

Programmable carbon nanotube/shape memory polymer composite fibers with both high actuation strain and stress

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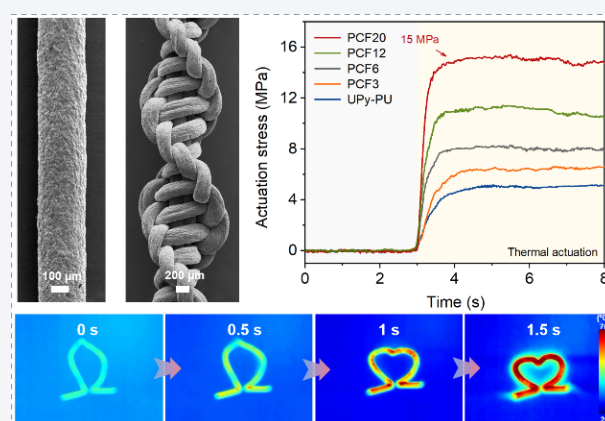
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ABSTRACT: Shape memory composite fiber actuators have been extensively studied due to their excellent flexibility, weavability, actuation performance, and low-cost continuous fabrication. However, integrating both high actuation strain and high actuation stress within a single fiber-based material system remains a key challenge. In this study, we developed a high-performance shape memory composite fiber using a scalable wet-spinning process. The fiber exhibited an actuation stress of 15 MPa and an actuation strain up to 76% within 1 s during thermal shrinkage, along with a high work capacity of 1339 J·kg⁻¹. The electrical actuation achieved through efficient Joule heating also demonstrated an actuation stress of 13 MPa. Mechanistic analysis revealed excellent interfacial bonding between carbon nanotubes (CNTs) and the polymer (polyurethane) matrix. Furthermore, the combined effect of CNTs and crystalline regions promoted tensile alignment of polymer chains, leading to improved mechanical and actuation properties of the fiber. This study demonstrated that the fiber structure enabled integrated actuation and programmed deformation in various two-dimensional/three-dimensional (2D/3D) configurations, with promising applications in future intelligent soft robotics, wearable devices, and smart textiles.

KEYWORDS: shape memory composite fibers, carbon nanotubes, actuation stress, actuation strain, pre-programmed deformation



1 Introduction

Shape memory materials are smart materials that can revert to their original and pre-set shape when exposed to an external stimulus. Shape memory polymers (SMPs) have gained wide attention due to their advantages such as high recoverable strain, multiple deformation modes, ease of processing, and biocompatibility [1, 2]. These strengths made them promising for fields like soft robotics [3, 4], self-deploying aerospace structures [5, 6], wearable devices

[7], and medical equipment [8]. However, the development of traditional SMPs, such as polyurethanes (PU), was hindered by several inherent drawbacks including the trade-off between actuation strain and stress, slow response caused by passive heating, and the lack of programmable complex deformation capabilities.

To overcome these bottlenecks, previous research mainly focused on enhancing the intrinsic properties of polymers and developing reinforced nanocomposites. Polymer properties could be significantly improved through cross-linked networks. For instance, Fu et al. proposed the incorporation of a precise ratio of chemical crosslinks into a supramolecular polymer network to create a high-performance shape memory polyurea relying on covalent-supramolecular synergy [9]. This dual crosslinking strategy, combining physical and chemical networks, enhanced the material's strength, toughness, recovery stress, and work capacity [10].

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However, the irreversible and fixed nature of chemical bonds often restricted chain mobility and limited the material's recovery strain. Consequently, dynamic covalent bonds (e.g., transesterification [11], disulfide bonds [12], metal coordination [13], and hydrogen bonds [14]) have attracted extensive development in recent studies. Leng et al. employed metal-phenolic coordination to construct polyurethanes with intrinsic photothermal effects, which provided efficient energy dissipation and significantly improved the toughness of the polymer network [15]. Light-driven shape memory enabled the deformation of multi-material heterogeneous structures. Although dynamic bonds offered high flexibility and reversibility, they often struggled to deliver high actuation stress. Thus, the nanocomposite approach had emerged as another effective strategy [16, 17]. Molecular design improved the intrinsic "genetics" of polymers, while the addition of nanofillers acted as structural reinforcements; the optimal approach often relied on the integration of both. In recent studies, carbon nanotubes (CNTs) were considered ideal nanofillers in nanocomposite actuators due to their high aspect ratio, exceptional mechanical strength, and electrical conductivity [18]. Liu et al. incorporated 0.1 wt.% CNTs into liquid crystalline elastomers to obtain elastomers with enhanced mechanical properties, achieving an actuation stress of 1.66 MPa [19]. While polymers incorporated with CNTs exhibited enhanced mechanical and shape memory properties, the effectiveness of these improvements was limited by challenges like filler agglomeration and weak interfacial bonding. Building on this, Baughman et al. fabricated a series of actuators based on CNT yarns by either guest infiltration into twisted yarns or yarn coating [20–22]. The continuous yarn network avoided dispersion problems. However, such composite strategies often resulted in trade-offs: Large-stroke actuation was achieved at the expense of reduced actuation stress and work capacity, or enhanced actuation stress came with decreased recoverable strain. In light of these advances, designing nanocomposite structures to maximize the reinforcing potential of nanofillers and to achieve both high actuation strain and stress output remained a critical research direction. Furthermore, the influence of key nanofillers such as CNTs on the crystallization alignment behavior of polymer matrices, which is a critical factor for achieving high-performance nanocomposite actuators, remained largely unexplored. And fiber actuators offered unique advantages due to their one-dimensional morphology, which enabled either individual or collective control for precise and complex motions [23]. Therefore, achieving large actuation strain and stress, high work capacity, and versatile programmability within a single fiber system was highly desired. Exploiting the unique interfacial interactions between CNTs and the polymer matrix provided a promising pathway to develop nanocomposites capable of delivering such integrated actuation performance.

