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Research Article

Phase-separation-enabled hollow silicon carbide composite nanofibers with multiscale interfaces for electromagnetic wave absorption over a wide temperature range

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Abstract:

Silicon carbide (SiC) nanofibers are attractive for electromagnetic wave (EMW) absorption in harsh environments, but the controllable construction of hollow architectures and the enhancement of dielectric loss remain challenging. Herein, phase-separation-enabled hollow SiC composite nanofibers with multiscale interfacial structures were developed to address these issues. By combining phase-field simulations with experiments, liquid-liquid phase separation was identified as the key factor governing radial compositional segregation and hollow-fiber formation, enabling controllable regulation of the phase constitution, defect density, and interfacial structure through the precursor ratio. The hollow architecture, together with the resulting multiscale interfaces and abundant defects, synergistically enhanced dielectric loss and improved impedance matching. At a PVP/PSN mass ratio of 2:1, the sample obtains a

minimum reflection loss of -56.11 dB at 1.53 mm, while the 1:1 hollow fiber delivered the broadest effective absorption bandwidth of 4.96 GHz at 1.39 mm. After incorporation into an AlPO_4 matrix, the optimized fibers enabled stable EMW absorption from 298 to 873 K, with a maximum effective absorption bandwidth of 4.08 GHz in the X-band. This work demonstrates a controllable route for constructing hollow silicon carbide-based fibrous absorbers and provides insight into multiscale interfacial design for EMW absorption over a wide temperature range.

Keywords: Hollow SiC composite nanofibers; liquid-liquid phase separation; electromagnetic wave absorption; multiscale interfacial structures; wide-temperature-range absorption.

1 Introduction

The rapid development of electronic information systems and radar detection technology imposes ever-increasing demands on electromagnetic wave (EMW) absorbing materials, which are expected to combine low density, thin matching thickness, broad absorption bandwidth, and strong attenuation capability with stable performance under harsh service conditions. This requirement is particularly critical for the skin structures of high-speed aircraft, which may experience drastic temperature fluctuations from room temperature to above 600 °C during operation. Under such conditions, absorbers must retain efficient EMW attenuation not only in the radar band but also over a wide temperature range [1,2]. However, conventional materials struggle to meet these demands: carbon-based materials suffer from oxidation and ablation at high temperatures, which degrades their dielectric loss capacity; ferrites and magnetic metals lose their magnetic properties above the Curie temperature, thereby eliminating magnetic loss; conductive polymers decompose directly, leading to a complete collapse of both conduction and absorption performance. In contrast, ceramic-based EMW

absorbing materials offer high-temperature resistance, high strength, strong absorption, and low density, positioning them as essential candidates for radar stealth technology [3,4].

Among ceramic-based EMW absorbing materials, SiC has emerged as an attractive candidate owing to its excellent physicochemical stability, yet its intrinsically low dielectric loss limits the absorption performance of pure SiC [5]. To overcome this limitation, several strategies have been explored. Morphological engineering enhances dielectric loss by extending propagation paths and introducing interfacial polarization through tailored architectures including nanoparticles [6], nanowires [7], foams [8], and aerogels, as exemplified by a gradient SiC/C aerogel achieving an 11.04 GHz effective bandwidth [9]. Compositional modification introduces highly conductive phases such as MXene or graphene derivatives to synergistically optimize impedance matching and reinforce interfacial polarization of the SiC network [10,11]. Beyond room-temperature optimization, the thermal and chemical stability of SiC also enables high-temperature EMW absorption. Kang et al. demonstrated this potential using electrospun submicron SiC fibers, which retained structural stability even at 1800 °C [12]. Furthermore, Gao et al. developed a SiC interphase within SiC_f/SiC/Mu composites and reported enhanced EMW absorption at elevated temperatures, achieving a bandwidth of 3.3 GHz [13]. Dai et al. reported stable X-band absorption from 298 to 873 K with SiC nanowire architectures [14]. Despite these advances, further structural and compositional design is still required to overcome the limited dielectric loss of SiC. Moreover, most existing studies focus on room-temperature absorption, whereas systematic investigations of absorption behavior over a wide temperature range remain limited.

Constructing multiscale interfacial structures is an effective strategy for improving the EMW absorption performance of SiC-based materials, because heterogeneous interfaces across different length scales can simultaneously optimize impedance matching and dielectric loss [15]. For instance, Zhang et al. reported SiO₂@SiC@C

microspheres with an $RL_{\min}$ of -64.2 dB and a 6 GHz EAB, benefiting from interfacial polarization at the multilayered boundaries [16], while Miao et al. achieved an $RL_{\min}$ of -47.96 dB and an EAB of 6.40 GHz by constructing hierarchical MnO_2/SiC heterointerfaces with oxygen vacancies [17]. Electrospinning offers a versatile platform for fabricating SiC nanofibers with intrinsic multiscale architectures [18], and various morphologies have been realized, including core-shell [19], hollow [20], and beaded [21] structures. Among these, hollow fibers are particularly attractive because the internal cavity introduces additional solid-void interfaces that improve impedance matching, as demonstrated by Yan et al., whose hollow SiC nanofibers delivered an EAB of 8.6 GHz [22]. Coaxial electrospinning, a conventional route to hollow fibers, suffers from complex equipment, difficult core-shell flow matching, and frequent needle clogging [23]. Tian et al. simplified this process by exploiting in-situ phase separation within a single spinning solution to produce hollow SiC fibers with a simple single-nozzle setup, although how solution properties and electrospinning parameters govern the phase separation behavior remains unclear, hindering precise structural control [24]. Current studies on electrospun SiC fibers focus predominantly on compositional modulation by introducing conductive or magnetic components, while structural regulation toward hollow fibers and its impact on EMW absorption remain largely unexplored.

While hollow SiC fibers fabricated by single-nozzle electrospinning via phase separation have been reported, this work focuses on the early-stage phase-separation mechanism and the resulting structural evolution of hollow fibers, and extends the study to wide-temperature EMW absorption. Specifically, combined phase-field simulations and experimental results reveal the liquid-liquid phase separation process underlying hollow structure formation, and clarify how the PVP/PSN mass ratio modulates the phase separation behavior and tailors the phase composition, hollow architecture, and multiscale interfacial structures. This composition-dependent structural modulation

enables synergistic optimization of dielectric loss and impedance matching, leading to strong EMW absorption and broad effective bandwidth. Moreover, the as-fabricated fibrous composite maintains stable EMW absorption across 298-873 K, with a maximum X-band effective absorption bandwidth of 4.08 GHz, demonstrating its potential for high-temperature EMW absorption scenarios.

2 Materials and methods

2.1 Raw materials

Polysilazane (PSN) was supplied by the Chinese Academy of Sciences (CAS). Polyvinylpyrrolidone (PVP, MW: ca. 1,300,000) was purchased from Macklin. N,N-dimethylformamide (DMF, $\geq 99.5\%$), anhydrous ethanol (EtOH, $\geq 99.5\%$), and alumina (Al_2O_3) powder ($\geq 99.9\%$) were supplied by Sinopharm Chemical Reagent Co., Ltd. Aluminum dihydrogen phosphate ($\text{Al}(\text{H}_2\text{PO}_4)_3$) solution (50 wt.% in H_2O) was purchased from Henan Borun New Materials Co., Ltd. Deionized (DI) water was produced in the laboratory.

2.2 Preparation of hollow SiC composite nanofibers

All spinning solutions were prepared at a fixed total solute concentration of 20 wt%, with their detailed compositions summarized in **Table S1**. Specifically, a solution labeled P1S1 was prepared by dissolving 1.0 g of PSN and 1.0 g of PVP in a mixed solvent comprising 5.6 g of ethanol and 2.4 g of DMF. The mixture was magnetically stirred at room temperature for 6 h to obtain a homogeneous spinning solution. To systematically investigate the effect of polymer composition, additional solutions were formulated at the same total solute concentration but with varying mass ratios of PVP to PSN (3:1, 2:1, and 1:2), while maintaining the identical solvent composition and total mass, and the resultant fibers were correspondingly named P3S1, P2S1, and P1S2. Prior to electrospinning, the solution was degassed under vacuum to eliminate entrapped air bubbles. The electrospinning was conducted using a 22G needle with an inner diameter

of 0.41 mm at 25-35 °C and 30-40% relative humidity. The tip-to collector distance was fixed at 12 cm, a positive high voltage of 21 kV was applied, and the syringe pump feeding rate was set to 0.01 mL min⁻¹. After 10 h electrospinning, a uniform white fibrous membrane precursor was obtained on the rotation drum.

