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Extreme Resilience: Scalable Electrospun Carbon Nanofiber aerogels with Ultrahigh Elasticity and Temperature Tolerance

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ABSTRACT

The rapid development of the field of aerospace engineering has driven the advancement of special materials with extreme temperature tolerance, mechanical durability, and the capability to monitor physiological signals under extreme environments. However, a significant challenge has emerged in striking a balance between thermal insulation and mechanical properties due to special materials' unstable lamellar structures and uncontrollable molding processes. Herein, we pioneer the scalable production of carbon nanofiber aerogels (CNFAs) using a volatilization-hygrosopicity synergistically induced phase-separation 3D electrospinning molding technology. Their advanced structures grant the CNFAs superb compression resistance (15 000 cycles), an extraordinarily low density (20.15 mg·cm⁻³), and ultra-low thermal conductivity (0.028 W·m⁻¹·K⁻¹). Thus, CNFAs can maintain their original morphology and mechanical behavior at temperatures ranging from -196 to 1500 °C. This breakthrough paves the way for designing and producing functional materials suitable for frontier fields such as space exploration.

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1. Introduction

The in-depth exploration of space has fostered the development of multifunctional specialty materials for spacecraft and spacesuits that can effectively withstand the extreme conditions of space while simultaneously providing protection for astronauts [1–3]. NASA has proposed the Artemis Program to establish a regularized presence on the Moon to pave the way for a future manned landing mission on Mars. Prolonged exposure to extreme environments places greater demands on specialty materials in terms of multiple metrics, requiring a light weight, durability, extreme temperature

tolerance and insulation, and even the ability to monitor and provide feedback on users' behavioral signals [4,5].

Carbon-based aerogels are extensively studied and widely employed in extreme environments such as aerospace and defense applications, owing to their low density, high porosity, low thermal conductivity, high electrical conductivity, chemical inertness, and exceptional thermal stability [6–9]. However, conventional carbon-based aerogels with typical layered structures are brittle and stiff, and unstable contacts at adjacent interfaces can trigger irreversible collapse of the three-dimensional (3D) structure when subjected to large strains [10–12]. Previous works have fabricated carbon-based aerogels with continuous structural units by adding cross-linking materials or constructing lamellar structures in anticipation of improving the material's mechanical properties [13–16]. Nevertheless, the contact mechanism between the cross-linking points/bonding surfaces and the lamellae is unclear and difficult to characterize, and the cross-linked materials lose their intrinsic

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mechanical properties in extreme environments; therefore, it is still a challenge to achieve large-scale preparation of carbon-based aerogels that possess both extreme temperature tolerance and super mechanical stability.

The assembly of special 3D structures from fiber networks is considered a rational and advanced approach in material structure design [17]. From the nano/micro scale to macroscopic complexity, these fiber network structures exhibit fascinating mechanical stability and thermal insulation properties. Although significant efforts have been made to develop carbon-based aerogels with 3D fibrous networks, achieving a balance between extreme temperature tolerance, mechanical durability, physiological signal-monitoring performance under extreme environments, and scalable fabrication methods remains difficult [18–21]. For example, fiber-reinforced carbon-based aerogels prepared via low-temperature freeze-drying exhibit high mechanical strength and durability, but the intolerance of the fiber material to a wide range of temperatures limits its potential application in extreme environments. The laser etching process allows for a wide-range of tuning of the mechanical properties of fiber-reinforced carbon-based aerogels, but the need for ordered structures poses a challenge for batch production [22]. Therefore, developing an efficient, controllable, and cost-effective scalable production strategy for preparing carbon-based aerogels with fiber structures that exhibit extreme temperature tolerance, mechanical durability, and the ability to monitor physiological signals in extreme environments is crucial for breakthroughs in specialty materials within the aerospace field.

Here, we present a volatilization–hygroscopicity synergistically induced phase-separation 3D electrospinning molding technology for the direct batch fabrication of large-area welded-reinforced carbon nanofiber aerogels (CNFAs) with porous interlayers and layer-fiber crosslinks. The urea incorporated into the spinning solution increases moisture retention during electrospinning, promoting phase-separation molding. Acetone (AC)'s high volatility accelerates the premature solidification of nanofibers. This synergistic molding process enables the spontaneous formation of a 3D nanofiber scaffold, eliminating the need for high-humidity conditions. The carbonization process promotes contraction, coiling,

and bending of the fibers in a 3D direction, creating more contact between the fibers and forming welded joints. This facilitates force dissipation when subjected to external forces, resulting in the CNFA's superior compression resistance of 15 000 cycles. In addition, a CNFA with an ultra-low density of $20.15 \text{ mg}\cdot\text{cm}^{-3}$ is not only lightweight but also provides support for wearable comfort. As a result, the CNFA exhibits an ultra-low thermal conductivity of $0.028 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, close to that of air, which provides the CNFA with extreme environmental resistance and stability from -196 to 1500°C . The CNFA still exhibits excellent elasticity, flexibility, and sensing properties in extremely hot and cold environments. We have integrated the CNFA into protective gloves that can monitor the movement of hand joints in real time in a simulated lunar environment (-183 to 128°C), demonstrating its potential for use in human safety alarm systems in space environments (Fig. 1(a)). A comparison of this work with recently reported carbon-based aerogels shows that the comprehensive performance of our CNFA-1200 is at a higher level (Fig. 1(b)) [23–27]. Its extreme temperature tolerance, mechanical durability, and capability to monitor physiological signals in extreme environments are of great significance in the insulating materials used in defense, military weapons, and specialty apparel.