Here, we investigated SMP/CNT fiber-shaped actuators and constructed a multilevel dynamic reinforcing network to reconcile the competing demands between actuation strain and stress. The CNT fillers served as "mechanical skeletons" and thermal/electrical conduction pathways, while the polyurethane matrix incorporating multilevel hydrogen bonds enabled an efficient actuation process. The synergistic effect between the two components significantly enhanced both the mechanical properties and actuation performance of the fiber. Furthermore, the polyurethane's large stretchability and cold-drawing deformation properties enable simple processing to pre-program the fibers into complex three-

dimensional (3D) structures, achieving multifunctional actuation modes. We provided a systematic study of the structure–property–function relationship in the fiber, elucidating the underlying mechanisms and offering a potential strategy for designing next-generation high-performance shape-memory fibers and functional textiles/fabrics.

2 Results and discussion

2.1 Preparation and characterization of composite fibers

Composite fibers were fabricated by wet-spinning and thermal densification (Fig. 1(a)), using a semi-crystalline thermoplastic polyurethane together with acid-treated multiwalled CNTs as raw materials. The polyurethane containing 2-ureido-4-pyrimidinone (UPy) as repeating units possessed a multilevel hydrogen-bonding network [24, 25], which underwent dissociation–sliding–reformation during stretching, thereby maintaining network integrity under high strain and exhibiting good stretchability. Acting as nanofillers, the uniformly dispersed CNTs promoted polymer chain alignment and increased physical crosslinking density, providing the fibers with robust and efficient pathways for stress and heat transfer. In addition, another crucial factor affecting composite performance was the introduction of pores and interfacial gaps during wet-spinning. In this work, pores left by solvent exchange in the coagulation bath were eliminated through thermal-assisted densification, leading to tight bonding between components (Fig. S1 in the Electronic Supplementary Material (ESM)). Figure 1(b) shows scanning electron microscopy (SEM) images of the surface and cross section of the fiber (252 μm in diameter). It could be observed that the densified fiber exhibited no porosity on either the surface or interior. Magnified SEM images clearly revealed uniformly dispersed CNTs embedded in the polyurethane matrix, with tight interfacial contact between the two components. The homogeneous distribution and interconnection of nanofillers within the polymer matrix ensured effective mechanical reinforcement and the formation of continuous conductive pathways. CNTs and polyurethane became entangled with each other, forming a nanocomposite fiber that deformed uniformly. Figure 1(c) shows the UPy-PU/CNT fiber (PCF) in both stretched (Fig. 1(c)(i)) and recovered (Fig. 1(c)(ii)) states. When the strain exceeded 300%, the fiber diameter decreased significantly due to the Poisson effect, and pronounced surface grooves appeared. These grooves originated from inherent microscopic inhomogeneities, such as local variations in diameter, polymer chain orientation, and crystallinity. After the axial strain contraction, these grooves disappear and the diameter returned to its original state. Notably, this microscopic heterogeneity did not impair macroscopic uniformity. As illustrated, the PCF also allowed for complex manipulations, including twisting, single-knot, double-knot, and high-density weaving (Fig. 1(d)), demonstrating its potential as a versatile material for high-performance fibers and functional textiles.

2.2 Mechanical properties and structure characterization of composite fibers

Using the same procedure, we prepared a series of composite fibers with varying CNT contents and systematically investigated the effect of CNT loading (0 wt.%–25 wt.%) on their mechanical properties, as well as the influence of temperature on structure. As