The fibrous membrane was dried in an oven at 80 °C for 12 h to remove the unevaporated solvent. Subsequently, pre-oxidation was performed in a muffle furnace under ambient atmosphere: the sample was heated from room temperature to 180 °C at a ramp rate of 1 °C min⁻¹, held isothermally for 2 h, and then cooled naturally. Finally, carbothermal reduction was carried out in a tube furnace under an Ar atmosphere: the pre-oxidized fibers were first heated to 1000 °C at 5 °C min⁻¹, then ramped to 1400 °C at 3 °C min⁻¹ and maintained for 2 h, then cooled to 1000 °C at 3 °C min⁻¹ and allowed to cool naturally to room temperature.

2.3 Preparation of SiC/AlPO₄ composite

The P1S1 powder was dispersed in ethanol, ultrasonicated for 30 min, and dried at 60 °C to obtain well-dispersed nanofibers for subsequent use. Then, 0.2 g of the as-prepared nanofiber powder was mixed with 2.0 g of liquid aluminum dihydrogen phosphate and 1 g of alumina, stirred for 1 h, poured into a mold, and cured at room temperature for 24 h. After demolding, the sample was further cured in an oven at 80 °C for 12 h. Finally, the cured composite bulk was transferred to a tube furnace, heated to 600 °C at a rate of 3 °C min⁻¹ under Ar atmosphere, held at this temperature for 1 h, and naturally cooled to room temperature, yielding the final SiC/AlPO₄ composite (denoted as SA).

2.4 Characterization

The structure and phase composition of the powder were characterized by X-ray diffraction (Empyrean, Panalytical) using a Cu-K α radiation ($\lambda = 1.54 \text{ \AA}$) with a scanning rate of 8°/min over the 2 θ range of 10-90°. The morphology of the materials

was observed by a scanning electron microscope (SEM, Tescan Amber, Tescan) equipped with energy disperse spectroscopy (EDS, Eultimately Max30, Oxford). The morphology and structure were further observed, and elemental composition was analyzed, using an in-situ multi-functional transmission electron microscope (TEM, Talos F200X, ThermoFisher). Raman spectra were acquired using a 532 nm laser in a tip-enhanced laser confocal Raman spectroscopy system (in Via-Reflex, Renishaw) to probe the lattice vibration characteristics of the SiC phase and the graphitization degree as well as defect features of the carbon species within the composite fibers. The chemical bond states of the material were studied by X-ray photoelectron spectrometer (XPS, Escalab 250Xi, ThermoFisher) using a monochromatic Al K α X-ray source. The particle distribution in the solutions was characterized by dynamic light scattering (DLS) using a Malvern instrument. The viscosity of the solutions was measured with a viscometer (NDJ-8S, Lichen), and the electrical conductivity was tested using a conductivity meter (DDS-11A Shanghai Leici). Fourier transform infrared (FTIR) spectroscopy was performed on a Thermo Fisher Scientific Nicolet iS50 spectrometer, using attenuated total reflection (ATR) mode for the solutions.

The electromagnetic parameters of the samples were measured by a vector network analyzer (VNA, 3671D, Ceyear) with the test frequency range of 2-18 GHz. For measurements, the powders were mixed with paraffin at an absorber loading of 40 wt.% in the paraffin matrix (absorber-to-paraffin mass ratio of 40:60), and the mixture was pressed into toroidal-shaped specimens with an inner diameter of 3.04 mm and an outer diameter of 7.00 mm. The composite was characterized via the high-temperature waveguide method. SA samples were processed into standard waveguide blocks with dimensions of 28.86 mm $\times$ 10.16 mm $\times$ 3.5 mm, the waveguide cavity was heated from room temperature to 873 K at a rate of 10 K $\cdot$ min⁻¹, with an isothermal hold of 10 min at every 100 K interval, to acquire the dielectric parameters at various temperatures.

Phase-field simulation implemented in COMSOL Multiphysics was used to

illustrate hollow-fiber formation during electrospinning. Electromagnetic simulation was carried out in CST Microwave Studio. A 100 mm $\times$ 100 mm absorber (2.7 mm in thickness) was mounted on a PEC backplane. A normally incident linearly polarized plane wave with electric-field oriented along the X-axis was applied. All computational boundaries were set to open to mimic free-space conditions, and the scattering result at 7.2 GHz was obtained for electromagnetic scattering analysis.

3 Results and discussion

3.1 Phase composition and microstructure

Fig. 1 illustrates the fabrication of hollow SiC composite nanofibers via alcoholysis-induced in situ phase separation and carbonization. During solution preparation, EtOH selectively cleaves the Si-N bonds of PSN, yielding low-molecular-weight fragments. The resulting solution appears macroscopically homogeneous but undergoes microscopic phase separation due to the immiscibility between alcoholized PSN domains and PVP. Electrospinning amplifies this phase separation through the combined effects of a strong electric field and high-speed stretching, producing as-spun fibers with a radial composition gradient, featuring a PVP-rich center and a PSN-rich periphery. Upon pre-oxidation and carbonization, the PVP-rich center decomposes to produce free carbon and release gaseous products that diffuse outward to create a hollow interior. This as-formed carbon reacts with the PSN-rich periphery, converting it into a continuous SiC phase and ultimately yielding hollow SiC fibers.

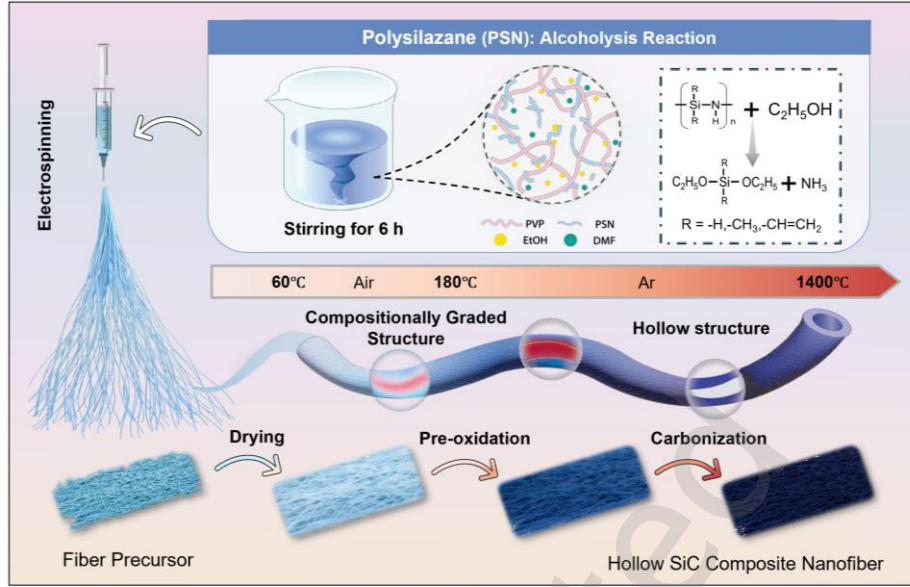


Fig. 1 Schematic illustration for the synthesis of hollow SiC composite nanofibers via alcoholysis-induced liquid-liquid phase separation and subsequent carbonization.

As depicted in **Fig. 2(a)**, the crystal composition and phase evolution of the electrospun composite nanofibers were investigated via XRD analysis. The characteristic diffraction peaks of all samples at 35.6° , 41.4° , 59.9° , 71.8° and 75.5° are indexed to the (111), (200), (220), (311) and (322) crystallographic planes of β -SiC; and the sharp peak at 33.5° is attributed to stacking faults, which are the predominant planar defects formed on the $\{111\}$ planes of β -SiC during the carbothermal reduction process [25]. Quantitatively, the $I_{SF}/I_{(200)}$ intensity ratio, an indicator of the stacking fault content in SiC, decreases remarkably with values of 1.85, 1.48, 1.25, and 0.89 as the PVP content decreases (**Fig. 2(c)**), a trend attributed to the diminishing free carbon released from the PVP during pyrolysis, which lowers the local carbon supersaturation and thereby suppresses the formation of stacking faults [26]. Consistently, for the P3S1, P2S1, and P1S1 samples, the broad 20° - 30° hump, characteristic of residual free carbon, gradually weakens, while SiC diffraction peaks grow progressively stronger. Notably, new diffraction peaks at 28.4° , 47.3° and 56.1° for P1S2 obtained at a reduced PVP/PSN mass ratio are assigned to cubic Si, which originates from Si enrichment under carbon-deficient conditions favoring Si-Si instead of Si-C bonding with

subsequent heating crystallizing Si-rich domains into nanoscale Si grains [27].

