arising from temperature gradients^[1–6]. Carbon/carbon (C/C) composites, featuring low density, high specific strength, low thermal expansion coefficient, and excellent mechanical properties, are among the few high-temperature structural materials capable of retaining high mechanical performance above 2000 °C, and even exhibiting counterintuitive strength enhancement under specific conditions. They are therefore considered as prime candidate materials for transpiration-cooled thermal protection structures^[7–8]. To fully harness their potential in such extreme environments, however, it is essential to simultaneously enhance their thermal conductivity and mechanical properties, thereby preventing local thermal-structural failure caused by strength degradation and oxidation-induced damage at ultra-high temperatures near sharp geometric features. Only through such comprehensive improvements can a reliable basis be established for evaluating the high-temperature strength and understanding the underlying damage mechanisms, which is of critical importance for the safe and durable design of next-generation thermal protection systems.

High-temperature mechanical performance is a key indicator for evaluating the suitability of C/C composites for transpiration-cooled load-bearing structures^[9–10]. Their mechanical response is collectively governed by fiber architecture, matrix composition, interfacial bonding state, and distribution of pores and cracks^[11–12]. Existing research has predominantly focused on conventional polyacrylonitrile-based carbon fiber reinforced C/C composites (C_{PAN}/C), providing valuable insights into their mechanical behavior at moderate to high temperatures. Song et al.^[13] demonstrated that the high-temperature tensile strength of C_{PAN}/C composite depends not only on intrinsic fiber properties but also on the evolution of graphite crystallites, fiber skin-core structure,

fiber/matrix interface, and pore/crack evolution. When the heat treatment temperature exceeds 2600 °C, the tensile strengths at 2000 and 2500 °C exceed room-temperature values, indicating that interfacial state and pore structure significantly influence high-temperature load-bearing behavior. Hatta et al.^[14] found that the compressive strength of C_{PAN}/C composite first increased with temperature, reached a maximum near 2026 °C, and subsequently declined due to matrix degradation. Yao et al.^[15] conducted transverse and longitudinal compression tests on fine-weave pierced C/C composites at temperatures above 2000 °C, and observed a variety of damage mechanisms at elevated temperatures, including fiber fracture, matrix cracking, fiber pull-out, and interfacial debonding. These studies collectively indicate that the high-temperature mechanical behavior of C/C composites is highly structure-sensitive, with failure modes typically governed by the interplay of fiber load-bearing, interfacial load transfer, crack deflection, and matrix degradation.

Compared with C_{PAN}/C composites, mesophase pitch-based carbon fiber reinforced C/C (C_{MP}/C) composites exhibit unique advantages in transpiration-cooled thermal protection and load-bearing structures owing to their higher graphitization degree, superior preferred orientation, and higher axial thermal conductivity^[16]. The highly oriented graphite crystallite structure of MPCF not only constructs continuous and efficient heat-transfer pathways within the composite, thereby reducing temperature gradients and thermal stress concentration during service, but also provides stable structural support under extreme thermal environments due to its high modulus characteristics^[17–18]. Previous studies have demonstrated that graphitization treatment at 3000 °C significantly improves the graphite crystallite ordering, texture orientation, and thermal conductivity of C_{MP}/C composites, resulting in superior thermophysical properties^[19]. However, while the highly graphitized and oriented structure enhances heat transport capability, it also modifies the thermal expansion behavior of fibers, thermophysical compatibility between the fiber and matrix, and inter-

facial stress state. Therefore, the influence of intrinsic structural differences between MPCF and conventional PAN-based carbon fibers on mechanical response and damage evolution remains to be further elucidated.

Currently, studies on C_{MP}/C composites have mainly focused on thermal conductivity enhancement, thermal expansion regulation, and ablation resistance improvement, whereas a systematic understanding of how different reinforcement fiber systems govern the mechanical behavior of C/C composites remains limited. In recent years, the ultra-high-temperature mechanical behavior of C/C composites has attracted increasing attention. Song et al.^[13] revealed the multi-scale evolution mechanism of C_{PAN}/C composites at temperatures up to 2500 °C; Wang et al.^[9] investigated the mechanical behavior of needle-punched C/C composites under time-varying thermo-mechanical coupling conditions; Li et al.^[20] reported the tensile properties of three-dimensional stitched C/C composites at temperatures up to 2500 °C. MPCF and PAN-based carbon fibers exhibit significant differences in graphitization degree, crystallite size, structural orientation, defect characteristics, and thermal expansion anisotropy. These intrinsic structural differences not only determine the mechanical response and high-temperature stability of the fibers themselves, but also further affect interfacial stress distribution, load transfer efficiency, crack evolution behavior, and final failure modes. Therefore, clarifying the influence mechanism of reinforcement fiber structure on damage evolution and macroscopic mechanical performance is essential for evaluating the service reliability of C_{MP}/C composites and promoting their application in load-bearing thermal protection structures.

In this study, a previously optimized 3000 °C-graphitized C_{MP}/C composite and conventional C_{PAN}/C composite as a benchmark were used to systematically investigate the tensile mechanical behavior and failure mechanisms at 2500 °C and 2800 °C. Through microstructural characterization, residual stress analysis, and finite element simulation, the differences in interfacial stress distribution, crack propagation paths,

and dominant failure modes between the two C/C composites are revealed. The relationships among fiber structural characteristics, interfacial load transfer behavior and ultra-high-temperature mechanical retention capability are established to advance fundamental understanding of ultra-high-temperature failure mechanism of C/C composites and offer theoretical guidance for material selection and reliability assessment of integrated thermal protection structures.

2 Experiment

2.1 Key materials

In this study, MPCF and polyacrylonitrile (PAN)-based carbon fiber (T700) were selected as reinforcements for the fabrication of C/C composites (Table 1). The T700 carbon fiber was supplied by Zhongfu Shenying Carbon Fiber Co., Ltd. (Lianyungang, China), and the MPCF by Hunan Toyi Carbon Materials Technology Co., Ltd. (Changsha, China). Prior to preform fabrication, the T700 fibers were desized by acetone rinsing followed by heat treatment at 450 °C to remove the surface sizing agent. Both C/C composites designated C_{MP}/C (MPCF-reinforced) and C_{PAN}/C (T700-reinforced) were fabricated by chemical vapor infiltration (CVI) followed by hot isostatic pressing (HIP). The preform features a fine-woven puncture structure, with the X/Y directions composed of stacked eight-harness satin weave carbon fabrics (lay-up density of approximately 8 layers/cm, arranged in a 0°/90° orthogonal stacking sequence). The Z-direction is reinforced by puncture fibers at a density of roughly 25 needles·cm⁻². The MPCF tow specification is 1K, while the T700 tow is 6K, and the overall fiber volume fraction is approximately 40%-45% (volume fraction). The CVI process was conducted to deposit pyrolytic carbon (PyC), with a deposition temperature of 950–1020 °C and a pressure of 1.0 kPa, to densify the preform to its initial densities (approximately 1.60 g·cm⁻³ for C_{MP}/C and 1.45 g·cm⁻³ for C_{PAN}/C). Subsequently, the preforms were subjected to HIP process assisted pitch impregnation and carbonization (impregnation temperature: 650–750 °C, impregnation pressure: 70–85 MPa), fol-

lowed by pyrolysis at 2300 °C, which further increased the density to approximately 1.85–2.0 g·cm⁻³[19]. Prior investigations have demonstrated that the graphitization significantly enhances the thermo-mechanical performance of C/C composites. Accordingly, both C/C composites were subjected to high-temperature graphitization at 3000 °C for 0.5 h.

Table 1 Main performance parameters of fibers

Carbonized fibers	Thermal conductivity/ W·m ⁻¹ ·K ⁻¹	Tensile strength/ GPa	Tensile modulus/ GPa	Elongation at break/%
MPCF	71.60	1.68	197.00	0.85
T700	9.40	4.90	235.20	2.10

2.2 Characterization

The morphology and microstructure of the composites were characterized using scanning electron microscopy (SEM, TESCAN MAIA3) and polarized light microscopy (PLM, BX-RLA2). Crack distribution was examined by high-resolution three-dimensional X-ray microscopy (Zeiss Xradia 515 Versa). Atomic-scale structures and defects were analyzed via transmission electron microscopy (FEI-Tecna F20). Phase composition, crystal structure, crystallinity, and crystallite parameters were investigated by X-ray diffraction (XRD, Rigaku SmartLab SE, $\lambda_{Cu} = 0.15406$ nm, 10°–60°, 10°·min⁻¹, step 0.02°). Defects, crystallinity, and amorphous carbon content were evaluated using Raman spectroscopy (WITec alpha300R, 532 nm). Surface elemental composition and chemical bonding states were determined by X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha). Tensile testing of individual carbon fibers was conducted on a fiber strength tester (XQ-1C) at 2 mm·min⁻¹.