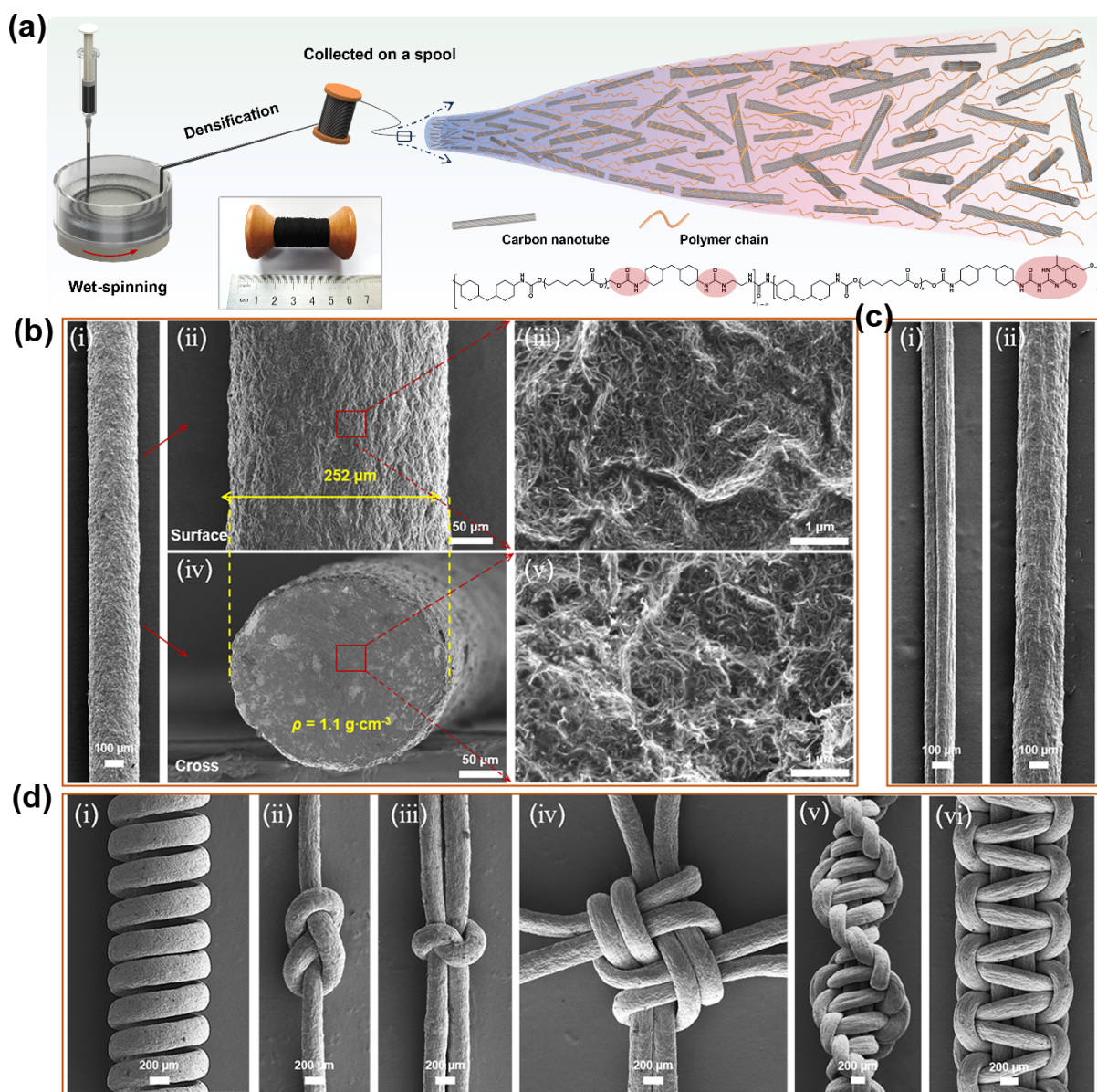


Figure 1 Preparation and structural characterization of PCFs. (a) Schematic illustration of the PCF preparation process. Wet spinning was followed by a thermal densification treatment at an optimized temperature. Inset shows a digital photograph of the PCF wound on a bobbin. (b) SEM images of the densified fiber. (i) The PCF after densification, showing uniform diameter. (ii) and (iii) Surface of the fiber and its corresponding high-magnification SEM image. (iv) and (v) Cross-section of the fiber and its corresponding high-magnification SEM image. (c) SEM images of the fiber in (i) the stretched and (ii) strain recovered states. (d) SEM images of the composite fibers in (i) twisted, (ii) single-knot, (iii) double-knot, and (iv)–(vi) multi-strand braided configurations.

shown in the tensile stress–strain curves (Fig. 2(a) and Fig. S2 in the ESM), when the CNT content increased from 0 wt.% to 25 wt.% (specific weight percentages were determined by thermal gravimetric analysis, Fig. S3 in the ESM), the yield strength of the fibers increased, while the fracture strain monotonically decreased. The fracture strength increased from 16 MPa for the pure PU (UPy-PU) fiber to 80 MPa for the fiber with 25 wt.% CNTs (PCF25), while the tensile strain dropped from 1160% to only 173%. These results thus confirmed the effective strengthening effect imparted by the CNTs. Toughness exhibited an initial increase followed by a decline (Fig. 2(b)), which can be attributed to increased filler agglomeration at higher CNT loading, restricting the plastic deformation capability of the polymer matrix. Therefore, material design can be optimized by adjusting the CNT content to meet different requirements for material strength or toughness in

practical applications. Nevertheless, the inherent advantages of CNTs as fillers, including their exceptionally high aspect ratio as well as intrinsic electrical and thermal conductivities, remained unaffected. Consequently, CNTs formed efficient percolation networks within the polymer matrix, providing rapid transport pathways for both electrons and phonons. Both the thermal and electrical conductivity of the fiber increased steadily with increasing CNT content (Fig. 2(c)). The electrical percolation threshold was observed at a CNT content of 20 wt.% (PCF20).

In addition, we observed an interesting phenomenon: The PCF maintained its fibrous morphology without disintegration or dispersion after immersion in the solvent *N,N*-dimethylformamide (DMF) (Fig. 2(d) and Fig. S4 in the ESM). To further investigate why the fibrous architecture did not collapse, we dispersed the annealed PCFs using an ultrasonic cell disruptor and examined

