

Thermostable and stretchable core-shell aerogel fibers for thermal insulation application

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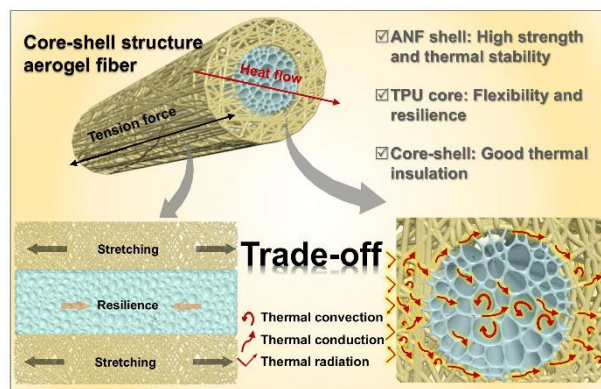
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A core-shell aerogel fiber with a rigid ANF network shell and flexible TPU foam core was fabricated via an ambient pressure drying assisted coaxial wet spinning to resolve the conflict between insulation and strength. It achieves ultralow thermal conductivity ($29.82 \text{ mW} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$), tensile strength of 8.9 MPa, 59.3% elongation, stretch recovery and thermal stability from -196°C to 200°C .

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ABSTRACT: Aerogels are among the most suitable thermal insulation materials for next generation textiles, yet they face an inherent structural conflict between high porosity for insulation and a dense network for mechanical strength. Herein, we propose a core-shell aerogel fiber architecture to resolve this conflict via a functional partitioning strategy. Using a coaxial wet-spinning method assisted by ambient-pressure drying, we fabricate aerogel fibers with a shell of aramid nanofiber (ANF) network and a core of thermoplastic polyurethane (TPU) foam. This hierarchical gradient porous structure integrates nano/micro-scale pores, endowing the core-shell aerogel fibers with an ultralow thermal conductivity of $29.82 \text{ mW} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ and superior thermal insulation performance from -196°C to 200°C . The TPU core provides flexible support and stretchability, delivering a tensile strength of 8.9 MPa , an elongation at break of 59.3% , and excellent stretch recovery performance to the aerogel fibers. Meanwhile, the thermostable ANF shell layer ensures thermal stability and structural integrity of the aerogel fibers under extreme thermal conditions. Therefore, the ANF@TPU core-shell aerogel fibers developed in this work achieve a favorable balance among thermal insulation, mechanical properties, and thermal stability, demonstrating promising potential in extreme temperature applications.

KEYWORDS: aerogel fibers; core-shell structure; thermal insulation; mechanical properties; thermostability

1 Introduction

Aerogels, distinguished by their intrinsic three-dimensional (3D) porous architecture, ultra-low density, and low thermal conductivity, represent a transformative advancement in thermal insulation materials [1-3]. The conversion into a fibrous form further endows them with weavability, facilitating the upgrade of aerogels from bulk materials to wearable textiles [4, 5]. In extreme environments such as aerospace and fire scenarios [6, 7], wearable materials are often required to possess multifunctional properties including thermal conductivity [8, 9] or insulation [10, 11], intelligence [12, 13], and multi-functionalization [14, 15]. Moreover, they must maintain excellent thermal insulation performance and mechanical stability under extreme temperature variations [16, 17]. These requirements are the key driver accelerating the development of aerogel fibers [18, 19]. However, the high porosity severely compromises the mechanical strength of aerogel fibers by minimizing the solid area available to bear loads [20-22]. Therefore, the central challenge for developing thermostable aerogel fibers is simultaneously achieving a porous structure for thermal insulation and a dense network for mechanical properties [23-25].

In recent years, aramid nanofiber (ANF) aerogels, produced

via the deprotonation and re-protonation of commercial Kevlar, have emerged as an effective solution to these challenges, owing to their rigid, oriented molecular chains, excellent thermal stability, and 3D nanofiber network structure [26, 27]. However, despite sharing an identical molecular structure with conventional Kevlar, the hydrogen bond interactions of ANF in nanofiber networks formed by re-protonation is still relatively weak, resulting in the mechanical properties of ANF aerogels failing to meet the requirements for practical applications [28-30]. For this reason, various methods such as chemical cross-linking [31, 32] or physical structure designs [33, 34] have been proposed to enhance the mechanical properties. For example, Sun et al. [35] adopted a slow-proton-release-modulating gelation and thermo-induced crosslinking strategy in the preparation of ANF aerogels, achieving a low thermal conductivity and high compressive recovery performance. Gong et al. [36] employed unidirectional freeze-casting-enabled microstructure orientation and acid-assisted hydrogen bond formation to facilitate nanofiber cross-linking during the preparation of ANF aerogels. The resultant framework demonstrated remarkable specific tensile strength ($89 \text{ MPa} \cdot \text{cm}^3 \cdot \text{g}^{-1}$), toughness ($1.3 \text{ MJ} \cdot \text{m}^{-3}$), and fracture energy ($7.36 \text{ kJ} \cdot \text{m}^{-2}$). Our group [37] also reported ANF aerogel fibers through Ca^{2+} crosslinking and rapid air-drying methods, achieving excellent tensile

strength (13.5 MPa) and toughness ($7.0 \text{ MJ}\cdot\text{m}^{-3}$) while retaining a low thermal conductivity. Nevertheless, the inherent structural weakness of the nanofiber network itself restricts the effectiveness of chemical cross-linking in strengthening the ANF aerogels.

To overcome this limitation, optimizing the physical structure of aerogels becomes the crucial factor [1, 38, 39]. Key strategies employed in current researches include improving molecular orientation [1], tailoring skin-core structures [38, 39], etc. For instance, Zhang et al. [1] employed liquid crystal spinning during the preparation of ANF aerogel fibers to enhance the orientation of internal ANF, which led to the production of aerogel fibers featuring extremely high mechanical strength (41.0 MPa) and excellent thermal insulation performance ($0.037 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$). Liu et al. [40] utilized freeze spinning to form a skin-core structure when preparing aerogel fibers, attaining high specific tensile strength ($775.8 \text{ MPa}\cdot\text{cm}^3\cdot\text{g}^{-1}$) and low thermal conductivity ($30.4 \text{ mW}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$). However, in terms of the tensile recovery performance required by aerogel fibers in practical applications, these above methods seem difficult to achieve it.

Core-shell structure design may be the key strategy to resolve the conflict between tensile recovery performance and thermal insulation in aerogel fibers [41, 42]. Currently, the literatures on core-shell structured aerogel fibers primarily focus on two aspects: one is leveraging the hierarchical pore architecture of the core-shell configuration to optimize the tortuosity of heat conduction pathways, thereby enhancing thermal insulation performance; the other is utilizing the dense shell layer to improve the mechanical properties of the aerogel fibers. However, despite their practical significance, the tensile elasticity of these fibers, an important property in real-world applications, has rarely been reported in existing studies. Zhuang et al. [41] and any other researchers engineered core-shell structure aerogel fibers, but most of them did not mention the tensile elasticity (Table S1). The core concept lies in the functional partitioning of mechanical properties and thermal insulation, enabling their synergistic enhancement. A typical example from nature is the polar bear hair, which exhibits a classic core-shell structure: the hollow core with high porosity effectively provides excellent thermal insulation, and the dense shell provides efficient mechanical protection. Inspired by this, Bai et al. [43] incorporated a core-shell structure into the aerogel fiber design. After coating aerogel fibers with thermoplastic polyurethane (TPU) shell layers. The TPU shell could prevent the porous structure of core layers from collapsing due to force or environmental erosion and endow aerogel fibers with super stretchability up to 1000% strain. Meanwhile, the aerogel fiber can retain its stable thermal insulation property after 10,000 stretching cycles at 100% strain. However, due to relatively low thermal stability of TPU, the utilization of TPU as shell layers rendered aerogel fibers unable to withstand high-temperature environments.

In this work, we report a thermostable and stretchable shell-core structural aerogel fiber with ANF as the shell and TPU as the core, prepared by coaxial wet spinning assisted by ambient pressure drying (APD). Using boric acid as the

coagulating bath and incorporating a small quantity of silane coupling agent promotes abundant hydrogen bonding, strengthening ANF networks and minimizing structural collapse of aerogel fibers during APD, with a shrinkage rate of merely 23%. Due to the hierarchical pore structure and excellent thermal stability of ANF shell layers, the obtained ANF@TPU aerogel fibers (ATAFs) exhibit a thermal insulation ability with low thermal conductivity of $29.82 \text{ mW}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ within the temperature range from -196 to 200°C . In an actual test at 200°C , the temperature difference across the single-layer textile can reach 67.8°C . More importantly, as the core layers, TPU endows aerogel fibers with excellent stretch recovery performance, tensile strength (8.9 MPa), and elongation at break (59.3%). Therefore, ATAFs integrate excellent thermal and mechanical properties, demonstrating significant potential in personal thermal management textiles for extreme temperature environments.

2 Experimental

2.1 Materials

Poly(p-phenylene terephthamide) (PPTA, Kevlar K49) fibers were offered by DuPont China. Thermoplastic polyurethane (TPU) pellets (Elastollan 1185A) were supplied by BASF Co., Ltd. Dimethyl sulfoxide (DMSO, AR, $\geq 99\%$) were supplied by Aladdin Biochemical Technology Co., Ltd. Potassium hydroxide (KOH, AR), boric acid (BA, AR, $\geq 99.5\%$), N, N-Dimethylformamide (DMF, AR, $\geq 99.5\%$), N-(3-(trimethoxysilyl) propyl)-2-ethanediamine (KH792, 95%), Ethanol (EtOH, AR, $\geq 99.7\%$) and n-hexane (AR, $\geq 99\%$) were procured from Sinopharm Chemical Reagent Co. Ltd. All chemicals were used as received without any further treatment.

2.2 Preparation of ANF and TPU Dispersion

ANF dispersion was synthesized by deprotonation of aramid microfibers (PPTA) according to the reported method. 6.24 g of Kevlar fibers, 9.36 g of KOH dissolved in 6 mL of DI water and 150 mL of DMSO were added to a 250 mL zirconia tan. Subsequently, the mixture was ball-milled in a planetary ball mill at 700 rpm for 4 h to obtain the dispersions of reddish-brown ANF with a concentration of 40 mg/mL. Then, the obtained dark red ANF dispersion was purified by high-speed centrifugation at 8000 rpm for 10 min to remove undissolved KOH. By keeping the volume of DMSO constant and controlling the weight of Kevlar fibers and KOH, ANF dispersions with concentrations of 20, 30 and 40 mg/mL were obtained for further use. Besides, TPU pellets were added to a mixture of DMF and DMSO (with a volume ratio of 1:1) and magnetically stirred at room temperature for 24 h until they were completely dissolved to obtain a uniform precursor solution with a concentration of 200 mg/mL.

2.3 Preparation of ANF@TPU Aerogel Fiber

Firstly, the ANF and TPU dispersions were extruded through a coaxial needle (model 15/22) into the coagulation bath (DI water) by a pump-controlled syringe at a spinning speed of 1 mL/min. Then the hydrogel fibers were sequentially immersed in ethanol and n-hexane for solvent replacement.

After that, the wet fibers were dried in an indoor environment at room temperature and ambient pressure to obtain aerogel fibers, which were named ATAF-H₂O. For comparison, when DI water was instead by the boric acid aqueous solution, the aerogel fibers were prepared in the same way and named ATAF-B. Besides, KH792 was added to the ANF solution and mixed evenly, and then the aerogel fibers were prepared in the same way of ATAF-B and named ATAF-BSi. In addition, ATAFs prepared with different concentrations of ANF are named ATAF_x, where x represents the concentration of ANF.

2.4 Characterizations

The morphologies of samples were observed by a JSM-6510 scanning electron microscope (SEM, acceleration voltage of 15 kV). The elemental distribution was characterized using an energy-dispersive X-ray spectrometer (JEOL Ltd.) in combination with SEM. The specific surface area and pore size distribution of aerogel fibers were determined with the Brunauer-Emmett-Teller (BET) method and the Barrett-Joyner-Halenda (BJH) nitrogen adsorption and desorption method, respectively (ASAP 2460, Micromeritics, USA). Fourier Transform infrared spectrometer (FTIR, NICOLET 6700) in the wave number range of 650-4000 cm⁻¹ and X-ray photoelectron spectrometer (XPS, Thermo ESCALAB 250XI) with Al K α anodes were used to record the chemical evolution of aerogel fibers.

Mechanical properties were tested in the tensile mode by a universal testing machine (AG-X Plus) with a controlled loading rate at 5 mm/min. To analyze the mechanism, the internal orientation of aerogel fibers before tensile tests, during tensile fracture, and after being placed for 7 days was studied by Wide-angle X-ray diffraction (WAXD) on a Bruker D8 Discovery X-ray setup equipped with a Cu-K α source, and the X-ray was perpendicular to the axial direction of fiber incidence.

Thermal conductivity was measured directly on a Hot Disk-2500S instrument using the transient planar source method. The surface temperature changes of individual aerogel fibers and textile samples woven from fibers during the thermal protection experiment were recorded by an infrared thermal imaging camera (E60, FLIR). The heat insulation capacity was studied by differential scanning calorimetry (DSC Q2000, TA Instruments) at a heating/cooling rate of 10 °C/min under a nitrogen atmosphere. Thermal stability was tested by thermogravimetric analysis (TG, Netzsch STA 209F3) in a nitrogen atmosphere with a heating rate of 20 °C/min.