2.3 Characterization and tests

2.3.1 Graphite microcrystals

The positions and shapes of XRD diffraction peaks reveal the lattice evolution of MPCF and C_{MP}/C composite during graphitization, enabling further analysis of how graphitization influences their microstructure and macroscopic properties. The interlayer spacing ($d_{(002)}$), crystallite dimensions (L_a and L_c), and degree of graphitization (g) were calculated using Eqs.

(1-4) to quantitatively characterize the graphitization extent and structural features.

$$d_{(002)} = \frac{\lambda}{2\sin\theta} \quad (1)$$

$$L_a = \frac{0.095}{d_{(002)} - 0.3354} \quad (2)$$

$$L_c = \frac{0.89\lambda}{\beta\cos\theta} \quad (3)$$

$$g = \frac{0.3440 - d_{(002)}}{0.3440 - 0.3354} \quad (4)$$

where θ is the diffraction angle of the (002) peak (°), λ is the X-ray wavelength (0.15406 nm), and β is the full width at half maximum (FWHM) of the (002) peak (rad).

2.3.2 Raman stress

In Raman spectroscopy, the shift of the G band is closely related to the in-plane stress. Based on this principle, the stress distribution in the material region can be obtained using the following equation^[21–22]:

$$\delta_{xx} = \frac{\Delta\omega E}{\omega_0\gamma(1-\nu)} = K\Delta\omega \quad (5)$$

where ω_0 is the original G -band position of the carbon material under stress-free conditions, γ is the Grüneisen parameter, ν is the in-plane Poisson's ratio, and E is the in-plane modulus.

2.3.3 Mechanical tests at elevated temperatures

To systematically evaluate the mechanical response of C/C composites under ultra-high-temperature conditions, tensile tests were conducted on C_{MP}/C and C_{PAN}/C composites using a high-temperature mechanical testing system (Fig. 1 and Fig. S1). This system comprises loading, temperature control, and strain measurement subsystems. The loading subsystem includes an electronic universal testing machine, an electrical heating system, water-cooled metal grips, and a high-strength loading head. Temperature was measured non-contact using an infrared pyrometer. Room-temperature tensile tests were performed on an electronic universal testing machine (MTS CMT6000) at a crosshead speed of 2 mm·min⁻¹. High-temperature tensile tests were conducted at 2500 and 2800 °C, with loading rate at 2 mm·min⁻¹. Using direct electric resistance self-heating, and the temperature was monitored in real time using an infrared pyrometry system to ensure uniformity and stability of the temperature

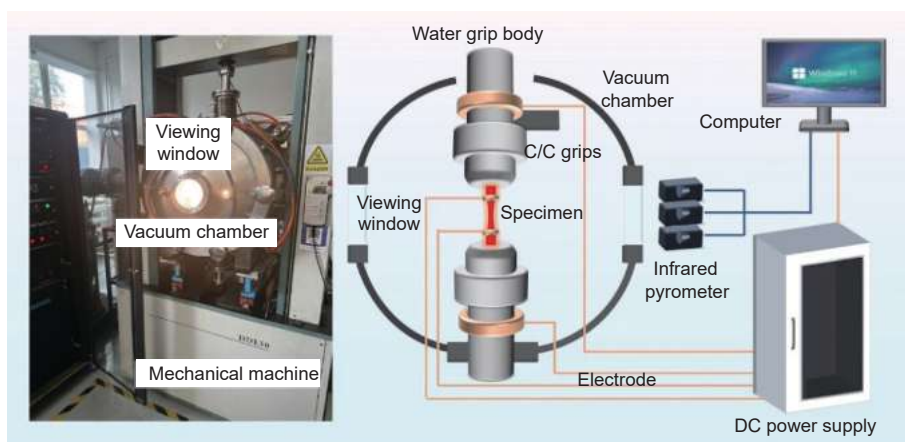


Fig. 1 Schematic diagram of high-temperature tensile process

distribution in the gauge section under an Ar atmosphere for protection. To minimize the adverse effects of performance variability inherent in C/C composites on the result analysis, three individual specimens from each group were tested.

3 Results and discussion

3.1 Structure and properties of carbon fibers

To comparatively analyze the structural differences of various reinforcing fibers after heat treatment at 3000 °C, the microstructures of the carbon fibers were characterized (Fig. 2). SEM cross-sectional morphology in Fig. 2a reveals that MPCF exhibits a highly developed “inner onion-skin and outer folded-radial” structure with graphitic layers highly oriented along the fiber axis, and the fiber surface displays pronounced groove-like features^[23]. Conversely, T700 retains a regular circular cross-section with a smaller diameter, with a comparatively smooth surface despite some grooves, likely related to the sizing agent and lacks the pronounced graphitic-layer structure seen in MPCF. XRD results further reveal the differences in graphitic crystallite development between the two fibers after 3000 °C treatment. As shown in Fig. 2b, T700 shows a distinct (002) diffraction peak, corresponding to a graphitization degree of ~ 47%, with in-plane crystallite size (L_a) and stacking height (L_c) of 20.68 and 9.10 nm, respectively. This indicates that although ultra-high temperature treatment promotes the transition from turbostratic carbon to an ordered layered structure, crystallite growth and inter-

layer stacking remain limited. In contrast, MPCF exhibits substantially larger L_a (129.25 nm) and L_c (34.75 nm) values under identical conditions, signifying larger graphitic crystallite dimensions, more regular interlayer stacking, and higher structural order. Raman spectroscopy was employed to evaluate the crystallite structure after graphitization (Fig. 2c). The spectrum of T700 exhibits characteristic D (~ 1358 cm^{-1}), G (~ 1597 cm^{-1}), A , and D' bands^[24–25]. The persistence of intense defect-related features after 3000 °C treatment indicates incomplete graphitization, consistent with the XRD results showing limited crystallite dimensions and graphitic ordering. Conversely, MPCF displays significantly weaker defect bands, reflecting a higher degree of structural ordering and more developed graphitic crystallites.

Fig. 2d presents the mechanical properties of MPCF and T700 after graphitization at 3000 °C. The tensile strength of T700 decreases from 4.90 to 2.13 GPa, whereas its tensile modulus increases from 235.20 to 360.81 GPa, indicating that ultra-high-temperature graphitization enhances structural stiffness at the expense of fiber strength. Conversely, MPCF exhibits a tensile strength of 3.05 GPa and a modulus of 594.25 GPa after identical treatment, both substantially exceeding the pre-graphitization values (1.68 and 197.00 GPa), demonstrating simultaneous improvement in strength and modulus. This disparity originates from their divergent precursor structures and crystallite evolution behaviors. For T700, graphitization promotes crystallite ordering, thereby increas-