2. Experimental methods

2.1. Preparation of the carbonized precursor (A)

PAN (Molecular weight = 150 000) from Shanghai Aladdin Biochemical Technology Co., Ltd., China was used as the main solute, and DMF and AC were used as solvents. Urea from China National Pharmaceutical Group Chemical Reagent Co., Ltd. was added as a structure–function modifier. The solid content of PAN in the spinning solution was 15%, and the mass ratio of DMF/AC was kept constant at 9.5/0.5. The content of Urea was controlled to vary between 1% and 5% to modulate the humidity response of the spinning solution. The above agents were mixed and stirred for more than 10 h until a homogeneous spinning solution was obtained. The self-developed needle-free electrospinning equipment enables the batch production of 3D nanofiber precursors. During the

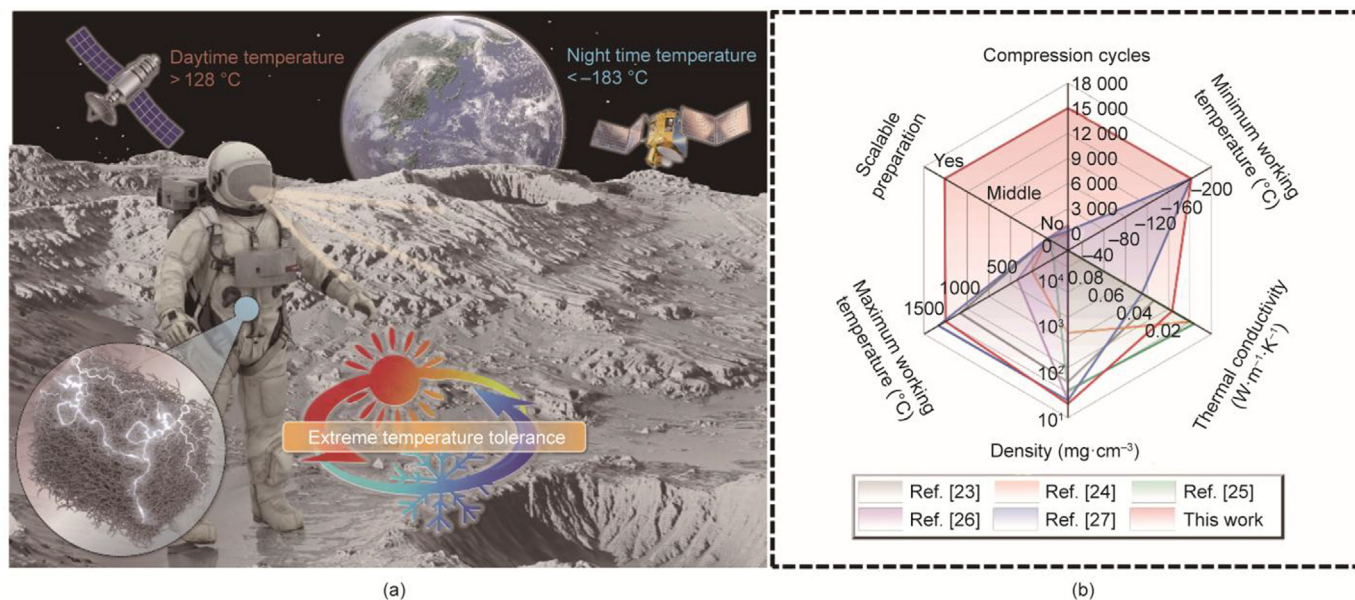


Fig. 1. Applications and performance of CNFAs. (a) Potential applications of CNFA-1200 in lunar environments. (b) Comparison of the CNFA-1200's combined properties, including extreme environmental tolerance, compression cycles, thermal conductivity, low density, and scalable production, with those of other reported carbon-based 3D structural materials.

spinning process, the spinning solution is suitable for a wide humidity range of 30%–90% relative humidity, with a temperature of $(22 \pm 5)^\circ\text{C}$. The voltage is controlled between 50 and 70 kV, and the spinning distance is 10–20 cm.

2.2. Preparation of CNFA (B)

The precursor (A) mentioned above was used for the preparation of the CNFA (B). First, A was pre-oxidized in a vacuum oven at 240°C for 4 h to enhance the mechanical properties and initial structural setting. Then, the pre-oxidized A was placed in air and cooled to room temperature. Subsequently, A was placed in a high-temperature tube furnace under nitrogen gas for high-temperature carbonization ($800\text{--}1400^\circ\text{C}$) at a temperature increase rate of $5^\circ\text{C}\cdot\text{min}^{-1}$. Finally, B was obtained by natural cooling after reaching the specified temperature, and its microstructure and properties were subsequently investigated.

2.3. Characterization

To investigate the microstructure of the CNFA, the microstructure was characterized by means of field-emission scanning electron microscope (FE-SEM; Hitachi SU-8010). Exploration of the dynamic microscopic morphology of the CNFA was carried out using *in situ* electron microscopy (American-FEI600I Dual-beam Electron Microscope; Gatan MTEST 2000ES, USA). A dynamic thermo-mechanical analyzer (DMA; USA-TA-Q800, America) was used to characterize the stress–strain of the CNFA in a high-temperature environment. Thermal conductivity was measured using a Hot Disk TPS 2500 S instrument (Hot Disk AB, Sweden). The hydrophilicity of the CNFA was characterized by water contact angle analysis (Beijing Insunxin Testing Instrument Co., Ltd., China). Its structural stability was demonstrated by testing the compressive strain properties of the CNFA with a microcomputer-controlled electronic universal material testing machine (CTM2050; Xieqiang Instrument Manufacturing (Shanghai) Co., China). The resistance signals of the materials were recorded in real time by a Keithley 2450 multimeter. Thermal images of samples were obtained using an infrared camera (FLIR; T60sc, China). Unless otherwise specified, tests were conducted at a temperature of $(24 \pm 2)^\circ\text{C}$ and a relative humidity of $(60\% \pm 3\%)$.

3. Results and discussion

3.1. Formation of a welding-reinforced ultra-elastic and extreme-temperature-tolerant CNFA

Fig. 2(a) shows the typical molding process of the 3D, fluffy, porous interlayered, weld-reinforced CNFA. Polyacrylonitrile (PAN), a high-quality carbon source material, was selected as the calcined precursor in this spinning solution. Urea was added as a structure–function-modification curing agent and stirred well to improve the hygroscopic properties of the spinning solution. *N,N*-Dimethylformamide (DMF) and AC with high volatility were mixed completely in a fixed ratio under stirring as solvents in the spinning system. The obtained spinning solution showed a homogeneous, clarified, and transparent state (Fig. S1 in Appendix A). During the electrospinning process, the high hygroscopicity of urea promotes the absorption of moisture from the surrounding air, accelerating the phase separation of the induced spinning jet. Meanwhile, the strong volatility of the AC facilitates the rapid solidification of the nanofibers (Fig. S2 in Appendix A). Taking single-needle electrospinning as a typical example, we show the spinning states of solutions with different urea contents to

demonstrate the advancement of the strategy (Fig. S3 in Appendix A). An increase in urea makes the straight-line section of the charge jet in the electrospinning process longer and reduces the diffusion area of the unstable region, both of which are conducive to the advanced molding and curing of the fibers. Corresponding spinning jet simulations were also performed to corroborate our conclusions (Fig. S4 in Appendix A). Three-dimensional nanofiber precursors were prepared via batchization using our previously reported industrialized electrospinning technique (Fig. S5 in Appendix A). The precursors were then pre-oxidized at 240°C to remove excess solvent and underwent initial consolidation and shaping. The 3D structure of the precursor was maintained after pre-oxidation (Fig. S6 in Appendix A). Finally, a CNFA with a thickness-tunable 3D structure different from those of conventional electrospinning products was obtained by means of carbonization (Fig. S7 in Appendix A). Notably, the CNFA can be recycled in a closed loop using vacuum pyrolysis activation, supercritical CO_2 cleaning, solvent-assisted redispersion, and freeze-drying technology.

