

Revealing the influence of composition and interface on the mechanical and dielectric properties of Si_3N_4 fiber-reinforced ceramic composites

Yingpeng Zhang, Zhilin Tian✉, Bin Li✉

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ABSTRACT: Si_3N_4 fiber-reinforced ceramic composites are promising candidates for high-temperature wave-transparent applications, yet the relationships among composition, interfacial characteristics, and mechanical performance remain inadequately understood. This study designs three kinds of composites, $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$, and systematically investigates their performances. The results reveal that the reaction between the BN precursors and silanol groups in the SiO_2 matrix during the fabrication of $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ enhances the chemical compatibility between the fiber and matrix, promoting elemental interdiffusion and forming a thicker interfacial diffusion region. Consequently, the interfacial shear strength of $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ is 1.86 and 2.35 times that of $\text{Si}_3\text{N}_4/\text{BN}$ and $\text{Si}_3\text{N}_4/\text{SiO}_2$, respectively. The stronger fiber-matrix bonding in $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ suppresses fiber pull-out, whereas the weaker bonding in the other two composites permits it. $\text{Si}_3\text{N}_4/\text{BN}$ primarily exhibits fiber bundle pull-out, whereas $\text{Si}_3\text{N}_4/\text{SiO}_2$ shows long single-fiber pull-out, indicating improved damage tolerance. In contrast, $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ displays a typical brittle fracture behavior with minimal fiber pull-out and degraded mechanical properties. The excessive interfacial bonding, together with the thermal residual stress arising from the thermal expansion mismatch between the Si_3N_4 fiber and the matrix, degrades the flexural and compressive strengths. Moreover, this excessive bonding restricts interfacial debonding and fiber pull-out, leading to a brittle fracture mode. Despite differences in interfacial microstructure, all three composites exhibit good dielectric properties. The use of SiO_2 and BN matrices effectively reduces both the dielectric constant and the dielectric loss tangent of Si_3N_4 fiber-reinforced ceramic composites. These findings provide valuable insights into the design of high-temperature wave-transparent composites operated in extreme environments.

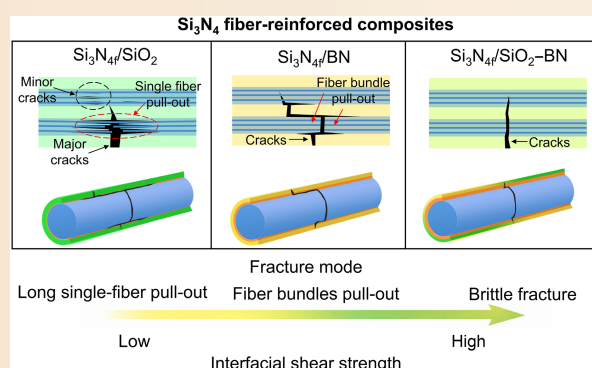
KEYWORDS: Si_3N_4 fibers; ceramic composites; composition; interface; dielectric properties

1 Introduction

High-temperature wave-transparent composites are critical for the development of radomes and antenna windows in hypersonic vehicles, enabling stable communication under extreme flight conditions [1]. Aerodynamic heating induced by high-speed flight presents significant challenges to the thermal and structural integrity of wave-transparent composites [2]. To perform reliably in such harsh environments, these materials must exhibit a low dielectric constant, a low dielectric loss tangent, high mechanical strength, and excellent thermal stability [3,4]. As a result, the development of advanced wave-transparent composites has become a major focus in current materials research.

The evolution of wave-transparent materials has gone through several generations. Polymer and bulk ceramic wave-transparent

materials are insufficient for high-temperature applications due to their low decomposition temperature and poor damage tolerance [5–12]. To overcome these limitations, ceramic fiber-reinforced ceramic matrix composites (CMCs) have been extensively studied [6,13,14]. The fibers play a pivotal role in enhancing the mechanical strength and damage tolerance of the composites [15]. However, the stringent requirements for a low dielectric constant and a low dielectric loss tangent in wave-transparent ceramics limit the choice of suitable fiber reinforcements to SiO_2 , BN, Al_2O_3 , and Si_3N_4 [16,17]. SiO_2 fibers tend to crystallize above 900 °C, leading to severe degradation of the mechanical properties [18,19]. BN fibers, despite their low dielectric constant, suffer from relatively low tensile strength (~0.6 GPa) and are prone to oxidation at temperatures above 900 °C in air [20]. In contrast, Si_3N_4 fibers offer outstanding high-temperature oxidation



School of Materials, Shenzhen Campus of Sun Yat-sen University, Shenzhen 518107, China.

✉ Corresponding authors. E-mail: Z. Tian, tianzhilin@mail.sysu.edu.cn; B. Li, libin75@mail.sysu.edu.cn

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resistance and mechanical properties [21–24]. Compared with SiO₂ fibers, Si₃N₄ fibers have a higher crystallization temperature and better strength retention at elevated temperatures [25,26]. In comparison with BN fibers, Si₃N₄ fibers not only provide superior mechanical properties but also show markedly improved high-temperature stability in air. These advantages make Si₃N₄ fiber an ideal reinforcement for high-temperature wave-transparent composites [26–31].

Previous studies have focused on Si₃N₄ fiber-reinforced Si₃N₄ matrix composites [32,33] and investigated their mechanical and dielectric properties. For instance, a Si₃N₄ fiber-reinforced Si₃N₄ matrix composite with a BN interface (Si₃N₄/BN/Si₃N₄) exhibits high mechanical strength but relatively poor dielectric properties, with a dielectric constant of approximately 5.0 and a dielectric loss tangent of 0.04 [32]. To address this, SiO₂ and BN are considered ideal matrix candidates because they possess inherently low dielectric parameters. SiO₂ and BN possess inherently low dielectric constants and low dielectric loss tangents, with reported values of 3.4 and 0.0004 for fused silica [34] and 4.5 and 0.0003 for BN [35], respectively. These characteristics suggest that using SiO₂ or BN as matrix materials could effectively reduce the overall dielectric constant and dielectric loss tangent. However, both SiO₂ and BN demonstrate inferior mechanical properties compared with Si₃N₄. While the dielectric performance can be enhanced through their incorporation, this improvement comes at the expense of mechanical properties, making it challenging to achieve the balanced performance required for wave-transparent applications. As demonstrated in previous studies [36,37], when fabricating wave-transparent materials via gel-casting with 15 wt% Si₃N₄ addition to BN–SiO₂ systems, the flexural strength increased from 57.43 to 108.72 MPa, while the dielectric constant concurrently rose from 4.35 to 6.43. The above examples highlight a persistent trade-off in matrix selection: Improving mechanical performance often compromises dielectric properties, and vice versa. Resolving this trade-off requires not only careful matrix design but also consideration of another equally critical factor—the fiber-matrix interface. Furthermore, the fiber-matrix interface serves as a mechanical fuse and plays a pivotal role in determining composite mechanical behavior. The optimal interfacial shear strength enables effective load transfer between the fibers and the matrix [38]. For example, when the interfacial shear strength increased from ~75 to ~125 MPa in SiC_f/SiC composites, their flexural strength rose markedly from ~100 to ~600 MPa. However, a further increase in interfacial shear strength to ~200 MPa caused the flexural strength to drop to ~300 MPa [38]. This demonstrates that moderate interfacial bonding facilitates effective load transfer and enhances flexural strength, whereas excessively strong bonding suppresses interfacial debonding and fiber pull-out, ultimately leading to brittle fracture [39]. In line with this, another study reported that for SiC_f/SiC composites, when the interfacial shear strength increased from 52.3±8.7 to 475.2±43.4 MPa, the flexural strength decreased significantly from 200 to 90 MPa, accompanied by markedly shorter fiber pull-out lengths in fracture surfaces. Therefore, achieving high-performance wave-transparent ceramic composites requires rational composition design and precise interface control [40]. Nevertheless, while the individual effects of matrix composition and interfacial strength on mechanical or dielectric properties have been separately documented, a systematic understanding of how matrix composition influences the interfacial structure and how they jointly determine the overall mechanical and dielectric performance of the composite remains lacking. This knowledge gap motivates the present study.

In this study, SiO₂ and BN with low dielectric constants and low dielectric loss tangents were selected as matrix components to optimize the dielectric performance of Si₃N₄ fiber-reinforced composites (Si₃N₄/BN, Si₃N₄/SiO₂, and Si₃N₄/SiO₂–BN). The microstructure and composition of the composites were investigated to reveal the distribution of the Si₃N₄ fibers and ceramic matrix. The fracture process and strain distribution of Si₃N₄/BN, Si₃N₄/SiO₂, and Si₃N₄/SiO₂–BN were explored by a digital image correlation (DIC) system to better understand the influence of composition and interface on the mechanical behavior. The fiber-matrix interface plays a crucial role in determining the mechanical properties of ceramic matrix composites. Therefore, transmission electron microscopy (TEM) was employed to observe the fiber-matrix interface and analyze the thickness of the diffusion region between the fiber and the matrix. In addition, fiber push-in tests were conducted to evaluate the interfacial shear strength within the composites. Their dielectric properties as a function of temperature were studied comprehensively, and the contribution of composition was discussed. The findings of this work provide theoretical and experimental guidelines for the optimization and design of high-temperature wave-transparent composites in extreme service environments.

2 Materials and methods

2.1 Materials

The Si₃N₄ fiber preform was supplied by Fujian Leadasia New Material Co., Ltd. The preform was woven using a 2.5-dimensional (2.5D) weaving method, with a fiber volume fraction of 44.65%. B₃N₃H₆ was used as the BN precursor and was provided by Feidu Aerospace Technology Co., Ltd. Silica sol was supplied by Hebei Jiashihongwei Technology Co., Ltd., with a density of 1.15 g·cm⁻³, a pH of 2.5, and a SiO₂ content of 25 wt%.

2.2 Preparation of Si₃N₄ fiber-reinforced ceramic composites

To remove the surface sizing agent, the Si₃N₄ fiber preform was first cleaned with acetone and then heated in air at 600 °C for 1 h. A Si₃N₄ fiber-reinforced BN matrix composite (Si₃N₄/BN) was fabricated using the precursor infiltration and pyrolysis (PIP) method, with B₃N₃H₆ as the precursor, as illustrated in Fig. 1(a). First, the Si₃N₄ fiber preform was placed in a sealable vessel, which was evacuated to create a vacuum environment. B₃N₃H₆ was then introduced into the vessel, ensuring full immersion of the Si₃N₄ fiber preform for 1 h to facilitate adequate infiltration. Subsequently, N₂ was introduced to restore atmospheric pressure. The preform, still immersed in the precursor, was transferred to an autoclave and heated gradually to 90 °C to initiate crosslinking of B₃N₃H₆. To ensure sufficient precursor loading, this infiltration and crosslinking process was repeated three times before pyrolysis. The infiltrated preform was then pyrolyzed at 800 °C for 1 h under N₂, at a heating rate of 10 °C·min⁻¹. This procedure was repeated three times to obtain dense Si₃N₄/BN.