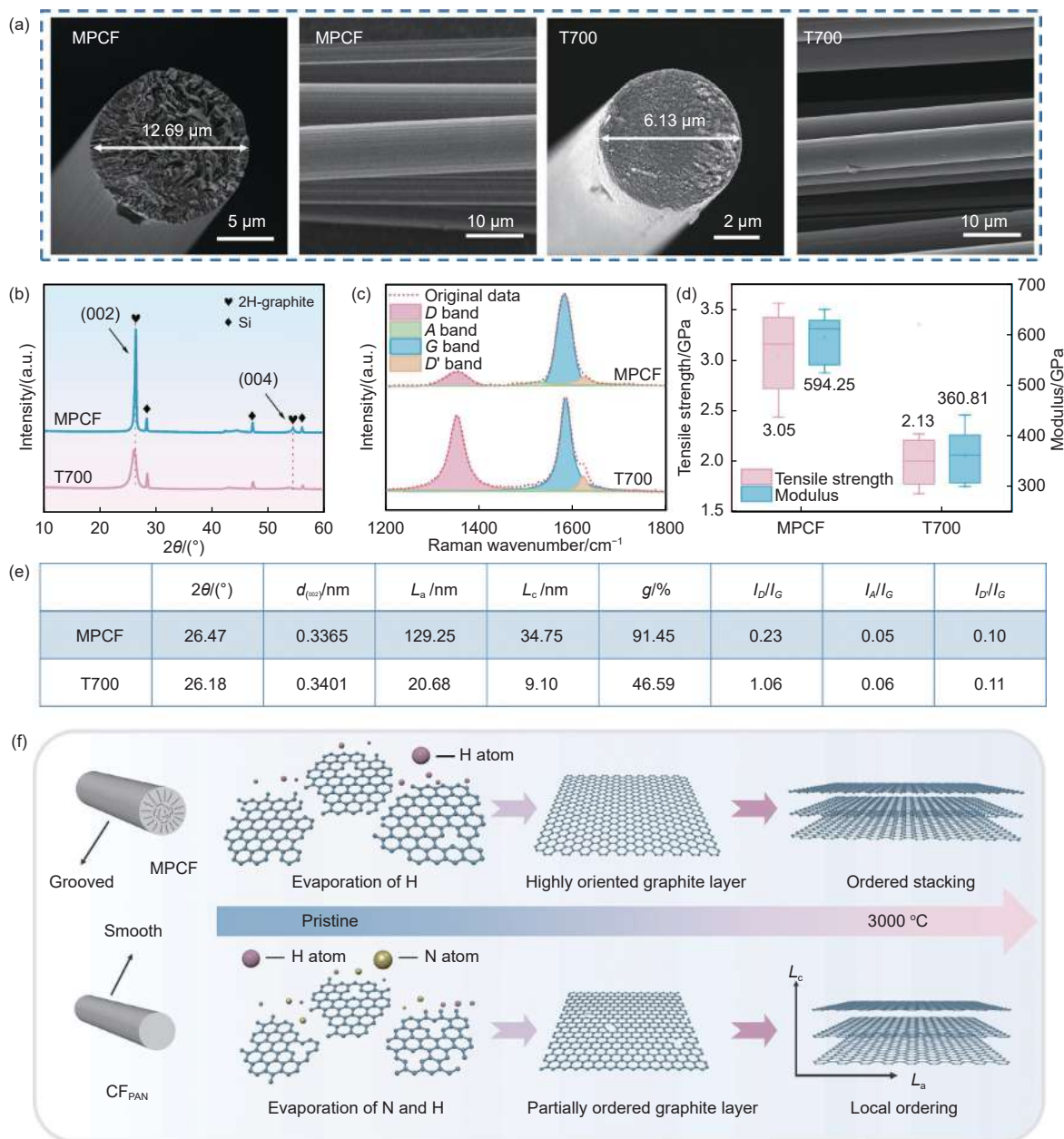


Fig. 2 Microstructure and properties of carbon fibers treated at 3000 $^\circ\text{C}$: (a) SEM, (b) XRD, (c) Raman, (d) strength and modulus, (e) crystallite parameters, and (f) mechanism of graphitization on crystallite development

ing the modulus. However, residual disordered structures and the skin-core architecture facilitate defect propagation and microcrack formation during high-temperature rearrangement, ultimately degrading tensile strength. By comparison, MPCF possesses higher initial orientation and superior graphitization potential, because it readily forms larger, highly oriented, and structurally complete graphitic crystallites, enhancing load-transfer efficiency along the fiber axis and concurrently improving both mechanical properties^[26–28].

T700 and MPCF undergo fundamentally distinct crystallite development pathways during graphitization (Fig. 2f), attributable to their different precursor structures, initial orientations, and microcrystal evolution mechanisms. Owing to the highly oriented aromatic layer structure of its mesophase precursor, MPCF follows an “inherited ordered evolution” pathway, wherein high-temperature treatment primarily promotes layer perfection and regular stacking via dehydrogenation, interlayer sliding, and coalescence. Consequently, MPCF readily develops highly oriented

graphitic crystallites with superior structural order. By contrast, T700 exhibits an orientationally dispersed turbostratic structure containing heteroatoms (N, H). Its crystallite development follows a “reconstructive growth” mechanism, requiring simultaneous heteroatom removal and carbon skeleton rearrangement^[29–30]. This extensive structural reorganization not only complicates microstructural reconstruction but also readily induces defects (including layer bending, dislocations, pores and microcracks), which ultimately compromise tensile strength^[31].

The HRTEM results in Fig. 3 further corroborate the above analysis. After heat treatment at 3000 °C, the internal graphitic layers of MPCF are highly ordered, exhibiting a continuous and regular layered

stacking structure without observable lattice defects. In Region A, the lattice fringes are straight, uniformly aligned and highly oriented, with an interlayer spacing of approximately 0.3388 nm, close to that of the typical graphite (002) plane. The corresponding selected area electron diffraction displays sharp spot-like diffraction patterns, indicating regular stacking of graphitic layers along the fiber axis and the formation of a relatively complete hexagonal graphite lattice structure. Geometric phase analysis (GPA) strain mapping further reveals uniform ε_{xx} and ε_{yy} distributions with only minor local fluctuations; the ε_{xy} shear strain remains weak, indicating minimal lattice distortion and a stable local strain field. This highly ordered, low-distortion microstructure facilitates effi-

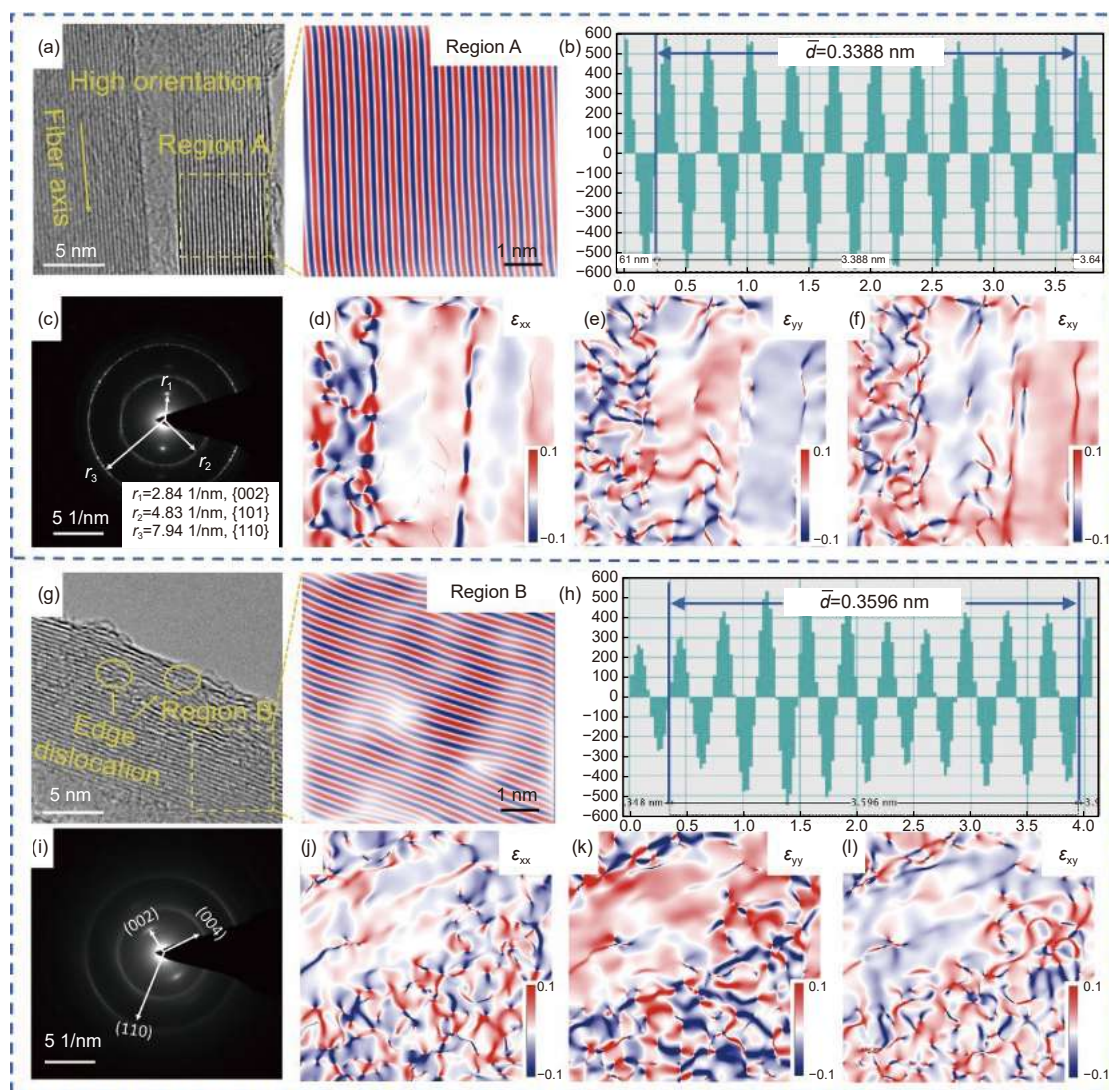


Fig. 3 TEM images of carbon fibers after graphitization at 3000 °C. For MPCF: (a) HRTEM image, (b) interlayer spacing, (c) SAED pattern, and (d–f) lattice distortion. For T700: (g) HRTEM image, (h) interlayer spacing, (i) SAED pattern, and (j–l) lattice distortion

cient load transfer along the fiber axis, underpinning the superior strength and modulus of graphitized MP-CF. By contrast, T700 develops only limited graphitic layering after 3000 °C treatment, with its layers exhibiting pronounced bending, misalignment, and edge dislocations—indicative of insufficient crystallite development. In Region B, lattice fringes are discernible yet exhibit substantially weaker continuity and uniformity compared with MPCF, with ordering confined to local domains. The interlayer spacing measures ~ 0.3596 nm, substantially exceeding that of ideal graphite (002), consistent with a lower graphitization degree. The corresponding FFT pattern displays diffuse spots, reflecting dispersed crystal orientation and insufficient structural order. GPA analysis reveals pronounced spatial fluctuations in the ε_{xx} and ε_{yy} distributions, with alternating local tensile and compressive strains. The ε_{xy} strain is markedly enhanced, particularly near dislocations and bent layers, evidencing severe local lattice distortion and stress concentration. These microstructural disparities dictate divergent load-bearing behavior. In T700, residual layer bending, dislocations, and local disorder generate pronounced lattice distortion and strain concentration. This microstructural inhomogeneity promotes crack initiation and propagation, ultimately accounting for the inferior mechanical performance of T700 and its composites relative to MPCF.