A range of CNFAs were prepared by controlling the carbonization at different temperatures, including 800°C (CNFA-800), 1000°C (CNFA-1000), 1200°C (CNFA-1200), and 1400°C (CNFA-1400). As shown in Figs. 2(b)–(d) and Fig. S8 in Appendix A, the micro-/nano-scale porous structure formed by intertwined layers of adjacent fibers can be clearly observed. It is noteworthy that the adjacent fibers exhibit a weld-like structure between them, which is visible in the microscopic view. This formation of welded joints can be attributed to the gradual transition of rigid PAN nanofibers into a flexible state when the temperature reaches PAN's melting point. At this stage, adjacent fibers undergo mutual fusion at their contact points, and the fused portions subsequently form welded joints upon cooling. As shown in Fig. S9 in Appendix A, the vertically aligned thermoplastic PAN nanofibers transform into thermosetting nanofibers during high-temperature carbonization, with multiple fibers coiling and bonding together to form welded structures. Separately, the fiber diameter of the PAN nanofibers is concentrated in the range of $0.500\text{--}0.575\ \mu\text{m}$, with an average fiber diameter of $(0.54539 \pm 0.04000)\ \mu\text{m}$. In comparison, the fiber diameter of the CNFAs are predominantly distributed between 0.4 and $0.5\ \mu\text{m}$, with an average diameter of $(0.46217 \pm 0.03000)\ \mu\text{m}$ (Fig. S10 in Appendix A). This results in a significant increase in carbon content, as detected by energy-dispersive X-ray spectroscopy (EDS), which is consistent with the X-ray photoelectron spectroscopy (XPS) results, confirming the carbonization of the PAN nanofibers (Fig. S11 in Appendix A) [28,29]. Correspondingly, we added schematic diagrams before and after carbonization showing that this phenomenon plays a positive role in enhancing the mechanical properties of the fibers and the inter-fiber mechanical properties (Fig. S12 in Appendix A). The stringent humidity requirements for electrospinning and the challenges in achieving large-scale production are major obstacles hindering the advancement of 3D electrospinning molding technology. Compared with previously reported methods, the characteristic feature of the volatilization–hygroscopicity synergistically induced phase-separation 3D electrospinning molding technology is that the charge jet can simultaneously achieve both solvent evaporation and the active adsorption of external water molecules during the fluid phase, causing the charge jet to solidify earlier in the fluid phase. The synergistic effect between solvent evaporation and water molecule adsorption enables this process to maintain essentially the same fiber quality across a wide range of humidity conditions. We prepared CNFAs under three representative relative humidity conditions (RH 30%, RH 60%, and RH 90%) and conducted compression stress–strain tests. The results demonstrate that the stress–strain curves of the CNFAs prepared under different humidity levels exhibit highly consistent trends, which further shows that the proposed 3D electrospinning molding technology is