A Si₃N₄ fiber-reinforced SiO₂ matrix composite (Si₃N₄/SiO₂) was prepared using a sol-gel method, as shown in Fig. 1(b). Initially, the Si₃N₄ fiber preform was placed in a sealable vessel and evacuated. The silica sol was introduced to fully immerse the Si₃N₄ fiber preform for 1 h to ensure complete infiltration. The infiltrated preform, together with the silica sol, was then transferred to a drying oven for gelation and condensation. This infiltration-gelation-condensation cycle was repeated four times,

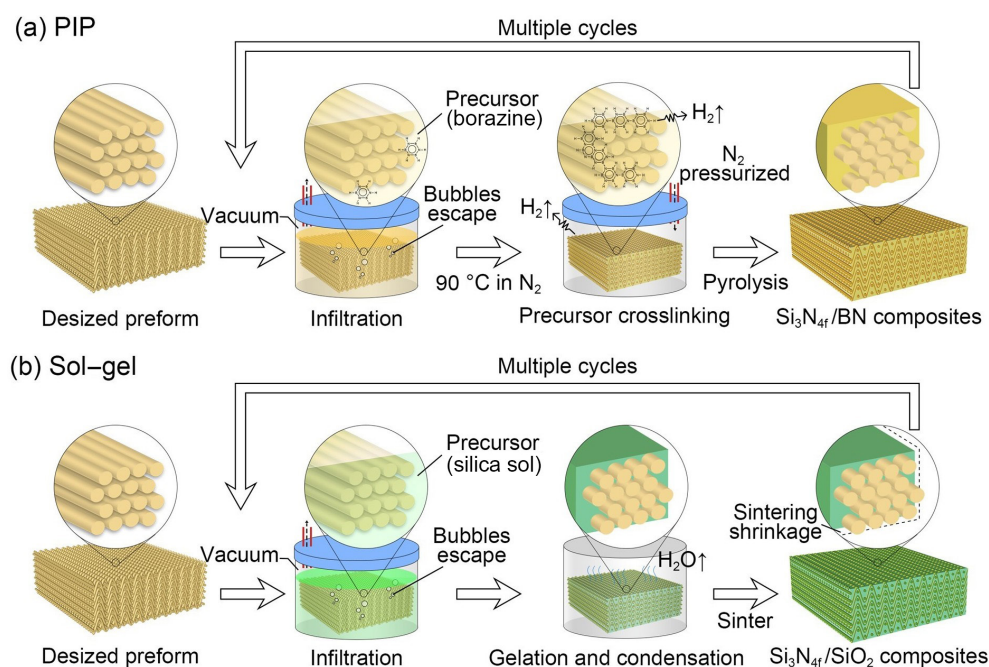


Fig. 1 Preparation procedure of (a) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ and (b) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$.

followed by sintering at 800 °C for 1 h under N_2 , at a heating rate of 10 °C·min⁻¹. This process was repeated three times to obtain dense $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$.

A Si_3N_4 fiber-reinforced SiO_2 and BN matrix composite ($\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$) was fabricated through a sequential combination of the sol-gel and PIP methods, using silica sol and $\text{B}_3\text{N}_3\text{H}_6$ to synthesize the SiO_2 and BN matrices, respectively. First, a sequence of three infiltration-gelation-condensation steps followed by one high-temperature sintering step was carried out to establish the SiO_2 matrix skeleton within the $\text{Si}_3\text{N}_4\text{f}$ preform. Subsequently, using the resulting $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ as the substrate, the BN matrix was introduced to backfill the residual pores. This was achieved by repeating a PIP cycle (consisting of three infiltration-crosslinking steps followed by one pyrolysis step) twice, finally yielding the fully densified $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composite.

2.3 Characterizations

The microstructure of the composites was observed using a field-emission scanning electron microscope (SEM; Sigma 300, Zeiss, Germany). Interfacial characteristics were analyzed by a transmission electron microscope (TEM; Talos F200X, Thermo Fisher, USA), and elemental distribution was examined using an energy-dispersive X-ray spectrometer (EDS; Quantax XFlash, Bruker, USA). Elemental distributions, particularly those of light elements, were further analyzed using an electron probe microanalyzer equipped with wavelength-dispersive spectrometer (EPMA; JXA-IHP200F, JEOL, Japan). The phase composition was determined by an X-ray diffractometer (XRD; Empyrean, PANalytical, Netherlands). The chemical composition of the composites was assessed by an X-ray photoelectron spectrometer (XPS; Thermo Fisher Scientific K-Alpha, USA) coupled with argon ion etching. Mechanical properties were evaluated using a universal testing machine (C42.104, Sintech, China), and flexural strength and compressive strength were measured according to GB/T 6569-2006 and GB/T 8489-2006, respectively. The elastic modulus of the Si_3N_4 fibers was determined using the fiber bundle tensile method based on GB/T 34520.4-2017. Surface strain fields during the flexural strength test were monitored *in situ* using a

DIC system (VIC-3D, Correlated Solutions, Germany). Before testing, a speckle pattern was applied to the surface, and the sample was then mounted in the mechanical testing machine. The camera lenses were adjusted to achieve proper focus, and image acquisition was synchronized with the mechanical loading, capturing images at 500 ms intervals. The recorded images were imported into VIC-3D software, where an appropriate subset size was set for correlation analysis. The density and open porosity of the composites were measured using the Archimedes method. The spatial distribution of fibers and matrix was further characterized using an X-ray computed tomography scanner (XCT; Xradia 610, Zeiss, Germany). The microhardness and reduced modulus of Si_3N_4 fibers and matrices were assessed using a nanoindenter (Hysitron TI 980, Bruker, USA), and the reduced modulus (E_r) obtained from nanoindentation reflects the combined elastic response of both the indenter and the tested material based on the Oliver-Pharr method and can be reasonably approximated as the material's actual Young's modulus [41]. The dielectric constant and dielectric loss tangent were measured from room temperature to 1100 °C using the high-Q cavity method (HQR08HT150, Enchi, China).

3 Results and discussion

3.1 Composition and structure

Figure 2 shows the XRD patterns of the $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composites. All patterns exhibit a broad amorphous band between 20° and 30°, with no detectable crystalline diffraction peaks. This suggests that both Si_3N_4 fibers and the matrix remain amorphous.

Figure 3 shows the microstructure and elemental distribution of the $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composites. The continuous Si_3N_4 fibers extend throughout the composites. The average diameter of the Si_3N_4 fibers is $14.2 \pm 1.2 \mu\text{m}$, and the fibers are well bonded to the matrix. As shown in Fig. 3(a1), the $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ composite exhibits a dense structure with no visible pores or cracks. The EDS elemental mapping reveals that Si is exclusively present within the fibers (Fig. 3(a2)), N is present in

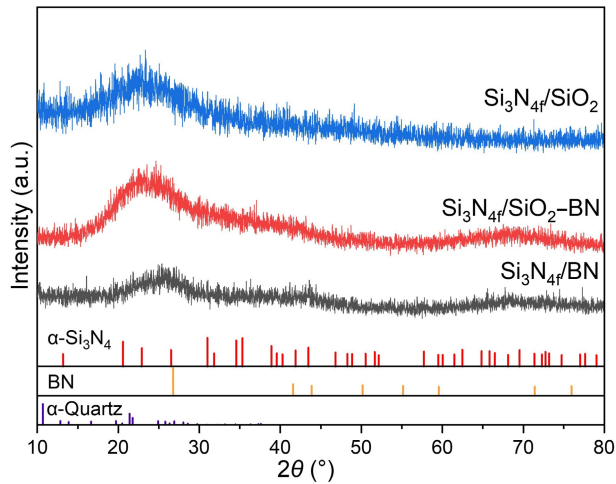


Fig. 2 XRD patterns of $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$.

both the matrix and fibers (Fig. 3(a3)), and while the B signal appears weak, its distribution is not clearly resolved (Fig. 3(a4)), owing to the low detection sensitivity of EDS for light elements such as boron.

In Fig. 3(b1), the cross-sectional image of $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ reveals several pores, suggesting minor porosity in the material. Si is distributed both in the Si_3N_4 fiber and the matrix (Fig. 3(b2)), with a higher concentration in the fibers. N is primarily located within the Si_3N_4 fibers (Fig. 3(b3)), while O is predominantly distributed in the SiO_2 matrix (Fig. 3(b4)).

Figure 3(c1) shows the cross-sectional image of the $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composite, which exhibits a dense microstructure with no visible pores. The presence of two distinct matrices results in differences in contrast. The EDS elemental mapping in Figs. 3(c2)–3(c5) indicates that Si is distributed in both the SiO_2 matrix and the Si_3N_4 fibers, with a notably higher concentration in the fibers and a significantly reduced signal in the regions between the fiber bundles (Fig. 3(c2)). N is distributed both within the Si_3N_4 fiber and between the fibers (Fig. 3(c3)). The B signal is similarly weak in the EDS map (Fig. 3(c4)). O is mainly concentrated in the SiO_2 matrix (Fig. 3(c5)). The simultaneous

decrease in Si and O, along with an increase in N in the regions between the fiber bundles, suggests localized BN formation in these areas. However, because the EDS maps for boron suffer from low detection sensitivity, the distribution of B cannot be reliably resolved. To obtain a clearer and more direct visualization of the boron distribution across all three composites, electron probe microanalysis (EPMA) was performed on their polished cross-sections. The results are shown in Fig. 4. Figure 4 presents the SEM images of the polished cross-sections and their corresponding EPMA elemental mappings for the three materials: $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ (Figs. 4(a1)–4(a4)); $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ (Figs. 4(b1)–4(b4)); and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ (Figs. 4(c1)–4(c5)). As presented in Fig. 4(c5), B is primarily distributed in the cracks present in the SiO_2 matrix. This indicates that during the fabrication process, $\text{B}_3\text{N}_3\text{H}_6$ infiltrates the preformed SiO_2 matrix, filling internal voids and cracks. Upon crosslinking and pyrolysis, BN preferentially forms in these defect-rich regions.

To further investigate the spatial distribution of the Si_3N_4 fibers and matrix phase within the composites, XCT was performed, and the results are presented in Fig. 5. All three composites exhibit similar overall structures and Si_3N_4 fiber distributions, owing to the same 2.5D weaving method. In these preforms, fiber bundles are predominantly oriented in the in-plane direction, with periodically inserted Z-binder yarns that improve through-thickness integrity while retaining planar reinforcement. The matrix phases in all three composites are well bonded to the Si_3N_4 fibers, thereby effectively filling the interstitial regions between the fiber bundles. Figures 5(c2) and 5(c3) show that the BN matrix in the $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composite preferentially infiltrated and filled the pores and cracks in the preformed SiO_2 matrix during fabrication. This process results in a discontinuous yet integrated composite structure, with the BN matrix embedded within the SiO_2 matrix, consistent with the SEM observations.

The spatial distribution of pores varies among the three composites. Figures 5(a4) and 6 illustrate that pores in $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ are mainly located in the BN matrix outside the fiber bundles, while only a few pores are present within the bundles. This is attributed to the multiple PIP cycles used to prepare the BN matrix. During the initial PIP cycle, the precursor preferentially

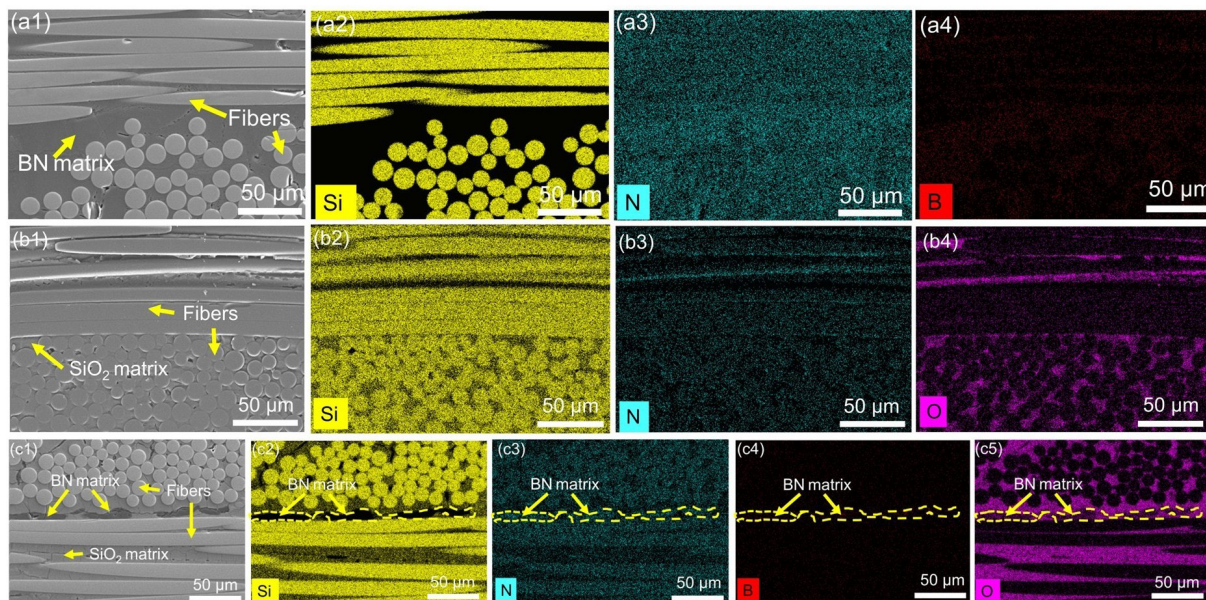


Fig. 3 SEM images of polished cross-sections and corresponding EDS elemental mappings for composites (a1–a4) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (b1–b4) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and (c1–c5) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$. (a1, b1, c1) display cross-sectional morphologies, while remaining panels show respective elemental mappings.