3 Results and discussion

3.1 Preparation of ANF@TPU Aerogel Fibers

To meet the demanding conditions of extreme temperature environments, textiles must possess key properties including high thermal insulation, broad-temperature stability, and flexibility with stretchability. Core-shell structured aerogel fibers, through functional zoning and coordinated structural design, offer a promising solution. In this work, the ANF network forms shell layers that maintain thermal stability and structural integrity across a wide temperature range of -

196 to 200 °C, effectively resisting extreme thermal cycling and minor impact. The inner core of TPU foam provides flexible support and ensures stretchability during joint movement. Crucially, the engineered core-shell gradient porous structure integrates ANF nanofiber networks with TPU microporous foams, creating a multi-scale porous architecture from nano- to micro-level. This structure significantly suppresses the thermal convection of air molecules, extends heat transfer paths and enhances phonon scattering, thereby delivering efficient thermal insulation for the aerogel fibers.

Figure 1a illustrates the scalable and continuous preparation process of ATAFs, including coaxial wet spinning, sol-gel transition, solvent exchange, and ambient pressure drying. Firstly, the ANF/DMSO dispersion (Figure S1) was prepared via ball milling-assisted deprotonation according to our previous work [44]. To reinforce the structural integrity of ANF networks, N- β -aminopropyl- γ -aminopropyl trimethoxysilane (KH792) was incorporated into the dispersion to enhance hydrogen bonding interactions. After that, the prepared ANF/DMSO dispersion and TPU/DMSO/DMF dispersion were simultaneously extruded as shell and core layers, respectively, into a saturated boric acid aqueous solution via a coaxial needle (Figure 1bi). Once in contact with coagulation bath, the shell ANF dispersion underwent rapid re-protonation, completing the sol-gel transition. During this process, the boric acid solution supplied abundant protons, which facilitated the reconstruction of hydrogen bonds between ANFs to achieve rapid re-protonation. Meanwhile, the silane coupling agent underwent rapid hydrolysis, generating abundant silanol groups. These groups then formed a dense hydrogen-bond network by interacting with both the amide groups on ANFs and the borate ions (Figure 1bii). Moreover, due to the encapsulating effect of the ANF shell layers, the aqueous solution gradually permeated into the core TPU dispersion, enabling controlled phase separation. Subsequently, the hydrogel fibers underwent sequential solvent replacement with low-surface-tension ethanol and n-hexane (Figure 1biii), which can effectively reduce collapse pressure according to Young-Laplace Equation, thereby preserving the porous architecture during drying [45]. By diminishing Laplace pressure and lowering surface tension, n-hexane facilitated diffusion along the nanofibers. Finally, the core-shell structured ANF@TPU aerogel fibers (ATAFs) with minimal shrinkage were successfully obtained by drying wet fibers at room temperature and ambient pressure (Figure 1biv). Due to the core-shell structure with strong shell layers and soft core layers, the ATAFs exhibit remarkable flexibility and strength, being capable of withstanding rolling, bending, and knotting (even at the micrometer scale) without any impairment (Figures 1c-d). Additionally, ATAF maintains favorable mechanical flexibility even at the extremely low temperature of liquid nitrogen (Figure 1e). Considering the continuity of wet spinning and the rapidity of ambient pressure drying, the ATAFs also have the potential for large-scale production and can be weaved (Figure 1f).

3.2 Structure and Morphology of ANF@TPU Aerogel Fibers

During the re-protonation process, the hydrogen bond network density directly determines the skeleton strength of ANF aerogels [46-48], thereby influencing shrinkage during ambient pressure drying and the final mechanical properties of aerogel fibers. Firstly, the hydrogen bond interactions between ANF and different proton donors were simulated via density functional theory (DFT, see S1. in Supplementary Material for details). The electrostatic potential maps of all molecules are presented in **Figure 2a**. Generally speaking,

regions with high electrostatic potential and those with low electrostatic potential are more prone to form hydrogen bonds and other interactions [49]. From the binding energy of ANF with H_2O , H_3BO_3 , and KH792 (Figure 2b, Table S2), the system containing both ANF, H_2O , H_3BO_3 , and KH792 (ANF- H_2O -BSi) exhibits the lowest binding energy among all combinations, demonstrating the strongest and most stable interaction among these components [33]. The independent gradient model based on Hirshfeld partition [50] (IGMH)

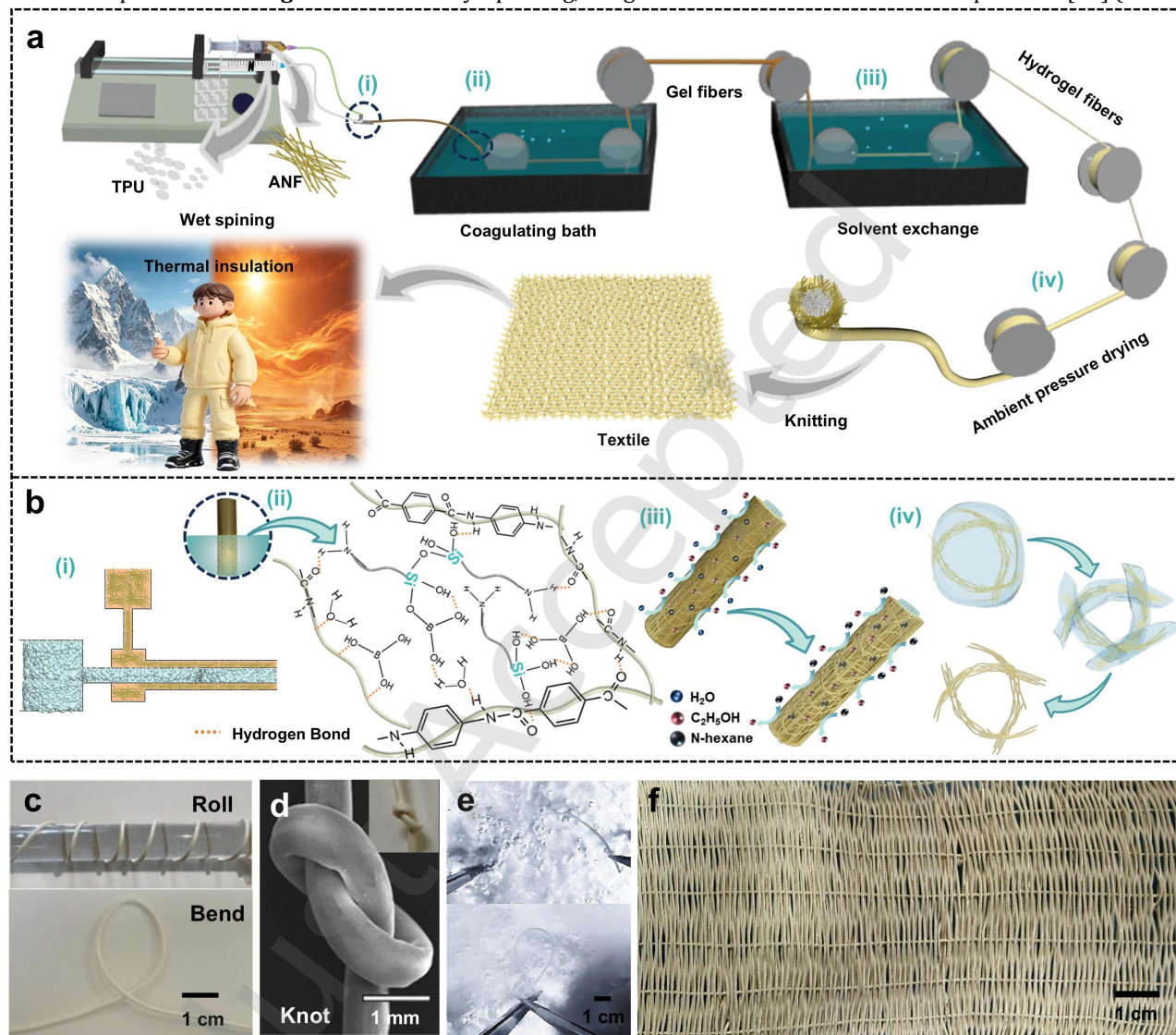


Figure 1. (a) Schematic illustration of preparation process of ATAFs and textile. (b) Schematic diagram of the formation mechanism of ATAFs. (c) Photographs of ATAF undergoing rolling and bending. (d) SEM image of ATAF undergoing knotting. (e) Photographs of ATAF maintaining flexible in liquid nitrogen, and (f) large-area fabrics woven from ATAFs by hand.

reveals that the ANF- H_2O -BSi system demonstrates stronger hydrogen-bonding interactions compared to ANF- H_2O and ANF- H_2O -B systems (Figure 2c). The detailed molecular cloud map reveals strong van der Waals interactions, primarily hydrogen bonding, between the ANF amide groups, borate ions, silanol groups, and amino groups of the silane coupling agent. Besides, the number of hydrogen bonds in the three systems was simulated and counted through molecular dynamics simulations (see S2. in Supplementary Material for details). Among them, the ATAF-BSi system had the highest number of hydrogen bonds compared with the ATAF- H_2O and ATAF-B system (Figure S2).

To confirm the hydrogen bonding interactions, the obtained

aerogel fibers are characterized by Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS). According to the FTIR spectra (Figure 2d), typical absorption peaks of ANF including -OH at 3445 cm^{-1} , N-H at 3324 cm^{-1} , C=O at 1690 cm^{-1} shift to lower wavenumbers when sequentially incorporating H_3BO_3 and KH792. From the high-resolution XPS spectra for C 1s and N 1s, it can be seen that the characteristic bond of C=O shifts from 287.9 to 288.2 eV and C-O bond shifts from 284.8 to 284.6 eV when sequentially incorporating H_3BO_3 and KH792 (Figure 2e). Meanwhile, the characteristic N-C bond shifts from 400.5 to 400.3 eV and the N-H bond shifts from 399.8 to 400.0 eV (Figure 2f). These spectral shifts confirm hydrogen bonding between the ANF amide groups and

proton donors [51, 52]. This interaction alters the electron cloud density of amide groups, leading to changes in vibrational modes and ultimately resulting in the splitting of the amide II band [46]. Besides, Si and B elements were also observed in XPS spectra (Figure S3). Through their high-resolution XPS spectra (Figure S4), it can be seen that the existence of bond interactions such as B-OH and Si-O indicates the hydrogen bonding interaction of boronic acid and silane coupling agents within the ANF network. Therefore, through theoretical calculations and experimental verification, we have confirmed the extensive hydrogen bond interactions formed during the re-protonation process of

aerogel fibers.

The morphology and microstructure of ATAFs were characterized by scanning electron microscopy (SEM). As depicted in **Figures 3a-c**, ATAFs present a typical core-shell structure with a highly porous nature, which is composed of ANF as the shell and TPU as the core. It exhibits a structure superior to that of the aerogel fibers prepared by freeze-drying (Figure S5). Meanwhile, as is evident from the elemental distribution map (Figure S6), each element is uniformly distributed within the aerogel fiber. It should be noted that the uniform Si distribution is likely attributable to the penetration of the silane coupling agent

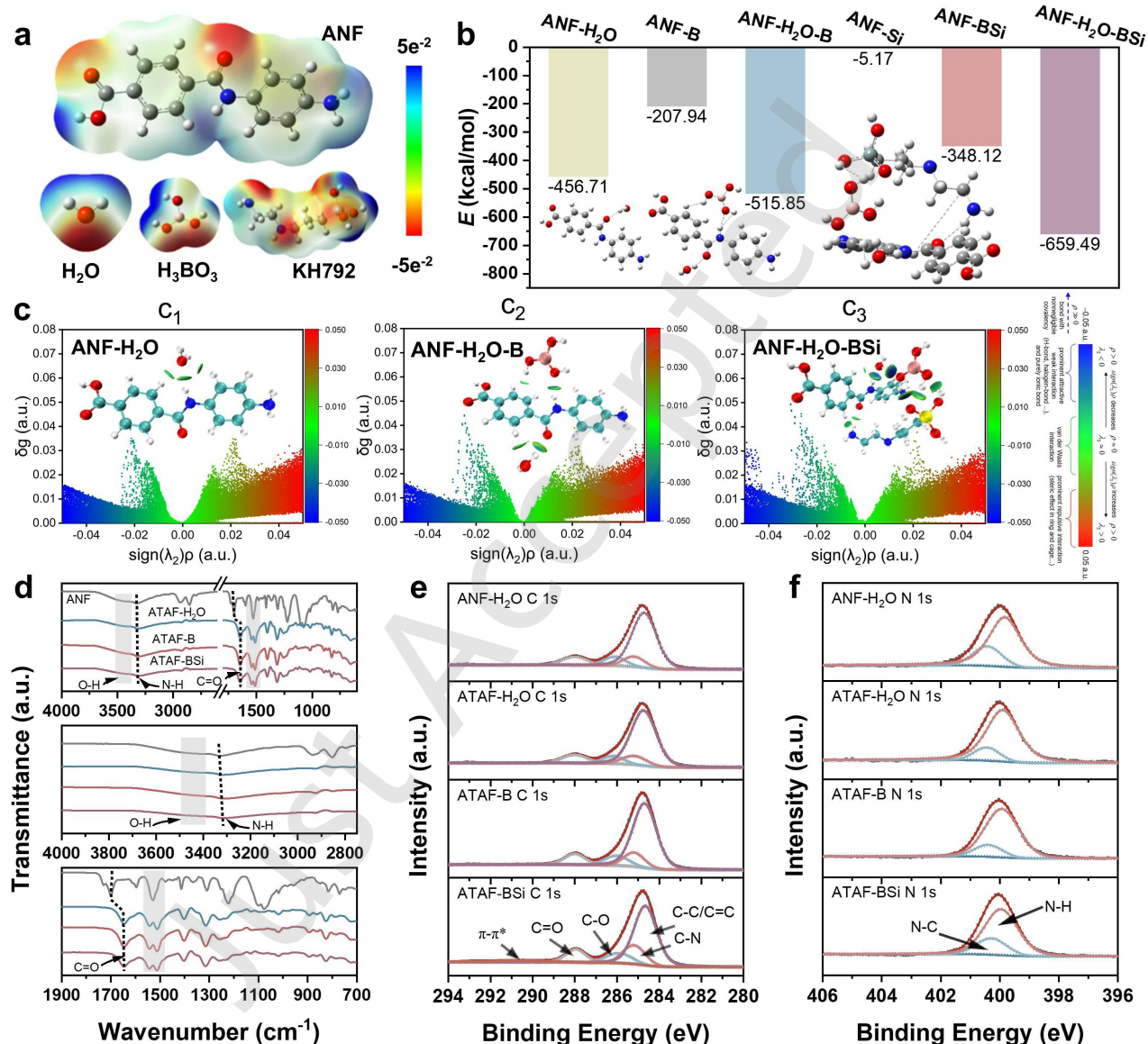


Figure 2. The formation mechanism of ATAFs. (a) Electrostatic potential distributions of ANF, H₂O, H₃BO₃, and KH792. (b) The binding energy (E) of ANF combined with H₂O, H₃BO₃, and KH792 respectively. (c) Diagram of the interaction and the integration results of IGMH (C_1) ANF and H₂O, (C_2) ANF, H₂O, and H₃BO₃, (C_3) ANF, H₂O, H₃BO₃, and KH792. (d) FTIR spectra of ANF, ATAF-H₂O, ATAF-B, and ATAF-BSi. High-resolution XPS spectra at C 1s (e) and N 1s (f) region of ANF, ATAF-H₂O, ATAF-B, and ATAF-BSi.