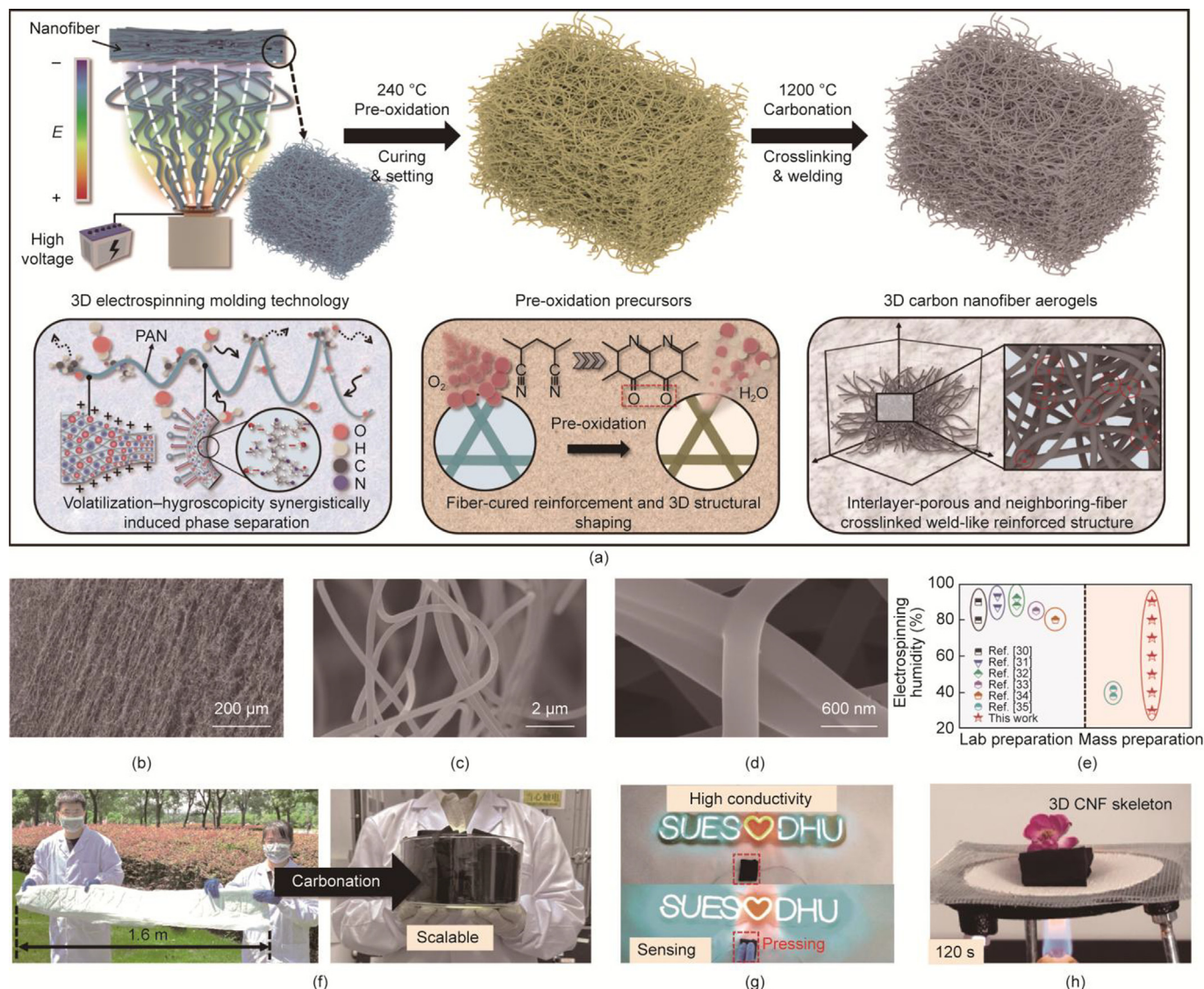


Fig. 2. Design and fabrication of the CNFA. (a) Demonstration of the 3D electrospinning molding technology for the direct fabrication of carbon nanofiber skeletons. (b–d) Scanning electron microscopy (SEM) images of the CNFA at various magnifications demonstrating its fiber-wound and welded structures. (e) The outstanding advantages of the volatilization-hygroscopicity synergistically induced phase-separation 3D electrospinning molding technology in the field of electrospinning 3D structures. (f) Photographs of the precursor nanofiber skeleton and the CNFA prepared at 1200 °C, showing large-scale preparation. (g) Optical image demonstrating the CNFA's high conductivity and sensing performance. (h) Demonstration of the CNFA's excellent thermal insulation performance.

suitable for a wide range of spinning humidity conditions (Fig. S13 in Appendix A). Moreover, mass production is possible with our self-developed equipment (Fig. 2(e)) [30–35]. Based on the mass preparation of calcined precursors, a large pyrolysis furnace can be used to realize CNFA batch preparation (Fig. 2(f)). CNFA-1200's porous interlayered structure and large number of micro-/nano-scale pores give it an extremely low density of 20.15 mg·cm⁻³ and an ultra-light weight (Fig. S14 in Appendix A). The intrinsic porous interlayered structure and welded structural properties of the CNFA-1200 allow it to fully recover after finger pressure and after 180° bending without damaging its structural and mechanical properties (Figs. S15 and S16 in Appendix A). The CNFAs also possess the intrinsically excellent electrical conductivity of carbon materials (Fig. S17 in Appendix A). As shown in Fig. 2(g), CNFA-1200 can be used as a conductor in a circuit to light up a light bulb; moreover, based on its excellent elasticity, it can realize piezoresistive sensing. We also demonstrate the CNFA-1200's water contact angle to show its excellent water repellency, which was main-

tained at 135° even after 5 min (Fig. S18 in Appendix A). Finally, the CNFA's inherent micro-/nano-scale porous channel structure effectively inhibits the thermal collision and heat conduction of gas molecules and thus gives the CNFA-1200 excellent thermal insulating properties. In a test with delicate flowers, the CNFA-1200 prevented the flowers from being destroyed under high-temperature flames (~500 °C) for more than 120 s (Fig. 2(h)).

3.2. Mechanical properties of the CNFA

Compared with previously reported carbon-based aerogels, our CNFA-1200 exhibits fascinating super-elastic properties, which can be attributed to its micro/nano-scale interwoven and welded fiber structure that facilitates force dissipation when subjected to external forces. As shown in the SEM images, the obtained CNFA-1200 displayed a unique structure (Fig. S19 in Appendix A). In contrast to the internal structure and fiber distribution of conventional low-dimensional fiber-based materials, the structure of the

CNFA-1200 showed a spatially structured and randomly entangled convoluted 3D fiber framework, including interlocked convoluted fibers and welded joints. This structure can be attributed to the cross-linking and fusion between adjacent fiber precursors that occur during carbonization and pre-oxidation. The stress (σ)-strain curves (ε) of the CNFA-1200 under loads ranging from 10% to 50% are further illustrated in Fig. 3(a). The stress-strain curves within the 10%–50% load range exhibit a high degree of overlap. In the compression loops corresponding to two adjacent compression strains (e.g., 10% and 20% strain), the peak of one compression loop is essentially located on the next compression loop, highlighting the superior elasticity of the CNFA-1200. At 800 and 1000 °C, the density of the carbonized CNFA is lower, and the maximum stress at 50% compressive strain is less than 4 kPa. At 1400 °C, the CNFA exhibits some local structural collapse, with stress below 2 kPa under a 50% compression strain (Fig. S20 in Appendix A).

We further tested the cyclic durability of the CNFA-1200 through loading and unloading cycles; the results show that the

CNFA-1200 maintains reversible stress-strain cycles even after 15 000 compression cycles (Fig. 3(b)). To provide more convincing evidence of the structural stability of CNFAs, three CNFAs (named CNFA 1, CNFA 2, and CNFA 3) were randomly selected for 15 000 compression cycle tests, with error comparisons conducted on their stress and strain. The results showed that the stress and strain of all three randomly selected CNFAs exhibited minimal errors, once again confirming that CNFAs possess exceptional compression cycle durability (Fig. S21 in Appendix A). In addition, *in situ* SEM images were used to further analyze the local structural evolution of a CNFA-1200 under compression-rebound (Fig. 3(c); Fig. S22 in Appendix A). Under an external force, the typical interwoven and welded structure of curly fibers inside the aerogel was gradually approached along the compression direction. Subsequently, at 50% of the compressive deformation, the curled fiber structure of the CNFA-1200 contracted slightly but without any damage. When the stress was removed, the curled fiber interwoven structure was fully restored to its initial position, highlighting