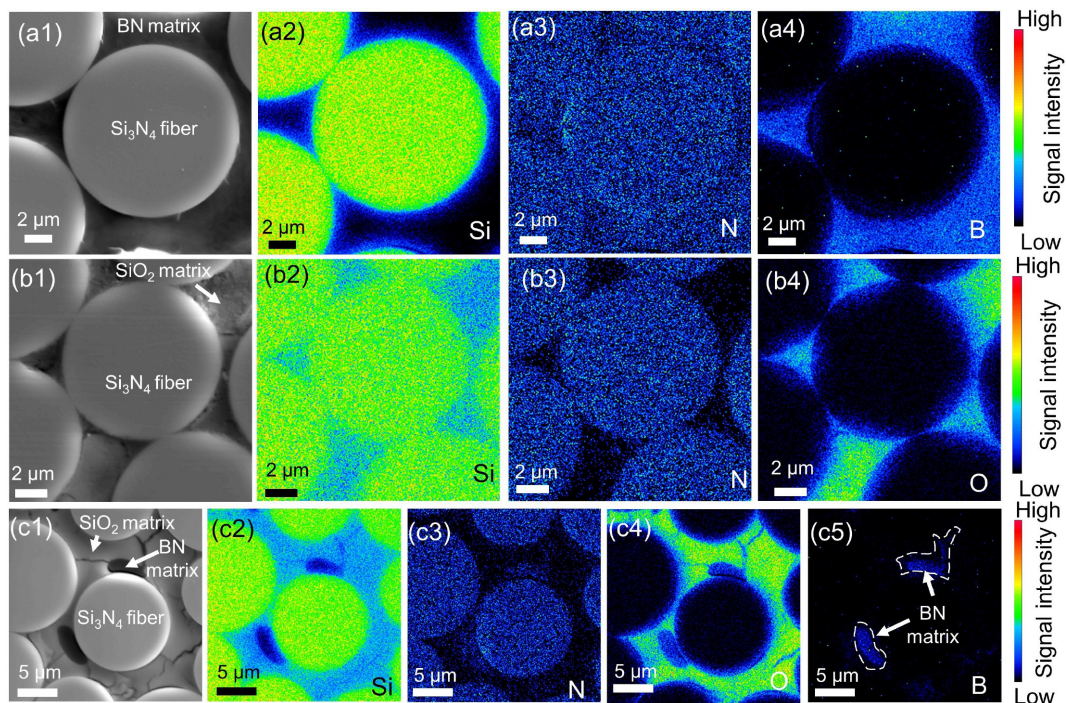


Fig. 4 SEM images of polished cross-sections and corresponding EPMA elemental mappings for composites: (a1–a4) $\text{Si}_3\text{N}_4/\text{BN}$, (b1–b4) $\text{Si}_3\text{N}_4/\text{SiO}_2$, and (c1–c5) $\text{Si}_3\text{N}_4/\text{SiO}_2\text{--BN}$.

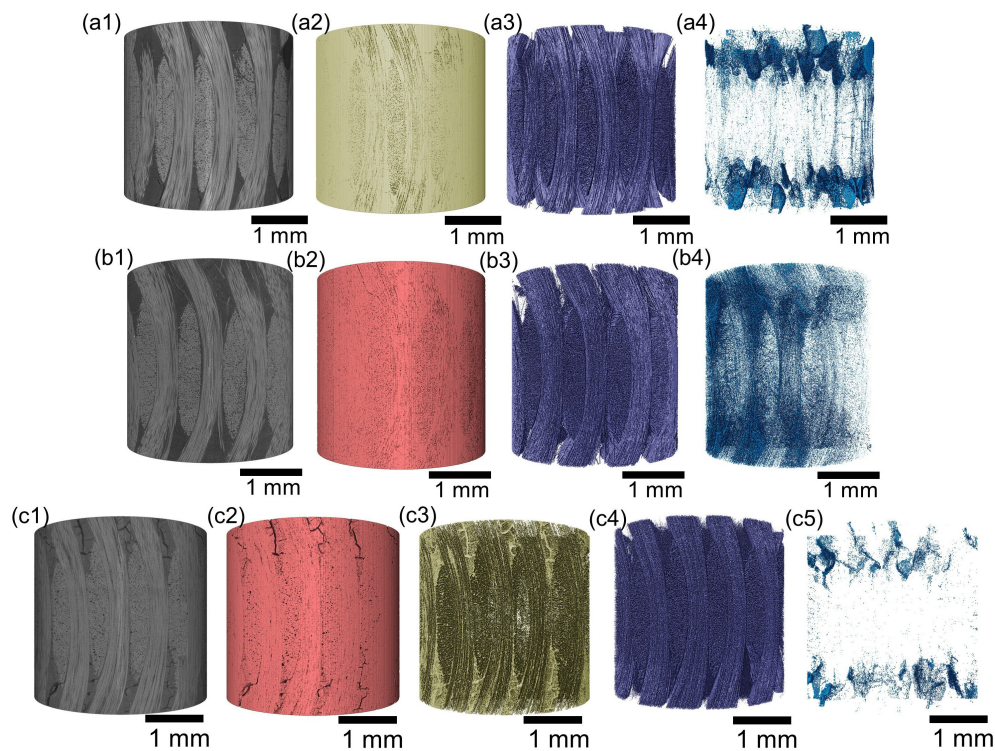


Fig. 5 Three-dimensional (3D) renderings of phase-segmented XCT data for composites: (a1–a4) $\text{Si}_3\text{N}_4/\text{BN}$, (b1–b4) $\text{Si}_3\text{N}_4/\text{SiO}_2$, and (c1–c5) $\text{Si}_3\text{N}_4/\text{SiO}_2\text{--BN}$. (a1, b1, c1) presents overall structures. Segmented phases are as follows: (a2, c3) BN matrix in yellow, (b2, c2) SiO_2 matrix in red, (a3, b3, c4) Si_3N_4 fibers in purple, and (a4, b4, c5) pores in blue.

infiltrates the fiber bundles, where it is pyrolyzed into BN, leaving voids in regions without fiber reinforcement. In the subsequent PIP cycles, the BN matrix tends to fill these initially unoccupied areas. However, interfaces are inevitably generated between the BN matrix from the first cycle and those from subsequent cycles, leading to the retention of pores. In contrast, Fig. 5(b4) shows that pores in $\text{Si}_3\text{N}_4/\text{SiO}_2$ are evenly distributed throughout the SiO_2

matrix, owing to the water released during the sol–gel process as the silica sol transforms into solid SiO_2 , which inevitably generates pores. These pores are uniformly distributed, indicating that the SiO_2 matrix formed during the first and subsequent sintering steps is well integrated, without noticeable interface formation. In the case of $\text{Si}_3\text{N}_4/\text{SiO}_2\text{--BN}$ (Fig. 5(c5)), the composite contains very few pores. This is due to the excellent infiltration capability of the

BN precursor, which effectively penetrates residual pores and cracks in the preformed SiO_2 matrix and is subsequently converted into BN during pyrolysis, significantly reducing the overall porosity. The pore distribution in the $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN resembles that of the $\text{Si}_3\text{N}_4/\text{BN}$, suggesting that interfaces and pores were also formed within the BN matrix during the PIP cycles.

The volume fractions of the matrix, Si_3N_4 fiber, and macropores in each composite are summarized in Table 1. Across all samples, the Si_3N_4 fiber content remains relatively constant (44.28%, 44.88%, and 44.57%), as does the matrix phase (55.45%, 54.80%, and 55.33%, with the latter comprising 9.49% BN and 45.84% SiO_2). The resolved porosities are 0.27%, 0.32%, and 0.1%, respectively, indicating that there are no significant macropores in any of the composites. Given that the XCT analysis was performed with a voxel size of $3\ \mu\text{m} \times 3\ \mu\text{m} \times 3\ \mu\text{m}$, this resolution is insufficient to precisely resolve and quantify the micropores within the composites. To obtain accurate porosity and density data, the Archimedes method was employed to measure the density and open porosity of $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN. The results are presented in Fig. 7.

The density and open porosity of $\text{Si}_3\text{N}_4/\text{BN}$ are $1.98 \pm 0.12\ \text{g}\cdot\text{cm}^{-3}$ and $0.42\% \pm 0.06\%$, respectively; those of $\text{Si}_3\text{N}_4/\text{SiO}_2$ are $1.91 \pm 0.22\ \text{g}\cdot\text{cm}^{-3}$ and $13.35\% \pm 3.87\%$; and those of $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN are $2.13 \pm 0.18\ \text{g}\cdot\text{cm}^{-3}$ and $0.51\% \pm 0.17\%$. The density of amorphous silica is $2.2\ \text{g}\cdot\text{cm}^{-3}$, that of amorphous boron nitride is $2.06\ \text{g}\cdot\text{cm}^{-3}$, and that of Si_3N_4 fiber is $2.25\ \text{g}\cdot\text{cm}^{-3}$. The theoretical density and closed porosity are calculated using Eqs. (1) and (2):

$$\rho_{\text{theory}} = V_f \cdot \rho_f + V_m \cdot \rho_m \quad (1)$$

$$P_c = \left(1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{theory}}}\right) \times 100\% - P_o \quad (2)$$

where ρ_{theory} represents the theoretical density; V_f and V_m are the volume fractions of the fibers and matrix, respectively; ρ_f and ρ_m are their corresponding densities. Additionally, P_c is the closed porosity, P_o is the open porosity, and ρ_{bulk} stands for the actual bulk density of the composites. Based on these parameters, the calculated theoretical density of the $\text{Si}_3\text{N}_4/\text{BN}$ composite is $2.14\ \text{g}\cdot\text{cm}^{-3}$, with a total porosity of 7.65% and a closed porosity of 7.23%. For $\text{Si}_3\text{N}_4/\text{SiO}_2$, the theoretical density is $2.22\ \text{g}\cdot\text{cm}^{-3}$,

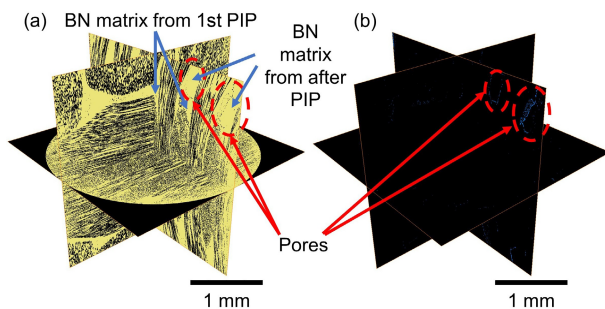


Fig. 6 XCT cross-sectional images of (a) matrix and (b) pores of $\text{Si}_3\text{N}_4/\text{BN}$.

Table 1 Volume fraction (%) of different components in the composites (Unit: %)

Composite	Resolved porosity	BN matrix	SiO_2 matrix	Si_3N_4
$\text{Si}_3\text{N}_4/\text{BN}$	0.27	55.45	—	44.28
$\text{Si}_3\text{N}_4/\text{SiO}_2$	0.32	—	54.80	44.88
$\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN	0.10	9.49	45.84	44.57

accompanied by a significantly higher total porosity of 14.06% and a lower closed porosity of 0.71%. The $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN composite exhibits a theoretical density of $2.21\ \text{g}\cdot\text{cm}^{-3}$, a total porosity of 3.57%, and a closed porosity of 3.06%.

The fiber-matrix interface plays a crucial role in determining the mechanical properties of ceramic matrix composites. To investigate the interfacial bonding and elemental distributions, the fiber-matrix interfaces of $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN were characterized, as shown in Figs. 8–11.