into the core layer during gelation. From the high-resolution SEM images, the ANF shell layers demonstrate a mutually interconnected 3D nanofiber network structure (Figures 3d-f). When only H₂O is used as coagulation bath (Figure 3d), the shell layer ANF networks display a relatively dense, highly branched morphology. The connectivity of ANF networks is exclusively governed by physical entanglement,

predominantly driven by dynamic reorganization of intra- and intermolecular hydrogen bonds during the sol-gel transition. Upon introduction of boric acid into the coagulation bath (Figure 3e), the shell layer ANF networks underwent structural loosening and developed localized small-scale arched structures due to the emergent chemical crosslinking between nanofibers. Further incorporation of

KH792 (Figure 3f), both the abundance and spatial extent of these arched structures significantly enhanced. These arched structures enhance the strength of ANF networks, enabling it to withstand the capillary pressure generated during APD. This resistance prevents the collapse of networks, thereby maintaining a relatively low network density. Moreover, increasing the ANF concentration results in the enhanced overall structural integrity of aerogel fibers (Figures S7-S9), accompanied by the progressive increase in shell layer ANF network density and shell thickness (Figure S10). The core layer TPU displays a honeycomb-like porous structure with a uniform pore size of 2.7 μm (Figure 3g). In contrast, directly

extruding the TPU dispersion into the coagulation bath leads to an uneven, large-pore structure due to the rapid phase separation (Figure S11). The formation mechanism of this entirely different structure is illustrated in Figure 3h. Briefly speaking, due to encapsulation by the ANF shell layers, water molecules gradually and uniformly penetrate the TPU core dispersion through slow diffusion. Unlike rapid phase separation which creates large and uneven pores, the gradual penetration of water slowly and uniformly replaces solvent in the TPU solution, ultimately forming small, uniform pores

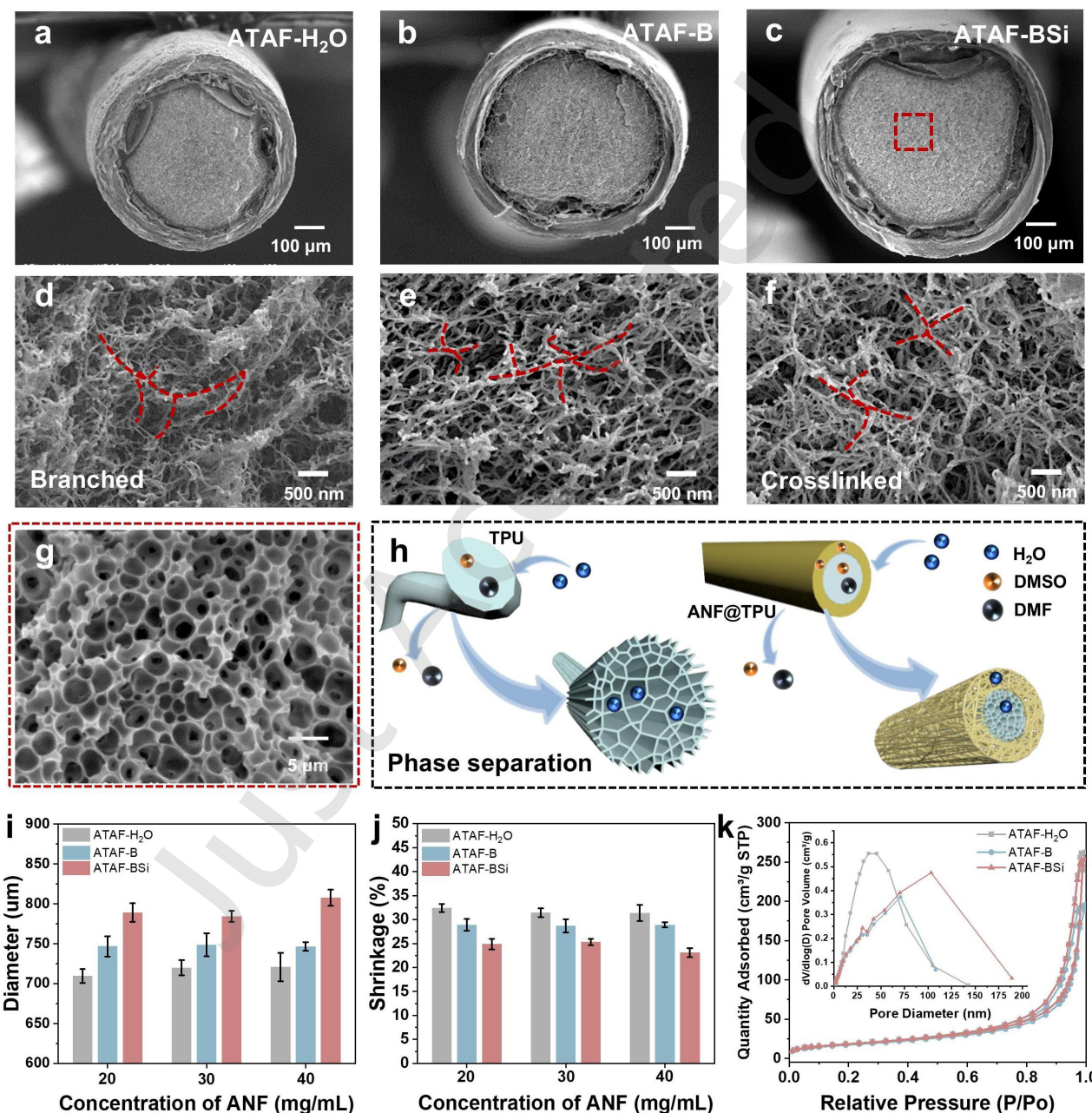


Figure 3. Cross-sectional SEM images of ATAF-H₂O (a), ATAF-B (b), ATAF-BSi (c), their corresponding SEM images of ANF shell layer (d-f), and TPU core layer (g). (h) The phase separation and structure formation mechanism diagram of TPU and ATAF. Graphs of the diameters (i) and shrinkages (j) of ATAF-H₂O, ATAF-B, and ATAF-BSi with different concentrations of ANF. (k) Nitrogen adsorption-desorption isotherms and pore size distribution curves of ATAF-H₂O, ATAF-B, and ATAF-BSi.

through non-solvent-induced phase separation. During the APD process, the collapse of porous structures significantly compromises the mechanical properties and

thermal insulation of aerogel fibers. In this work, by incorporating boric acid and silane coupling agents into ANF networks to introduce extensive hydrogen bonding

interactions, the network's strength was enhanced, and the shrinkage of aerogel fibers was thus suppressed. Taking ATAF₄₀ as an example, the aerogel fiber diameter gradually increases from 721 to 808 μm , accompanied by a corresponding decrease in the diameter shrinkage rate (see S3. in Supplementary Material for details) from 31.4 to 23.1% (Figures 3i, j, and Figure S12 (a) and (b)). Besides, increasing the ANF concentration effectively enhances the diameter of aerogel fibers, owing to the increased density of shell fiber networks. As the diameter increases, the porosity of aerogel fibers increases accordingly, reaching a maximum of $\sim 80\%$ (Figure S13). As expected, the estimated specific surface area shows the same trend as the aerogel fiber diameter, with a maximum value reaching $68 \text{ m}^2/\text{g}$. Besides, according to the Brunauer-Emmett-Teller (BET) results presented in Figure 3k and Figure S14, the isotherms of these aerogel fibers exhibit a typical IV characteristic. Within the relative pressure range of $P/P_0 = 0.6-1$, a significant hysteresis loop emerges, indicating that the material features a regular mesoporous channel structure. From the pore size

distribution curves depicted in Figure 3k, it is evident that an increase in crosslinking density induces a shift in the peak toward the larger pore size region. This observation demonstrates that the dominant pore size range of ATAF expands with increasing crosslinking density, which is consistent with the morphological variations reflected in the corresponding SEM images (Figures 3d and e).

3.3 Mechanical Properties of ANF@TPU Aerogel Fibers

Mechanical properties are of vital importance for the practical application of aerogel fibers, but are often compromised by their inherent high porosity [28, 53, 54]. In this work, compared with freeze-drying, the fibers obtained through APD have a tightly wrapped core-shell structure, which exhibits excellent mechanical properties (Figure S15). The shell layers, composed of ANFs with extensive hydrogen bond crosslinking, provide structural strength of aerogel fibers, while the porous TPU core layers ensure stretchability. Meanwhile, taking into account the balance between tensile

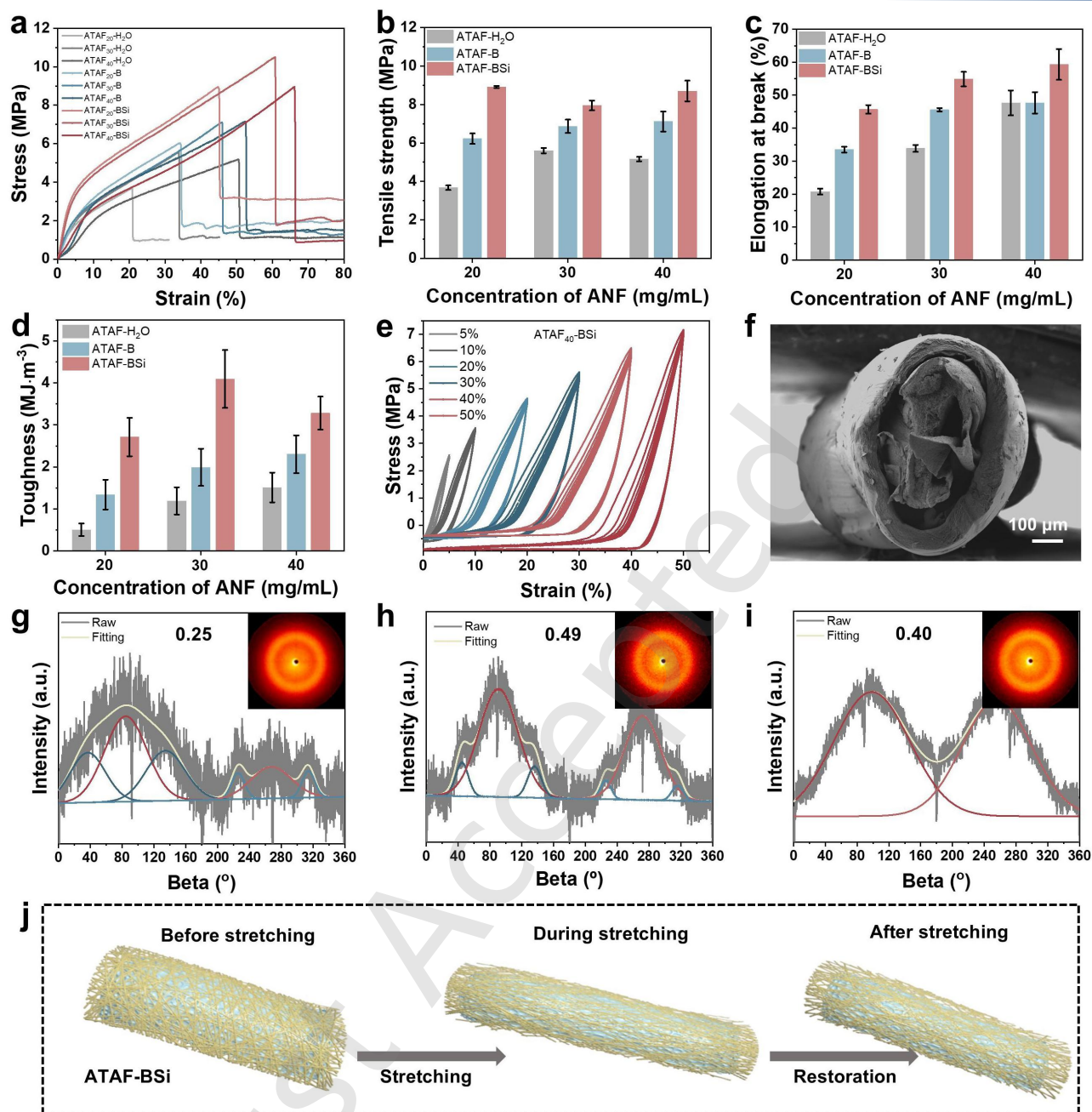


Figure 4. (a) Stress-strain curves, (b) tensile strength, (c) elongation at break, and (d) toughness of ATAFs with different ANF concentrations. (e) Fatigue tensile properties of a single fiber (ATAF₄₀-BSi) at different elongation rates. (f) SEM image of ATAFs about stretched fracture section. WAXD patterns obtained with the incident X-ray beam perpendicular to the ANF layer of ATAFs about (g) before stretching, (h) during stretching, (i) after stretching and leaving them for a period of time, and (j) the mechanisms corresponding to each stage.