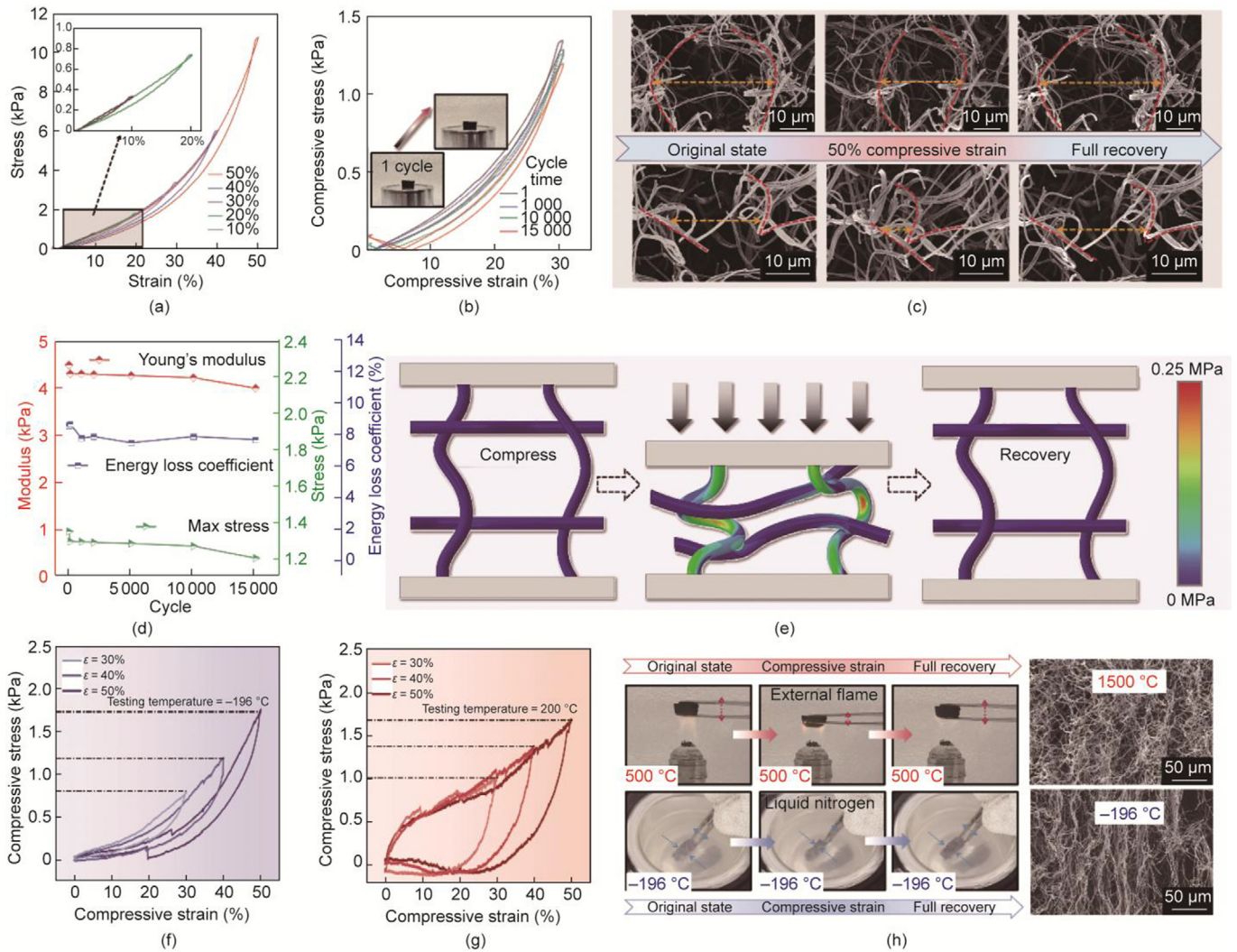


Fig. 3. Characterization of the mechanical properties of the CNFAs under various extreme environments. (a) Stress-strain curves of the CNFA under various compressive strains. (b) Compressibility of the CNFA-1200 at 30% strain for 15 000 cycles. (c) *In situ* SEM observation of the microstructural evolution of the CNFA-1200. (d) History of the Young's modulus, maximum stress, and energy loss coefficient of the CNFA-1200 as a function of the compressive test cycles. (e) Finite-element simulation results for the microstructure evolution of the CNFA-1200 during compression-recovery. (f, g) Stress-strain curves of the CNFA-1200 at various compressive strains under (f) -196 °C and (g) 200 °C. (h) Demonstration of compression performance in extreme environments (flame and liquid nitrogen) and SEM image after carbonization at 1500 °C and freezing at -196 °C.

the structural stability of the CNFA-1200. During the first 500 compression cycles, the structural stability of the CNFA-1200 decreased slightly, with the stress dropping from 1.36 to 1.31 kPa before stabilizing. Impressively, the energy loss coefficient of the CNFA-1200 remained below 10% throughout the entire 15 000-cycle test, further confirming the robustness of the CNFA-1200 structure (Fig. 3(d)).

We selected part of the 3D structure of the CNFA-1200 for equivalent modeling. Mechanical analysis of the compression process was carried out through finite-element modeling (FEM), as shown in Fig. 3(e). When 50% of the compression deformation is applied, it can be clearly observed from the figure that the pressure on the welding point is greater than that on the other positions, indicating that the welding point effectively shares the pressure deformation during the compression process and thus enables the CNFA-1200 to have a good recovery. When the external force is withdrawn, the CNFA-1200 returns to its initial state, in line with our experimental results.

A dynamic mechanical property analysis (DMA) was performed to characterize the thermomechanical stability of the CNFA-1200 over a wide temperature range. The compressive stress–strain curves of the CNFA-1200 in the range of $T = -196$ – 200 °C are shown in Figs. 3(f) and (g). The structure is fully recovered at $\varepsilon = 50\%$ strain, indicating that the CNFA-1200 has elasticity regardless of whether it is at extremely low or extremely high ambient temperature. Among these, the stress–strain curve of the CNFA-1200 under low-temperature conditions exhibits a slight sudden change, which may be attributed to liquid nitrogen seeping into the interior of the CNFA-1200 during compression and thereby affecting the stress distribution. Alternatively, the reversible sudden change in the CNFA-1200 may originate from nanoscale elastic buckling and interface dynamic adjustment, rather than permanent structural damage. These phenomena confirmed that the CNFA-1200 has very good temperature inertness under both low and high temperatures. Therefore, we placed the CNFA-1200 in *in situ* compression experiments in a high-temperature flame (500 °C) and under liquid nitrogen (-196 °C); the results show that the aerogel recovered to its initial geometry after the external stresses were removed (Fig. 3(h); Video S1 in Appendix A). Additionally, the SEM images on the right side of the figure demonstrate that the CNFA-1200 can operate in an inert environment at temperatures up to 1500 °C and at cryogenic temperatures down to -196 °C without significant damage to its microstructure. Furthermore, the CNFA-1200 maintained a regular compressive response after being exposed to a high temperature of 1500 °C for 0.5 h (Fig. S23 in Appendix A).