Figure 8 presents the overall TEM morphologies of the fiber-matrix interfaces in the three composites. In $\text{Si}_3\text{N}_4/\text{BN}$ (Fig. 8(a)), a well-defined interfacial region is observed between the Si_3N_4 fiber and the BN matrix. The interface in $\text{Si}_3\text{N}_4/\text{SiO}_2$ (Fig. 8(b)) appears narrow and distinct. In comparison, $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN (Fig. 8(c)) exhibits a noticeably wider and more diffuse interfacial zone, suggesting enhanced elemental interdiffusion during fabrication.

For $\text{Si}_3\text{N}_4/\text{BN}$, as shown in Fig. 9(a), a distinct transition zone is observed between the fiber and the matrix, indicating elemental interdiffusion at the interface. The diffusion region spans approximately $37.73 \pm 2.43\ \text{nm}$ in width. The EDS line scan in Fig. 9(b) shows that N is present in both the fiber and the matrix. The line scan results further reveal that Si is present in the fiber but not in the matrix. A transition region exists between the fiber and the matrix where the Si content gradually decreases. B and O are absent in the fiber and present in the matrix. Across the transition region, the B content gradually increases. The presence of O in the matrix indicates that the BN matrix underwent partial oxidation during fabrication. Elemental mapping in Figs. 9(c)–9(f) further confirms this observation, showing spatial distributions consistent with the line scan data. Collectively, these observations indicate that a distinct amorphous interdiffusion zone exists between the fiber and the matrix.

For $\text{Si}_3\text{N}_4/\text{SiO}_2$, the high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) image in Fig. 10(a) shows a distinct interface between the Si_3N_4 fiber (left) and the SiO_2 matrix (right), with a diffusion region approximately $9.41 \pm 1.02\ \text{nm}$ in width. The EDS line scan in Fig. 10(b) confirms this narrow diffusion region, while the elemental mapping results in Figs. 10(c)–10(e) illustrate the overall elemental distributions near the interface, revealing a distinct interfacial boundary with limited elemental interdiffusion.

In contrast, $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN exhibits a significantly wider and more gradual interface, as shown in Fig. 11(a), with a diffusion region approximately $53.09 \pm 0.85\ \text{nm}$ in width. The EDS line scan in Fig. 11(b) shows gradual variations in elemental intensities across this region, while the elemental mappings of O and N in

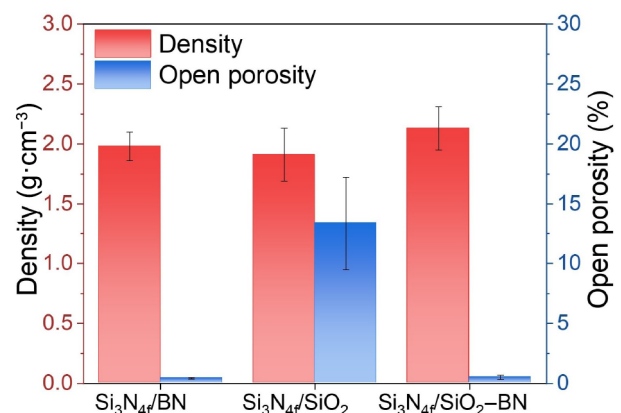


Fig. 7 Density and open porosity of $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2$ -BN.

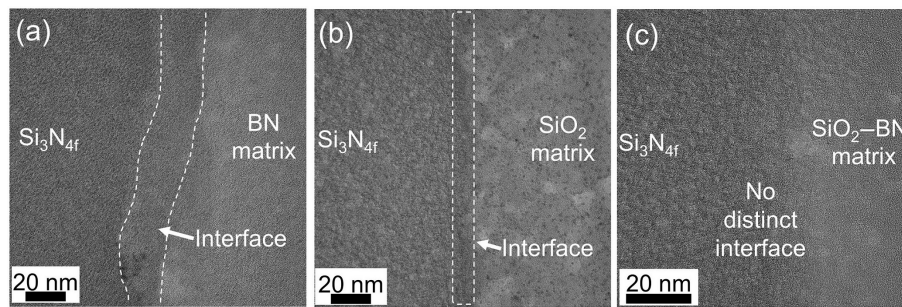


Fig. 8 TEM images of interfacial regions in composites: (a) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (b) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and (c) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$.

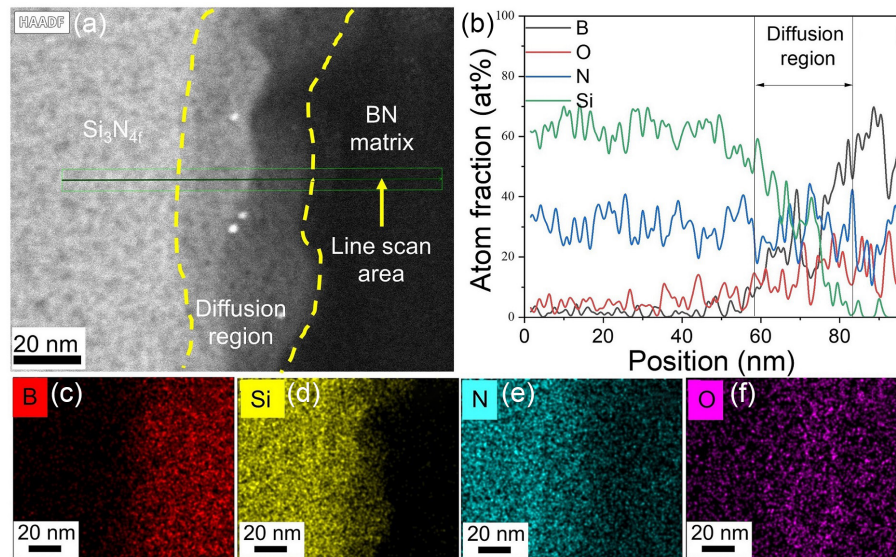


Fig. 9 (a) HAADF-STEM image of fiber-matrix interface in $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (b) EDS line scan of B, N, and Si along marked path, and (c-f) corresponding elemental mappings of B, Si, N, and O.

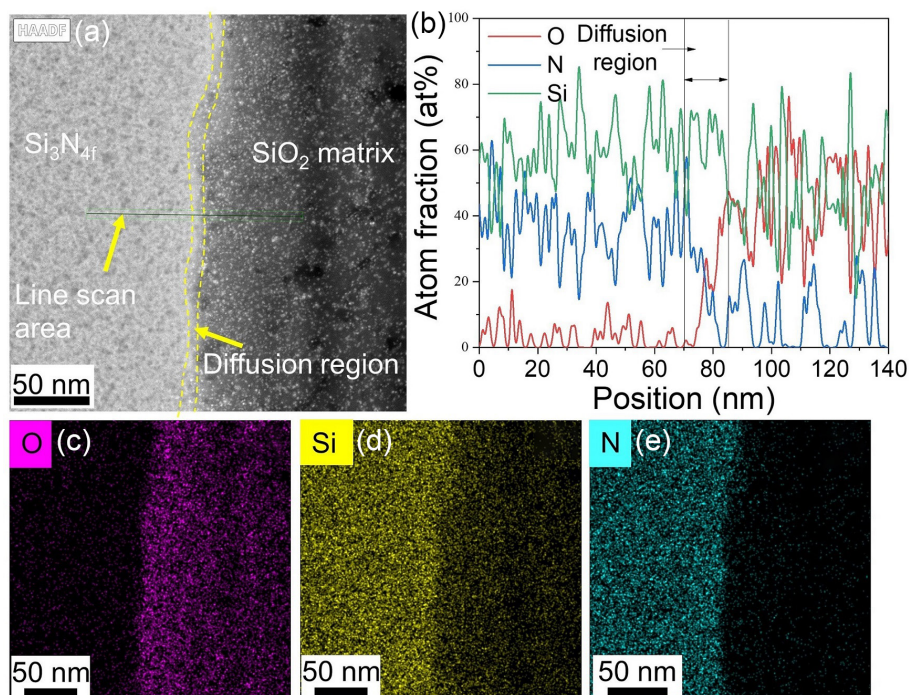


Fig. 10 (a) HAADF-STEM image of fiber-matrix interface in $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, (b) EDS line scan of O, N, and Si along marked path, and (c-e) corresponding elemental mappings of O, Si, and N.

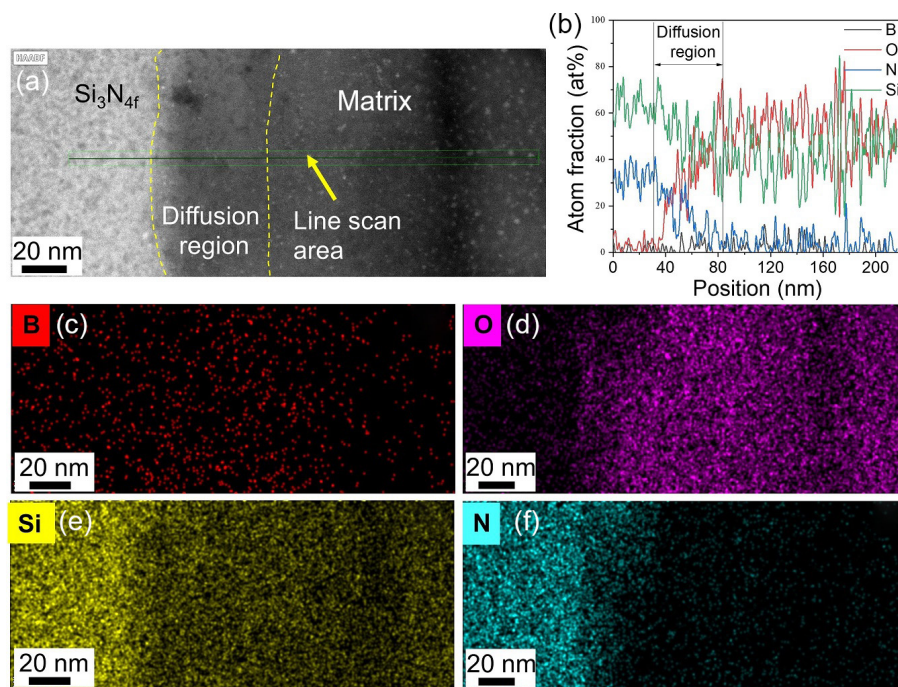


Fig. 11 (a) HAADF-STEM image of fiber-matrix interface in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$, (b) EDS line scan of B, O, N, and Si along marked path, and (c–f) corresponding elemental mappings of B, O, Si, and N.

Figs. 11(c)–11(f) further confirm the presence of a wide interfacial diffusion zone, which is noticeably broader and more diffuse than those observed in the other two composites.

The expanded diffusion region in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ is likely a consequence of its fabrication process, in which a BN matrix is introduced into a preformed SiO_2 matrix via precursor infiltration pyrolysis. During this process, the residual silanol groups (Si-OH) in the SiO_2 matrix react with the highly reactive BN precursor ($\text{B}_3\text{N}_3\text{H}_6$), thereby improving the chemical compatibility between the fiber and matrix [42]. This enhanced chemical affinity facilitates greater elemental diffusion at the interface. Interfacial diffusion regions form during high-temperature sintering due to atomic migration between the fiber and matrix. The thickness of the interfacial diffusion region between the fiber and matrix in the three composites follows the order $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN} > \text{Si}_3\text{N}_{4f}/\text{BN} > \text{Si}_3\text{N}_{4f}/\text{SiO}_2$.

XPS was employed to investigate the chemical bonding states in the three composites. The results are shown in Fig. 12. As illustrated in Fig. 12(a), the B 1s spectrum of $\text{Si}_3\text{N}_{4f}/\text{BN}$ exhibits two characteristic peaks at 190.55 and 192.81 eV, corresponding to B–N and B–O bonds, respectively [43]. The B–N bond originates from the BN matrix, while the B–O bond is attributed to partial oxidation of the BN precursor during the fabrication of the BN matrix. In $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ (Fig. 12(b)), the B–N peak remains at 190.55 eV, confirming the presence of BN. However, in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$, the B–O peak shifts to 192.98 eV, compared with 192.81 eV in $\text{Si}_3\text{N}_{4f}/\text{BN}$ (Fig. 12(a)). This shift suggests that in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$, the B–O bonds form not only from partial oxidation of the BN precursor but also from reactions between the BN precursor and surface silanol groups (Si-OH) in the SiO_2 matrix. The additional reaction pathway alters the local chemical environment of boron and leads to the observed shift toward higher binding energy.