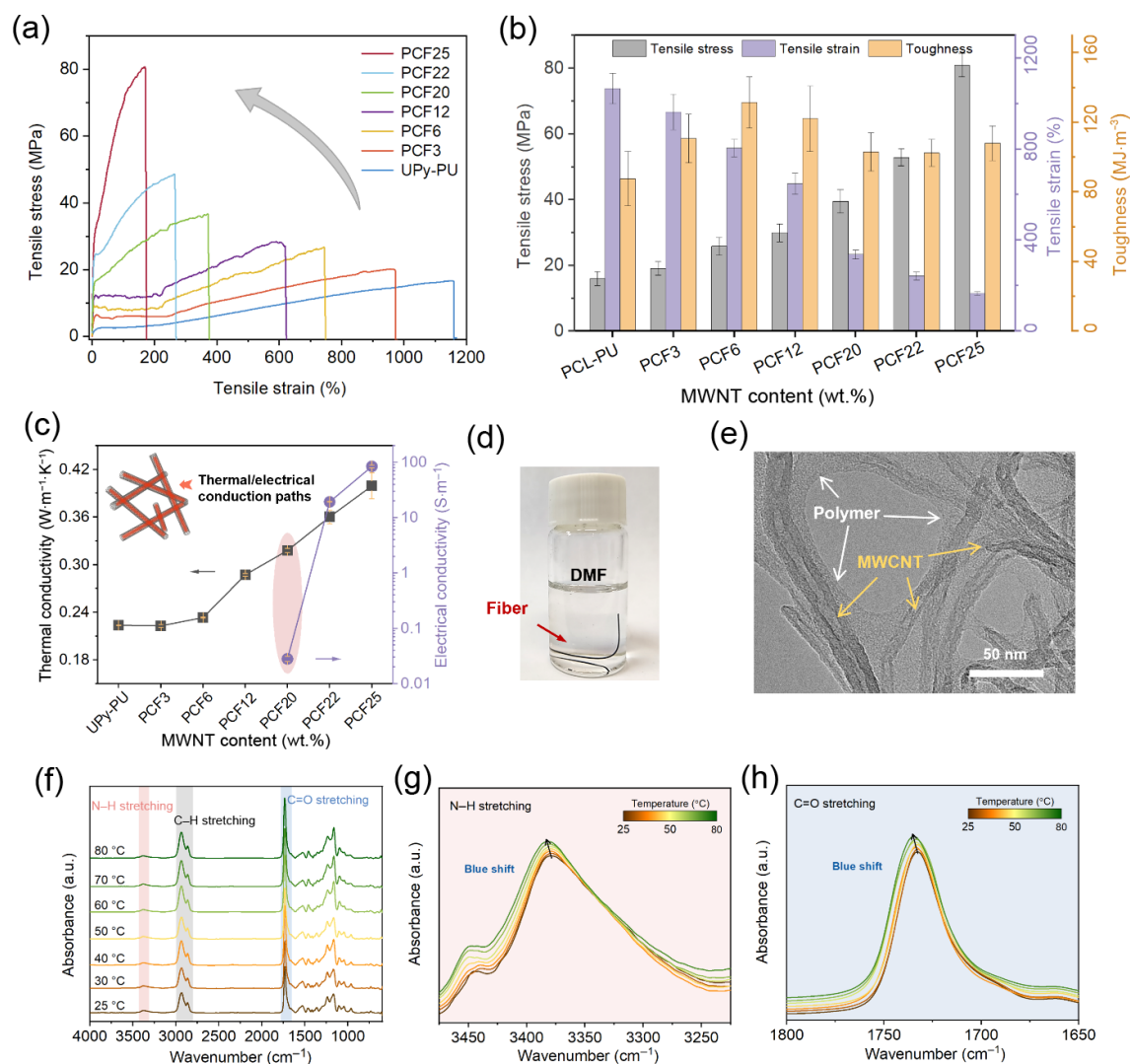


Figure 2 Mechanical properties and structural characterization of PCFs. (a) Stress–strain curves of pure UPy-PU fiber and PCFs with different CNT loadings (0 wt.%–25 wt.%). PCF3 denoted the fiber sample with 3 wt.% CNT loading. (b) Fracture stress, strain, and toughness of neat UPy-PU fiber and PCFs with varying CNT contents (the average and error bar of each data point were plotted from 10 fiber samples). (c) Electrical and thermal conductivity of UPy-PU fiber and PCFs with different CNT loadings (0 wt.%–25 wt.%). Electrical conductivity became measurable above a CNT loading of 20 wt.%. Inset shows a schematic of the electron and Joule heat transfer pathways along the CNT network. (d) Photograph of the fiber maintaining its structural integrity after immersion in DMF. (e) TEM image of the fiber after DMF immersion. Arrows indicate the CNT skeleton and the polymer coated on it. (f) *In situ* variable temperature Fourier transform infrared (FTIR) spectroscopy spectra of PCF20. (g) Absorption peak corresponding to N–H stretching vibration at 3379 cm^{-1} . (h) Absorption peak corresponding to C=O bending vibration at 1732 cm^{-1} .

them by transmission electron microscopy (TEM). These results revealed that even though most of the UPy-PU matrix was dissolved by the solvent, a small amount remained wrapped around the CNT framework, preserving the overall fiber structure (Fig. 2(e)). We inferred that this structural stability arose from the combined effects of van der Waals forces and hydrogen bonding. The oxygen-containing functional groups (such as nitro and carboxyl groups) on the surface of acid-treated CNTs formed strong hydrogen-bond interactions with the polar groups on polyurethane chains. Although DMF solvent molecules partially disrupted these hydrogen bonds, the polymer chains mechanically entangled with CNTs could hardly detach and diffuse into the solvent. The CNT network created substantial steric hindrance, preventing full solvent penetration and restricting mobility of polymer chains. This synergistic confinement also contributed to improved mechanical properties of PCF.

To further elucidate the effect of DMF immersion on the

composite fibers, the mass change and mechanical properties were systematically evaluated before and after solvent treatment. A noticeable mass loss was observed after DMF immersion, indicating the partial removal of the polymer component from the composite (Fig. S5(a) in the ESM). Correspondingly, the stress–strain curves revealed distinct changes in the mechanical response after treatment, reflecting the structural evolution of the fiber (Fig. S5(b) in the ESM). These results suggested that DMF immersion altered the internal microstructure, which was consistent with the TEM observations discussed above.

The thermal performance of composite fibers was as critical as their mechanical properties for practical applications. As shown in the differential scanning calorimetry (DSC) curves, the incorporation of CNTs reduced the glass transition temperature (T_g) of the fibers (Fig. S6(a) in the ESM). With increasing CNT content, the T_g gradually decreased from -55.3 (UPy-PU) to -57.7 $^{\circ}\text{C}$ (PCF25). This behavior was mainly attributed to the

enhanced thermal conductivity provided by CNTs, which rendered the PCFs more sensitive to temperature changes. It is also possible that CNTs introduced minor defects and increased the free volume within polymer network, facilitating chain segment mobility at lower temperatures and thereby lowering the T_g . In addition, hydrogen bonding interactions gradually weakened with increasing temperature. The major hydrogen bond donors and acceptors in PCFs were the N–H and C=O groups within symmetric urea units and quadruple hydrogen-bond structures. Therefore, changes in characteristic peaks of these groups were monitored by *in situ* variable-temperature infrared spectroscopy (Figs. 2(f)–2(h) and Fig. S6(b) in the ESM). During heating of PCF20, both N–H and C=O peaks exhibited significant blue shifts. As temperature increased from room temperature (25 °C) to 80 °C, the N–H stretching peak shifted from 3379 to 3383 cm^{-1} , and the C=O stretching peak shifted from 1732 to 1735 cm^{-1} . This behavior mainly resulted from hydrogen bond weakening or dissociation, which reduced constraints on N–H and C=O groups, increasing their force constant (k). According to Hooke's law, an increase in k leads to higher vibrational frequency, resulting in the observed blue shift. The observation that hydrogen bonding persisted at low temperatures but weakened gradually upon heating was consistent with the thermally responsive behavior of hydrogen bonds reported in literature.