3.2 Microstructure of C/C composites

Fig. 4 presents the microstructure of C/C composites after 3000 °C graphitization. Both composites exhibit a typical dual-phase architecture comprising PyC surrounding the fibers and pitch carbon infiltrating the interbundle regions. Under polarized light, the pitch carbon displays optical anisotropy with a streamline texture, whereas the PyC region appears relatively uniform. Notably, pronounced matrix cracks are evident, particularly within the pitch carbon, suggesting divergent shrinkage behaviors among the carbon phases during carbonization and graphitization that induce local stress concentration and thermal expansion mismatch. SEM images (Fig. 4b) reveal that cracks in the C_{MP}/C composite are predominantly con-

fined to the matrix, while the PyC/fiber interface remains relatively intact. This is attributed to the rough, groove-like surface of MPCF, which enhances mechanical interlocking and thereby inhibits continuous interfacial crack propagation^[32]. In contrast, cracks in the C_{PAN}/C composite propagate not only through the matrix but also extensively along fiber bundles and around individual fibers, with some regions exhibiting a penetration tendency. These observations highlight distinct interfacial bonding states and structural continuity between the 2 composites, which may differentially influence their high-temperature mechanical response and thermal stability. Raman spectroscopy further characterizes the structural ordering of individual carbon phases (Fig. 4c). The spectral profiles differ markedly among the phases, reflecting variations in structural order. MPCF exhibits the highest degree of graphitic order ($R = 0.25$), followed by pitch carbon and PyC, whereas T700 shows the lowest ($R = 1.04$), establishing a pronounced structural gradient across the composite constituents. The lower R value of PyC in the C_{MP}/C composite is attributed to the templating effect of highly oriented MPCF on PyC deposition during CVI^[33–34]. XRD patterns (Fig. 4d) further differentiate the graphitic crystallite structures. Both composites exhibit a distinct (002) diffraction peak near $2\theta \approx 26.4^\circ$, confirming the development of graphitic structures. However, the C_{MP}/C composite displays a sharper (002) peak, indicating more complete crystallite development and regular interlayer stacking. Conversely, the broader (002) peak of C_{PAN}/C suggests increased stacking defects and limited graphitization. Moreover, both the (002) and (004) peaks of C_{MP}/C shift to higher angles with increased intensity and narrowing, while its graphitization degree increases from 81.87% to 86.38%, exceeding that of C_{PAN}/C .

The XPS survey was further employed to characterize the carbon bonding states of the 2 composites, as depicted in Fig. 4e. C_{MP}/C exhibits a higher sp^2 carbon content and more pronounced $\pi-\pi^*$ shake-up features than C_{PAN}/C , with concomitantly lower sp^3 content. This indicates more complete transformation of

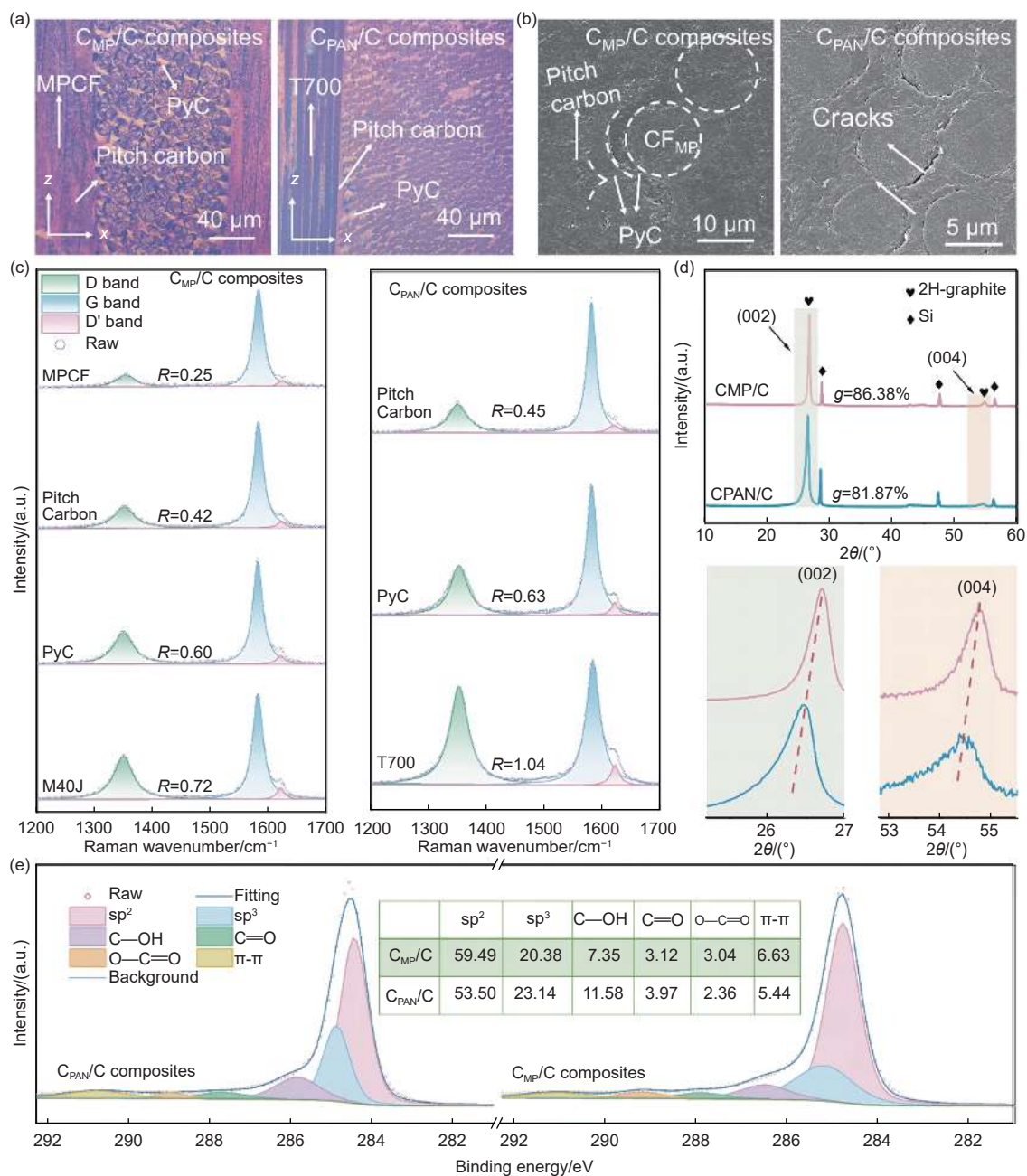


Fig. 4 Microstructure of C/C composites after treatment at 3000 °C: (a) PLM images, (b) SEM images, (c) Raman spectra, (d) XRD patterns, (e) XPS spectra

disordered sp^3 carbon into ordered sp^2 graphitic networks in C_{MP}/C . The enhanced π - π^* interaction further reflects greater electron delocalization and superior structural order. Notably, residual sp^3 carbon at graphitic layer edges and defects constitute preferential active sites for oxidation, facilitating the formation of oxygen-containing functionalities ($-OH$, $C=O$, $-COOH$). In summary, both composites develop multiphase carbon architectures after 3000 °C graphitization. However, C_{MP}/C exhibits superior graphitization, structural continuity, and interfacial

stability, whereas C_{PAN}/C retains more defects, crack pathways, and interfacial discontinuities. These microstructural differences govern the mechanical response and failure behavior of the 2 composites in ultra-high-temperature environments.