strength and fracture elongation, a 15/22 with suitable core-shell ratio needle was selected. Figure 4a depicts the stress-strain curves of ATAFs with different crosslinking. It is evident that the aerogel fibers generally demonstrate superior properties with tensile strength of 3.7-10.5 MPa and fracture strain of 20.9-66.3%. The introduction of boric acid and silane coupling agents can sequentially enhance the strength and elongation at break (fracture toughness) of aerogel fibers, due to the increase in hydrogen bond crosslinking in ANF shell layers (Figures 4b-d, Figure S12(c) and S17). As the ANF network density of shell layers increases, the optimal mechanical properties of ATAF can be enhanced to 8.9 MPa of strength, 59.3% of strain and 3.3

MJ·m⁻³ of toughness. Furthermore, after being exposed to temperature of 200 °C and -196 °C, ATAF₄₀-BSi still exhibited approximately the same tensile strength and elongation at break (Figure S18), demonstrating its excellent temperature resistance. Notably, on the tensile stress-strain curve, all aerogel fibers show a sharp stress drop upon reaching the failure strain, yet maintain a residual tensile strength of about 0.9-3.2 MPa (Figure 4a). This behavior is attributed to the rupture of ANF shell layers at failure, while TPU core layers persist in sustaining low-stress deformation. This characteristic implies that even when the aerogel fibers are damaged by a large external force in practical applications, the textile still retains protective capability. For aerogel

fibers, stretch recovery is a critical feature in practical applications where joint movements cause large deformations, as it allows the textile to withstand pulling forces and return to its original shape. In this work, the inherent viscoelasticity of TPU endows ATAFs with a certain degree of elastic recovery. The tensile recovery tests (Figure 4e) reveal that a single fiber can withstand strains ranging from 5% to 50%, and exhibits a certain elastic recovery after stress release. In comparison, traditional and hollow ANF aerogel fibers do not possess the elastic recovery property upon stretching (Figure S19). Furthermore, the aerogel fiber was subjected to 1,000 cyclic tensile fatigue tests at varying strain levels. After testing, it maintained appreciable tensile recovery performance (Figure S20(a)) without any obvious performance degradation and structural damage (Figure S20(b)). Therefore, the single aerogel fiber is capable of lifting a 500 g weight and stretching at large strain (Figure S21).

To explore the stretching mechanism, the fracture morphology and structural evolution of aerogel fibers during stretching are investigated using SEM and wide-angle X-ray diffraction (WAXD). As shown in Figure 4f, the fracture surface shows a relatively smooth shell layer section and an extremely rough core layer section, corresponding to the brittle fracture of ANF shell and the ductile tearing of TPU core, respectively. Figures 4g-i present the WAXD spectra and fitting results of the shell layer ANF during different stretching stages, in which the orientation of ANF can be calculated (see S4. in Supplementary Material for details) [55]. According to the calculations, Herman's orientation factor is 0.25 before stretching, increases to 0.49 at 50% strain, and recovers to 0.40 after elastic recovery. Based on these results, the stretching mechanism of ATAF is proposed in Figure 4j. Before stretching, the shell ANF networks are randomly arranged. During stretching process, the nanofibers within ANF networks gradually align along the direction of applied force. During this process, structural rearrangement in ANF networks contributes to the absorption of a substantial amount of stretching energy, which is reflected on the stress-strain curve as a gradual increase in stress with strain. Upon removal of the external force, the elastic recovery of porous TPU core layers enables the stretched and oriented ANF shell networks to gradually revert to a relatively random state. Consequently, the ATAF retains excellent mechanical strength and toughness while maintaining notable stretch-recovery elasticity.

3.4 Thermal Insulation Performance of ANF@TPU Aerogel Fibers and Textiles

Traditional ANF aerogel fibers exhibit promising thermal insulation properties owing to their porous structure, but suffer from inherent limitations [28, 56]. Specifically, the rigid molecular chains and oriented chain alignment result in relatively high thermal conductivity in the ANF skeleton [8, 57]. Moreover, the 3D nanofiber network forms an open-cell architecture, which facilitates intense thermal convection[58]. In contrast, the core-shell structure presented in this work features a synergetic thermal insulation mechanism (Figure 5a): the TPU skeleton in core layers possesses a lower thermal conductivity, attributed to its flexible molecular chains, and the closed-cell structure

formed via phase separation more effectively suppresses thermal convection. Furthermore, the insulation effect of core layers causes heat to transfer along a tortuous path within the shell layers, thereby impeding heat conduction. To validate this mechanism, finite element analysis was performed to analyze the heat transfer behavior (see S5. in Supplementary Material for details). As illustrated in Figure 5b, unlike the uniform heat transfer observed in traditional ANF fiber networks (Figure S22), the core-shell structured ATAF exhibits a heat transfer retardation effect, which is primarily attributed to the TPU core's ability to inhibit thermal conduction throughout the aerogel fiber.

Subsequently, the cross-linking strategy was optimized to minimize shrinkage and enhance porosity, further improving the thermal insulation capability of ATAF. As depicted in Figure 5c, the optimal ATAF exhibits an extremely low thermal conductivity of $29.82 \text{ mW}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, which is lower than that of the traditional ANF aerogel fibers with 3D nanofiber networks (Figure S23). Furthermore, the thermal insulation performance of ATAF can be further optimized by adjusting the shell-to-core ratio. As shown in Figure S24, increasing the proportion of the core layer leads to a further reduction in thermal conductivity, highlighting the critical role of the porous TPU core layer in thermal insulation. The statistical analysis reveals that higher porosity, larger pore diameter, and greater shell thickness all correlate with reduced thermal conductivity in the aerogel fibers (Figure S25). To further verify the influence of ANF shell network densities on thermal insulation performance, the thermal diffusion behavior of individual aerogel fibers with different ANF concentrations is recorded using infrared cameras. As shown in Figure 5d and Figure S26, a gradual decline in the surface temperature of ATAFs is observed with increasing ANF concentration in the shell layers. At a hot plate temperature of 200°C , the temperature difference (ΔT) between a single ATAF₄₀ and the hot plate could be 10.9°C . Moreover, this thermal insulation performance was largely retained even after 1000 cyclic fatigue testing (Figure S27). This result is mainly attributed to the higher ANF concentration, which leads to a lower shrinkage rate of ATAF, resulting in a larger diameter and higher porosity, and thus slower heat diffusion and enhanced insulation performance.

In practical applications, the flexible and robust ATAF fibers can be woven into a series of flexible wearable textiles. To confirm their thermal insulation ability, these textiles were placed on a hot plate with a temperature range of 50 to 200°C , and the variations in the textile's surface temperature were recorded through an infrared camera (Figure S28). As shown in Figures 5e-g, a substantial ΔT is observed between the textile surface and the hot plate, and this ΔT further increased with the increasing of ANF network densities. Taking a hot plate at 200°C as an example, the equilibrium surface temperatures of the ATAF₂₀, ATAF₃₀, and ATAF₄₀ textiles are 162.1 , 143.5 and 119.5°C respectively, and the corresponding ΔT are 25.2 , 43.8 , and 67.8°C . Moreover, the ATAF textiles exhibit excellent thermal stability after being tested at 200°C , showing no signs of degradation (Figure S29). Besides, to highlight the advantages of ATAF in terms of thermal insulation performance, the cotton, conventional ANF aerogel textile,

and ATAF textile were placed on a hot plate to observe their surface temperatures. As shown in Figure S30, the ATAF textile shows the lowest surface temperature. These results demonstrate that the ATAF textiles possess excellent

insulation capabilities to resist high temperatures in practical applications.

The performance stability is of critical significance for the

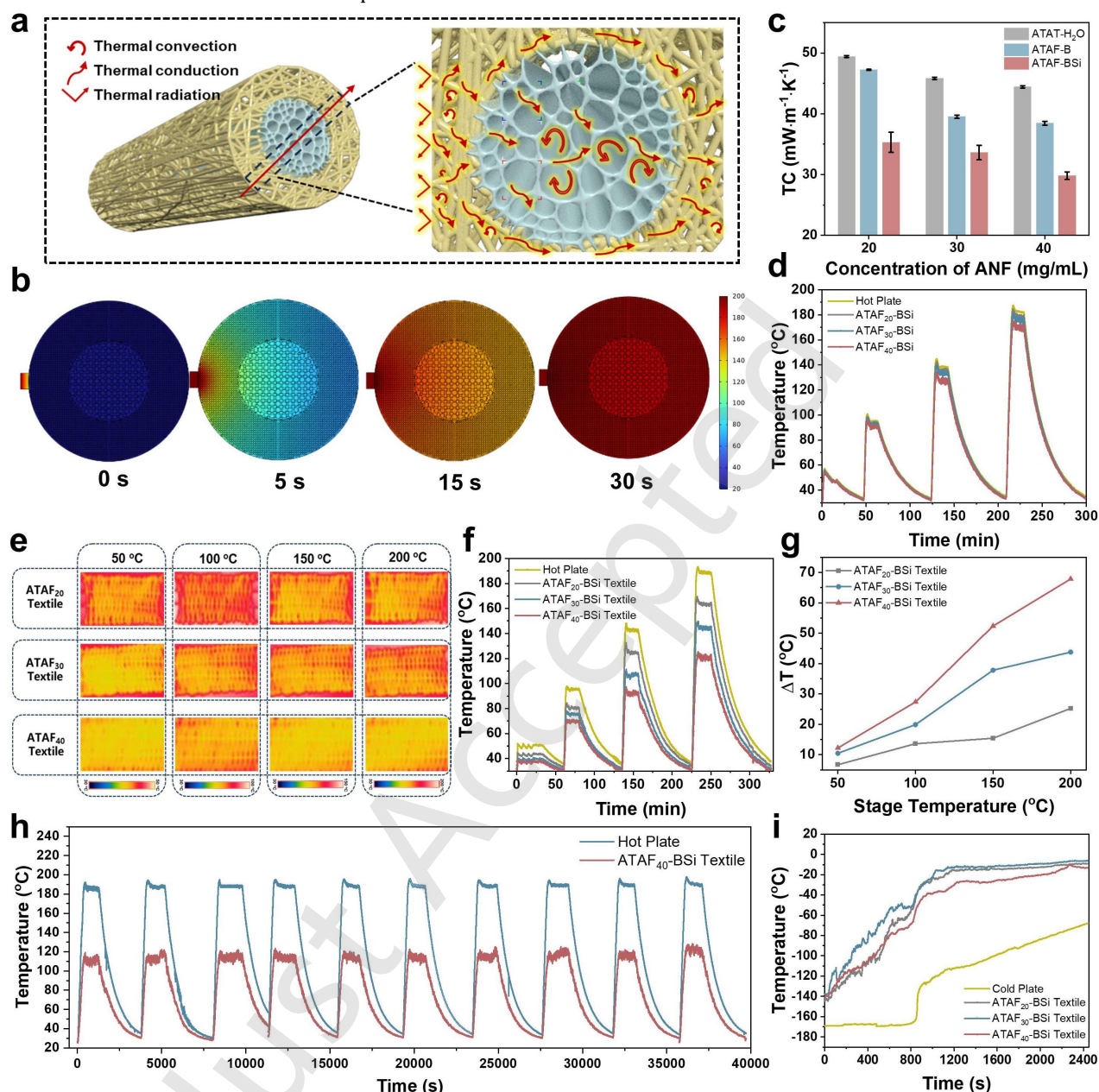


Figure 5. (a) Schematic diagram of thermal insulation mechanism about ATAFs. (b) The heat transfer results of the finite element simulation for ATAFs. (c) Thermal conductivities of ATAFs with different ANF concentrations. (d) Temperature-time curves of ATAFs with different ANF concentrations under 50-200 °C. (e) Thermal infrared images at equilibrium state under for ATAF-BSi textiles on the hot plate of 50-200 °C. (f) Temperature-time curves and (g) temperature differences (ΔT) between textiles surface and hot plate of different ATAF-BSi textiles at different hot plates. The surface temperature distribution curve of ATAF₄₀-BSi textile under cyclic on/off cycle (h) exposure at 200 °C hot plate. (i) Temperature-time curves of ATAF-BSi textile at -196 °C.

practical application of ATAFs as a thermal insulation material. As shown in Figure 5h and Figure S31, whether in the cyclic high-temperature test or in a long-term (>2 h) high-temperature insulation test, the ATAF textile maintains a stable insulation effect with a ΔT of approximately 67 °C. Moreover, after the long-term test, there are no signs of degradation on the ATAF textile (Figure S32). This phenomenon emphasizes the excellent and stable thermal insulation performance of ATAF textile. In addition to its

exceptional high-temperature insulation properties, the textile also exhibits outstanding low-temperature insulation performance. When the ATAF textile is placed in an environment with a low temperature of -196 °C, the surface of the textile exhibits a significantly higher temperature than that of surrounding environment (Figure 5i). Moreover, the ATAF textile exhibits stable low-temperature insulation performance throughout the cyclic test. (Figure S33). Therefore, given its exceptional thermal insulation across an

extreme temperature range (-196 °C to 200 °C), this ATAF textile holds significant promise for personal thermal management, as it can prevent overheating in high temperatures and retain body heat in cold environments.