2.3 Mechanism study: Influence of CNTs on the crystallization behavior during deformation

CNTs not only served as nanoscale reinforcements for the fibers but also played a crucial role in influencing the crystallization behavior of the UPy-PU soft segments. In this section, the overall

crystallization behavior of PCFs and the dynamic evolution of crystalline domains during tensile deformation were investigated using DSC and *in situ* two-dimensional wide-angle X-ray diffraction (2D-WAXD). These analyses revealed the mechanism by which CNTs enhanced the actuation output performance of UPy-PU. Crystalline regions in polyurethane facilitate molecular chain orientation and assist shape fixation during cold drawing, serving as an essential component of the shape memory mechanism. The DSC thermograms of fibers with varying CNT contents are shown in Fig. 3(a). Results indicated that as CNTs mass fraction increased, melting enthalpy (ΔH_m) decreased from 44 (UPy-PU) to 18 $\text{J}\cdot\text{g}^{-1}$ (PCF25). Therefore, the overall weight-based crystallinity was calculated to decrease from 32% to 19%. This result indicated that the incorporation of CNTs reduced the overall crystallinity of the fibers to some extent. However, this did not imply a performance loss but rather reflected the natural outcome of spatial confinement imposed by the CNT network. The interconnected CNT framework penetrating the polymer matrix suppressed the development of large crystalline domains and promoted the formation of numerous smaller crystallites. These microcrystals acted as uniformly distributed physical crosslinking points, which more effectively locked in and stored the entropic elasticity generated during deformation, leading to higher actuation stress during recovery.

The role of CNTs during the stretching process of the fibers was also vital. They were not merely fillers but participated in the microscopic mechanisms of deformation and energy storage. *In situ* 2D-WAXD further revealed the microstructural evolution of PCFs under dynamic stretching. Figure 3(b) shows the 2D diffraction patterns of PCF20 and pure UPy-PU fiber during stretching (Fig.

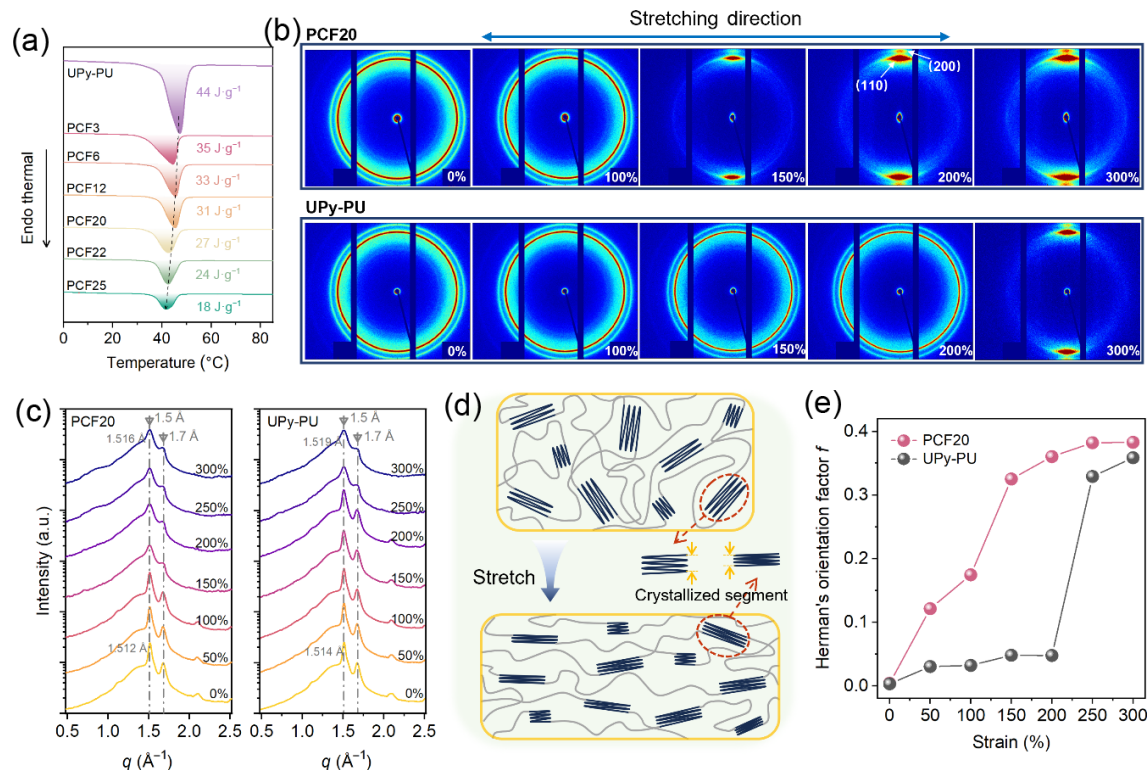


Figure 3 Crystallization behavior of PCFs and analysis of the CNT-induced mechanism. (a) DSC results for pure UPy-PU fiber and PCFs with different CNT loadings (0 wt.% to 25 wt.%). (b) *In situ* 2D-WAXD patterns of PCF20 and UPy-PU fiber. The tensile strain ranges from 0% to 300%. (c) 1D-WAXD curves of PCF20 and UPy-PU fiber. (d) Schematic illustration of the dynamic changes in polymer lattice spacing during stretching. (e) Calculated f for UPy-PU fiber and PCFs under different strain conditions.