Fig. 2(b) shows the Raman spectra of composite nanofibers synthesized with different precursor ratios. The peaks at 792 cm^{-1} and 972 cm^{-1} are assigned to the transverse optical (TO) and longitudinal optical (LO) phonon modes of β -SiC [28], while the bands at 1345 cm^{-1} and 1590 cm^{-1} correspond to the D and G modes characteristic of a disordered carbon phase. A distinct peak at 507 cm^{-1} assigned to crystalline Si is observed in sample P1S2, while P3S1, P2S1 and P1S1 samples exhibit only the characteristic signals of β -SiC and amorphous carbon. To quantitatively assess the structural evolution of the carbon phase, the D and G bands were analyzed, where the D band corresponds to interfacial carbon defects, and the G band represents in-plane vibration of graphitic carbon. The I_D/I_G intensity ratio is generally employed to characterize the graphitization degree and structural defects of amorphous carbon, where a lower ratio indicates a higher degree of structural ordering in the carbon network, and the calculated values are 1.57, 1.48 and 1.41 for samples P3S1, P2S1 and P1S1 (**Fig. 2(c)**). The monotonic decline reflects a reduction in defect concentration and a concomitant improvement in local graphitic ordering, consistent with decreasing carbon content and enhanced structural ordering during pyrolysis.

XPS spectra were employed to characterize the elemental composition of the SiC composite nanofibers. The survey spectra shown in **Fig. 2(d)** reveal that Si, C, and O are the predominant elements across all samples. The high-resolution C 1s spectrum in **Fig. 2(e)** was deconvoluted into three different components: the peak at 283.2 eV assigned to C-Si bonds in the β -SiC phase, the peak at 284.6 eV attributed to C-C bonds in carbon, and the peak at 285.3 eV ascribed to C-O bonds. In **Fig. 2(f)**, the Si 2p spectrum shows two dominant peaks at 103.2 and 101.3 eV, corresponding to Si-O and Si-C bonds, respectively. Notably, a weak signal at 99.3 eV ascribed to Si-Si bonds is additionally detected in the P1S2 sample, corroborating the presence of crystalline Si identified by XRD. The O 1s spectrum (**Fig. S1**) shows a dominant peak at 532.6 eV

corresponding to the Si-O/C-O bond, confirming the formation of an amorphous SiOC phase as a minor interfacial component during raw material conversion [29,30]. Based on the above analysis, the composite nanofibers are composed mainly of crystalline β -SiC as the dominant phase, along with a certain amount of free carbon and a small fraction of amorphous SiOC. With increasing silane content, β -SiC shows improved crystallinity and reduced stacking-fault content. For the carbon phase, enhanced structural ordering is accompanied by an elevated degree of graphitization. The abundant heterogeneous interfaces formed by these phases, together with additional polarization centers from lattice defects in SiC and structural defects in the carbon network, collectively strengthen interfacial and dipole polarization, generate multiple dielectric relaxation processes, and benefit EMW absorption [31].

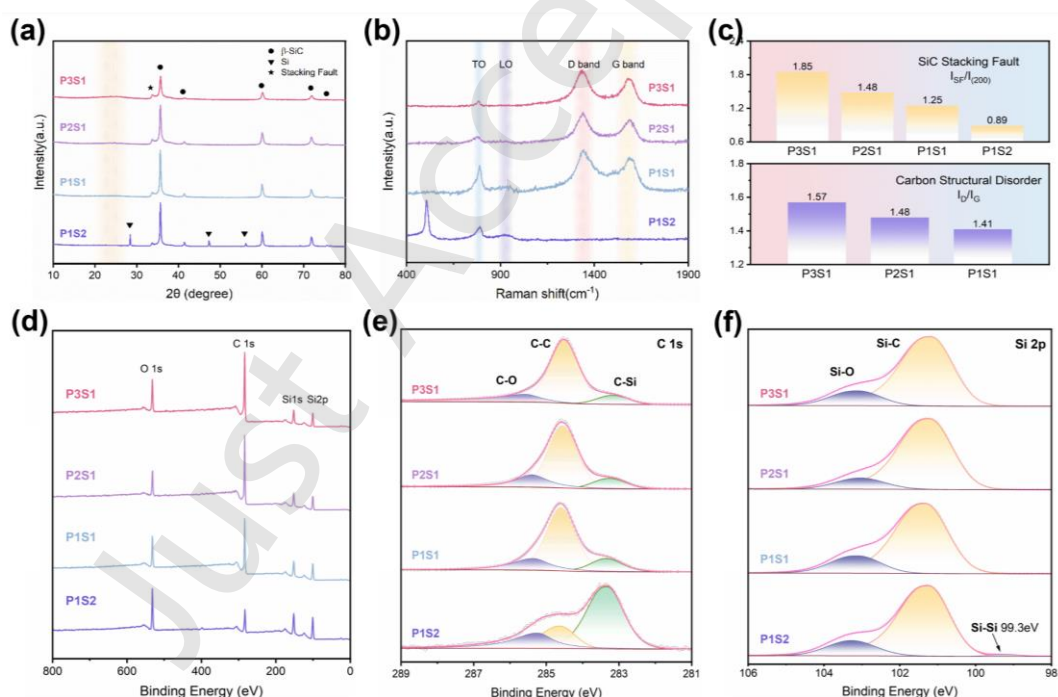


Fig. 2 Phase structure and chemical bonding characterization of P3S1, P2S1, P1S1 and P1S2. (a) XRD patterns. (b) Raman spectra. (c) Relative defect intensity of SiC stacking faults and carbon. (d) Full-range XPS survey spectra. (e) High-resolution C 1s spectra. (f) High-resolution Si 2p spectra.

Fig. S2 shows the smooth surface morphology of the as-spun precursor nanofibers and **Fig. S3** summarizes the evolution of fiber diameter both before and after carbonization, revealing a systematic decrease with diminishing PVP content at constant solute concentration, from 3.16 μm (P3S1), 2.06 μm (P2S1), 1.05 μm (P1S1) to 1.01 μm (P1S2), due to reduced polymer chain entanglement and solution viscosity [32]. Upon carbonization, all samples undergo substantial radial shrinkage, resulting in final diameters of 1.89 μm (P3S1), 1.26 μm (P2S1), 0.96 μm (P1S1), and 0.79 μm (P1S2). Meanwhile, the originally smooth surfaces become rough, as evidenced by SEM images in **Figs. 3(a1-a4)**. Specifically, P3S1 exhibits uniform, long fibers that form an interconnected network, while P2S1 yields shorter fibers. P1S1 develops a well-defined hollow architecture with a wall thickness of approximately 200 nm and a continuous channel along the fiber axis, featuring a smooth, intact surface free of collapse or delamination. In contrast, P1S2 retains only vestiges of fibrous morphology, with severe fragmentation dominating and leaving isolated wall fragments with exposed fracture surfaces. These results demonstrate that the PVP/PSN mass ratio serves as a precise lever for tailoring fiber microstructure, driving a progressive morphological transition from continuous fibers to disconnected short segments, then to robust hollow structures, and ultimately to fragmented remnants with discontinuous walls [33]. This transition is dictated by the carbon residue from PVP decomposition: at high PVP content, the abundant carbon binds SiC domains into a continuous, dense structure; as the carbon supply decreases, the weakening inter-domain connections first cause fiber fracture and then, at a moderate carbon level, yield well-defined hollow channels, where the residual carbon preserves wall integrity but is insufficient to fill the interior; with further carbon depletion, the framework loses cohesion and disintegrates into scattered fragments [34].