3.5 Weather resistance of ANF@TPU aerogel fibers

In practical applications, aerogel textiles often face various harsh environmental conditions [59, 60]. As a derivative of aramid fibers, ANF shares similar heat resistance, UV resistance, and chemical corrosion resistance [61]. Therefore, in this work, the ANF shell provides effective encapsulation and protection for the core TPU layers. **Figures 6a-c**

compare the thermal properties of TPU, ANF, and ATAF tested by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). It can be observed that the TGA curves of aerogel fibers fall between those of TPU and ANF, and the first two decomposition stages correspond to TPU, whereas the final stage is attributed to ANF. In contrast, using highly stable ANF as the protective shell layer effectively enhances both the thermal decomposition temperature (>300 °C) and the residual yield (~20%) of the aerogel fibers. DSC analysis reveals that the aerogel fibers exhibit stable thermal behavior

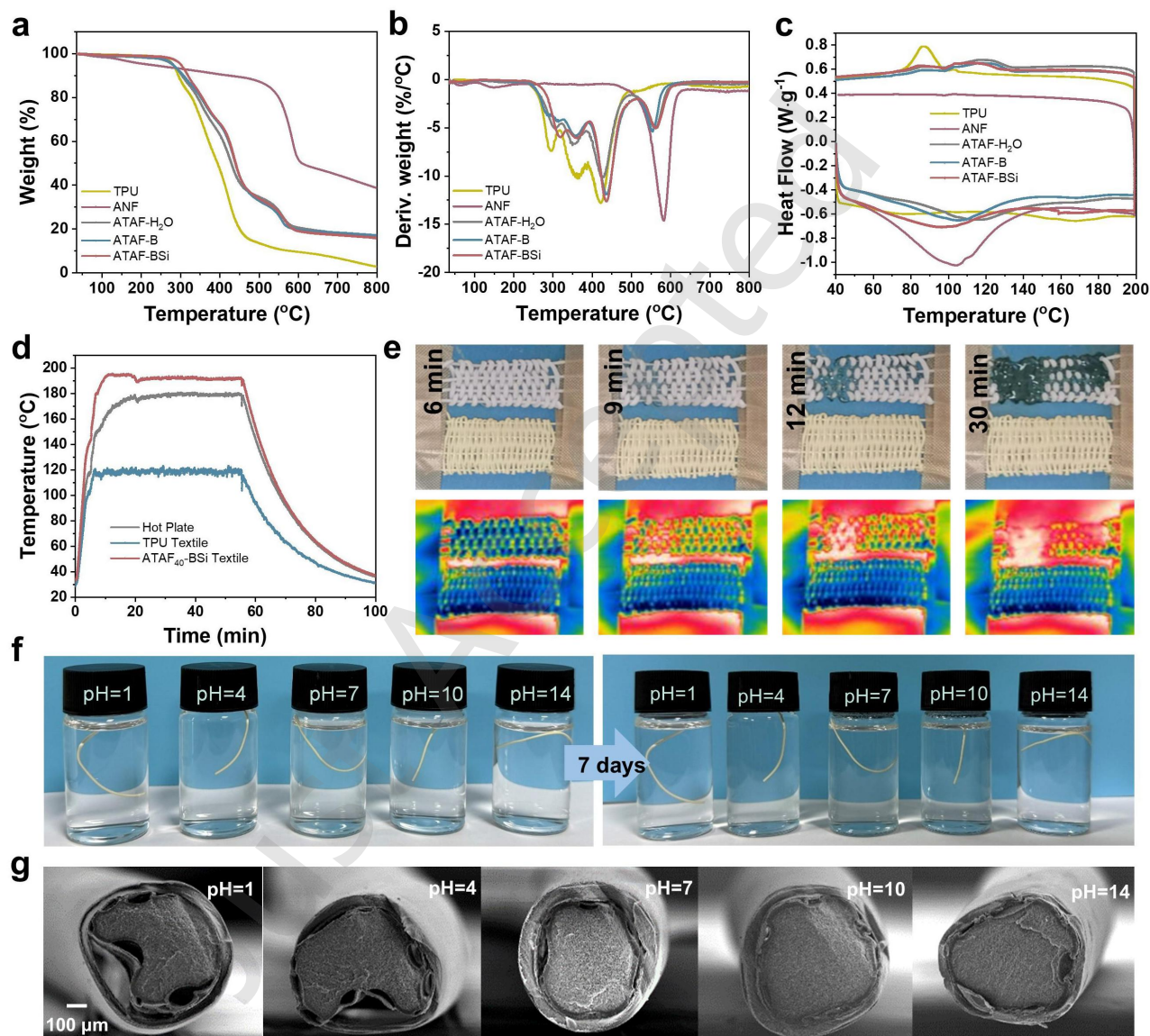


Figure 6. (a) TGA and (b) DTG curves of TPU, ANF, and ATAFs. (c) DSC curves of TPU, ANF, and ATAFs. (d) Temperature-time curves and (e) photographs and corresponding thermal infrared images between textiles surface and hot plate of TPU and ATAF textiles at 200 °C. (f) photographs and (g) SEM images of ATAFs after being immersed in different pH solutions for a week.

between 40 °C and 200 °C, excluding an initial exothermic peak at 100-120 °C attributed to moisture release (Figure 6c). Furthermore, the ANF shell layers effectively suppress the crystallization of TPU hard segments during cooling.

To more intuitively demonstrate thermal stability, pure TPU and ATAF aerogel textiles were placed on a 200 °C hot plate to simulate the heat resistance performance of the fibers in an actual high-temperature environment. As shown in Figures 6d and e, the ATAF textile exhibits excellent thermal

insulation and shape stability, achieving a temperature reduction of 70.1 °C. In contrast, the TPU aerogel textile melts at high temperatures and loses its insulating function, resulting in a surface temperature nearly equal to that of the hot plate. This outcome clearly demonstrates that the ANF aerogel network, as an outer shell, effectively provides thermal protection to the core TPU layers, thereby preventing its thermal melting and functional failure. Simultaneously, the acid and alkali resistance of ATAFs was

also assessed. After immersion in solutions of different pH values for one week, as shown in Figure 6f, the aerogel fibers exhibited no significant damage compared to their initial state. The cross-sectional morphology of the fibers after immersion in acid and alkali solutions reveals that no significant structural changes were detected (Figure 6g). Besides, its mechanical properties have remained largely intact when after immersion in acid solutions. The elongation at break has decreased slightly after immersion in acid and alkali solutions because the number of hydrogen bonds in the system decreases after the boric acid is consumed. (Figure S34). Furthermore, leveraging the high thermal stability and char residue of the ANF shell, the ATAF exhibits flame retardancy and self-extinguishing properties, which are further enhanced by crosslinking with boric acid and silane coupling agents (Figure S35). In conclusion, beyond its fundamental thermal insulation and mechanical properties, the ATAF exhibits enhanced environmental resistance due to its unique core-shell architecture, ensuring practicality in harsh conditions.

4 Conclusions

In summary, we have developed a core-shell structured ANF@TPU aerogel fiber (ATAF) via a scalable coaxial wet-spinning and ambient-pressure drying strategy. The introduction of boric acid and silane coupling agents can strengthen the ANF nanofiber network by constructing abundant hydrogen bonds, effectively inhibiting structural collapse and reducing the shrinkage rate to only 23%. The engineered multi-scale porous architecture with a high porosity of 79.2% integrates a high-strength ANF nanofiber network shell with a flexible TPU microporous core. This design achieves an exceptional combination of properties: high tensile strength (up to 8.9 MPa), toughness (strain up to 59.3%), and remarkable elastic recovery. Crucially, the ATAF exhibits ultralow thermal conductivity ($29.82 \text{ mW}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) and maintains stable thermal insulation across a broad temperature range from -196 to 200 °C. The protective ANF shell also imparts outstanding thermal stability, chemical resistance, and flame retardancy to the fibers. These integrated characteristics make the weavable ATAF a highly promising candidate for advanced protective textiles, particularly in the extreme thermal and mechanical conditions.

Electronic Supplementary Material: Supplementary material (theoretical calculation method for DFT and molecular dynamics simulations, porosity and orientation, and finite element simulation, photo of dispersions, high-resolution XPS spectra, elemental distribution map, cross-sectional SEM images, statistics of the shell-layer thickness and porosity, nitrogen adsorption-desorption isotherms, stress-strain plot, tensile strength and elongation at break, photographs of ATAFs during tensile tests, thermal conductivity, temperature difference of ATAFs, schematic diagram of fabric insulation test, photo of ATAF textiles, surface temperature curve of ATAF textile under long-term exposure, photos of ATAF textile before and after the high-temperature cycling test, photos of ATAF in flame retardant

test.) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94909241>.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

Jin Gao: Data curation, project administration, validation, writing manuscript, experimental design. Gaojie Han: Resources, Writing-review & editing, methodology. Yining Lei: Investigation, software. Shuai Wang: Data curation. Jingwen Dong: Formal analysis, validation. Bing Zhou: Data curation, supervision. Chuntai Liu: Conceptualization, methodology. Yuezhan Feng: Resources, project administration, funding acquisition, conceptualization. All the authors have approved the final manuscript.

Use of AI statement

None.

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Thermostable and stretchable core-shell aerogel fibers for thermal insulation application

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INFORMATION ABOUT ELECTRONIC SUPPLEMENTARY MATERIAL. Theoretical calculation method for DFT and molecular dynamics simulations, calculation methods of porosity and orientation, finite element simulation method, comparison with the performance of previous studies, number of hydrogen bonds in ATAF-H₂O, ATAF-B, and ATAF-BSi, the hydrogen bond formation situation of ATAF-BSi, photograph of ANF/DMSO dispersions, high-resolution XPS spectra, Elemental distribution map, cross-sectional SEM images, statistics of the shell-layer thickness and porosity, nitrogen adsorption-desorption isotherms, stress-strain plot, tensile strength and elongation at break, photographs of ATAFs during tensile tests, thermal conductivity, temperature difference of ATAFs, schematic diagram of fabric insulation test, photograph of ATAF textiles, surface temperature curve of ATAF textile under long-term exposure, photos of ATAF textile before and after the high-temperature cycling test, photos of ATAF in flame retardant test.

S1. Theoretical Calculation Method for DFT

The density functional theory (DFT) computations were carried out using the B3LYP exchange correlation functional [1, 2], together with the 6-311G(d,p) [3] basis set for all atoms. All computations were performed using the Gaussian 09 software suite [4]. The geometrical optimizations were performed in the gas phase. The solvent effect was taken into consideration based on the Polarizable Continuum Model (PCM) [5]. The tight criteria were used for the optimization and the frequency calculation was carried out to confirm the nature of the stationary points. The DFT calculations using the B3LYP/6-311G(d,p) basis set revealed that conformer (ANF-H₂O-BSi) in alignment is more stable than conformer (ANF-H₂O) and conformer (ANF-H₂O-B) in the gas phase, respectively (Figure 2b; Table S1). The interaction between each component can be calculated by the following formula:

$$E(\text{interaction}) = E(AB) - E(A) - E(B) \dots\dots\dots (1)$$

S2. Theoretical Calculation Method for Molecular Dynamics Simulations

The molecular dynamics simulations were carried out using Materials Studio software. H₂O, H₃BO₃, KH792 and ANF molecules were simulated. Parameter fitting was performed using the density functional theory (DFT) under B3LYP exchange-correlation functional. The system was constructed using the Amorphous Cell. The box size of these systems was limited in 5 × 5 × 5 nm³. The MD simulation was firstly performed in the NPT ensemble at temperature (298 K) by using 0.1 ps time step for 10 ps. Then equilibrium simulation is performed in the NPT ensemble at constant pressure (1 bar) and temperature (298 K) in a cubic box with periodic boundary conditions in all xyz Cartesian directions by using 0.1 ps time step for 10 ps. Finally, the production simulation is performed in the NPT ensemble at temperature (298 K) by using 0.1 ps time step for 200 ps.

S3. Calculation Method of the Shrinkage about Aerogel Fibers

The method for calculating the shrinkage is based on the diameters of coaxial needle and aerogel fibers. The calculation formula is as follows:

$$\text{Shrinkage} = \frac{d_f}{d_n} \times 100\% \dots\dots\dots (2)$$

where d_f is the diameter of the aerogel fibers after drying determined by SEM images, d_n is the diameter of coaxial needle. In this experiment, the model 15/22 coaxial needle was used. Based on the polarizing microscope image (Figure S1) of hydrogel fibers, the original diameter is approximately 1250 μm. After removing the thickness of the core layer needle, the actual diameter should be approximately 1050 μm. Therefore, d_n was regarded as approximately 1050 μm.



Figure S1. The polarizing microscope image of hydrogel fibers.

S4. Calculation Method of Orientation Degree about Aerogel Fibers

The method for calculating the orientation degree is based on the methods described in literature[6]. WAXD (2D-WAXD, D8 Discover, Germany) was used to examine the orientation degrees of crystalline region, which can be respectively calculated by formula:

$$f = \frac{360^\circ - H}{360^\circ} \times 100\% \quad (3)$$

where H is the sum of full width at half-maximum.