3.3 Mechanical behaviors of C/C composites at ultra-high temperatures

Tensile tests were conducted on C_{PAN}/C and C_{MP}/C composites at 2500 and 2800 °C to assess their ultra-high-temperature mechanical behaviors. Detailed procedures and temperature stability data are

given in Supporting Material (Fig. S2). Fig. 5a shows distinctly different fracture morphologies between room temperature (RT) and 2800 °C. At RT, C_{MP}/C exhibits crack deflection and branching along the fiber/matrix interface, producing a stepped, rough fracture surface with limited fiber pullout, indicating that moderate interfacial bonding promotes crack deflection and energy dissipation. At 2800 °C, increased ordering of pitch carbon and MPCF structures alters interfacial bonding; cracks tend to penetrate the interface, causing concurrent fiber and matrix fracture and a flatter surface with a more concentrated fracture zone. In contrast, C_{PAN}/C at RT shows extensive long-bundle pullout, suggesting crack propagation mainly along the weak fiber/matrix interface. At 2800 °C, the damage zone near the fracture surface expands markedly, with shorter and more dispersed fiber bundle ends, implying greater matrix cracking, interfacial debonding, and localized damage growth under high-temperature tension. The stress-strain curves (Fig. 5b) reflect temperature-dependent mechanical response. At RT, both composites have steep initial slopes (stiffness-dominated). At 2500 °C, slope and peak stress decrease, but curves remain nearly linear, indicating persistent stiffness-dominated behavior despite strength loss. At 2800 °C, pronounced nonlinearity, lower peak stress, and higher failure strain appear, showing reduced load capacity yet enhanced deformability. This marks a transition from stiffness-controlled failure to damage accumulation via fiber fracture, interfacial sliding, and crack growth. Compared with C_{PAN}/C , C_{MP}/C maintains higher stress levels and more stable deformation at elevated temperatures, demonstrating superior load transfer and structural stability.

Fig. 5c further validates the above analysis. For the C_{MP}/C composite, the tensile strengths at room temperature, 2500 and 2800 °C are 236.7 ± 13.1 , 186.6 ± 12.5 and 160.9 ± 13.7 MPa, with corresponding moduli of 97.8 ± 11.0 , 75.7 ± 9.2 and 43.1 ± 5.2 GPa, respectively. The strength retention rates at 2500 and 2800 °C are 79% and 68%, and the modulus retention rates are 77% and 44%. By comparison, the C_{PAN}/C

composite exhibits much lower tensile strengths (119.6 ± 7.8 and 103.7 ± 9.3 MPa) and moduli (48.0 ± 9.1 and 16.8 ± 4.3 GPa) at 2500 °C and 2800 °C respectively, indicating that C_{MP}/C outperforms other publicly reported C/C composites (Table S1). These results indicate that although both composites degrade in strength and modulus at ultra-high temperatures, C_{MP}/C shows a smaller reduction, suggesting superior structural stability. This is attributed to the higher graphitization degree and axial orientation of MPCF, enabling effective load transfer along the continuous ordered graphitic structure even at elevated temperatures. Meanwhile, the interface retains partial cooperative load-bearing capability, thereby delaying unstable crack propagation. Conversely, the T700 fibers and their interfacial structure in C_{PAN}/C are more prone to high-temperature degradation, leading to reduced load-transfer efficiency and exacerbated modulus decay. Fig. 5d reveals distinct internal damage characteristics in the two composites after tensile testing at 2800 °C. In C_{MP}/C , cracks primarily propagate along interlaminar regions and between fiber bundles with limited penetration depth, although some eventually penetrate into bundles and cause fiber fracture. This indicates a combined failure mode involving interfacial cracking, crack deflection, and fiber fracture. In contrast, C_{PAN}/C exhibits preferential crack propagation along interlaminar regions and bundle interfaces, accompanied by longer fiber pullout and localized through-cracks, suggesting interface-dominated damage propagation. Quantitative analysis of the XRM datasets was further performed (Figs. S3, S4 and Table S2). The crack density is 2.15 mm^{-1} for C_{PAN}/C , and the crack orientation distribution histograms reveal that more than 80% of the cracks in C_{PAN}/C are oriented along the fiber-bundle direction, forming a percolating interfacial crack network, whereas the cracks in C_{MP}/C are short and isolated, mainly confined within the matrix regions without a preferred percolation direction. Fig. 5e shows that the R value of C_{MP}/C minimizes at 2800 °C, reflecting intense in-situ graphitization of MPCF and a substantial reduction in defect density. The enhanced graphitization

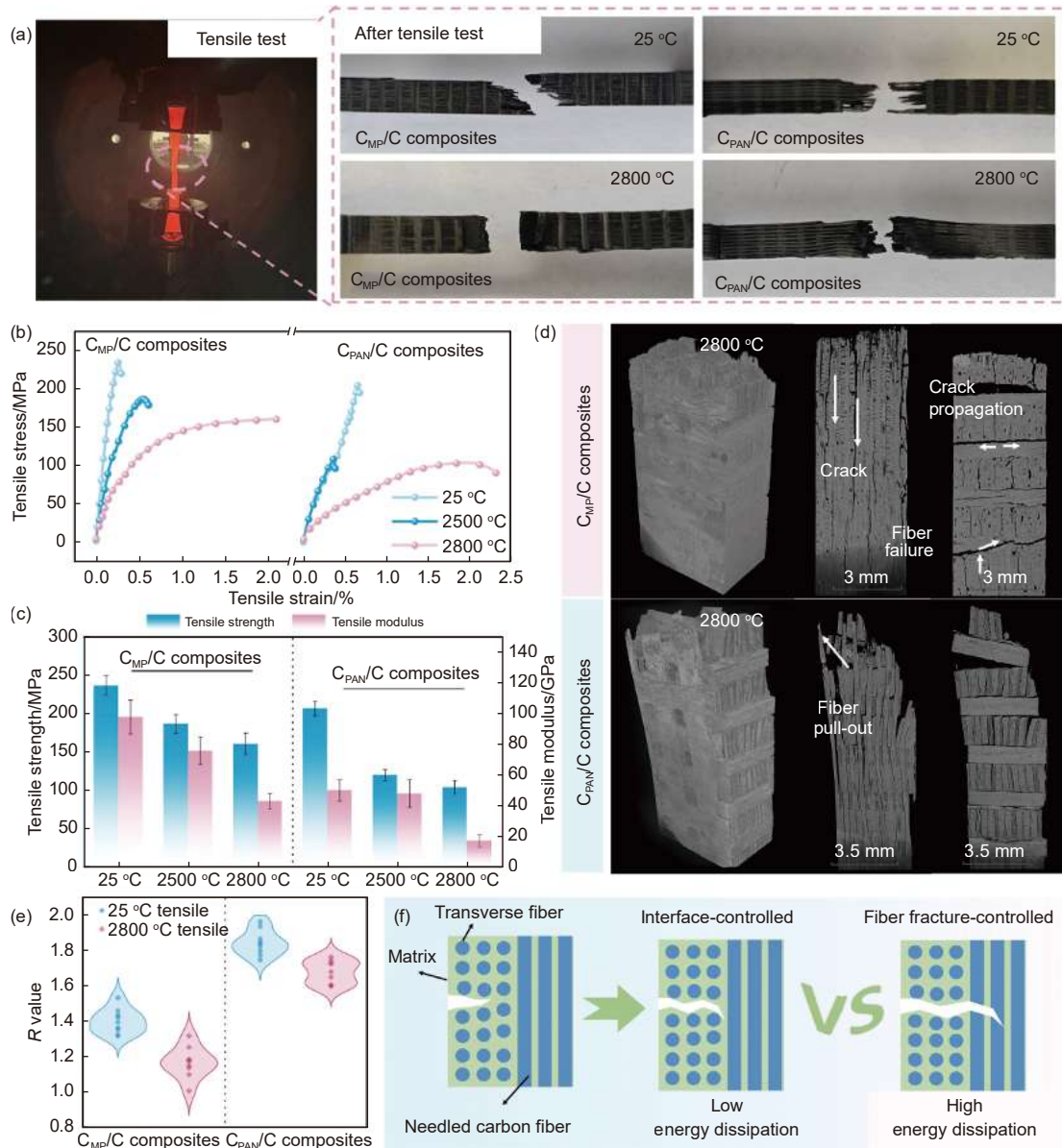


Fig. 5 Mechanical behaviors of C/C composites: (a) macroscopic fracture morphology, (b) stress-strain curve, (c) tensile strength and modulus, (d) crack propagation path, (e) R -value distribution on the fractured fiber cross-section, and (f) failure mode

amplifies the thermal expansion mismatch between fibers and matrix, thereby strengthening interfacial constraint^[20]. Consequently, cracks cannot effectively deflect at the interface during propagation, instead of penetrating directly into fiber bundles and inducing fiber fracture. Fig. 5f schematically illustrates the contrasting failure mechanisms. C_{PAN}/C fails via crack initiation and propagation along weak interfaces between transverse fibers, needled fibers, and the matrix, exhibiting predominantly interface-dominated behavior; although C_{MP}/C contains interconnected pores and interfacial weak zones, crack propagation involves ex-

tensive fiber fracture and path deflection, enabling a high-energy-dissipation failure mode involving MP-CF fracture.