S7 in the ESM). In the initial unstretched state, both fibers showed two uniform diffraction arcs corresponding to the (110) and (200) crystal planes, respectively. As shown in Fig. 3(c), the one-dimensional WAXD (1D-WAXD) curves revealed the crystalline domain size and the evolution of lattice spacing during stretching. For the (110) crystal plane, q -values of $1.5 \text{ \AA}^{-1}$ correspond to lattice spacings of $4.18 \text{ \AA}$. Notably, when tensile strain increased from 0% to 300%, the q -value of the pure UPy-PU fiber increased from 1.514 to $1.519 \text{ \AA}^{-1}$, corresponding to a decrease in d -spacing from 4.15 to $4.13 \text{ \AA}$. Similarly, the composite fiber exhibited a minor increase in q value (from 1.512 to $1.516 \text{ \AA}^{-1}$), indicating progressive lattice contraction during stretching. A schematic illustration of the domain evolution during stretching is shown in Fig. 3(d). In addition, 2D-WAXD further revealed the role of CNTs during the stretching of the PCF. During stretching, the diffraction arcs in Fig. 3(b) progressively evolved into two oriented streaks. These sharp diffraction streaks indicated that mechanical deformation promoted local reorganization and alignment of polymer chains along the fiber axis. Notably, at a tensile strain of 150%, the PCF already showed significant splitting in its diffraction pattern, a phenomenon not observed in the pure UPy-PU fiber. It followed that the presence of CNTs imposed spatial confinement effects, enhancing fiber orientation at moderate strains. Overall fiber orientation was quantified by the Hermann's orientation factor (f) obtained from azimuthal integration, which increased with applied strain (Fig. 3(e)). Compared to pure UPy-PU fibers, the PCF showed significantly higher orientation levels, indicating that CNTs promoted stretch-induced polymer chain alignment. Higher orientation allowed polymer chains to store more conformational entropy under external force, which enhanced the recovery stress and work capacity of the fiber.

2.4 Actuation properties of composite fibers

Actuation stress output was a critical indicator of the actuation performance in shape memory composite fibers. Similar to the pure UPy-PU fiber, the presence of multilevel hydrogen bonds (including single, double, and quadruple types) enabled the PCF to be cold-drawn and fixed under external tensile strain (Fig. S8 in the ESM). When exposed to thermal stimulation, these hydrogen bonds dissociated, allowing the CNTs and polymer chains to return to their original disordered state, leading to a full recovery of the macroscopic strain (Fig. 4(a) and Fig. S9 in the ESM). Cyclic uniaxial tests under different strains showed that PCF20 exhibited excellent cyclic stretchability (Fig. S10(a) in the ESM). Based on strain fixation behavior, pre-strain could be designed for subsequent actuation performance. Meanwhile, the recovery strain and response rate were also crucial for shape memory materials. Figure 4(b) shows the shape recovery rate (R_r) and recovery time of PCF20 with the same pre-strain (300%) under different stimulation temperatures (Fig. S10(b) in the ESM). At a stimulation temperature of $55 \text{ }^\circ\text{C}$, the fiber achieved the maximum recovery rate (80%) and the shortest recovery time ($< 1 \text{ s}$). Further increasing the temperature to $60 \text{ }^\circ\text{C}$ did not significantly alter the recovery rate or time. Therefore, with the stimulation temperature set at $55 \text{ }^\circ\text{C}$, we directly measured the actuation stress of the PCF during recovery using an *in situ* mechanical testing setup. This setup measured the actuation stress by constraining the strain recovery and converting the resulting retraction tendency into a macroscopic force, which was recorded as a data curve [26].

The thermal actuation performance of the PCFs was quantitatively evaluated under a pre-strain of 300% (Fig. 4(c) and

Fig. S10(c) in the ESM). A pronounced positive correlation was observed between the actuation stress and the CNT content. The stress increased progressively from 4 MPa for the neat UPy-PU fiber to 15 MPa for the PCF with the highest CNT loading (PCF25). Moreover, all samples achieved rapid shape recovery within 1 s, outperforming most nanofiller-reinforced shape memory polyurethane composites reported previously. This confirmed that the presence of CNTs facilitated polymer chain orientation during stretching, allowing greater conformational entropy storage and thereby leading to higher output stress upon recovery. To investigate the effect of pre-strain on actuation stress, PCF20 fiber was selected for comparison under different pre-strain levels (Fig. 4(d) and Fig. S10(d) in the ESM). It was demonstrated that higher pre-strain resulted in greater actuation stress. At larger strains, polymer chains were more highly extended, leading to a greater entropy change during recovery. Concurrently, more dynamic hydrogen bonds formed between molecular chains, collectively contributing to enhanced actuation stress.

Furthermore, the CNT network within the UPy-PU matrix enabled electro-induced shape memory actuation through the Joule heating effect. First, the electrothermal heating effect was visualized by monitoring the temperature distribution along PCF25 using an infrared camera (Fig. S11(a) in the ESM). The optimal stimulus temperature of approximately $55 \text{ }^\circ\text{C}$ was reached when the current was increased to about 3 mA. Second, to determine a suitable pre-strain range for PCF25, its electrical properties were measured during stretching until fracture (Fig. 4(e)). The results showed that the resistance rose steadily with increasing strain until fracture. Based on this, a pre-strain range of 40%–120% was selected for electrical actuation tests under a constant current of 3 mA. As shown in Fig. 4(f), the electrical actuation stress increased with pre-strain and reached a maximum value of 13 MPa at a pre-strain of 120% (Fig. 4(g) and Fig. S11(b) in the ESM), which was comparable to that achieved under thermal actuation. Although the nonuniform temperature distribution induced by Joule heating led to a slightly lower electrical actuation stress compared to direct thermal actuation, this remote and controllable electrical actuation capability established a foundation for developing next-generation intelligent actuation systems. Figure 4(h) and Table S1 in the ESM compile reported data on the maximum recovery rate and actuation stress of human skeletal muscle and various composite actuators from Refs [19, 27–41].