S5. Finite Element Simulation Method for Heat Conduction

The simulation work was completed with the help of the finite element software COMSOL Multiphysics. Transient solution method was carried out for all models with the same loading sub-steps of 30 steps and total time of 30 s. The first and second types of boundary conditions were used as the upper and lower boundaries, respectively. The governing equation is:

$$\rho C_p \frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(k \frac{\partial T}{\partial x} \right) + \frac{\partial}{\partial y} \left(k \frac{\partial T}{\partial y} \right) \quad (4)$$

$$\text{Initial conditions: } T|_{t=0} = T_0 \quad (5)$$

$$\text{Temperature boundary condition: } T|_{\Gamma} = T_1 \quad (6)$$

$$\text{Heat flow boundary conditions: } -k \frac{\partial T}{\partial n} \Big|_{\Gamma} = q \quad (7)$$

Among them, ρ represents density, C_p is specific heat capacity, and k stands for thermal conductivity. Once the temperature profile is determined, the total heat flow can be calculated by numerical integration on the boundary of the applied temperature according to the heat flow calculation equation (Fourier equation):

$$k_{eff} = \frac{q}{\frac{\Delta T}{\Delta x}} \quad (8)$$

where q is the heat flux, k_{eff} is the thermal conductivity of composites, ΔT and Δx are the temperature gradient and distance between the two sides of model, respectively.

Supplementary Table

Table S1. Comparison with the performance of previous studies

Previous studies	Mechanical recovery	Thermal stability	Thermal conductivity (mW·m ⁻¹ ·K ⁻¹)	Fabrication scalability
Ref. [7]	Compression	-20 °C-200 °C	28.4-32.1	√
Ref. [8]	×	-20 °C-200 °C	37.2	√
Ref. [9]	×	-15 °C-200 °C	28.3	√
Ref. [10]	stretch	-20 °C-40 °C	26.9	√
Ref. [11]	×	-20 °C-80 °C	52.4	√
Ref. [12]	×	-20 °C-100 °C	32.0	√
This work	stretch	-196 °C-200 °C	29.8	√

Table S2. Electronic energy of the components of the studied compounds and their energy difference in the gas phase.

Components	Electronic energy (Hartree)	<i>E(interaction)</i> (kcal/mol)
ANF	-875.927385	
H ₂ O	-75.971502	
H ₃ BO ₃	-252.480316	
KH792	-824.969073	
ANF+H ₂ O	-952.626694	-456.7054428
ANF+H ₃ BO ₃	-1128.739072	-207.9382848
ANF+H ₃ BO ₃ +H ₂ O	-1205.201268	-515.8531861
ANF+KH792	-1700.888223	-5.167536615
ANF+KH792+H ₃ BO ₃	-1953.931537	-348.1187754
ANF+KH792+H ₃ BO ₃ +H ₂ O	-2030.399237	-659.4874861

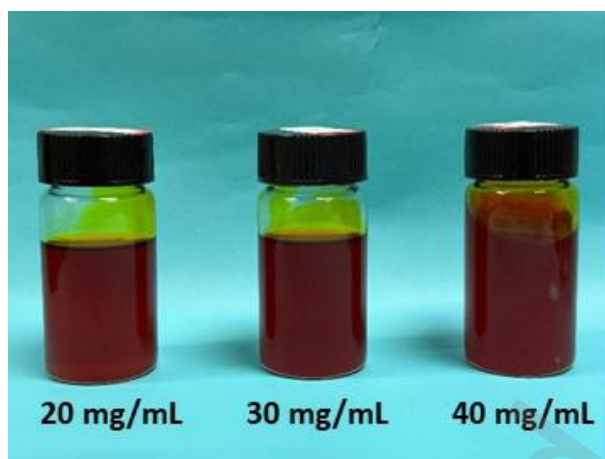


Figure S1. The photograph of ANF/DMSO with ANF of different concentrations.

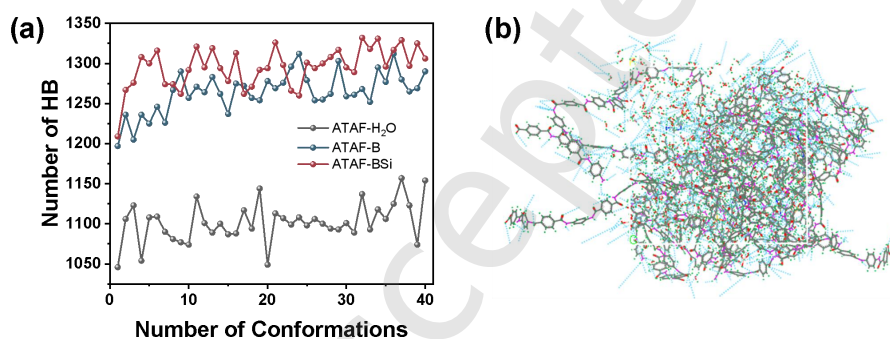


Figure S2. (a) Number of hydrogen bonds in ATAF-H₂O, ATAF-B, and ATAF-BSi. (b) The hydrogen bond formation situation of ATAF-BSi.

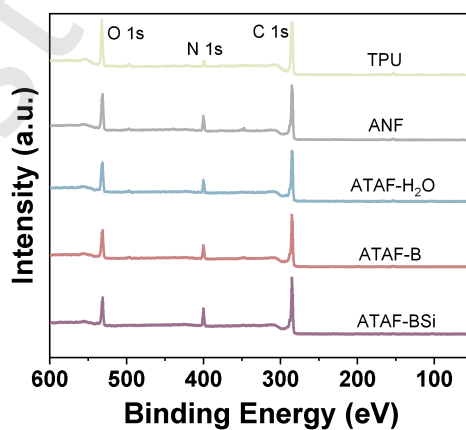


Figure S3. XPS spectra of TPU, ANF, ATAF-H₂O, ATAF-B and ATAF-BSi.

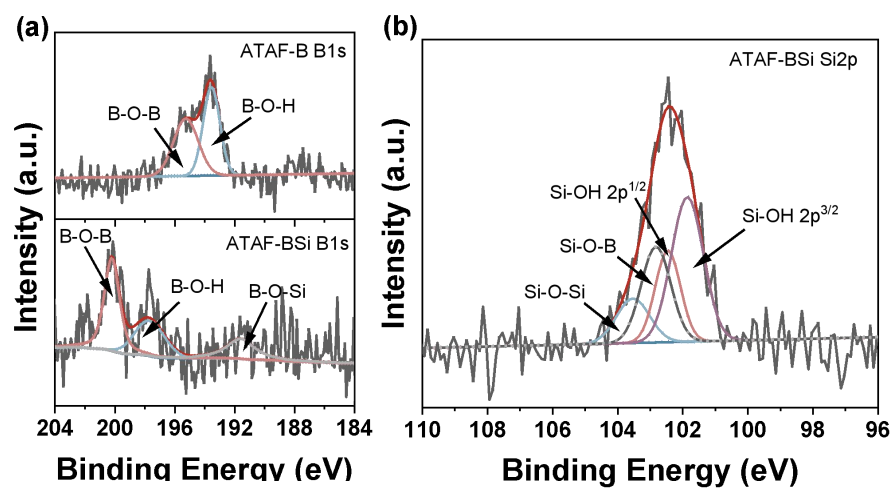


Figure S4. High-resolution XPS spectra at B 1s of ATAF-B and ATAF-BSi (a) and Si 2p of ATAF-BSi.

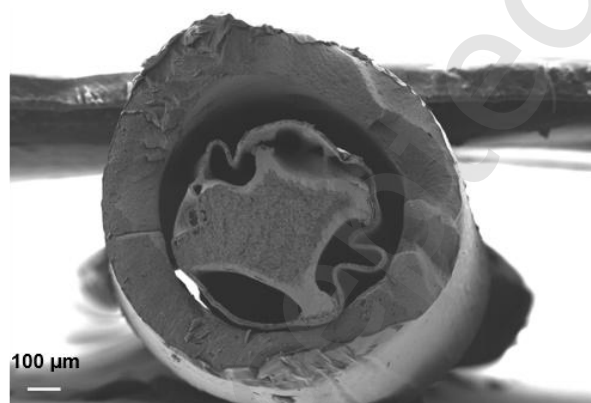


Figure S5. SEM image of ANF@TPU aerogel fibers prepared by freeze-drying.

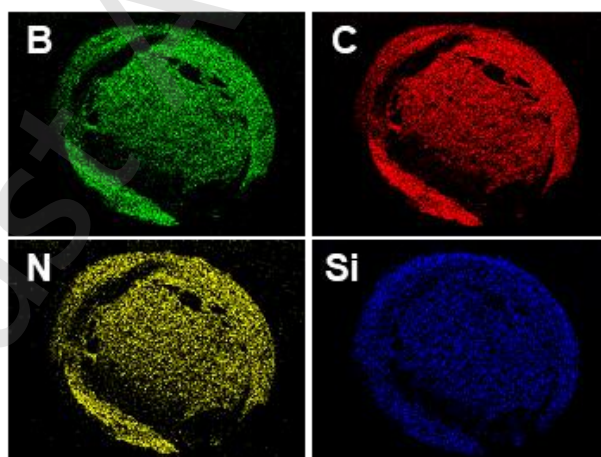


Figure S6. Elemental distribution mapping of the cross-section of ATAF-BSi.

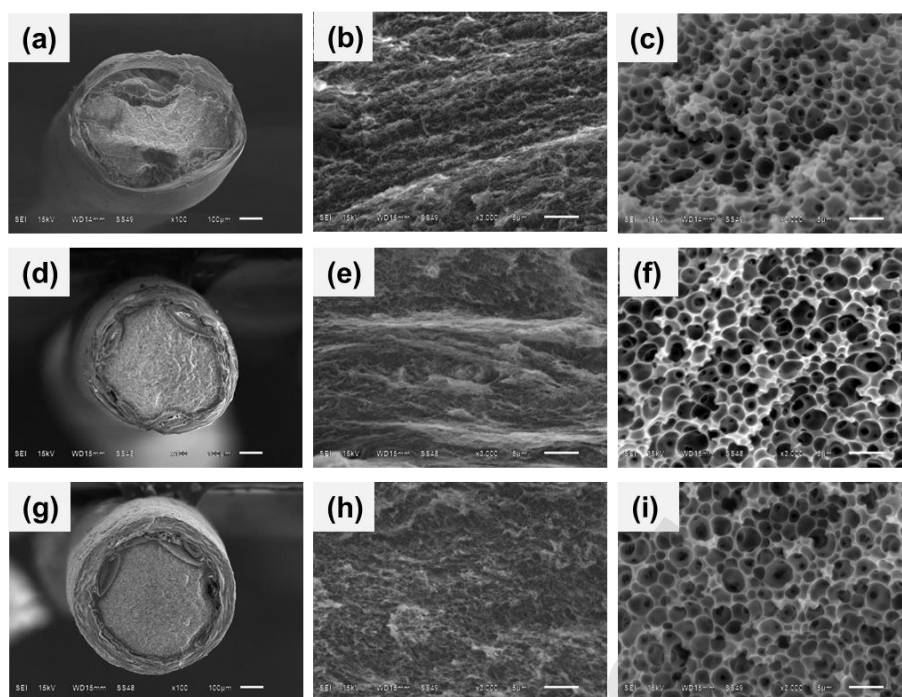


Figure S7. Cross-sectional SEM images of ATAF-H₂O about the overall morphology, ANF shell layer and TPU core layer with ANF concentrations: 20mg/mL (a-c), 30mg/mL (d-f) and 40mg/mL (g-i).

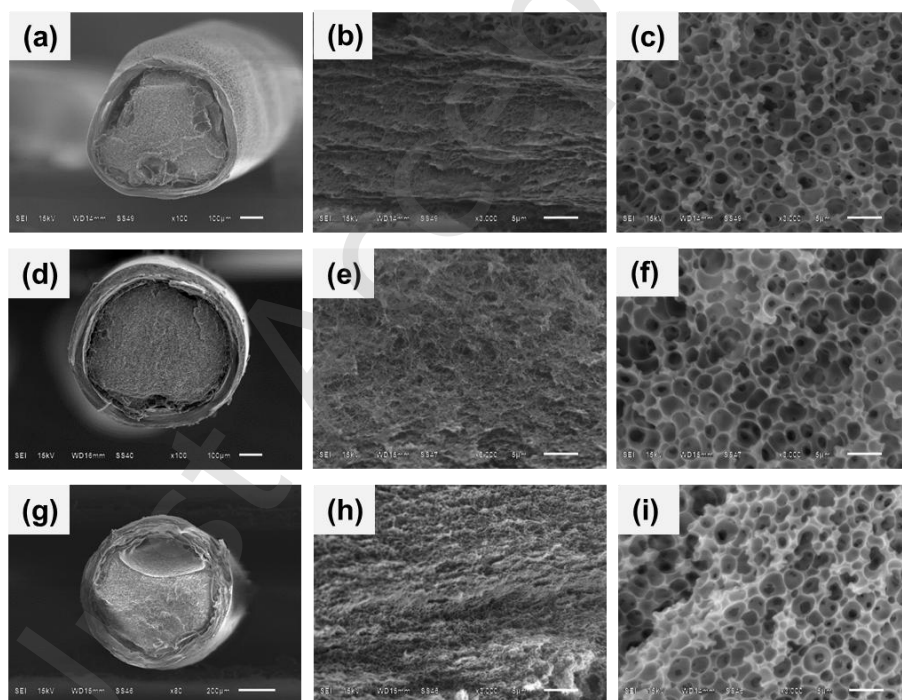


Figure S8. Cross-sectional SEM images of ATAF-B about the overall morphology, ANF shell layer and TPU core layer with ANF concentrations: 20mg/mL (a-c), 30mg/mL (d-f) and 40mg/mL (g-i).