The Si 2p spectra further highlight the variations in the chemical environment among the composites. As shown in Figs. 12(c) and 12(d), a peak at 102.11 eV, corresponding to Si–N bonds, is consistently observed in both $\text{Si}_3\text{N}_{4f}/\text{BN}$ and $\text{Si}_3\text{N}_{4f}/\text{SiO}_2$,

indicating the presence of Si_3N_4 fibers [44]. In $\text{Si}_3\text{N}_{4f}/\text{SiO}_2$ (Fig. 12(d)), an additional peak at 103.88 eV is assigned to the Si–O bonds of the SiO_2 matrix [45]. The Si 2p spectrum of $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ (Fig. 12(e)) is fitted with two peaks at 102.11 eV (Si–N) and 103.69 eV (Si–O). Compared with the Si–O peak in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2$ (103.88 eV), the Si–O peak in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ appears at a lower binding energy of 103.69 eV. This shift indicates a change in the chemical environment of Si atoms in the hybrid matrix, consistent with the reaction between the BN precursor and residual silanol groups. Together with the B 1s evidence change in Fig. 12(b), these spectral features can be attributed to the reaction between $\text{B}_3\text{N}_3\text{H}_6$ and Si-OH . On one hand, this reaction improves the chemical compatibility between the fiber and the matrix; on the other hand, it introduces defects into the silica network that facilitate elemental diffusion. The synergistic effects promote the formation of a broader interfacial diffusion region in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$.

3.2 Mechanical properties

To investigate the properties of individual constituents in the composite, nanoindentation tests were performed to determine the hardness and reduced modulus of the fibers and matrices, as summarized in Table 2.

The BN matrix in both $\text{Si}_3\text{N}_{4f}/\text{BN}$ and $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ exhibits comparable hardness and modulus. However, the SiO_2 matrix in $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ shows significantly higher hardness and modulus than those of $\text{Si}_3\text{N}_{4f}/\text{SiO}_2$. This enhancement arises from two synergistic mechanisms. First, during the PIP process for introducing the BN matrix, the BN precursor effectively infiltrates and fills the open pores and voids within the sol-gel-derived SiO_2 matrix. As a result, $\text{Si}_3\text{N}_{4f}/\text{SiO}_2\text{-BN}$ exhibits a much lower matrix porosity (3.57%) than $\text{Si}_3\text{N}_{4f}/\text{SiO}_2$ (14.06%), and this densification directly increases the stiffness and hardness of the SiO_2 matrix. Second, the BN precursor ($\text{B}_3\text{N}_3\text{H}_6$) reacts with residual silanol groups (Si-OH) in SiO_2 , introducing B and N into the silica network and forming a more highly crosslinked, denser amorphous phase, potentially resembling a borosilicate-like

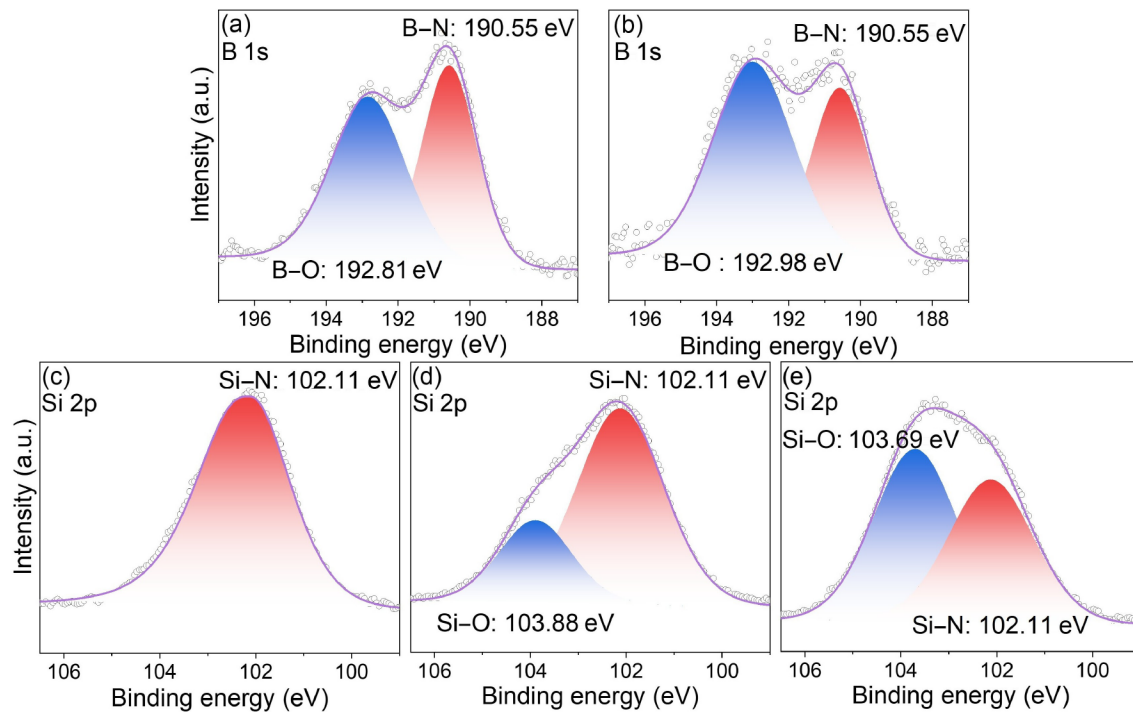


Fig. 12 XPS spectra of B 1s for (a) Si₃N₄f/BN and (b) Si₃N₄f/SiO₂-BN and Si 2p spectra for (c) Si₃N₄f/BN, (d) Si₃N₄f/SiO₂, and (e) Si₃N₄f/SiO₂-BN.

Table 2 Hardness and E_r of different components in the composites

Composite	Component	Hardness (GPa)	E_r (GPa)
Si ₃ N ₄ f/BN	Si ₃ N ₄ fiber	13.54±0.24	144.89±3.36
	BN matrix	1.27±0.12	19.95±1.58
Si ₃ N ₄ f/SiO ₂	Si ₃ N ₄ fiber	13.74±0.20	152.73±2.62
	SiO ₂ matrix	1.97±0.06	20.48±0.40
Si ₃ N ₄ f/SiO ₂ -BN	Si ₃ N ₄ fiber	14.39±0.26	164.72±1.24
	SiO ₂ matrix	4.06±0.14	48.43±1.71
	BN matrix	1.14±0.04	16.98±0.34

structure. This chemically reinforced network further enhances the hardness and modulus of the matrix. The combined physical pore-filling and chemical network-reinforcement effects are consistent with the observed highest density and lowest porosity of Si₃N₄f/SiO₂-BN among the three composites, providing a coherent explanation for the marked improvement in the mechanical properties of the SiO₂ matrix.

To investigate the interfacial bonding between the fiber and the matrix, fiber push-in tests were conducted to quantify the interfacial shear strength between the Si₃N₄ fiber and the surrounding matrix, which directly affects crack deflection and fiber pull-out behaviors. As illustrated in Fig. 13(a), the test involves applying a localized load to a single fiber using a flat punch indenter. The corresponding push-in force-displacement curves of Si₃N₄f/BN, Si₃N₄f/SiO₂-BN, and Si₃N₄f/SiO₂ are presented in Fig. 13(b). The interfacial shear strength (τ_{SL}) between the fibers and the composites was calculated using Eq. (3) [30], where S_0 is the average stiffness in the initial linear region of the push-in curve; P_0 is the critical force that causes fiber-matrix debonding; r is the radius of the Si₃N₄ fiber; E_f is the elastic modulus of Si₃N₄ determined by tensile testing.

$$\tau_{SL} = \frac{S_0 P_0}{2\pi^2 r^3 E_f} \quad (3)$$

The calculated interfacial shear strength for the Si₃N₄f/BN,

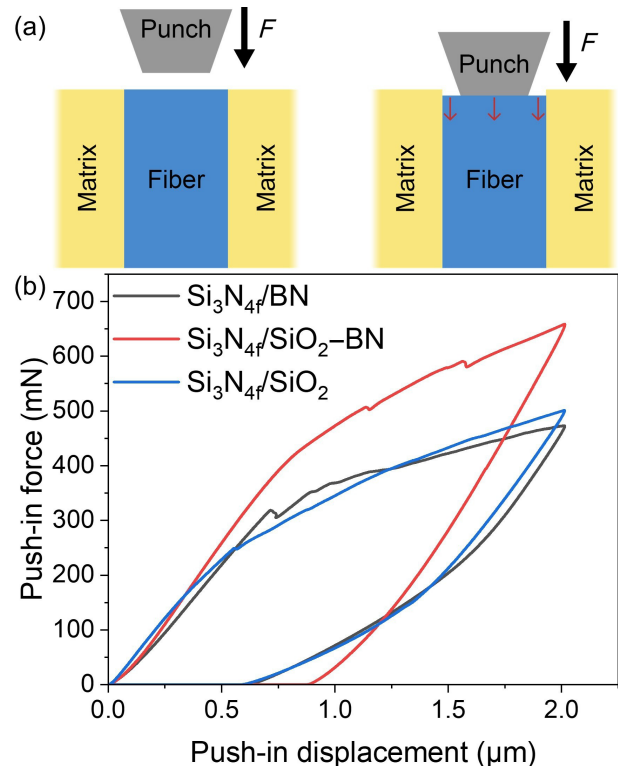


Fig. 13 (a) Schematic diagram of fiber push-in process and (b) push-in curves of Si₃N₄f/BN, Si₃N₄f/SiO₂-BN, and Si₃N₄f/SiO₂.

Si₃N₄f/SiO₂, and Si₃N₄f/SiO₂-BN composites is 238.15±28.21, 188.80±14.02, and 443.68±20.12 MPa, respectively. Notably, the interfacial shear strength in Si₃N₄f/SiO₂-BN is approximately 1.26 times that in Si₃N₄f/BN and more than 2.35 times that in Si₃N₄f/SiO₂. This trend shows a clear positive correlation with the thickness of the fiber-matrix diffusion region observed by HAADF-STEM and EDS line scan: The thickest diffusion region in Si₃N₄f/SiO₂-BN corresponds to the highest interfacial shear

strength, while the thinnest in $\text{Si}_3\text{N}_4/\text{SiO}_2$ corresponds to the lowest. This strong positive correlation can be explained by the chemical reactions occurring during composite fabrication. During the fabrication of $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$, the BN precursor ($\text{B}_3\text{N}_3\text{H}_6$) infiltrates the preformed SiO_2 matrix and reacts with residual silanol groups (Si-OH). This reaction promotes the interdiffusion of elements between the fiber and the matrix through two synergistic mechanisms: (i) The introduction of N into the SiO_2 network improves the chemical compatibility between the Si_3N_4 fiber and the SiO_2 matrix [46]; (ii) the incorporation of boron into the SiO_2 network disrupts the regular connectivity of the original SiO_4 tetrahedra, increasing the degree of structural disorder and the defect concentration and thereby providing additional pathways for the interdiffusion of Si, N, and O [47,48]. The synergy of the enhanced chemical compatibility and the increased number of diffusion pathways leads to a significantly thicker interfacial diffusion zone in $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ than those in $\text{Si}_3\text{N}_4/\text{BN}$ and $\text{Si}_3\text{N}_4/\text{SiO}_2$. A thicker diffusion zone provides a larger region of chemical bonding between the fiber and the matrix, transforming the interface from limited mechanical contact into extensive chemical bonding. As a result, the interfacial shear strength is raised to the measured level.