Fig. 6a presents the fracture surface morphology after 2800 °C tensile testing. The C_{MP}/C surface appears coarser with carbon microsphere deposition; however, the layered fiber bundle architecture remains discernible. High-magnification images reveal fine granular deposits ($\sim 11 \mu\text{m}$). By contrast, C_{PAN}/C exhibits large-scale agglomerated carbon microspheres ($\sim 163 \mu\text{m}$), showing the secondary deposition characteristics. Considering the previous micro-

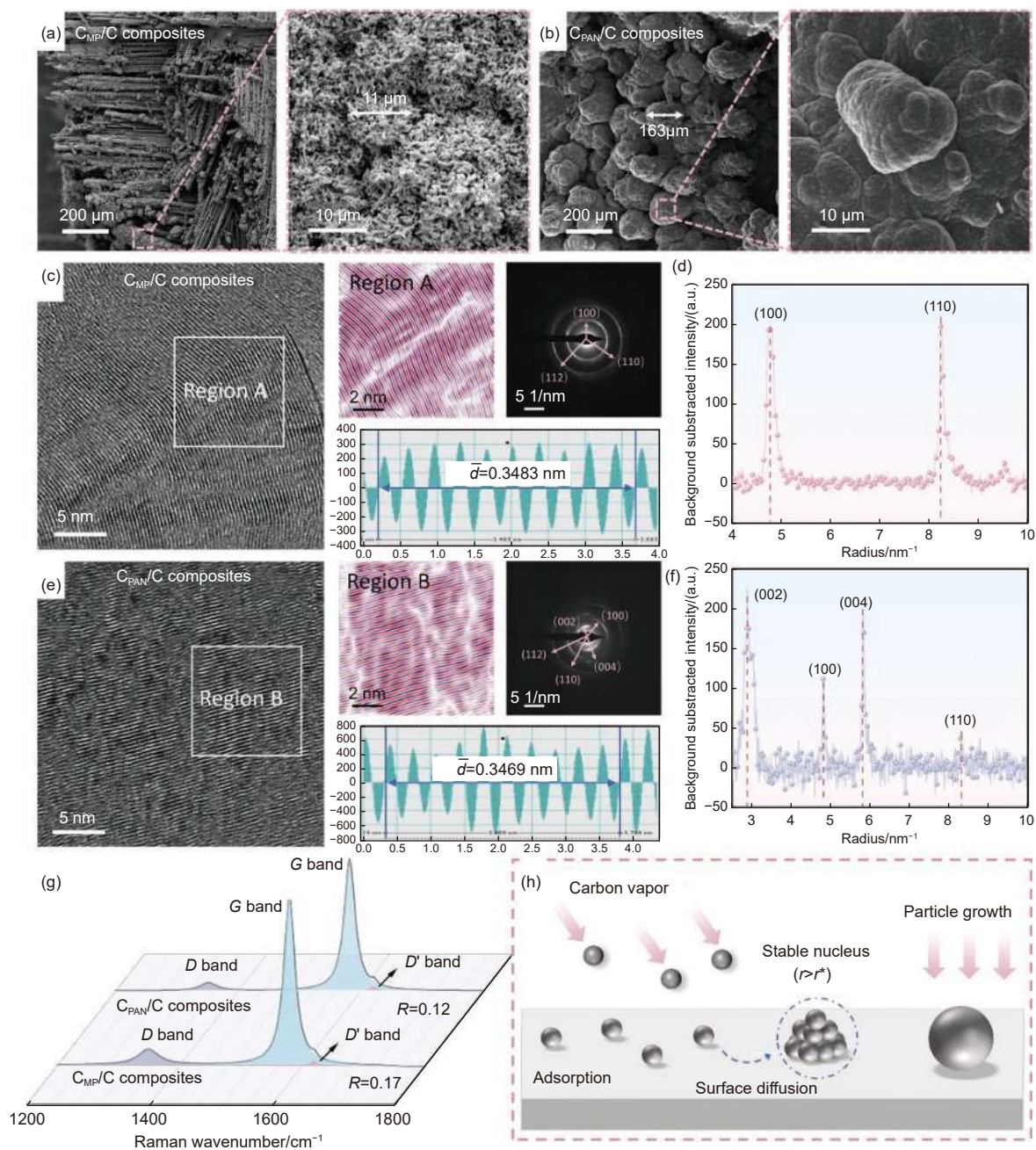


Fig. 6 Microstructure of C/C composites: (a) fracture morphology, (b) fracture morphology, (c) HRTEM image of surface carbon microspheres, (d) integral curve of C_{MP}/C composite; (e) HRTEM image of surface carbon microspheres, (f) integral curve of C_{PAN}/C composite; (g) Raman spectra of carbon microspheres; (h) deposition process of carbon microspheres

structural analysis, they mainly contain three carbon phases: carbon fibers, PyC, and pitch carbon. Among these, the matrix carbon near the fracture, particularly the PyC and pitch carbon regions with poor continuity and more defects around cracks, is more susceptible to exposure to high temperature and stress concentration during tensile testing at 2800 °C, leading to preferential sublimation. Moreover, the C_{PAN}/C composite originally contains the crack network in the

matrix, providing favorable conditions for the generation, migration, and local enrichment of gaseous carbon species. Further SEM observations of the fracture and tail regions of C_{PAN}/C composite (Figs. S5 and S6) and C_{MP}/C composite (Figs. S7 and S8) show that relatively intact fiber bundles and regular fiber cross-sections are still clearly observable on the tail surface, indicating that carbon fibers are not the primary source of initial gaseous carbon and instead mainly serve as

deposition substrates. Consequently, the spherical deposits likely originate from preferential matrix carbon sublimation, migration, and redeposition within the fracture zone. This suggests that failure during 2800 °C tensile testing involves not only fiber fracture, interfacial damage, and matrix cracking, but also matrix carbon sublimation-redeposition.

To further clarify the failure mechanism, carbon microspheres deposited near the fracture surface were selectively extracted by FIB and characterized by TEM. The microspheres on both composites exhibit distinct graphitic layering (Fig. 6c-f), albeit with differing crystallite development. In C_{MP}/C , the lattice fringes are relatively straight and locally oriented, with an interlayer spacing of ~ 0.3483 nm in Region A. The corresponding FFT pattern shows (100) and (110) diffraction spots, indicating preferred orientation of the graphitic layers. By contrast, the microspheres on C_{PAN}/C exhibit a more distinct internal graphitic structure with superior lattice-fringe continuity. The interlayer spacing in Region B is ~ 0.3469 nm, approaching the ideal graphite (002) spacing. The FFT pattern additionally reveals (002) and (004) reflections alongside the (100) and (110) spots, indicating more complete interlayer stacking along the c-axis. Moreover, the radially integrated intensity shows more pronounced diffraction peaks, suggesting a more complete graphitic lattice and higher graphitization degree. Raman spectra (Fig. 6g) corroborate this assessment. Both *D* and *G* bands are evident, with the *G* band predominant, indicating well-developed sp^2 graphitic layers. The persistent *D* band indicates residual edge defects and disordered structures. Notably, the microspheres on C_{PAN}/C exhibit slightly higher structural order than those on C_{MP}/C . Collectively, these results indicate that the carbon microspheres constitute a redeposited phase with relatively high graphitization, while retaining some defects and turbostratic characteristics. Fig. 6h illustrates the formation mechanism of surface carbon microspheres. During 2800 °C tensile testing, local sublimation of either carbon fibers or matrix carbon generates gaseous carbon species that migrate to low-energy re-

gions and adsorb at matrix surfaces, defect sites, and interfaces. Adsorbed carbon atoms aggregate via surface diffusion and nucleate heterogeneously once the critical nucleus size is exceeded. Continued carbon deposition drives particle growth via Ostwald ripening, wherein smaller particles coalesce into larger ones^[35]. Surface energy minimization drives the redeposited carbon to evolve into regularly shaped microspheres^[36–37]. This sublimation-migration-redeposition sequence constitutes the primary mechanism of carbon microsphere formation during high-temperature tensile failure.