3.3. Thermal insulation and heat-transfer mechanisms of the CNFA

In addition to the exceptional mechanical properties mentioned above, the CNFA-1200 exhibits excellent thermal insulation and flame resistance. We compared the thermal conductivity of the CNFAs at room temperature with that of raw textile materials for everyday wear and highly insulating polyurethane (PU) foam (Fig. 4(a)). The CNFA-1200 exhibited an extremely low thermal conductivity close to that of air, at $0.028 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, which is also significantly lower than the thermal conductivity of PU foam and textiles. It is worth noting that the thermal conductivity remained at $0.0309 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ even at 100 °C (Fig. S24 in Appendix A). Moreover, due to the excellent temperature inertness of carbon nanofibers, the structure and properties of the CNFA-1200 did not change when it was placed under liquid nitrogen or in flame conditions for more than 180 s (Fig. S25 in Appendix A). As a result, the CNFA-1200 provides an excellent barrier to heat transfer in both high- and low-temperature environments (Fig. 4(b)). This heat-rejection performance is above average [36–38].

For aerogels, thermal conductivity is determined by the thermophysical properties of their constituent units and the geometric configuration of their aggregates. To further elucidate the thermal insulation mechanism of the CNFA-1200, we have provided detailed heat-transfer schematics (Fig. 4(c); Fig. S26 in Appendix A) to facilitate understanding. In this study, the synergistic effect between the porous interlayered structure and the fiber-stacking architecture provides the CNFA-1200 with thermal insulation by effectively restricting heat transfer. Notably, when the intrinsic pore diameter of the material is less than 1 mm, natural convection under ambient pressure becomes negligible due to the low Grashof coefficient. Benefiting from its unique 3D fluffy and porous interlayered structure, the CNFA-1200 can efficiently trap air while simultaneously suppressing all three heat-transfer pathways—namely, solid-phase conduction, gas convection, and radiation—giving it remarkable advantages in thermal insulation applications. Specifically, the layered structure of the CNFA-1200 forces heat to propagate along tortuous paths through stacked arrangements, significantly prolonging the phonon travel distance. Moreover, the CNFA's low solid content and intricate micro-nanofiber pore channels, coupled with its strong phonon-scattering effects, collectively suppress heat conduction through the solid skeleton. The nano-scale pores formed between the fibers not only reduce the possibility of macroscopic relative motion of gas-phase molecules but also scatter electromagnetic waves, thereby extending radiation pathways and reducing thermal conductivity [23,27]. Additionally, owing to the inherent chemical stability of carbon nanofibers, the CNFA-1200 exhibits exceptional flame-retardant properties, even under extreme conditions. When flowers were placed on asbestos mesh and heated underneath with an alcohol lamp, they burned rapidly (Fig. 4(d); Video S2 in Appendix A). However, when a piece of CNFA-1200 was placed between the flowers and the asbestos mesh, the flowers were kept for 120 s under the same heating conditions without morphological changes, demonstrating once again the excellent thermal insulating properties of the CNFA-1200. FEM results also confirm the advanced nature of the nanofiber structure in this work.

Furthermore, the heat transfer of the CNFA-1200 under different temperatures was analyzed by means of finite-element simulations. A local equivalent model of the CNFA-1200 was first constructed, as shown in Fig. 4(e). Then, a set extreme temperature was assigned to the lower end of the CNFA-1200. From the temperature distribution graphs, it can be seen that the multilayer fiber structure of the CNFA-1200 effectively blocks the temperature transfer under both 1000 °C heat-source and -200 °C cold-source conditions, and the results are consistent with the experimental findings. In addition, the varying degrees of *in situ* bending of the CNFA-1200 under extreme temperatures demonstrate its exceptional flexibility, making it a promising material for specialized applications in fields such as aerospace and defense (Fig. 4(f); Video S3 in Appendix A).

3.4. Potential applications of CNFAs in extreme environments

Due to the limited tolerance of traditional sensing materials to extreme environments, the challenge of monitoring astronauts' physiological signals under conditions such as those on the Moon has remained unsolved. CNFAs demonstrate exceptional conductivity and mechanical stability under extreme conditions, positioning them as a promising candidate for the monitoring of human physiological signals in such environments. Sensors ($1.2 \text{ cm} \times 0.6 \text{ cm}$) were prepared by connecting two copper wires as electrodes on one side of the CNFA-1200 and applying a high-temperature-resistant graphite glue to minimize the contact resistance (Fig. S27 in Appendix A). The fabricated sensors were then placed