In addition to high actuation stress, large actuation strain, and rapid response, the composite fibers also exhibited reversible actuation behavior. Under a fixed pre-strain condition, the fibers quickly released actuation stress when subjected to thermal stimulation. Once the external heat stimulus was removed, the actuation stress dropped sharply and stabilized. Upon reapplying the heat stimulus, the fibers were able to regenerate actuation stress, and this process could be repeated for over 20 cycles (Fig. S12(a) in the ESM). This was due to the synergistic reconstruction mechanism of the internal crystalline regions and dynamic hydrogen bonding network. In the cyclic tests, the maximum reversible thermal actuation stress of the PCF20 composite fibers reached 14 MPa. In the electrical actuation mode, reversible actuation behavior was similarly achieved due to the Joule heating effect from the CNT conductive network, with the maximum reversible electrical actuation stress being approximately 12 MPa (Fig. S12(b) in the ESM). The thermal/electrical cyclic test results demonstrate that the composite fibers exhibit both cyclic stability.

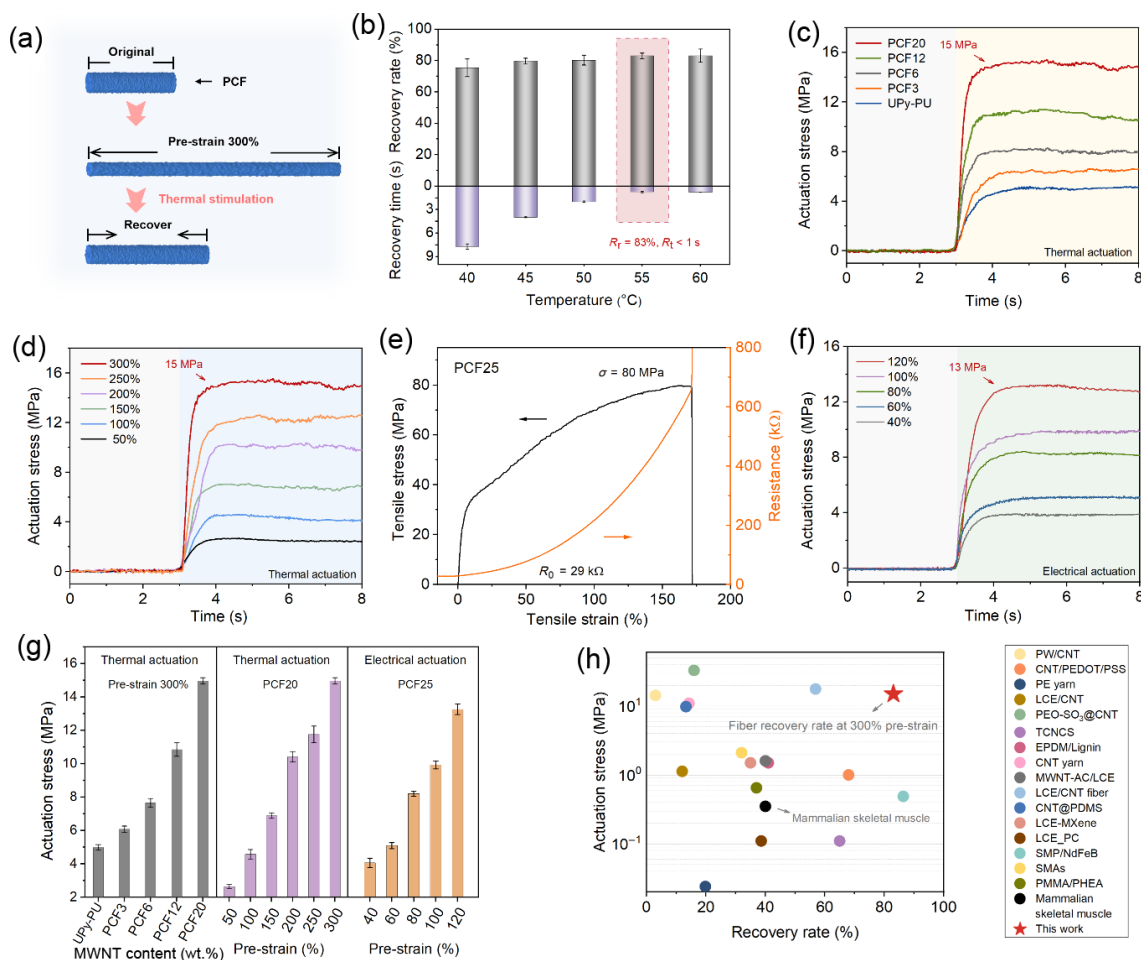


Figure 4 Measurement of actuation stress of PCFs. (a) Schematic illustration of the recovery process for a pre-strained fiber (300%) under thermal stimulation. (b) Thermal recovery rate and recovery time of PCF20 at different temperatures (40–60 °C). (c) Thermally actuated stress of UPy-PU fiber and PCFs (with different CNT loadings) under a pre-strain of 300%. (d) Thermally actuated stress of PCF20 under different pre-strain (0%–300%). (e) Changes in stress and electrical resistance of the PCF25 during tensile testing. (f) Electrically actuated stress of PCF25 under different pre-strain (40%–120%). (g) Statistical summary of the actuation stresses presented in (c), (d), and (f). (h) Comparison of the actuation stress between our fiber and recently reported composite materials.