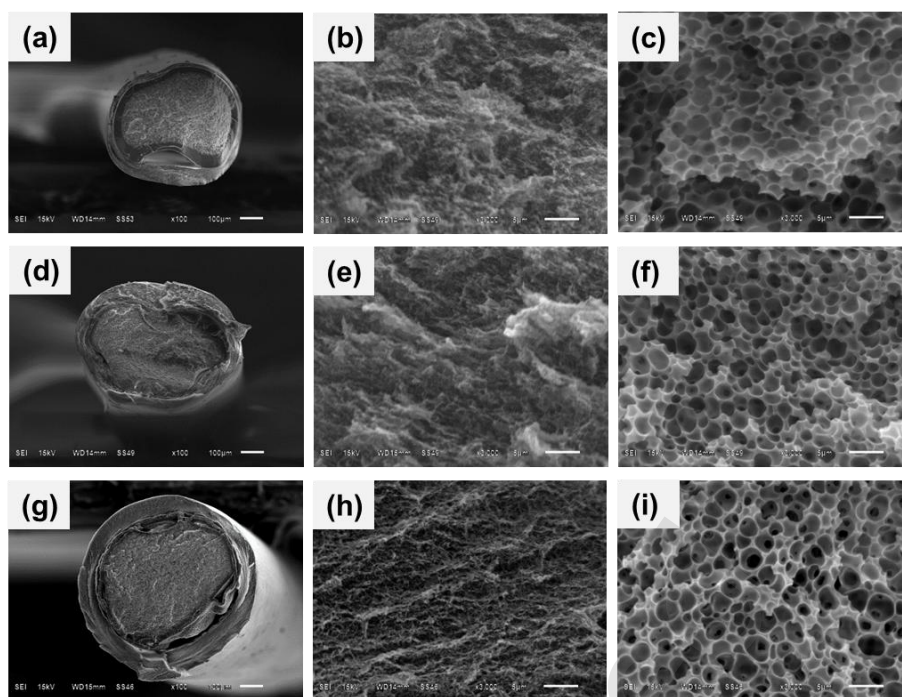


Figure S9. Cross-sectional SEM images of ATAF-BSi about the overall morphology, ANF shell layer and TPU core layer with ANF concentrations: 20mg/mL (a-c), 30mg/mL (d-f) and 40mg/mL (g-i).

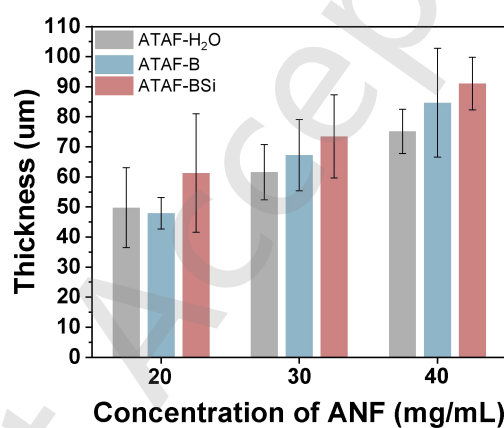


Figure S10. Shell-layer thickness about ATAF-H₂O, ATAF-B and ATAF-BSi with different concentrations of ANF.

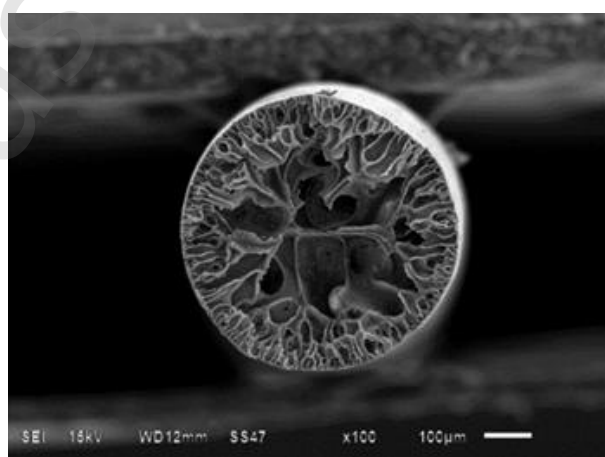


Figure S11. SEM image of cross-section morphology of pure TPU aerogel fiber.

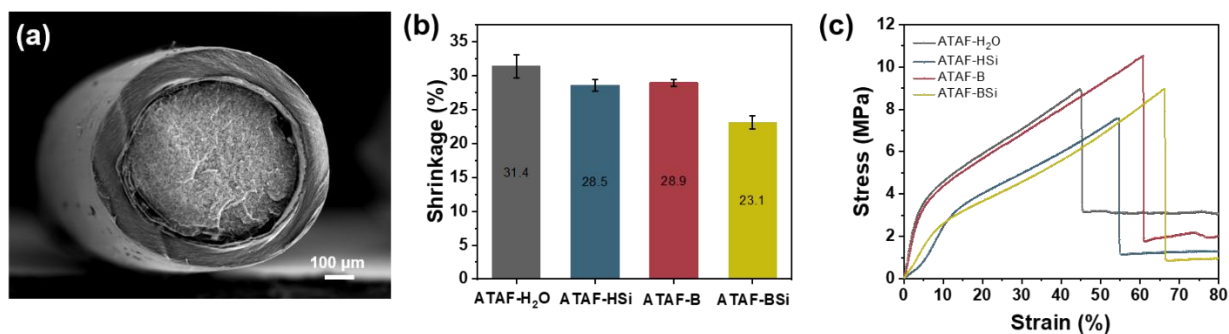


Figure S12. (a) SEM image of ATAF-HSi. (b) Shrinkage of ATAF₄₀. (c) The stress-strain curve of ATAF₄₀. (ATAF-HSi used ANF as the shell layer and TPU as the core layer. KH792 was added to ANF. H₂O was used as the coagulating bath. The aerogel fibers were prepared using the method described in the manuscript.)

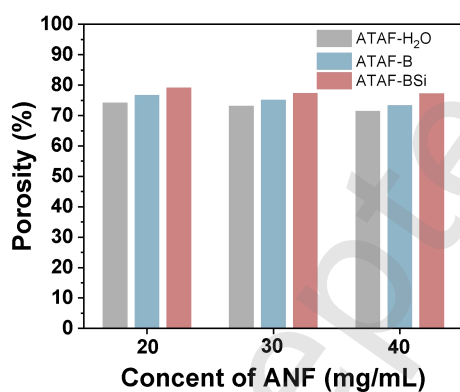


Figure S13. The porosity of ATAF-H₂O, ATAF-B and ATAF-BSi with different concentrations of ANF.

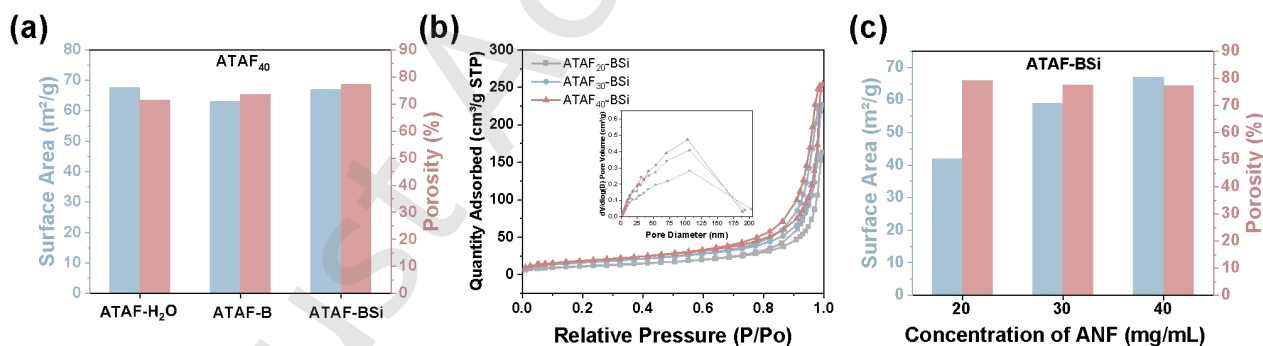


Figure S14. (a) Surface area and porosity of ATAF prepared by the different coagulation bath. (b) Nitrogen adsorption-desorption isotherms and (c) surface area and porosity of ATAF-BSi with different concentrations of ANF.

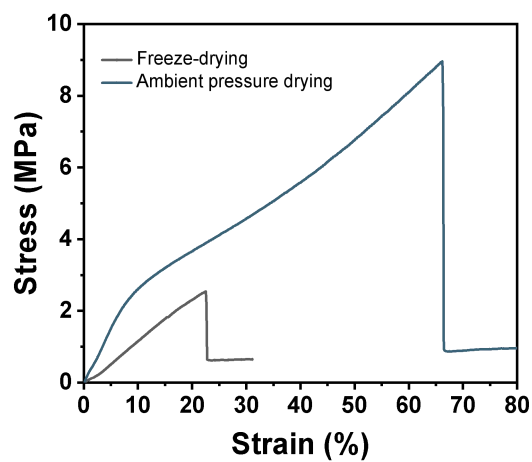


Figure S15. Stress-strain curves of ANF@TPU aerogel fibers prepared by freeze-drying and ambient pressure drying.

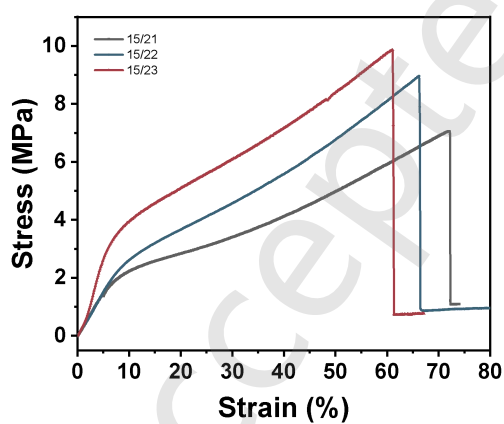


Figure S16. Stress-strain curves of aerogel fibers prepared by different needles (core-shell ratio).

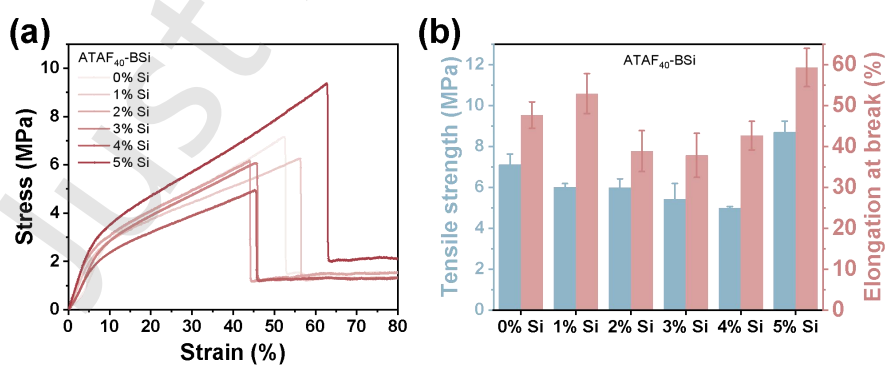


Figure S17. (a) Stress-strain curves, (b) tensile strength and elongation at break of ATAF-BSi with different concentrations of KH792.

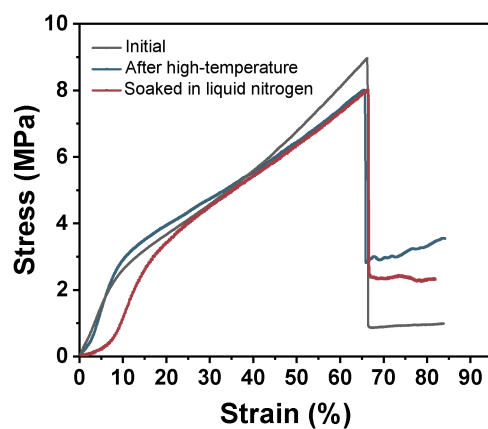


Figure S18. Stress-strain curves of aerogel fibers before and after high and low temperature treatment.

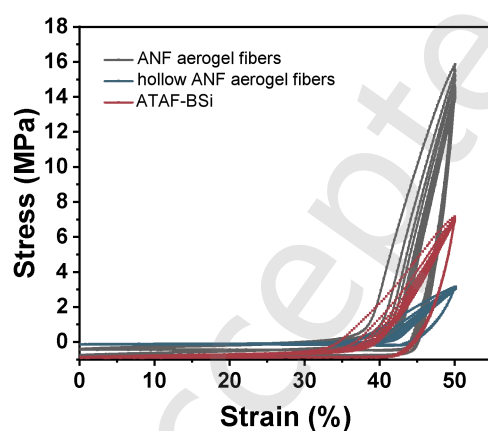


Figure S19. Fatigue tensile properties of an aerogel fiber with different structure.

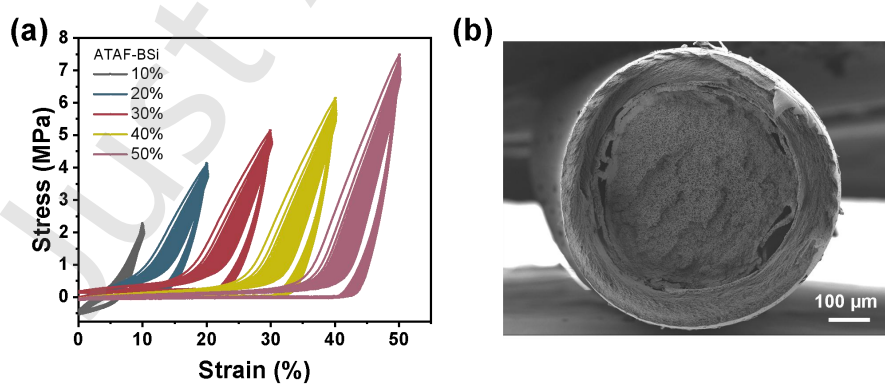


Figure S20. (a) Fatigue tensile properties of an aerogel fiber with different strain. (b) SEM image of the aerogel fiber after fatigue tensile.