The markedly higher interfacial shear strength of $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ indicates excessively strong fiber-matrix bonding that effectively locks the fiber and the surrounding matrix together. When cooled from the processing temperature to room temperature, the Si_3N_4 fiber and the matrix experience different degrees of thermal contraction due to their mismatched coefficients of thermal expansion [49]. If unconstrained, the fiber and the matrix contract independently (Fig. 14(b)). However, the strong interfacial bonding established through the thick diffusion zone prevents any relative sliding or debonding that could accommodate this differential contraction. Consequently, the matrix is forced into residual compression, while the fiber is subjected to residual tension (Fig. 14(c)). These thermally induced tensile stresses are locked into the fiber and cannot be relieved. Moreover, the higher stiffness of the SiO_2 matrix in $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ amplifies the magnitude of the thermal residual stress under a given coefficient of thermal expansion (CTE) mismatch. The resulting residual stress within the composite reduces its effective load-bearing capacity. Furthermore, this excessive bonding also suppresses interfacial debonding during

failure, thereby inhibiting fiber pull-out and leading to a brittle fracture mode. This mechanistic framework provides the basis for understanding the mechanical behavior discussed below.

To evaluate how the varying interfacial bonding states influence the load-bearing performance of the three composites, their flexural and compressive strengths were measured. The stress-displacement curves under flexural and compressive loading, as well as the corresponding flexural and compressive strengths, are shown in Fig. 15.

Figure 15(a) presents the stress-displacement curves of $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ composites under flexural loading. All three composites initially exhibit a linear elastic region, indicating elastic deformation. For $\text{Si}_3\text{N}_4/\text{BN}$, microcracks began to form at point A1 as the stress increased, causing the curve to deviate from linearity. At the peak stress (point B1), extensive cracking occurred, resulting in a sharp load drop. For $\text{Si}_3\text{N}_4/\text{SiO}_2$, microcracks initiated at point A2, also causing the curve to deviate from linearity. After reaching the peak stress at point B2, the load dropped gradually, indicative of progressive and significant fiber pull-out, suggesting superior damage tolerance. $\text{Si}_3\text{N}_4/\text{SiO}_2$ shows a lower peak stress but a greater displacement than $\text{Si}_3\text{N}_4/\text{BN}$, implying that more cracks were generated during deformation. In contrast, for $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$, the curve reached its peak stress within the linear region at point A3, followed by a steep load drop without a preceding nonlinear region caused by quick crack propagation, suggesting a typical brittle fracture behavior.

Figure 15(b) displays the stress-displacement curves of the three composites under compressive loading. At the initial stage, all curves exhibit a nonlinear region up to points O1 ($\text{Si}_3\text{N}_4/\text{BN}$), O2 ($\text{Si}_3\text{N}_4/\text{SiO}_2$), and O3 ($\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$). This initial nonlinearity is primarily attributed to the closure of processing-induced microcracks within the composites [50–52]. Notably, $\text{Si}_3\text{N}_4/\text{SiO}_2$ exhibits the largest displacement in this region, which is consistent with its higher porosity, and the abundant pores likely acted as stress concentrators that promoted a greater density of microcracks during fabrication. Following the nonlinear stage, the curves display a linear region between points O and A (O1–A1 for $\text{Si}_3\text{N}_4/\text{BN}$, O2–A2 for $\text{Si}_3\text{N}_4/\text{SiO}_2$, and O3–A3 for $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$), indicating linear-elastic deformation after crack closure. Beyond point A, the deviation from linearity signifies the onset of further damage, such as matrix cracking or propagation

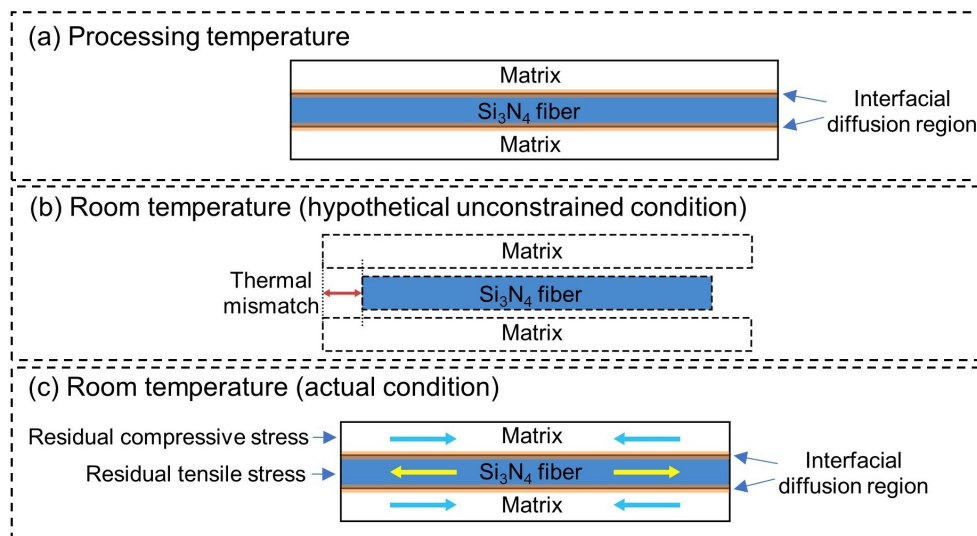


Fig. 14 Schematic illustration of residual thermal stress generation in composite due to strong interfacial bonding.

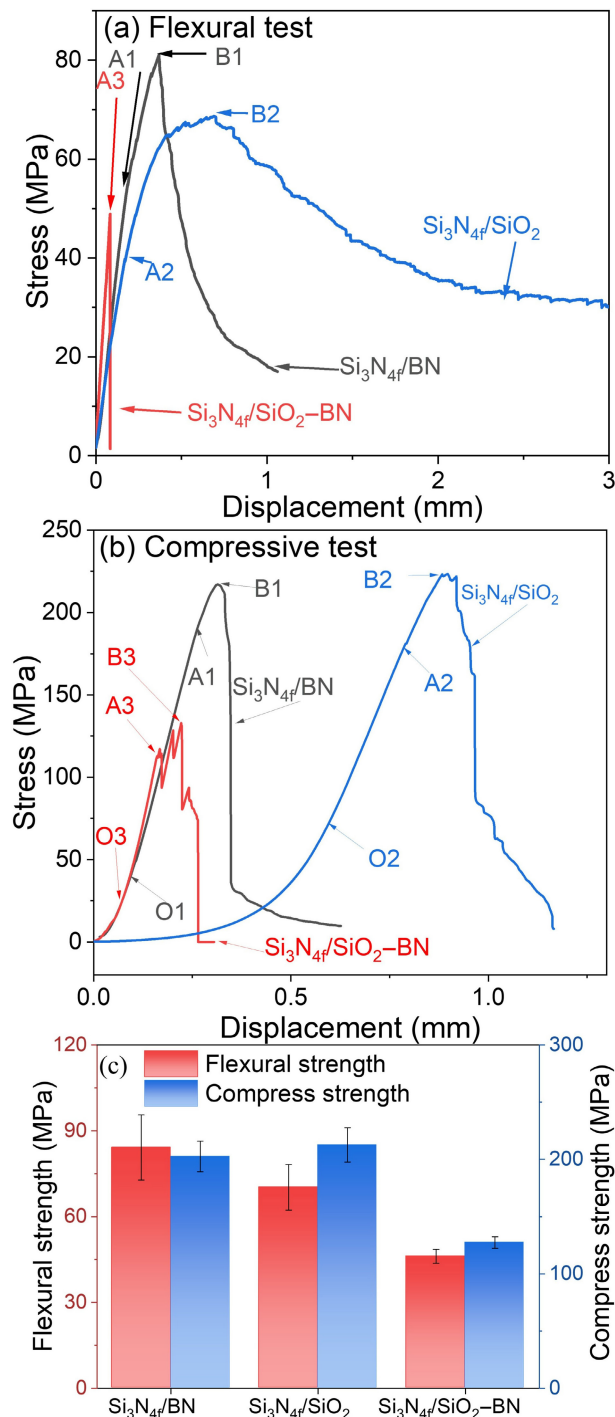


Fig. 15 Stress–displacement curves of $\text{Si}_3\text{N}_4/\text{BN}$, $\text{Si}_3\text{N}_4/\text{SiO}_2$, and $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ under (a) flexural and (b) compressive loading. (c) Comparison of flexural strength and compressive strength of three composites.

of preexisting microcracks. For $\text{Si}_3\text{N}_4/\text{BN}$, the curve reaches peak stress at point B1 and then drops sharply, indicating sudden failure. In contrast, $\text{Si}_3\text{N}_4/\text{SiO}_2$ exhibits a gradual load drop post-peak, suggesting progressive damage and superior compressive strength, as well as the largest overall displacement among the three materials. $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ shows a rapid increase to peak stress at point B3, followed by a steep decline, indicating limited compressive deformation capacity.

As shown in Fig. 15(c), $\text{Si}_3\text{N}_4/\text{BN}$ demonstrates the highest flexural strength, followed by $\text{Si}_3\text{N}_4/\text{SiO}_2$, while $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ exhibits the lowest flexural strength. In terms of compressive

strength, the order is $\text{Si}_3\text{N}_4/\text{SiO}_2 > \text{Si}_3\text{N}_4/\text{BN} > \text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$. Regarding displacement at failure, $\text{Si}_3\text{N}_4/\text{SiO}_2$ shows the largest displacement under both flexural and compressive loading, followed by $\text{Si}_3\text{N}_4/\text{BN}$, with $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ showing the smallest displacement. The large displacement of $\text{Si}_3\text{N}_4/\text{SiO}_2$ is attributed to the combined effect of its highest porosity and its lowest interfacial shear strength. Under flexural loading, the high porosity provides sites for crack deflection, while under compressive loading, the closure of microcracks contributes to an additional displacement during the initial loading stage. In both loading modes, the low interfacial shear strength permits fiber-matrix debonding and fiber pull-out, further increasing the displacement before failure. In contrast, the lowest porosity and the highest interfacial shear strength in $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ suppress these mechanisms, resulting in the smallest displacement at failure.

DIC was utilized to obtain the full-field strain distribution during the flexural tests. Sequential images were captured and analyzed using DIC software to calculate the strain fields. The normal strain components (ϵ_{xx} and ϵ_{yy}) and shear strain component (ϵ_{xy}) were extracted at different deformation stages, offering insights into crack initiation and propagation, as well as the deformation and failure mechanisms of the composites under flexural loading.

As shown in Figs. 16(a1)–16(a4), strain localization in $\text{Si}_3\text{N}_4/\text{BN}$ occurred primarily on the tensile surface during flexural loading. In the ϵ_{xx} and ϵ_{yy} fields (Figs. 16(a1) and 16(a2)), tensile strain initially concentrated at the center of the tensile surface, opposite the loading point, indicating crack initiation. As loading increased, strain propagated from the surface into the interior and toward the compressive side, following the fiber bundle orientation. The ϵ_{xy} field (Fig. 16(a3)) showed prominent shear strain on the tensile side, suggesting that failure occurred predominantly through slippage in this region. The post-fracture morphology (Fig. 16(a4)) revealed step-like features and crack deflection, consistent with the observed strain distribution.

In Figs. 16(b1)–16(b4), the $\text{Si}_3\text{N}_4/\text{SiO}_2$ composite displayed a more distributed strain pattern. The ϵ_{xx} field (Fig. 16(b1)) showed progressive tensile strain on the tensile surface and compressive strain on the loading surface. The ϵ_{yy} field (Fig. 16(b2)) exhibited strain localization expanding from the tensile surface toward the compressive side, forming a high-strain zone near the left side of the loading point, while the strain on the right side remained comparatively lower. This left–right asymmetry in the strain field under the indenter suggests that fiber-matrix debonding initiated on one side of the loading point. The ϵ_{xy} field (Fig. 16(b3)) revealed opposite shear strains on the tensile and compressive surfaces, indicating internal sliding between layers. The asymmetric strain distribution can be understood as follows: Once debonding occurs on one side of the loading point, the interface loses its ability to transfer stress. In these debonded regions, the load is carried predominantly by the fibers, which undergo greater deformation. This leads to larger relative displacements between DIC speckle points and thus higher measured strain. In the adjacent regions that remain bonded, the fiber and matrix continue to deform together, resulting in lower strain. This contrast between the debonded side and the bonded side directly produces the left–right asymmetric strain field observed in the DIC results. The post-fracture image (Fig. 16(b4)) revealed significant fiber pull-out and numerous minor cracks along the fiber direction, reflecting a damage-tolerant fracture mode.