The samples were further characterized by transmission electron microscopy (TEM) coupled with energy-dispersive X-ray spectroscopy (EDS), as shown in **Figs.**

3(b₁-b₄) and **Fig. S4**. P3S1 exhibits a dense, fully solid fibrous morphology with a homogeneous distribution of Si, C, and O. P2S1 displays emergent contrast differentiation in brightness between the fiber periphery and central region, accompanied by localized Si enrichment at the wall. This brightness contrast intensifies markedly in P1S1, which features thinner walls, well-defined hollow cavities. In contrast, P1S2 shows severe structural degradation, including wall rupture, discontinuous grain boundaries, and loss of morphological coherence. **Figs. 3(c-d)** present TEM bright-field image and corresponding elemental mapping of P1S1, where the atomic fractions of C, Si, and O are 50.83%, 47.41%, and 1.75%, indicating a matrix predominantly composed of β -SiC, with minor free carbon and amorphous SiOC phases. The HRTEM image (**Fig. 3(e)**) reveals the microscopic phase composition of the fiber, which is dominated by β -SiC crystallites with intrinsic stacking fault defects, alongside limited localized amorphous SiOC around the grains and turbostratic free carbon embedded within these amorphous zones. The lattice spacings of the turbostratic free carbon and β -SiC nanocrystals in **Fig. 3(e)** were quantified, and the results are shown in Fig. S5. The adjacent amorphous regions show faint curved and band-like lattice features with an average interplanar spacing of 0.354 nm, characteristic of graphitic-like stacking in turbostratic carbon, while the β -SiC nanocrystals exhibit an interplanar spacing of 0.253 nm, consistent with the (111) crystal plane of SiC [35]. **Fig. 3(f)** shows the magnified HRTEM image of the stacking fault area in **Fig. 3(e)**. The corresponding FFT pattern (inset) exhibits distinct streaking tails perpendicular to the β -SiC (111) planes, a characteristic signature of stacking fault-induced translational disorder in SiC [36]. Although these fibers show sub-micrometer outer diameters, they possess abundant nanoscale structural features such as thin hollow walls, heterogeneous phase boundaries and lattice defects. These nanoscale features are key to their electromagnetic wave absorption performance.

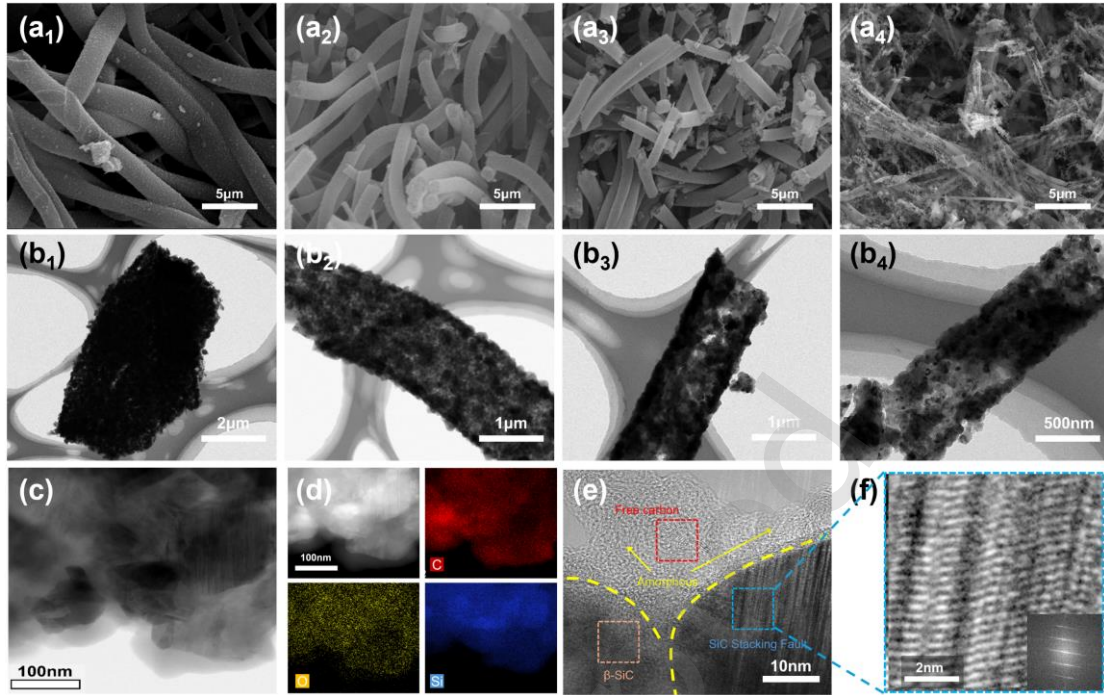


Fig. 3 Morphological and microstructural characterization of the SiC composite nanofibers. (a₁-a₄) SEM images and (b₁-b₄) TEM images of P3S1, P2S1, P1S1, and P1S2. (c) TEM bright-field image of P1S1. (d) EDS elemental mapping of panel (c). (e) HRTEM image of P1S1. (f) Enlarged HRTEM image of the stacking-fault region in panel (e) with the corresponding FFT pattern shown in the inset.

3.2 Formation mechanism of the hollow fiber morphology

The formation of hollow SiC fibers relies on a radial composition gradient in the as-spun precursor, with PVP enriched at the fiber center and PSN concentrated at the periphery. To understand how this segregation arises, the chemical evolution and solution properties of the PSN/PVP blends were investigated. In the EtOH/DMF mixed solvent, EtOH cleaves the Si-N bonds of PSN via alcoholysis, yielding small ethoxysilane molecules, while DMF enhances dissolution and spinnability. FTIR spectra (**Fig. 4(a)** and **Fig. S6**) confirm this transformation: the Si-N peak at 1159 cm^{-1} weakens after EtOH addition, and new Si-O-Si and Si-O-C peaks appear; upon PVP addition, the Si-H and Si-N peaks vanish, and the O-H band shifts and broadens, indicating hydrogen bond formation that promotes further alcoholysis [37]. DLS

measurements (**Fig. 4(b)** and **Fig. S7**) reveal that pure PVP exhibits a monodisperse distribution, whereas pure PSN displays a bimodal profile from alcoholysis-produced small silane molecules and their aggregates. All blends exhibit relatively high polydispersity (PDI) values above 0.3, suggesting pronounced compositional heterogeneity within the blend solution [38]. As the PVP content increases, the distribution transitions from bimodal to unimodal, indicating that PVP disrupts PSN aggregates and stabilizes the dispersed phase [39]. These structural differences translate into contrasting solution properties in **Fig. 4(c)**: pure PVP exhibits high viscosity (5398 mPa·s) and low conductivity (4.69 $\mu\text{S}/\text{cm}$), whereas pure PSN shows low viscosity (4.86 mPa·s) and higher conductivity (9.78 $\mu\text{S}/\text{cm}$). With increasing PSN content, viscosity declines as alcoholized PSN aggregates disrupt the PVP network. Conductivity remains above that of pure PVP but decreases gradually, suggesting that dispersed PSN aggregates hinder ionic mobility while introducing additional mobile ions. The obvious differences in viscosity and conductivity reflect poor intermolecular compatibility between PSN and PVP. According to the Flory-Huggins theory, such molecular incompatibility gives rise to a tendency toward thermodynamic immiscibility, even though the solution appears macroscopically transparent and may still possess molecular-scale compositional heterogeneity. During electrospinning, rapid solvent evaporation and jet stretching amplify local concentration differences, driving radial redistribution: the higher-viscosity PVP concentrates in the center while the lower viscosity PSN migrates to the surface, forming a gradient frozen upon solidification [40, 41]. This radially segregated architecture, with a PVP-rich center and a PSN-rich periphery, provides the structural template for the hollow SiC fibers obtained after thermal treatment.

To investigate the liquid-liquid phase separation behavior on the fiber cross-section, we performed COMSOL phase-field simulations using the Flory-Huggins free energy as the thermodynamic driving force. In the **Electronic Supplementary**

Materials, we conducted parameter sensitivity analyses for the thermodynamic parameter, interface mobility, and interface width (**Figs. S8-S10**), and determined appropriate values for these parameters. Based on this, three typical samples P2S1, P1S1, and P1S2 were selected for simulation. The simulation time was set to 2 ms after extrusion because the electrospinning jet remains fluidic during the first 2 ms after leaving the needle. This time window is critical for phase separation and is consistent with the electrospinning jet travel time [42].

The phase-separation evolution was quantified by the time-dependent order parameter variance (**Fig. S11**), which reflects the extent of compositional fluctuation during the process. P1S1 is the closest to the critical composition and therefore is expected to exhibit the strongest thermodynamic tendency for phase separation. Consistent with this expectation, P1S1 shows the most pronounced and fastest evolution, whereas P2S1 and P1S2 exhibit weaker and delayed evolution, respectively. At the same time, the actual evolution rate is also modulated by the viscosity of the dominant phase. As shown in **Fig. 4(d)**, P1S1 combines a near-critical composition with moderate viscosity, leading to the fastest development of radial compositional redistribution within 1 ms. In contrast, P2S1 shows only weak evolution even at 2 ms, mainly because the high viscosity of the dominant PVP phase retards phase separation. P1S2 behaves differently: although its thermodynamic tendency for phase separation is weaker, the lower viscosity of the dominant PSN phase still allows delayed evolution, which becomes more evident by 2 ms. As solvent evaporation proceeds, the increasing viscosity gradually freezes the radial compositional redistribution and arrests further phase separation. Taken together, the blend composition governs the thermodynamic tendency, while the viscosity of the dominant phase controls the kinetics, and these two factors jointly determine the radial segregation process [43].