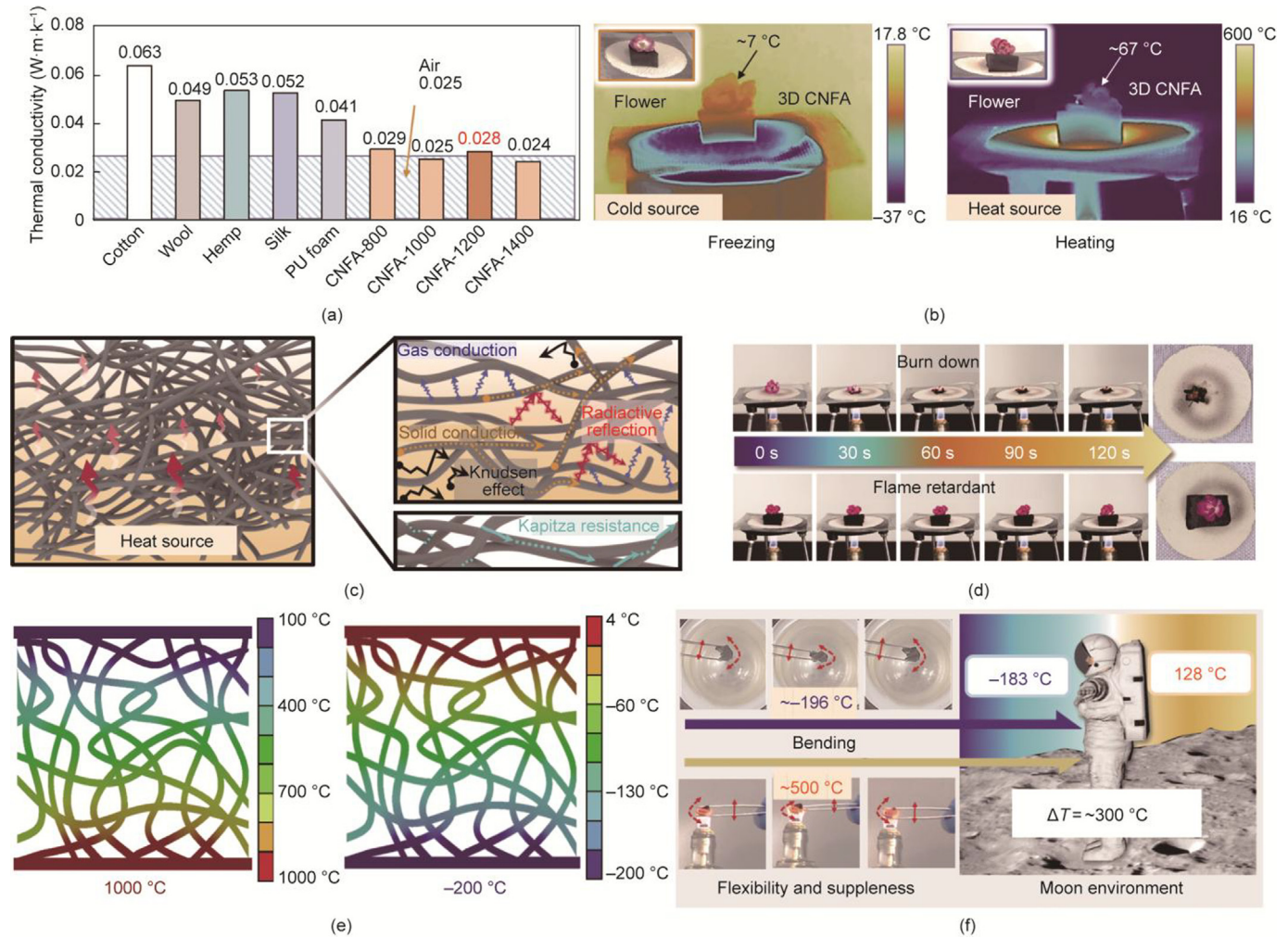


Fig. 4. Temperature insulation and flame-retardant properties of the CNFAs. (a) Thermal conductivities of common textiles used for apparel, PU foam, and CNFAs. (b) Infrared image of the CNFA's hindering effect on temperature. (c) Schematic diagram showing the factors promoting the CNFA-1200's adiabatic capacity. (d) Thermal insulation demonstration: a flower on asbestos net without and with a layer of CNFA-1200 heated by a spirit lamp. (e) CNFA-1200 temperature distribution based on FEM. (f) The CNFA-1200 demonstrates flexibility in extreme environments and shows potential for application in certain special situations such as space environments.

on our self-built compression sensing platform for testing. As shown in Fig. 5(a), the resistance and compressive strain exhibited a good linear relationship when the sensor was compressed. This result is due to the formation of more conductive bridges in the fiber network when the CNFA-1200 is compressed; it is also consistent with our previous observation under *in situ* electron microscopy. An equivalent schematic model upon compression is also shown to complement our conclusions (Fig. S28 in Appendix A). When the sensor is compressed sequentially from 12% to 96%, its resistance shows a clear distinction at different levels of compression (Fig. 5(b)). The current–voltage curves corresponding to the strain states display a uniform shift from 10% to 50% (Fig. S29 in Appendix A). The response and recovery times of the CNFA-1200 reach 20 and 30 ms, respectively (Fig. 5(c)), indicating that the 3D structure of the fibers prevents hysteresis of the compression rebound as much as possible. After 10 000 compression cycles, the resistance of the sensor remained almost unchanged (Fig. S30 in Appendix A). When exposed to air, the resistance of the sensor remained unchanged for up to 180 days (Fig. S31 in Appendix A). These results confirm the CNFA-1200's excellent durability and stability.

The performance of the sensor in extreme environments was explored (Videos S4 and S5 in Appendix A). Firstly, when placed

on a high-temperature flame, the resistance of the sensor dropped rapidly; it then returned to its initial state when placed in a room-temperature environment, which indicates that the high temperature did not change the intrinsic structure and pathways of the CNFA-1200 (Fig. S32 in Appendix A). The sensor was also placed in liquid nitrogen, where its resistance rose rapidly (Fig. S33 in Appendix A). Again, when placed in a room-temperature environment, the resistance returned to its initial state. These exceptional properties triggered the exploration of potential applications of the CNFA-1200 in a simulated lunar environment (Fig. 5(d)). We first conducted compression sensing tests on the CNFA-1200 under flame exposure and liquid nitrogen freezing conditions in a controlled environment with a relative humidity of $(60\% \pm 3\%)$ and a temperature of (24 ± 2) °C. The resulting curves exhibited favorable performance (Fig. 5(e)). The CNFA-1200 had a stable and reliable electrical response even after 1000 cycles of loading at extreme temperatures (Fig. S34 in Appendix A). This stability indicates that the CNFA-1200 holds great potential as a wearable sensor device in extreme environments.

As shown in Fig. 5(f), the CNFA-1200 was further integrated into a protective glove, and we conducted wearable sensing tests in simulated lunar day and night environments. The CNFA-1200 in the glove accurately distinguished and collected signals at different