3.4 Interfacial analysis of failed C/C composites

Raman mapping was performed on the regions near the fracture surface and the tail section to visualize the residual stress distribution around the fiber/matrix interface. As shown in Fig. 7a, the two composites exhibit markedly different local stress states between the fracture and tail regions. For the C_{MP}/C composite, the tail region displays typical confined fiber stress characteristics. Specifically, the Raman peaks within the fibers shift to higher wavenumbers (blue shift), signifying a marked concentration of compressive stress, whereas the adjacent matrix carbon exhibits a progressive red shift, indicative of a low-magnitude tensile or transitional stress^[21]. The fiber, interface, and matrix zones are clearly distinguishable, indicating relatively intact interfacial bonding in this region and effective constraint of the fiber by the matrix. Conversely, the stress field near the fracture surface becomes more discrete; the originally continuous compressive stress concentration is weakened and locally transformed into tensile stress or irregular low-stress distribution, suggesting local stress release and redistribution near the interface after ultra-high-temperature tensile testing. However, no large-scale abnormally high stress zone extending continuously from the fiber to the matrix is formed near the fracture surface of C_{MP}/C , indicating that the good thermophysical compatibility between MPCF and the matrix carbon is conducive to suppressing the propagation of interface mismatch stress. For C_{PAN}/C composite, it exhibits less uniform stress distribution

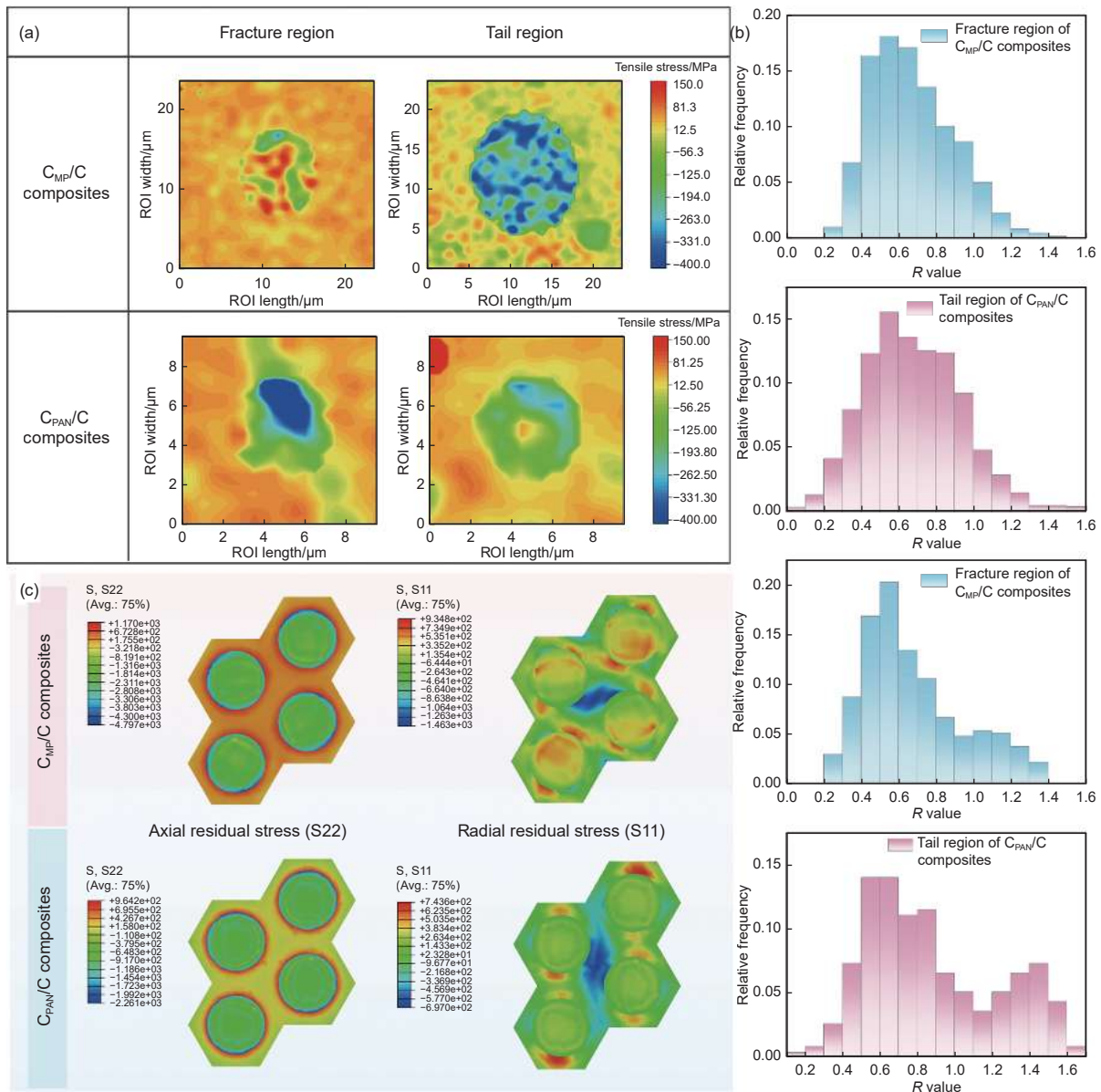


Fig. 7 Analysis of the interfacial state of C/C composites: (a) distribution of interfacial residual stress, (b) R-value distribution, (c) finite element simulation results of the residual stress

in the tail section. While the fiber, interface, and matrix remain distinguishable, stress fluctuations near the interface indicate a pronounced thermomechanical mismatch between T700 and the matrix. Near the fracture surface, this mismatch intensifies, generating a compressive stress concentration zone within the fiber that extends into the adjacent matrix and forms a broad high-stress band. This indicates stress perturbation propagation from the fiber across the interface. Furthermore, the high-stress zone displays irregular, asymmetric distribution, suggesting the stress originates not only from confined fiber stress but also from

interfacial debonding, microcrack propagation, and local matrix degradation. Overall, the greater thermal expansion anisotropy of MPCF raises interfacial residual stress, but its highly oriented graphitic structure permits slight slippage to dissipate radial stress and provides a continuous path for stress transfer. Consequently, C_{MP}/C shows a more continuous interfacial stress distribution with smaller local gradients. Fig. 7b highlights the contrasting structural uniformity near the interface. C_{MP}/C exhibits a narrow R-value distribution in both the fracture and tail regions, indicating stable defect distribution and preserved interfacial

continuity after high-temperature tension. Conversely, C_{PAN}/C displays a broader distribution, particularly in the tail region, with elevated R -values reflecting pronounced defect fluctuations and greater local heterogeneity. This is consistent with the interfacial stress concentration and weaker bonding observed in C_{PAN}/C .

To reveal the spatial distribution of residual thermal stress in C_{PAN}/C and address the limitation of surface-sensitive Raman measurements, a mesoscale fiber-bundle/matrix phase-separation model was established (Fig. S9) to simulate local stress/strain evolution during high-temperature tension. Fig. 7c depicts the axial and radial residual stress distributions. In C_{MP}/C , both stress components concentrate at the fiber periphery and interface, decaying gradually into the matrix. The axial residual stress forms a continuous annular concentration zone around the fiber, indicating that axial deformation mismatch between MPCF and matrix carbon during post-tension unloading and cooling drives interfacial stress accumulation. Radial residual stress also concentrates at the interface and in inter-fiber matrix regions, revealing that interfacial constraint restricts not only axial contraction but also radial deformation compatibility. The high graphitization degree and strong anisotropy of MPCF yield a low axial thermal expansion coefficient, whereas the matrix carbon contracts isotropically during cooling, generating a pronounced thermal contraction mismatch. Moreover, the high elastic modulus of MPCF sustains strong interfacial constraint during unloading and cooling, producing elevated residual stress near the C_{MP}/C interface. These results indicate that despite good interfacial integrity after high-temperature tension, the interface remains a critical locus for thermomechanical mismatch energy accumulation and stress transfer.

By contrast, residual stress in C_{PAN}/C also concentrates at the fiber/matrix interface and narrow inter-fiber matrix regions, yet its distribution exhibits stronger local heterogeneity. Axial residual stress concentrates near the interface, indicating significant displacement mismatch between T700 fibers and the matrix during cooling. Radial residual stress is pro-

nounced at the fiber periphery and in inter-fiber matrix regions, particularly between adjacent fibers, suggesting that interfacial constraint and geometric confinement jointly amplify local stress concentration. This behavior is attributed to the lower graphitization degree, higher defect density, and lower thermal expansion anisotropy of T700 fibers. During cooling, deformation mismatch generates significant interfacial residual stress, which is further intensified by local matrix heterogeneity. Combined with the Raman stress results, the finite element simulation reveals a similar distribution of stress concentration, indicating that residual stress is governed primarily by fiber/matrix thermophysical mismatch and interfacial constraint. The following assumptions were adopted in the finite element model: (i) the fibers and matrix phases are treated as linearly elastic; (ii) the fiber/matrix interface is perfectly bonded, without debonding or damage initiation; (iii) crack propagation and stress relaxation are not considered; (iv) the cooling process is quasi-static; and (v) the phase properties are approximated as temperature-independent. The model therefore represents an elastic upper bound of the residual stress distribution and is used to identify the locations and trends of stress concentration rather than the absolute post-fracture stress level. Accordingly, the simulated values are systematically higher than the Raman-derived values, because Raman measurements capture partially relaxed stresses after fracture and interfacial damage, whereas the model corresponds to the intact state without accounting for these stress-relief processes. The two approaches are thus compared only in terms of the qualitative consistency of stress-concentration locations and distribution trends, rather than being regarded as mutual quantitative validation.