Fig. 4(e) presents the SEM image and elemental line scans of the P1S1 fiber precursor cross-section, revealing a radial composition gradient with Si content

increasing from the center toward the edge and C showing the opposite trend. To trace the structural evolution from precursor to hollow fiber, the thermal decomposition of P1S1 and pure PSN was compared by TG/DSC in **Fig. 4(f)**. Pure PSN shows a single minor weight loss and a smooth DSC curve, with a weak exotherm above 800°C corresponding to its amorphous-to-crystalline transition. P1S1 exhibits multi-stage behavior: a weak endotherm at 50-400 °C from decomposition of PVP functional groups [44], followed by rapid weight loss near 450 °C with an exotherm from bulk PVP decomposition and interfacial reaction with PSN-derived silicon species. Above 800°C, the TG curve plateaus, indicating complete release of volatiles and a residual mass of silicon-based inorganic phases and free carbon. At this stage, the PVP-rich center has largely decomposed to create a hollow interior, while the PSN-rich periphery forms an inorganic framework with relatively thick walls, as confirmed by the SEM image of the 800 °C-treated fiber (**Fig. S12**). Upon further heating to 1400 °C, the free carbon reacts with the surrounding silicon-based phase to form SiC, leading to gradual wall thinning.

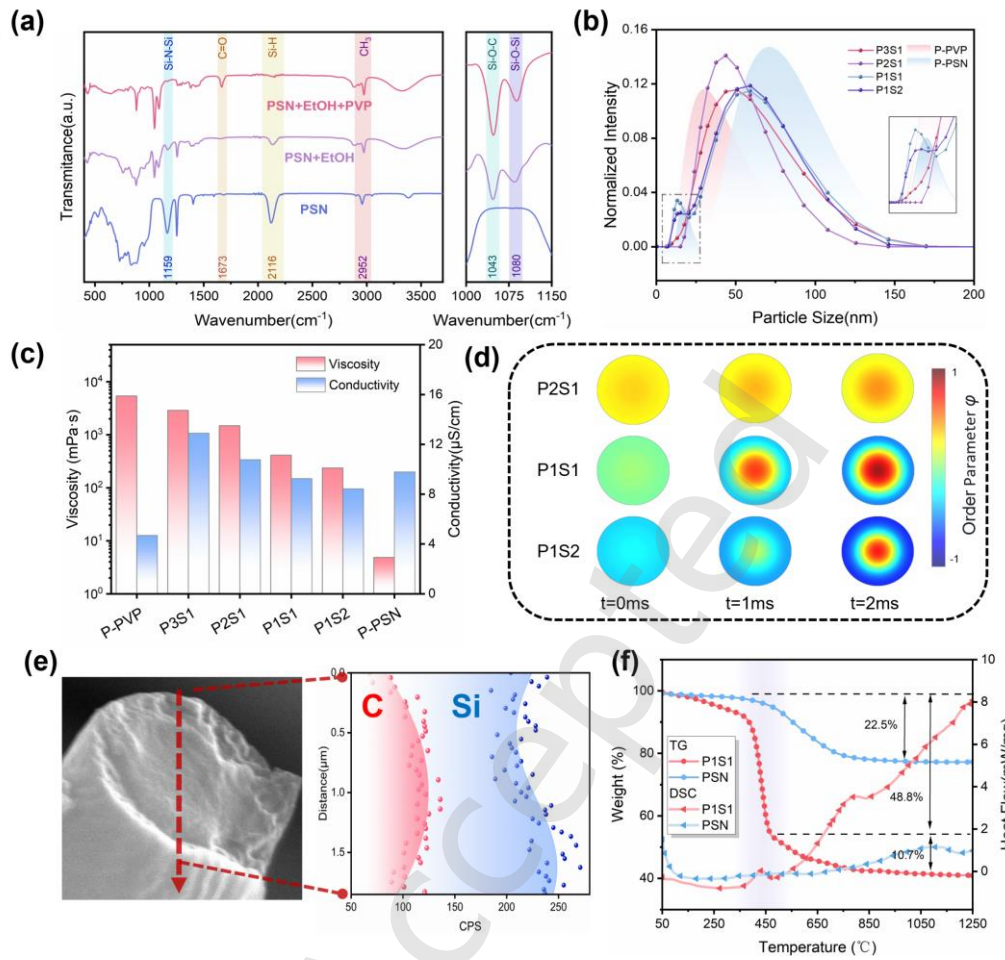


Fig. 4 Formation mechanism analysis of hollow fibers. (a) FTIR spectra of different solutions. (b) DLS size distributions of different PVP/PSN ratio. (c) Viscosity and conductivity of the solutions. (d) Phase-field simulation of fiber cross-sections in the jet region ($\phi=1$ for PVP phase, $\phi=-1$ for PSN phase). (e) Line scan results of the fiber precursor. (f) TG and DSC curves of P1S1 and PSN.

3.3 EMW absorption performance

The electromagnetic parameters of the four samples were measured using a vector network analyzer (VNA) over the frequency range of 2-18 GHz. The EMW absorption performance at thicknesses from 0.5 to 5.5 mm was calculated based on Eq. (1) and Eq. (2).[45]

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left(j \frac{2\pi f d}{c} \sqrt{\epsilon_r \mu_r} \right) \quad (1)$$

$$RL = 20 \lg \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right| \quad (2)$$

Herein, Z_{in} denotes the input impedance of the absorber, Z_0 is the characteristic impedance of free space, ϵ_r and μ_r represent the complex relative permittivity and permeability, respectively, f is the frequency of the incident wave, c is the velocity of microwaves, and d is the thickness of the material.

Figs. 5(a1-d1) show the evolution of RL with thickness and frequency for different composite nanofibers. The P3S1 sample exhibits poor absorption, with a minimum RL of only -20.14 dB at 1.11 mm and narrow bandwidth. P2S1 achieves an RL_{min} of -56.11 dB at 1.53 mm, and P1S1 reaches -53.91 dB at 2.66 mm. However, P1S2 requires a much larger matching thickness (5.43 mm) to achieve an RL_{min} of -50.96 dB, indicating a performance degradation. This behavior follows the quarter-wave cancellation mechanism [46], where the optimal RL of P1S2 at different thicknesses aligns with the $1/4\lambda$ and $3/4\lambda$ curves, necessitating large thickness for efficient absorption. In contrast, P3S1, P2S1 and P1S1 all work well at low thicknesses. The EAB extracted from **Figs. 5(a2-d2)** provides further insight. Over the 0.5-5.5 mm thickness range, P3S1 achieves a maximum EAB of 3.11 GHz, with the effective band mainly covering 7-18 GHz. By varying the matching thickness, P2S1 and P1S1 can achieve effective absorption across nearly the entire 2-18 GHz range. At a fixed thickness, P1S1 attains a higher maximum EAB (4.96 GHz at 1.39 mm) than P2S1 (3.55 GHz at 1.15 mm). In comparison, the EAB of P1S2 is only 2.11 GHz, which is the lowest among all samples. Impedance matching analysis (**Fig. S13**) reveals the origin of these performance differences. The normalized input impedance $|Z_{in}/Z_0|$ varies with microstructural evolution: P1S1 possesses an impedance value closest to 1, while P3S1 and P1S2 deviate far below and above 1, respectively, indicating severe mismatch. The Smith charts (**Fig. 5(e)**) at 1.4 mm corroborate this: P3S1 fluctuates away from the center, P2S1 shows improved convergence, and P1S1 displays the broadest, most centered trajectory, accounting for its improved absorption performance [47]. P1S2,

though concentrated near the center, covers a narrow region, limiting its bandwidth. Furthermore, the RCS of SiC composite nanofiber samples at 7.2 GHz was simulated to evaluate their angular-dependent EMW attenuation. Angle-dependent curves in **Fig. 5(f)** reveal that all samples exhibit significantly lower RCS than the PEC reference, indicating efficient energy consumption and scattering suppression. As summarized in **Table S3**, P1S1 and P2S1 compare favorably with most reported SiC-based absorbers in terms of matching thickness, reflection loss, and effective absorption bandwidth.