In Figs. 16(c1)–16(c4), the $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ composite exhibited lower strain levels than the other composites. With increasing stress, an obvious strain concentration point indicating crack

initiation appeared on the tensile surface in the ε_{xx} field (Fig. 16(c1)), and the crack rapidly propagated through the interior. The ε_{yy} field (Fig. 16(c2)) revealed positive strains at the crack locations, reflecting local opening due to vertical crack propagation. The ε_{xy} field (Fig. 16(c3)) showed shear strain primarily on the tensile surface, indicating that interfacial sliding and deformation occurred there during fracture. Compared with $\text{Si}_3\text{N}_4/\text{BN}$ and $\text{Si}_3\text{N}_4/\text{SiO}_2$, $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$ exhibited smaller strain magnitudes in all directions, suggesting the formation of smaller cracks with limited deflection but rapid penetration throughout the composite. This interpretation is further supported by the relatively straight crack paths observed in the post-fracture sample (Fig. 16(c4)), in contrast to the more tortuous paths observed in the other two composites. The corresponding DIC videos showing

the full-field strain evolution for all three composites are available in Videos S1–S9 in the Electronic Supplementary Material (ESM).

The fracture morphologies of the flexural-tested sample are presented in Fig. 17. In Figs. 17(a1) and 17(a2), $\text{Si}_3\text{N}_4/\text{BN}$ exhibits fiber bundle pull-out, with only a small amount of individual filament pull-out (Fig. 17(a3)). Cracks propagate primarily along the fiber bundle direction. As shown in Figs. 17(b1) and 17(b2), $\text{Si}_3\text{N}_4/\text{SiO}_2$ displays extensive fiber pull-out, with fiber bundles unraveling into individual filaments. Conversely, Figs. 17(c1) and 17(c2) show a relatively smooth fracture surface for $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$, indicating brittle fracture behavior. In addition, no clear fiber-matrix interface is observed on the fracture surface (Fig. 17(c3)), which is consistent with the diffuse interfacial region

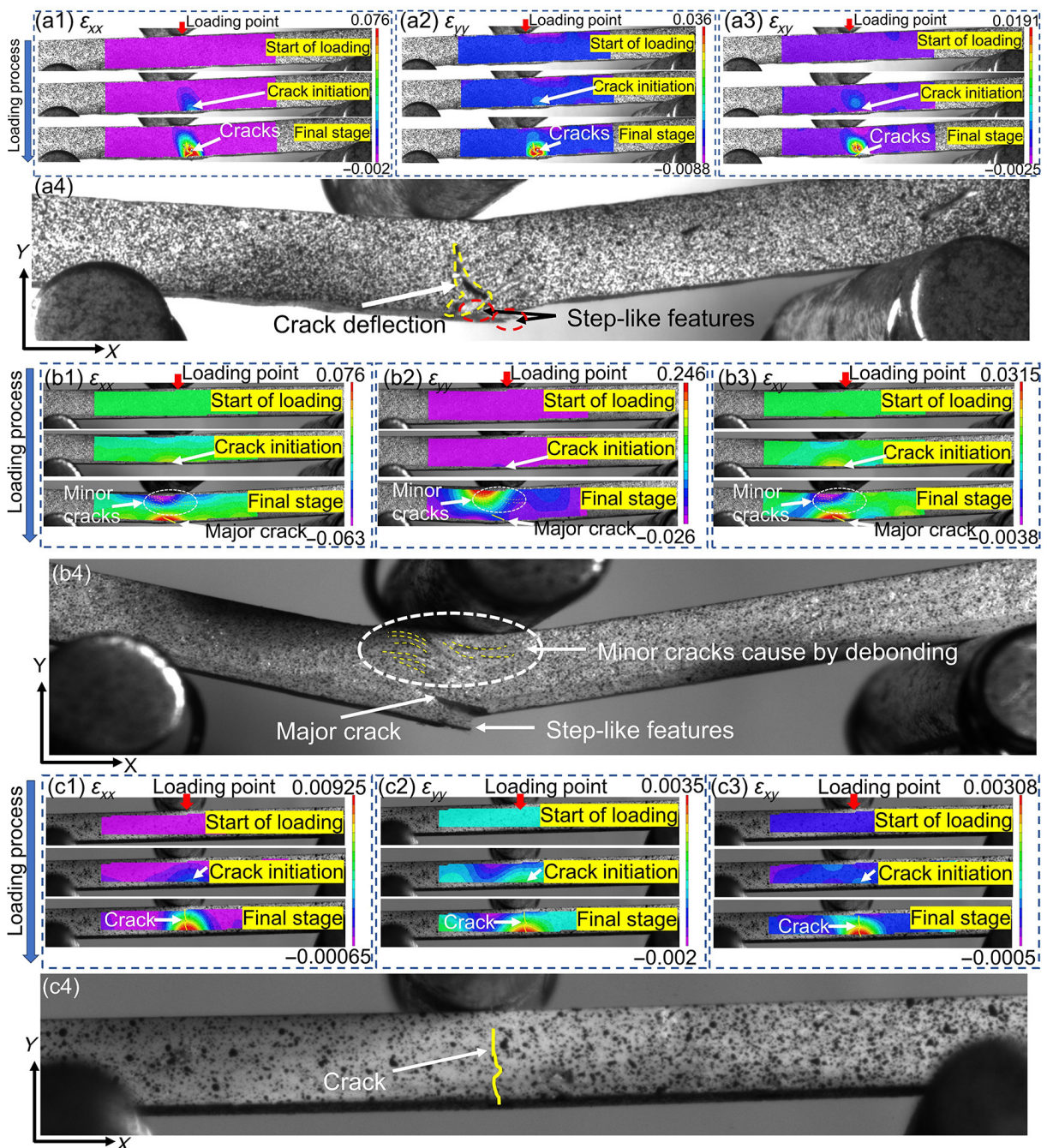


Fig. 16 DIC-derived strain fields during flexural testing and post-fracture morphologies of composites: (a1–a4) $\text{Si}_3\text{N}_4/\text{BN}$, (b1–b4) $\text{Si}_3\text{N}_4/\text{SiO}_2$, and (c1–c4) $\text{Si}_3\text{N}_4/\text{SiO}_2\text{-BN}$. For each composite, the first three panels show strains ε_{xx} , ε_{yy} , and ε_{xy} , respectively, while the fourth panel displays post-fracture sample.

observed by TEM (Figs. 8(c) and 11(a)) and suggests strong interfacial bonding. These observations are consistent with the stress–displacement curves and DIC strain field results discussed above. Furthermore, they are substantiated by the high interfacial shear strength determined via fiber push-in tests. Such strong interfacial bonding effectively restricts crack deflection and suppresses fiber pull-out, ultimately governing the macroscopic brittle failure of Si₃N₄/SiO₂–BN.

The differences in fracture behavior are related to the distribution of the matrix and porosity. As illustrated in Fig. 5, 3D renderings of phase-segmented XCT data visualize the spatial distribution of the fibers, matrices, and pores within the composites. In Fig. 5(a4), porosity in Si₃N₄/BN is mainly located in the BN matrix in the interbundle regions, while the intrabundle regions are relatively dense. This nonuniform structure weakens the interbundle regions. Consequently, under applied stress, cracks tend to initiate and propagate along these regions, leading to fiber bundle pull-out with limited filament pull-out (Figs. 17(a1)–17(a3)). Conversely, Fig. 5(b4) demonstrates that in Si₃N₄/SiO₂, the microporosity formed during the sol–gel process is more uniformly distributed in the SiO₂ matrix. Combined with relatively weak interfacial bonding, this promotes crack deflection between individual filaments and the matrix. This results in extensive individual fiber pull-out (Figs. 17(b1) and 17(b2)). As illustrated in Fig. 5(c5), during the fabrication of Si₃N₄/SiO₂–BN, borazine (B₃N₃H₆) fills the pores in the SiO₂ matrix and forms a dense hybrid matrix with low porosity. Together with strong interfacial bonding, this limits crack deflection in both the matrix and the fiber–matrix interface. Therefore, cracks propagate directly

through the matrix and fibers. This leads to the relatively flat fracture surface shown in Figs. 17(c1) and 17(c2), which is typical of brittle fracture.

Based on a comprehensive analysis of the stress–displacement behavior, the fracture modes of Si₃N₄/BN, Si₃N₄/SiO₂, and Si₃N₄/SiO₂–BN during flexural testing were proposed, as schematically illustrated in Fig. 18. In Fig. 18(a1), during the failure process of Si₃N₄/BN, cracks propagate along the fiber bundle direction under flexural loading. The fiber bundles are pulled out and fractured as a unit, with minimal pull-out of individual filaments and relatively short pull-out lengths (Fig. 18(a2)). In contrast, for Si₃N₄/SiO₂, in addition to the primary crack initiating on the tensile surface and propagating into the interior of the sample, multiple minor cracks form along the fiber alignment direction within the matrix (Fig. 18(b1)). Post-fracture observations reveal significant fiber pull-out with extended pull-out lengths, suggesting an energy-dissipating, damage-tolerant fracture mode (Fig. 18(b2)). For Si₃N₄/SiO₂–BN, cracks generated on the tensile surface propagate rapidly through the sample with minimal deflection (Fig. 18(c1)). The fracture surface appears smooth with limited fiber pull-out, indicative of typical brittle fracture behavior (Fig. 18(c2)). Taken together, the mechanical behavior of the three composites correlates closely with their interfacial characteristics: Weak or moderate interfacial bonding (Si₃N₄/SiO₂ and Si₃N₄/BN) allows fiber pull-out and higher damage tolerance, whereas the excessively strong bonding in Si₃N₄/SiO₂–BN, compounded by thermal residual stress, suppresses toughening mechanisms and leads to brittle failure with reduced strength.

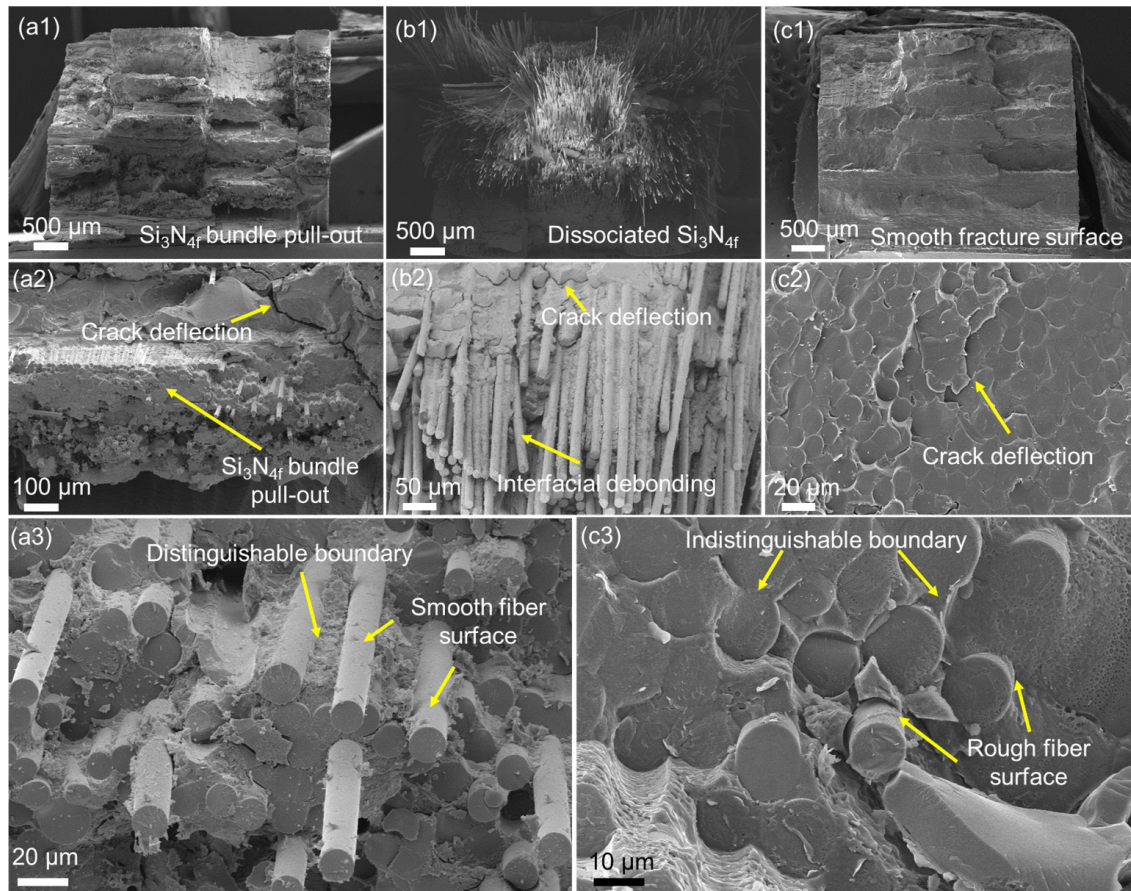


Fig. 17 Fracture surface morphology of (a1–a3) Si₃N₄/BN, (b1, b2) Si₃N₄/SiO₂, and (c1–c3) Si₃N₄/SiO₂–BN.