3.5 Tensile failure mechanisms of C/C composites at ultra-high temperatures

Based on the foregoing analysis, we propose that at room temperature, the intrinsic mechanical properties of the fibers dictate the upper limit of the mechanical performance of C/C composites, constituting the primary source of the strength disparity between the

two material systems. At 2500 °C, the efficiency of interfacial load transfer governs the utilization rate of fiber strength, and the divergence in mechanical properties is predominantly attributable to differences in the residual stress state at the interface and the load transfer efficiency. At 2800 °C, the superimposed damage arising from sublimation and redeposition of the matrix carbon emerges as a critical factor responsible for the degradation in mechanical performance. Moreover, independent evidence from two complementary aspects, namely the interfacial residual stress distribution and the fracture morphology, corroborates that the disparity in intrinsic fiber properties fun-

damentally determines the gap in strength upper limits, whereas interfacial factors govern the difference in strength retention at elevated temperatures. Both contributions are of comparable significance and cannot be overlooked.

Fig. 8 illustrates that the tensile failure of C/C composites at ultra-high temperatures results from the coupled effects of interfacial thermal mismatch, crack propagation, localized carbon sublimation, possible redeposition, and degradation of structural integrity. During tensile loading, the combined effects of interfacial thermal mismatch and the applied tensile stress promote crack initiation and propagation, exposing

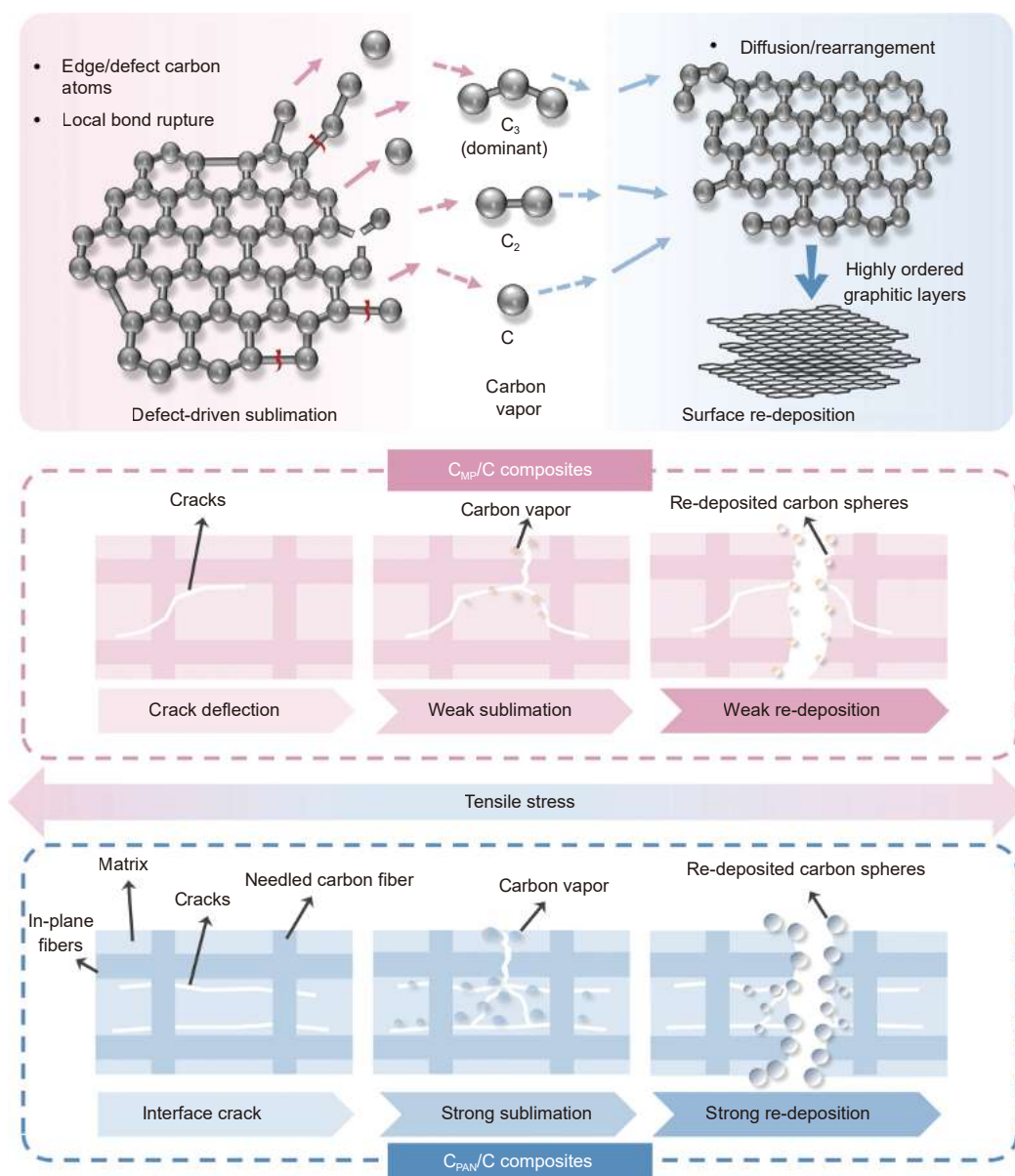


Fig. 8 Schematic diagram of the failure mechanism of C/C composites under high-temperature tensile

defect-rich matrix regions and newly generated fracture surfaces. Gas release from crack tips and pores reduces the local pressure, thereby lowering the temperature threshold for carbon sublimation. Theoretical calculations under vacuum conditions indicate that significant carbon sublimation occurs at approximately 2700 °C, close to the 2800 °C testing temperature employed in this study, providing thermodynamic support for localized sublimation within crack regions^[38–39]. Under these conditions, carbon atoms at edge sites and structural defects preferentially sublime as gaseous C_3 species, accompanied by smaller amounts of C_2 and C_1 species^[40–41]. These gaseous carbon species may subsequently redeposit on relatively cooler crack surfaces, gradually forming graphitic redeposition products. Meanwhile, continuous matrix sublimation progressively degrades the local structural integrity and facilitates crack propagation and coalescence, establishing a positive feedback process of “crack propagation-carbon sublimation-possible redeposition-structural degradation”, which ultimately accelerates the tensile failure of C/C composites under ultra-high-temperature conditions.

The performance disparity between C_{MP}/C and C_{PAN}/C stems primarily from divergent crack propagation paths and their consequences for load transfer. In C_{MP}/C , cracks tend to deflect along tortuous paths and induce needled-fiber fracture, impeding the formation of a continuous interfacial crack network. Consequently, localized sublimation-redeposition is relatively limited, and the interface retains partial load-transfer capacity prior to failure, yielding a higher high-temperature tensile strength retention. For comparison, cracks in C_{PAN}/C propagate readily along the fiber/matrix interface, forming a well-developed interfacial crack network that rapidly degrades interfacial load-transfer capacity. Meanwhile, increased matrix carbon exposure to the high-temperature environment promotes carbon sublimation and establishes a local carbon-rich atmosphere, inducing more pronounced redeposition. Thus, larger and more abundant carbon microspheres form near the fracture surface, reducing high-temperature strength retention. In summary, the

key to the performance difference lies not only in interfacial thermal mismatch stress but also in crack propagation mode, local carbon sublimation, and redeposition behavior. These factors collectively govern damage evolution and load-bearing capacity retention under extreme temperatures.

4 Conclusions

This study investigates the mechanical behavior and failure mechanisms of C_{MP}/C and C_{PAN}/C composites under ultra-high temperature conditions, revealing the differences in tensile mechanical behavior, damage characteristics, and failure modes between the two materials. The specific conclusions are as follows:

(1) After tensile testing at 2800 °C, C_{PAN}/C exhibited abundant spherical carbon deposits (up to ~ 163 μm), whereas C_{MP}/C showed only limited granular deposits (~ 11 μm). The more severe carbon deposition in C_{PAN}/C is closely associated with the formation of continuous interfacial crack networks. Based on the microstructural observations and previous studies, the deposited carbon is likely related to carbon sublimation and subsequent vapor-phase redeposition under ultra-high-temperature conditions.

(2) C_{MP}/C retains 79% and 68% of room-temperature tensile strength at 2500 and 2800 °C, outperforming C_{PAN}/C (~ 58% and 50%), owing to the higher graphitization degree and axially oriented crystalline structure of MPCF versus the lower graphitization and weaker bonding of T700, indicating fiber structure as the dominant factor governing strength retention.

(3) C_{PAN}/C fails via interface-dominated crack propagation with extensive fiber pullout and massive redeposition. Conversely, C_{MP}/C undergoes high-energy-dissipation fracture via crack deflection and cooperative fiber-matrix fracture, impeding continuous interfacial damage.

(4) Despite the greater thermal-expansion anisotropy of MPCF, its highly oriented graphitic structure enables stress relief via interlayer slippage and continuous axial stress transfer, yielding stable interfacial stresses in C_{MP}/C . In contrast, the thermal expansion

mismatch between T700 and the matrix, combined with its high defect density, causes concentrated interfacial stresses in C_{PAN}/C , showing that interfacial stress state, not magnitude alone, governs crack initiation.

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