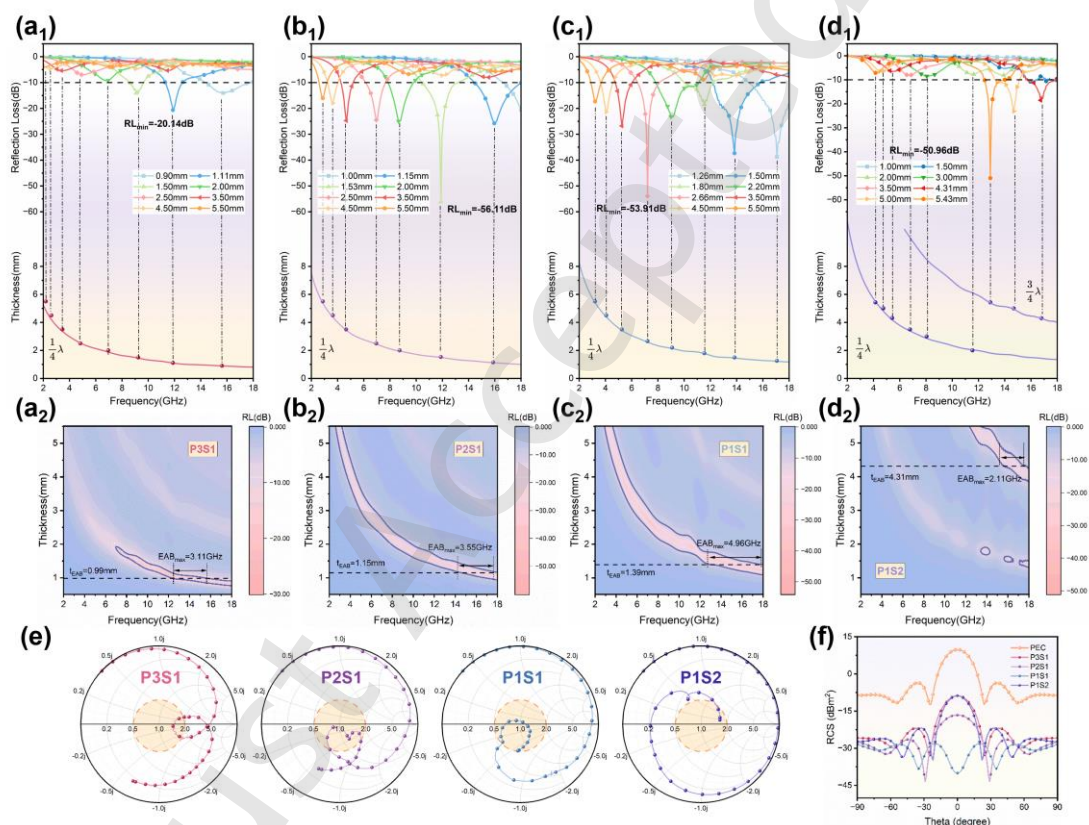


Fig. 5 EMW absorption performance of P3S1, P2S1, P1S1 and P1S2. (a1-d1) One-dimensional RL curves with quarter-wavelength matching lines. (a2-d2) RL contour maps. (e) Smith charts at a thickness of 1.4 mm. (f) Simulated radar cross-section (RCS) of different samples at 7.2 GHz.

To investigate the intrinsic mechanism for the difference in EMW absorption properties, the relevant electromagnetic parameters were analyzed. Given the absence of magnetic components in this system, the real and imaginary parts of the relative

permeability are nearly constant around 1 and 0, allowing the real permittivity (ϵ') and the imaginary part (ϵ'') to be the focus. As shown in **Figs. 6(a-b)**, all four samples present high permittivity, benefiting from the high intrinsic dielectric constant of SiC and the continuous fiber network for charge migration and accumulation. P3S1 has the highest permittivity due to its complete fiber structure. The gradually intensified hollow characteristic across P2S1, P1S1 and P1S2 reduces the effective dielectric medium and progressively lowers permittivity. Furthermore, severe structural fragmentation of P1S2 further depresses both ϵ' and ϵ'' , yielding the lowest permittivity among all samples. In the 8-18 GHz range, the ϵ'' curves of all samples do not show a continuous downward trend with increasing frequency, but exhibit obvious fluctuations and multiple relaxation peaks, indicating the presence of interfacial polarization loss [48].

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \quad (3)$$

$$\alpha = \frac{\sqrt{2}\pi f}{c} \times \sqrt{(\epsilon''\mu'' - \epsilon'\mu') + \sqrt{(\epsilon''\mu'' - \epsilon'\mu')^2 + (\epsilon'\mu'' + \epsilon''\mu')^2}} \quad (4)$$

The microwave attenuation ability was further evaluated using the dielectric loss tangent ($\tan\delta$) and attenuation constant α , as calculated from Eq. (3) and Eq. (4). Plots of the $\tan\delta$ versus frequency are presented in **Fig. 6(c)**. For P3S1, P2S1, and P1S1, $\tan\delta$ decreases with increasing frequency in the low-frequency range (2-8 GHz), with P3S1 showing the highest initial value and steepest decline. At high frequencies, their responses converge, fluctuating narrowly between 0.3-0.4. **Fig. 6(d)** displays the attenuation constant α as a function of frequency for all samples: P3S1 shows the highest values, while P2S1 and P1S1 follow nearly identical fluctuating trends. Notably, P1S2 exhibits the lowest values in both $\tan\delta$ and α across the entire measured range.

The Cole-Cole plots in **Figs. 6(e-h)** exhibit multiple distinct semicircles. Each semicircle ideally represents a polarization relaxation process, with its shape and size reflecting the relaxation strength and characteristic frequency [49]. The low-frequency tail is characteristic of conduction loss: a larger slope indicates stronger conduction loss

from free carrier migration, while the slight tilt arises from residual polarization loss. The slopes indicated by the arrows in the figure follow the order P3S1, P2S1, P1S1, indicating that conduction loss is strongest in P3S1 and becomes nearly negligible in P1S2. Apart from the conductive tail, all samples exhibit a large low-frequency semicircle and several small high-frequency semicircles, corresponding to multiscale polarization relaxations in which larger microscale interfaces dominate the low-frequency response [50,51]. The low-frequency semicircle reflects the Maxwell-Wagner-Sillars (MWS) polarization at submicron interfaces including fiber agglomerates, fiber/matrix boundaries and fiber wall/cavity interfaces, and such polarization weakens as fiber continuity and structural integrity decline [52]. The small high-frequency semicircles correspond to two distinct nanoscale relaxation processes: interfacial polarization among β -SiC, free carbon and amorphous SiOC, and defect dipole polarization originating from lattice and carbon defects.

Overall, the dielectric loss mainly consists of conduction loss and polarization loss. To quantitatively evaluate the contributions, a modified Cole-Cole model incorporating a broadening factor γ was employed to decompose the ε'' [53]:

$$\varepsilon^*(\omega) = \varepsilon_{\infty} + \frac{\varepsilon_s - \varepsilon_{\infty}}{1 + (j\omega\tau)^{\gamma}} + \frac{\sigma}{j\omega\varepsilon_0} \quad (5)$$

where ε^* is the complex permittivity, ε_{∞} and ε_s are the high-frequency and static permittivity limits, respectively, τ is the relaxation time, and σ is the electrical conductivity. The first two terms describe polarization loss, while the third term accounts for conduction loss. Based on this decomposition, the conduction loss (ε_c'') decreases with increasing frequency in **Fig. 6(i)**, which is consistent with following the order of the low-frequency tail slopes in the Cole-Cole plots and the measured DC conductivity (**Fig. S14**). The polarization loss (ε_p'') fluctuates between 1 and 4 across the measured frequency range in **Fig. 6(j)**, with distinct relaxation peaks indicating multi-scale interfacial polarization. The broadening factor γ reflects the distribution

width of relaxation times and serves as an indicator of interfacial heterogeneity: a smaller γ indicates a broader relaxation-time distribution and stronger interfacial heterogeneity. P1S1 yields the lowest γ (0.5453) among all samples, which is primarily attributed to the additional wall/cavity interfaces introduced by its hollow architecture, combined with multiple phase boundaries and lattice defects [54]. In the low-frequency region (2-8 GHz), polarization from submicron interfaces weakens with declining fiber continuity, yet the extra interfaces in P1S1 raise its polarization above P2S1. In the high-frequency region (8-18 GHz), P3S1, P2S1, and P1S1 show comparable polarization responses, which may arise from the combined contribution of nanoscale interfacial polarization and defect-related dipolar relaxation [55,56]. P1S2 exhibits the weakest response due to severe fiber fragmentation and the similar dielectric properties of SiC and Si, which limit interfacial charge accumulation. **Fig. 6(k)** tracks the ratio of polarization loss to total dielectric loss, showing a progressive transition from conduction loss dominant to polarization loss dominant behavior as the PVP/PSN mass ratio in raw material decreases. As frequency increases, the conduction-loss contribution decays more rapidly because free carriers cannot fully follow the alternating electric field. Meanwhile, interfacial polarization and dipolar relaxation remain active within the measured range, so polarization loss becomes progressively dominant at higher frequencies [15,57].