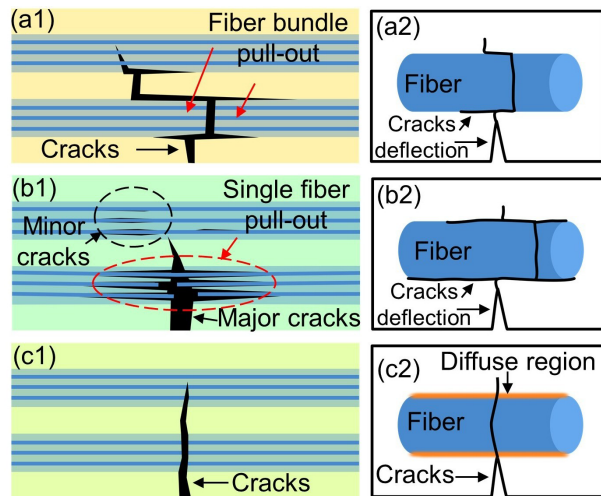


Fig. 18 Schematics of crack propagation in (a1, a2) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (b1, b2) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and (c1, c2) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$.

3.3 Dielectric properties

Dielectric properties are crucial parameters for wave-transparent materials. Figure 19 shows the dielectric constants and dielectric loss tangent values of the $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ composites in the 7–18 GHz frequency band from room temperature to 1100 °C.

At room temperature, all three composites exhibit excellent dielectric stability across the 7–18 GHz frequency range. The average dielectric constant of $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ is 4.03, with a dielectric loss tangent of 3.3×10^{-3} . For $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, the corresponding values are 3.81 and 4.2×10^{-3} , while $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ exhibits values of 4.13 and 2.7×10^{-3} , respectively. These results indicate that all three composites maintain low dielectric constants (< 4.5) and low dielectric loss tangents (< 0.005), with subtle variations influenced by the matrix composition. At room temperature, the dielectric constant follows the order $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN} > \text{Si}_3\text{N}_4\text{f}/\text{BN} > \text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$. The lowest dielectric constant of $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ is attributed to its highest porosity, as the dielectric constant of air in the pores is approximately 1, which reduces the effective dielectric

constant of the composite. In contrast, the dielectric loss tangent follows a slightly different trend: $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2 > \text{Si}_3\text{N}_4\text{f}/\text{BN} > \text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$. The relatively higher dielectric loss tangent of $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ is likely due to its high porosity (and correspondingly large specific surface area) coupled with the presence of residual silanol groups (Si-OH) in the SiO_2 matrix, which were introduced during sol-gel processes. These polar groups tend to absorb moisture under ambient conditions, leading to enhanced dipolar relaxation and interfacial polarization, which increase the dielectric loss tangent [53–56].

As shown in Fig. 19, when the test temperature increases from room temperature to 1100 °C, the dielectric constants of the three composites exhibit a mild temperature dependence. Specifically, the average dielectric constant of $\text{Si}_3\text{N}_4\text{f}/\text{BN}$ increases from 4.03 to 4.37; $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ increases from 3.81 to 4.08; and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ increases from 4.13 to 4.39. The slight increase in dielectric constant with temperature is primarily due to the enhanced thermal motion of molecules at higher temperatures, which facilitates dipole orientation in the applied electric field, thereby increasing polarization intensity and, consequently, the dielectric constant [57]. Notably, the dielectric loss tangents of the three composites remain very stable from room temperature to 1100 °C, with fluctuations of less than 0.001. Equation (4) [58] is commonly used to evaluate the temperature dependence of the dielectric constant.

$$\kappa = \frac{\Delta \epsilon_r}{\epsilon_r \Delta T} \quad (4)$$

where κ is the temperature coefficient, T is the temperature, and ϵ_r represents the dielectric constant. According to Eq. (4), the values of κ for $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$ are 7.9×10^{-5} , 6.3×10^{-5} , and $5.4 \times 10^{-5} \text{ } ^\circ\text{C}^{-1}$, respectively, all of which fall within the 10^{-5} range. This indicates that the three materials exhibit excellent dielectric stability at high temperatures. Thus, the three composites maintain stable dielectric responses over a wide frequency and temperature range, demonstrating promising potential for applications as high-temperature wave-transparent materials. At elevated temperatures ($\geq 200 \text{ } ^\circ\text{C}$), the dielectric loss tangent follows this trend: $\text{Si}_3\text{N}_4\text{f}/\text{BN} > \text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN} > \text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$. The reduced dielectric loss tangent in $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$ is

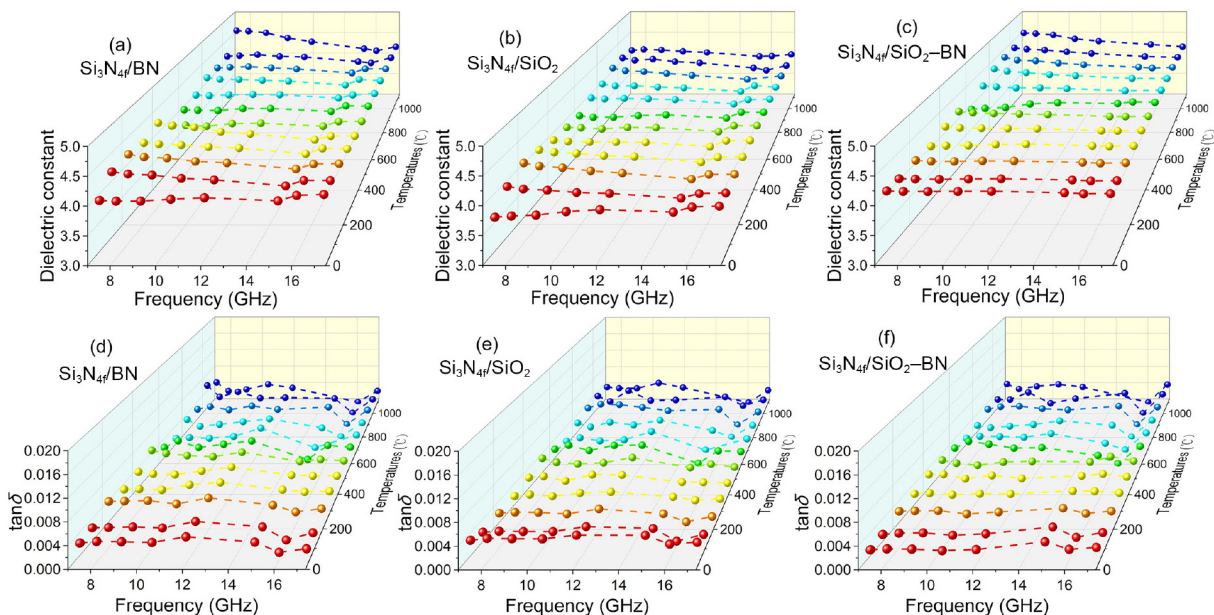


Fig. 19 Dielectric constant of (a) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (b) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and (c) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$; dielectric loss of (d) $\text{Si}_3\text{N}_4\text{f}/\text{BN}$, (e) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2$, and (f) $\text{Si}_3\text{N}_4\text{f}/\text{SiO}_2\text{-BN}$.

likely attributed to the desorption of moisture previously absorbed by silanol groups at elevated temperatures [59]. For Si₃N₄/BN, a noticeable increase in the dielectric loss tangent is observed above 1000 °C, which can be ascribed to the intrinsic rise in the dielectric loss of the BN matrix at such high temperatures [60]. As a hybrid system, Si₃N₄/SiO₂-BN exhibits an intermediate dielectric loss level between those of Si₃N₄/SiO₂ and Si₃N₄/BN at elevated temperatures. The introduction of the BN matrix via PIP fills the open pores within the SiO₂ matrix, thereby reducing the overall porosity. At the same time, reactions between the BN precursor and SiO₂ consume residual silanol groups. The combined effect of lower porosity and fewer polar silanol groups suppresses moisture absorption from the environment, leading to more stable dielectric loss values from room temperature up to 200 °C. The overall temperature stability of the dielectric response of the three composites is primarily governed by the intrinsically low polarizability of the constituent covalent ceramics and the persistence of this low-polarizability state at elevated temperatures. Specifically, Si₃N₄, SiO₂, and BN are all strong covalent compounds. These robust covalent bonds tightly constrain electron cloud distortion and ion displacement, resulting in a low macroscopic polarizability. Crucially, this strong constraint remains highly effective even at high temperatures, endowing these constituent materials with extremely low temperature coefficients of permittivity. The composites inherit this intrinsic trait, thereby maintaining stable dielectric performance across a wide temperature range. While pores and fiber-matrix interfaces inevitably exist within composites, their impact on temperature stability is limited. The pores within the composites are filled with air, whose dielectric constant is approximately 1 and essentially independent of temperature, thus introducing no additional temperature-dependent polarization mechanisms. Furthermore, the fiber-matrix interfacial regions, which are essentially diffusion zones, although varying in thickness among the three composites, are compositionally boron-silicon-oxygen-nitrogen amorphous phases with covalent bonding characteristics comparable to those of the fibers and matrices. Consequently, no enhanced interfacial polarization arises from the diffusion region, and its effect on the high-temperature dielectric response is negligible. These microstructural factors contribute only to the subtle differences in dielectric properties among the three composites, without altering their overall temperature stability. Notably, while the fiber-matrix interface significantly influences mechanical properties, its effect on dielectric performance is minimal within the studied systems.

4 Conclusions

The mechanical properties, fracture behaviors, and dielectric performance of the three Si₃N₄ fiber-reinforced composites (Si₃N₄/BN, Si₃N₄/SiO₂, and Si₃N₄/SiO₂-BN) were systematically investigated to understand the influence of matrix composition and fiber-matrix interfacial structure. The Si₃N₄/SiO₂-BN composite possessed the highest interfacial shear strength due to its thickest diffusion region, which resulted in excessively strong fiber-matrix bonding. Combined with the thermal expansion mismatch between the fiber and the matrix, this induced significant residual stress and brittle fracture, leading to lower flexural and compressive strengths. In contrast, Si₃N₄/BN exhibited the highest flexural strength, while Si₃N₄/SiO₂ demonstrated superior damage tolerance due to its higher porosity and extensive fiber pull-out. Despite differences in mechanical performance and interfacial microstructures, all three composites maintained low dielectric constants (< 4.5) and low dielectric loss tangents (< 0.005) up to 1100 °C, demonstrating

excellent high-temperature wave transparency. The use of BN and SiO₂ as matrix materials effectively reduced the dielectric constant and the dielectric loss tangent of the Si₃N₄ fiber-reinforced ceramic composites, while the influence of the fiber-matrix interface on the dielectric properties was minimal. These findings provide guidance for designing ceramic composites with balanced dielectric and mechanical properties.

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Author contributions

Yingpeng Zhang: writing — original draft, investigation, and conceptualization. Bin Li: writing — review and editing, supervision, funding acquisition, resources and conceptualization. Zhilin Tian: writing — review and editing, supervision, resources, and conceptualization.

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.

Electronic Supplementary Material

Supplementary materials (DIC videos showing the full-field strain evolution for the three composites (Videos S1–S9)) are available in the online version of this article at <https://doi.org/10.26599/JAC.2026.9221338>.

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