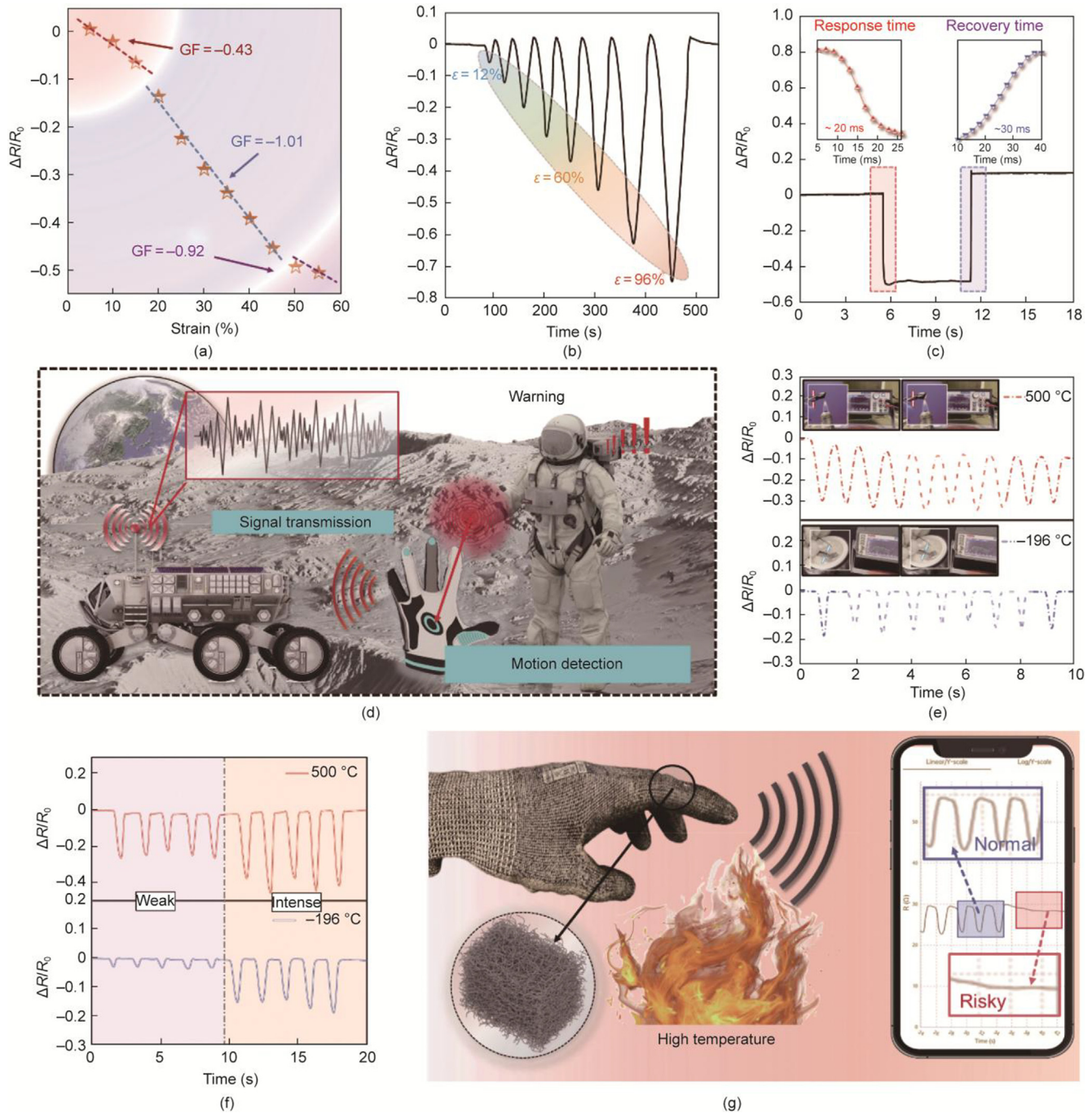


Fig. 5. Sensing performance of the CNFA-1200 at ambient and extreme temperatures. (a) Gauge factor (GF) of the CNFA-1200 under different compression strains. (b) Resistance response of the CNFA-1200 from 12% to 96% compression strain. (c) Response recovery time of the CNFA-1200. (d) CNFA-1200 can be used in the lunar environment for advanced applications such as astronaut body signal detection and physical health warnings. (e) Sensors made of CNFA-1200 were tested in simulated extreme environments (-196 and 500 °C). (f) CNFA-1200 was attached to a glove to detect finger bending under different bending angles at -196 and 500 °C. (g) Integrating the CNFA-1200 with a wireless transmission module allows remote monitoring of the user's body signals in extreme environments.

extreme temperatures: The user first slightly flexes the finger, at which point the CNFA-1200 signal peaks in the figure are small. As the user performs a larger finger-flexing motion, the CNFA-1200 peaks increase significantly. This feature of CNFA-1200 can be used to monitor the safety status of astronauts in real time. As shown in Fig. S35 in Appendix A, when a user works normally in a simulated lunar environment, the sensor signals in the glove show regular changes. When the user is in danger, the disappearance of the motion signal indicates the need for rescue. By connect-

ing the sensor to a wireless Bluetooth module, real-time monitoring of the user's status can be achieved on a mobile phone (Fig. 5(g)). Moreover, the CNFA-1200 reported in this work has excellent water repellency, with no change in resistance even in underwater environments, such that it can be tested for compression sensing underwater (Figs. S36 and S37 and Video S6 in Appendix A). Therefore, CNFA-1200 may be an excellent candidate for thermal protection and smart wear in extreme environments such as aerospace and space exploration.

4. Conclusion

To summarize, we have pioneered a novel volatilization–hygroscopicity synergistically induced phase-separation 3D electrospinning molding technology to batch-prepare CNFAs without the need for strict humidity control. The obtained CNFA-1200 structure shows 3D fluffiness, interlayer porosity, interlocking cross-linking between fibers, and the formation of a weld-like, point-like reinforcement structure, which endow it with superb compression resistance and enable it to remain stable after 15 000 compression cycles. The unique 3D structure and the chemically inert synergistic interactions of the constituent units enable the CNFA-1200 to adapt to an exceptionally broad temperature range (−196 to 1500 °C). The large number of internal micro- and nano-scale pore structures give the CNFA-1200 an ultra-low near-air thermal conductivity ($0.028 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) and excellent conductive paths, making it suitable for temperature protection and signal monitoring in a simulated lunar environment (−183 to 128 °C). This fascinating CNFA-1200, with its variety of excellent properties, is expected to be used as a high-performance insulating and sensing material under extreme hazardous conditions, providing an important support in the future of human cosmic exploration and residence in outer space.

CRedit authorship contribution statement

Shouzhi Yan: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Conceptualization. **Xinyang He:** Writing – review & editing, Writing – original draft, Methodology, Conceptualization. **Zhilin Teng:** Methodology. **Wendi Liu:** Methodology. **Xuepeng Ni:** Methodology. **Zhen Li:** Methodology. **Ding Zhang:** Methodology. **Jinhao Xu:** Methodology. **Binjie Xin:** Writing – review & editing, Validation, Supervision, Resources. **Dahua Shou:** Writing – review & editing, Validation, Supervision, Resources, Funding acquisition. **Liming Wang:** Writing – review & editing, Writing – original draft, Validation, Supervision, Resources, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.eng.2025.11.013>.

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