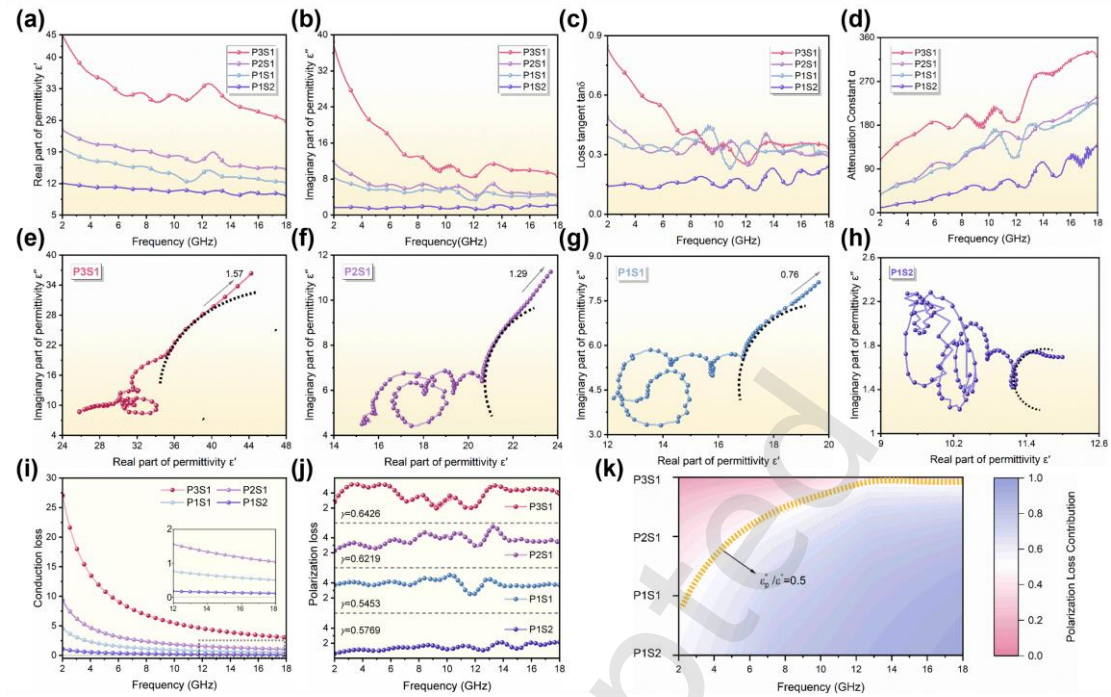


Fig. 6 Dielectric properties and loss mechanisms of P3S1, P2S1, P1S1 and P1S2. (a) Real part of permittivity. (b) Imaginary part of permittivity. (c) Dielectric loss tangent. (d) Attenuation constant. (e-h) Cole-Cole plots. (i) Conduction loss. (j) Polarization loss (k) Ratio of polarization loss to total dielectric loss.

P1S1 exhibits excellent EMW absorption performance and is therefore selected for high-temperature testing. The porous AlPO_4 matrix was adopted for its high-temperature stability and low dielectric permittivity (**Fig. S15**). These properties ensure that the measured electromagnetic response at elevated temperatures originates from the embedded SiC nanofibers rather than the matrix itself. SEM (**Fig. S16**) reveals that the SiC fibers are uniformly distributed within this porous network and XRD (**Fig. S17(a)**) confirms the formation of crystalline SiC and an AlPO_4 matrix.

Figs. 7(a-b) present high-temperature X-band (8.2-12.4 GHz) waveguide measurements of the SiC/ AlPO_4 composite. These conditions differ from prior room-temperature coaxial-ring tests on SiC/paraffin, so their dielectric parameters cannot be directly compared and show different frequency dependences. At room

temperature, the ε' shows a relaxation-type decrease with frequency, while ε'' increases slightly, raising the dielectric loss tangent. This behavior arises from interfacial polarization at the SiC/AlPO₄ boundaries. Compared with SiC/paraffin, the AlPO₄ system provides stronger interfacial polarization with a longer relaxation time and a lower-frequency relaxation peak, leading to an increase in ε'' with frequency in the X-band. With increasing temperature, both ε' and ε'' increase significantly while maintaining similar frequency dependence, and $\tan\delta$ rises monotonically. This gradual enhancement aligns with the dielectric behavior observed in SiC-based EMW absorption materials [58,59], where elevated temperatures promote charge carrier migration, electron hopping in free carbon, and interfacial polarization, collectively boosting the dielectric response without altering the frequency trend.

In the SiC/AlPO₄ composite (SA), the primary electromagnetic loss phase is the SiC composite nanofibers with multiscale interface structure, while the AlPO₄-based matrix mainly serves as a wave-transparent, shape-forming, and high-temperature stabilizing support. As temperature increases from 298 K to 873 K, both ε' and ε'' show a progressive enhancement. The increase is most pronounced between 373 K and 473 K, where ε' rises by ~5% and ε'' by ~12%, reflecting thermal activation of additional polarization processes at heterointerfaces. From 473 K to 773 K, both parameters continue to rise but at a slower rate. At 873 K, ε' still increases, whereas ε'' exhibits a slight decline in the low-frequency region but remains comparable to the 773 K level at higher frequencies, suggesting that polarization-related losses are largely sustained even at the highest temperature. This behavior may be associated with the oxidation-induced depletion of the carbon phase, as supported by the structural evolution of SA before and after high-temperature treatment in air (**Fig. S17**), which shows that the SiC nanofibers are retained, while the carbon-related signal decreases and the O signal increases. Since the carbon phase plays a major role in the conduction-loss pathway, its thermal evolution leads to a reduction in the low-frequency ε'' , whereas the high-

frequency ε'' , which is mainly governed by polarization loss, is less affected. Overall, the progressive enhancement of permittivity with temperature confirms that thermally activated polarization across multiple heterointerfaces governs the dielectric response over the entire temperature range [60].

To further clarify the underlying mechanism, the Cole-Cole plots are analyzed in detail, as shown in **Fig. 7(c)**. Multiple semicircles shift to the upper right with increasing temperature, arcs extend longer and become more dispersed, indicating dominant polarization relaxation with multiple relaxation types at elevated temperatures. The fiber/matrix interfaces, together with the intrinsic multiscale interfaces within the fibers, contribute to these diverse relaxation processes. The relaxation factor κ is introduced:

$$\kappa = \frac{\partial \tan \delta}{\partial \omega} \quad (6)$$

Fig. 7(d) presents a temperature-frequency contour map of κ , where κ values are visualized via color gradients. In this representation, dashed lines marking $\kappa=0$ denote transitions between different relaxation states, and each peak-valley pair in the κ distribution corresponds to an individual relaxation process [61]. As temperature increases, more $\kappa=0$ transition boundaries gradually emerge across the measured frequency range, indicating that additional relaxation state transitions are triggered at elevated temperatures. Meanwhile, the difference between the peaks and valleys in the κ map grows with rising temperature, indicating intensified relaxation strength. This enhancement reflects that higher temperatures activate charge carriers at the abundant multi-scale heterointerfaces within the composite, enabling stronger polarization responses at the existing relaxation sites and thereby intensifying the relaxation processes manifested in the κ distribution. Collectively, these thermally activated interfacial relaxation effects contribute to electromagnetic wave attenuation over a broad temperature range [62].

$$Z_n = \sqrt{\frac{\mu_r}{\varepsilon_r}} \quad (7)$$

Fig. 7(e) illustrates the temperature-dependent evolution of the average attenuation constant and normalized intrinsic impedance (Z_n), which is defined in Eq. (7). The attenuation constant increases markedly with rising temperature, which arises from the progressive activation of multi-scale interfacial relaxation and the enhancement of dielectric losses, leading to more efficient electromagnetic energy dissipation. The attenuation constant declines at 873 K, likely due to high-temperature-induced surface oxidation that suppresses some interfacial relaxation and conductive loss pathways. Meanwhile, the normalized intrinsic impedance decreases gradually with increasing temperature, as permittivity rises significantly while permeability changes little, causing the impedance to deviate further and leading to a slight degradation in impedance matching. However, the average normalized impedance remains above 0.36 across the temperature range, still sufficient for effective wave incidence, and the interplay between this stable impedance matching and the enhanced attenuation from thermally activated interfacial polarization collectively governs the high-temperature absorption performance.