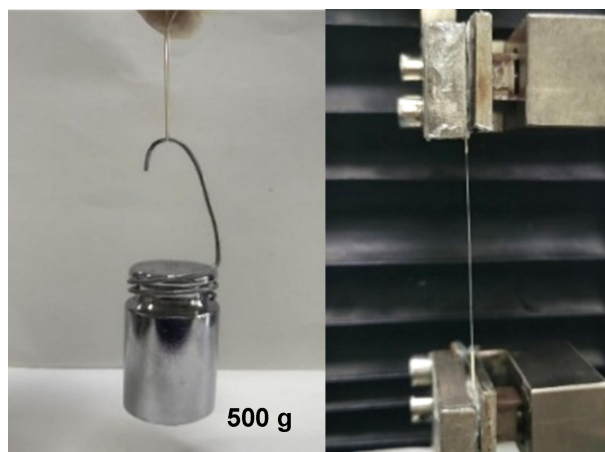


Figure S21. Photographs of single ATAF with a tensile load of 500 g and the state of a single fiber during tensile test.

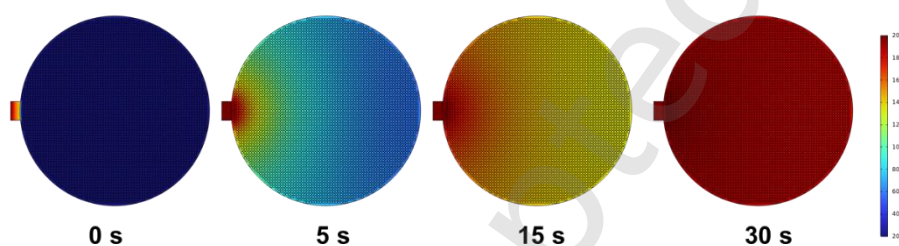


Figure S22. Heat transfer result of the finite element simulation for ANF aerogel fiber.

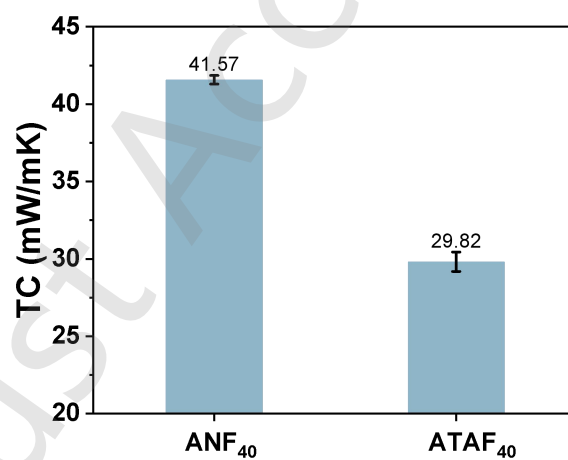


Figure S23. Thermal conductivity of ANF₄₀ and ATAF₄₀ textiles.

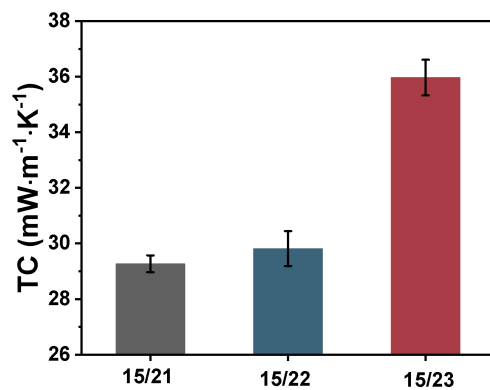


Figure S24. Thermal conductivity of ATAF₄₀-BSi textiles prepared by different needles (core-shell ratio).

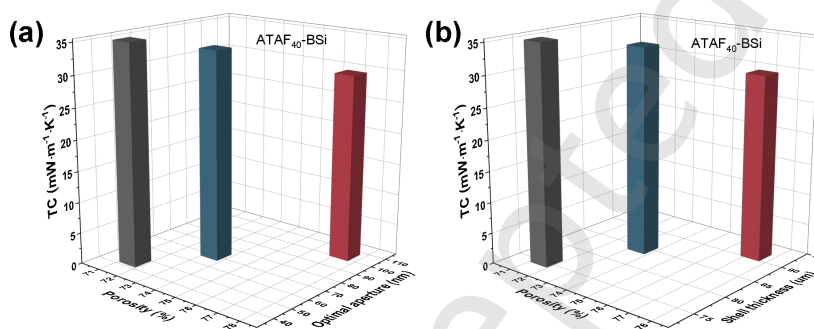


Figure S25. (a) The relationship between thermal conductivity and porosity as well as pore size distribution. (b) The relationship between thermal conductivity and porosity as well as pore closure degree.

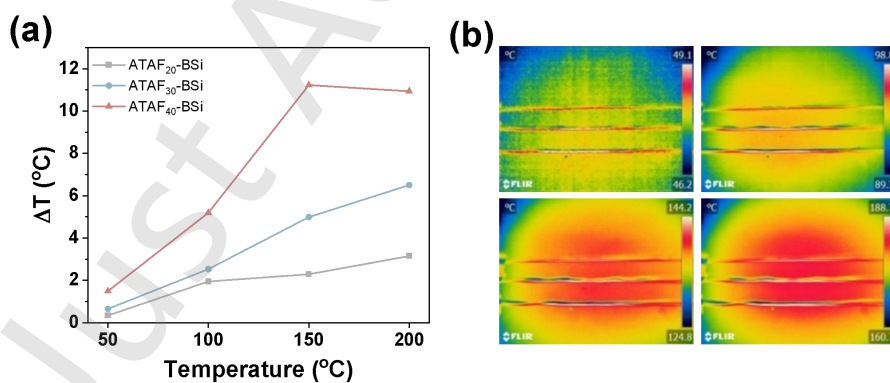


Figure S26. (a) Temperature difference at equilibrium under 50-200 °C of ATAF₂₀, ATAF₃₀ and ATAF₄₀. (b) Infrared thermal images of ATAFs at equilibrium.

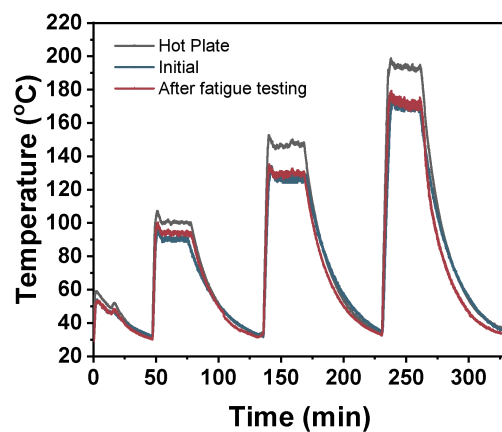


Figure S27. Temperature-time curves of ATAFs before and after the fatigue test under 50-200 °C.

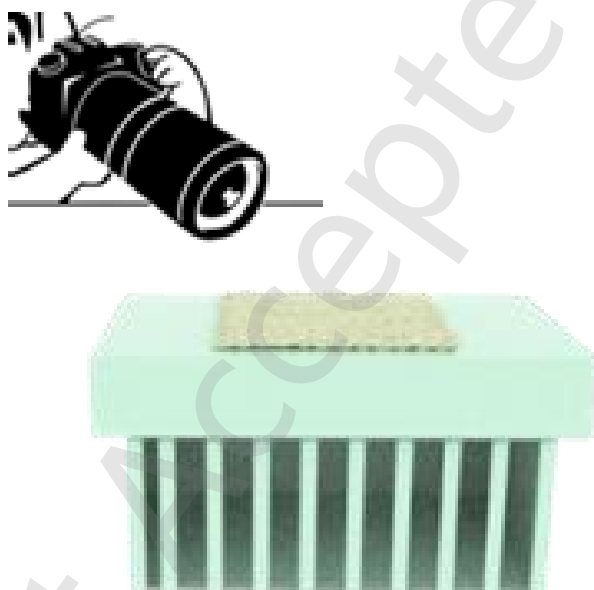


Figure S28. Schematic diagram of fabric insulation test.



Figure S29. The photograph of ATAF₂₀, ATAF₃₀ and ATAF₄₀ textiles after the test on a hot plate at 200 °C.

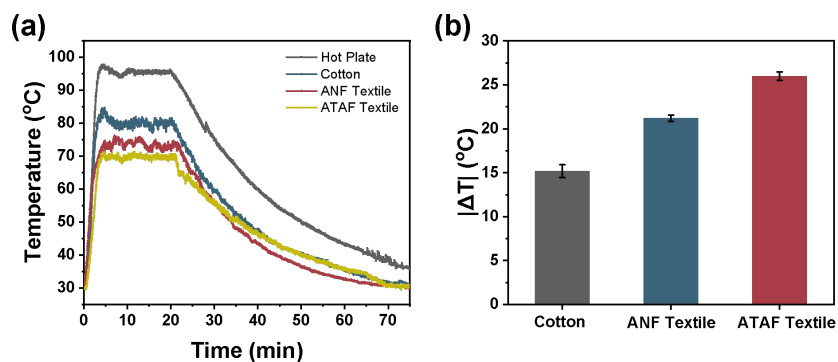


Figure S30. (a) Temperature-time curves and (b) temperature differences (ΔT) between textile surface and hot plate of different textiles.

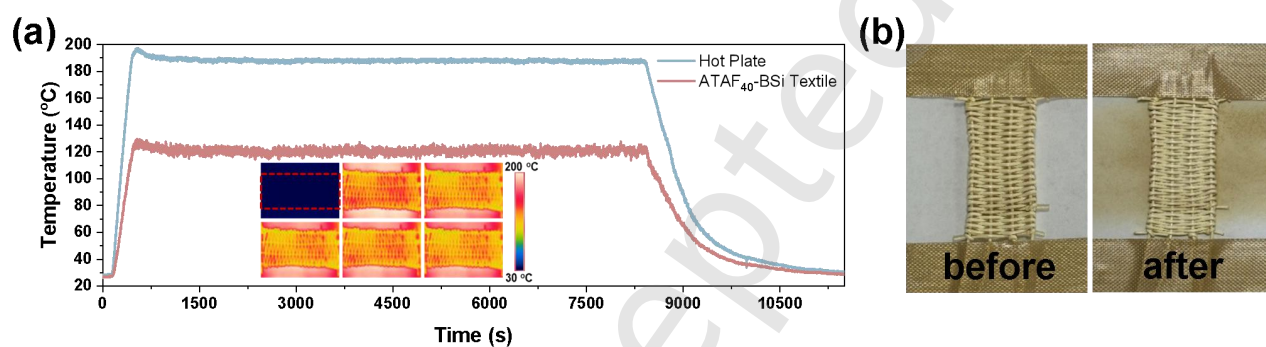


Figure S31. (a) Surface temperature evolution of ATAF textile under long-term exposure at 200 °C hot plate. (b) Photographs of ATAF textile before and after the test.

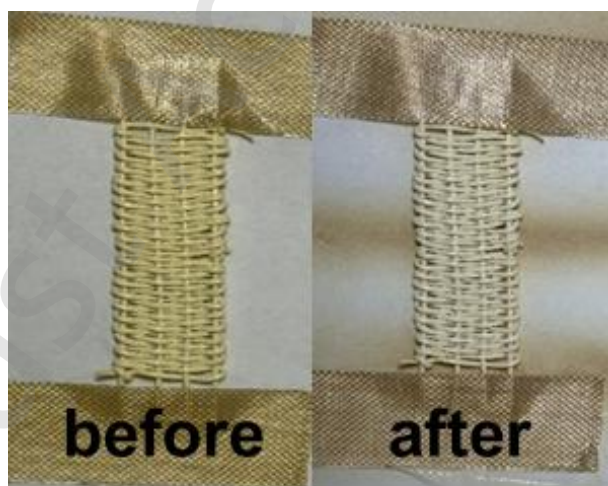


Figure S32. Photographs of ATAF textile before and after the high-temperature cycling test.

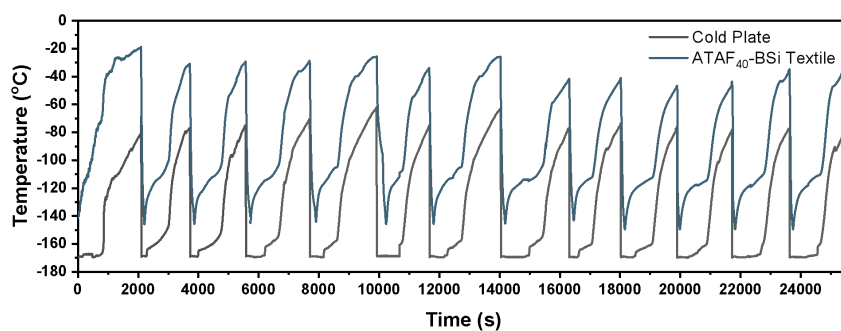


Figure S33. Temperature-time curves of ATAF40-BSi textile in cycling stability test at -196 °C.

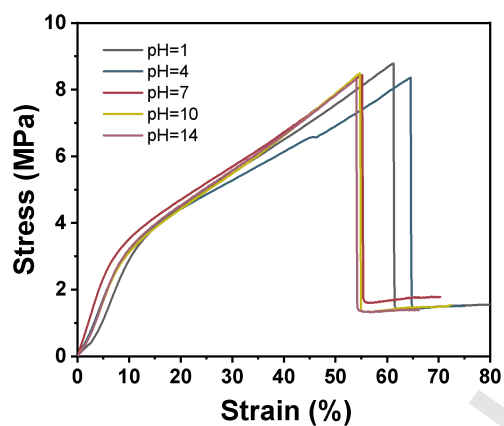


Figure S34. Stress-strain curves of aerogel fibers after immersion in acid and alkali solutions.



Figure S35. Photographs of ATAFs in flame retardant test, ATAF-H₂O (top), ATAF-B (middle), and ATAF-BSi (bottom).

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