Based on the measured dielectric parameters at each temperature, the RL curves were calculated after thickness optimization. **Figs 7(f-g)** present the optimized EMW absorption performance of the sample at different temperatures. Within the wide temperature range of 298-873 K, the SA composite maintains stable EMW absorption performance at matching thicknesses of 2.6-2.8 mm and shows a gradual improvement with increasing temperature, with only a slight decline at 873 K. The optimized RL_{min} reaches -82.14 dB at 673 K, and the EAB expands to 4.08 GHz at 773 K, nearly covering the entire X-band. Such temperature-induced performance enhancement stems from optimized electromagnetic parameters, which improve impedance matching and reduce matching thickness. Although a slight decay in performance is observed at 873 K, the composite still retains an RL_{min} of -63.29 dB and an EAB of 3.63 GHz at this temperature, indicating stable EMW absorption over a wide temperature range. After

two-month ambient storage, the stored sample exhibits nearly identical complex permittivity to pristine SA and retains an EAB of 3.12 GHz at 3.16 mm (Fig. S18), confirming its environmental stability and long-term durability. As summarized in Fig. 7(h), the SA composite delivers advantages in high-temperature performance, $RL_{\min}$, and $EAB_{\max}$ compared with recently reported absorbers [63-69], demonstrating its potential as a high-performance high-temperature electromagnetic wave absorbing material.

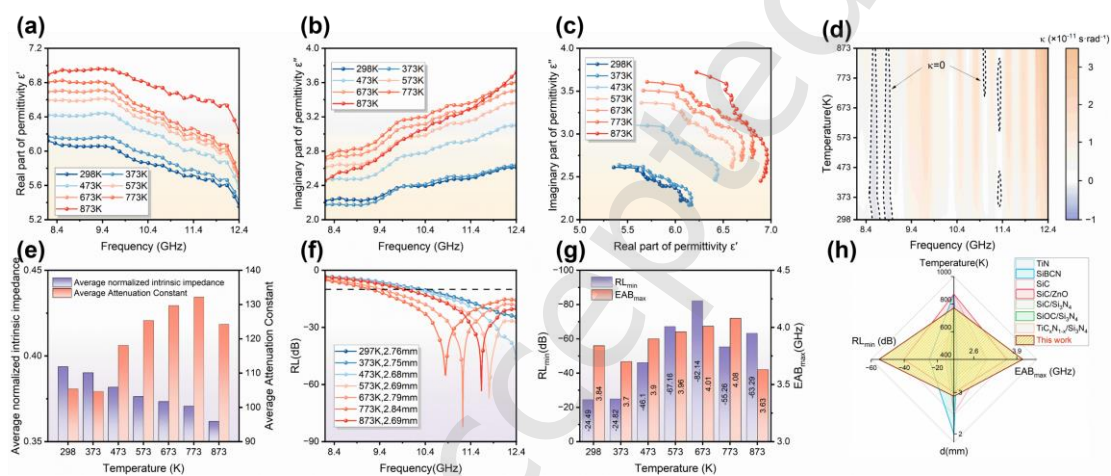


Fig. 7 High-temperature EMW absorption performance of SA. (a-b) Temperature-dependent permittivity. (c) Cole-Cole plots. (d) Relaxation factor κ . (e) Average of attenuation constant and normalized intrinsic impedance. (f) RL curves at different temperature. (g) Performance summary. (h) Radar chart for comprehensive performance comparison against reported high-temperature absorbers.

Fig. 8 illustrates the multiscale interfacial design and EMW absorption mechanism of hollow SiC composite nanofibers. To optimize impedance matching, multiscale interfaces across micron, nano and atomic scales are introduced to deliver diverse loss pathways. The interwoven micron-scale fiber network extends the propagation path via multiple scattering, facilitating efficient coupling of incident electromagnetic waves into the material interior, and provides conductive loss through electron migration across the inter-fiber carbon-containing network; at the nano scale, the hollow fiber

architecture with SiC as the dominant phase, accompanied by minor amorphous SiOC and free carbon, generates interfacial polarization from charge accumulation at phase boundaries; at the atomic scale, lattice stacking faults in SiC and structural defects in carbon induce dipole polarization that further enhances high-frequency loss. These multiscale interfaces constitute the material basis for broadband absorption over a broad temperature range. With increasing temperature, interfacial polarization relaxation is progressively activated, increasing both the number and intensity of relaxation processes and establishing polarization as the dominant loss mechanism, while the stable fiber architecture ensures efficient absorption from room temperature to elevated temperatures.

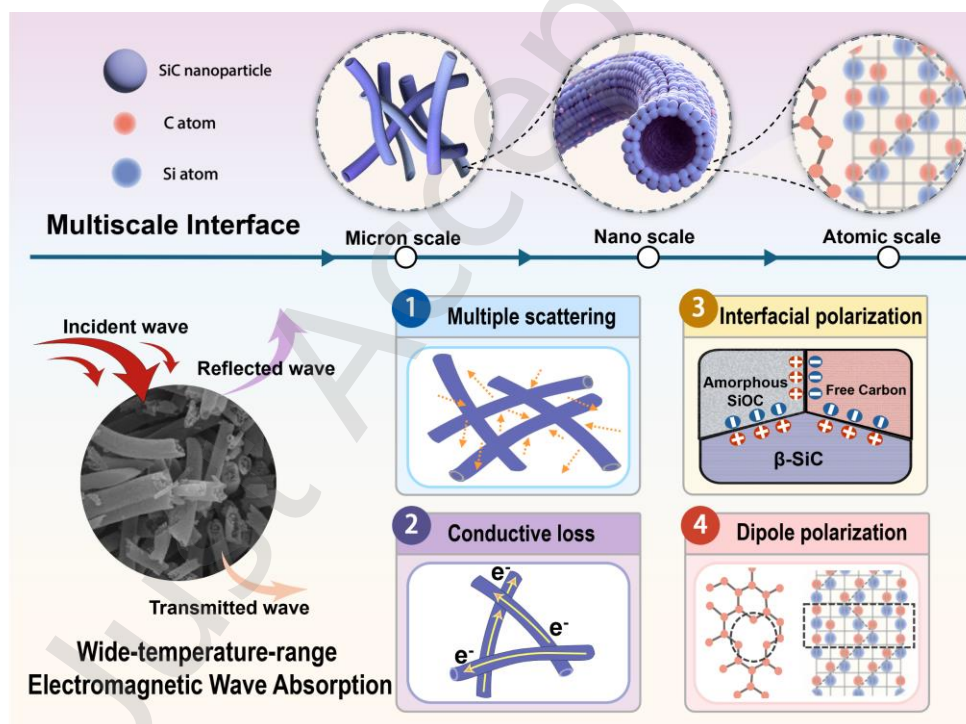


Fig. 8 Schematic illustration of multiscale-interface design and EMW absorption mechanism for hollow SiC composite nanofibers.

4 Conclusions

Alcoholysis of PSN and its immiscibility with PVP drive liquid-liquid phase separation during electrospinning. Phase-field simulations and experiments show that

the PVP/PSN ratio controls this separation, producing a radial gradient with a PVP-rich center and a PSN-rich periphery. After carbothermal reduction, the final structure evolves from dense fibers to hollow fibers and finally to fragmented remnants as the PVP/PSN mass ratio decreases. This ratio also tailors the phase composition and defect structure of these fibers. β -SiC is the dominant crystalline phase, with amorphous SiOC and free carbon as minor phases, and the densities of SiC stacking faults and carbon-related defects decrease with decreasing PVP content. Within the fibrous network, these phases and defects create multiscale interfaces that improve impedance matching and provide multiple loss pathways, leading to efficient electromagnetic energy dissipation and a room-temperature EAB of 4.96 GHz at a thickness of 1.39 mm. At elevated temperatures, thermally activated interfacial polarization further contributes to electromagnetic attenuation, while the stable fibrous architecture ensures consistent absorption from 298 to 873 K, delivering an EAB of 4.08 GHz and an $RL_{\min}$ of -82.14 dB in the X-band. Overall, this study provides a controllable strategy for constructing hollow silicon carbide-based fibrous absorbers and offers insight into the phase evolution and structure-property relationship governing EMW absorption over a wide temperature range.

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Lan Wang, Lin Zhu: Software, Formal analysis, Validation;
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Jinyu Wang, Yingzhi Liu, Haobo Shi: Investigation, Methodology, Formal analysis;
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Availability of data and materials

The data that support the findings of this study are available in this article.

Competing interests

The authors declare no conflict of interest

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