2.5 Applications of composite fibers in weight lifting

Work capacity, as a key indicator for evaluating actuator performance, was also systematically investigated. The work capacity and power density of the PCFs were further examined through weight lifting experiments. Both thermal and electrical actuation of the PCFs induced pronounced contractile strain, enabling them to lift weights ranging from 1 to 5 g, which corresponded to approximately 2000–10,000 times their own mass. When stimulated by a heat gun, a PCF20 fiber lifted a load 2000 times its own weight (equivalent to an external stress of 0.2 MPa) within 0.8 s (actuation rate of 108 %·s⁻¹), achieving a maximum actuation strain of 76% (Figs. 5(a) and 5(b), and Movie S1 in the ESM). Even under a load of 10,000 times its weight, the fiber still generated an actuation strain of 30% within 1 s. Based on these results, the maximum work capacity and power density for PCF20 under thermal actuation were calculated to be 1339 J·kg⁻¹ and 1408 W·kg⁻¹, respectively (Fig. 5(c)). These values well surpassed the established performance limits of mammalian skeletal muscle (39 J·kg⁻¹ and 50 W·kg⁻¹). For electrical actuation, a constant current source was used, and samples were connected via thin silver wires to minimize the influence of electrode weight (Fig. 5(d)). Under this setup, the fiber (PCF25) achieved an actuation strain up to 60% at a

recovery rate of 70 %·s⁻¹ (Fig. 5(e)). Both the work capacity and power density increased with the external load, reaching maximum values of 385 J·kg⁻¹ and 444 W·kg⁻¹, respectively (Fig. 5(f)). To further advance practical applicability, multiple fibers were integrated to cooperatively lift heavier weights beyond the capability of a single fiber. The combined actuation stress from multiple fibers enabled synchronous lifting of larger loads (Fig. S13 in the ESM). For example, ten fibers with a pre-strain of 300% collectively lifted 20 g of weight under thermal stimulation, delivering an overall actuation strain of 80% and a maximum recovery rate of 100 %·s⁻¹. These characteristics supported the creation of actuators with customized performance, from discrete devices to woven integrated systems.

The performance of this fiber actuator was compared with that of other reported polymer-based actuators [42–56]. Five key parameters were evaluated (Fig. 5(g)): actuation strain (76%), recovery rate (108 %·s⁻¹), actuation stress (15 MPa), work capacity (1339 J·kg⁻¹), and power density (1408 W·kg⁻¹). Overall, the PCF exhibited superior comprehensive performance, particularly in terms of actuation strain and work capacity (Fig. 5(h) and Table S2 in the ESM). Although its actuation stress was slightly lower than that of some shape memory polyurea materials, its excellent

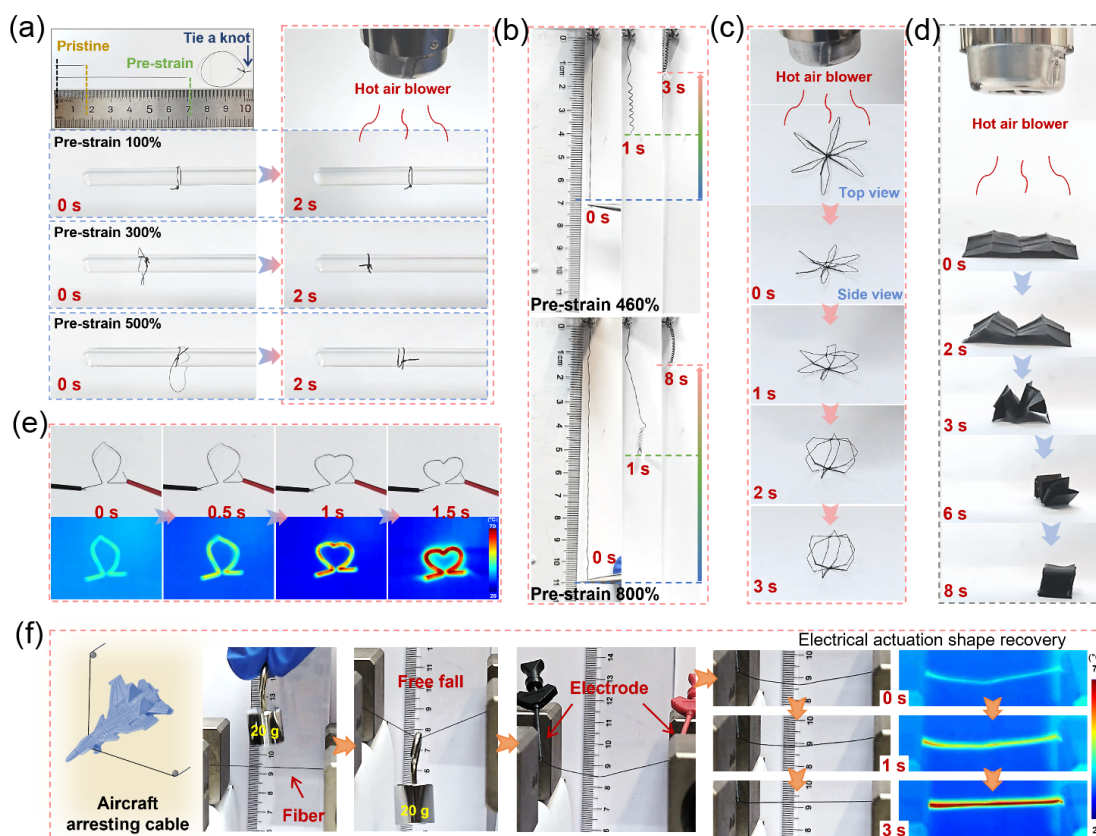


Figure 6 Application demonstration of programmable spatial deformation of PCFs. (a) Series of photographs showing PCFs with pre-strains of 100%–500% contracting and fastening around a glass rod within 2 s under thermal stimulation. (b) Photographs of a hollow spring shaped PCF showing thermally induced shape and strain recovery under different pre-strains. (c) Shape memory recovery of a pre-designed 3D polygonal structure under thermal stimulation. (d) A sequence of images showing the folding process of the composite in film form triggered by heat. (e) Sequential photographs of the shape change into a heart-like structure under electrical stimulation, along with corresponding infrared thermal images. (f) Sequential snapshots captured during the interception of a free-falling weight by the fiber and during its subsequent electrically actuated shape recovery, with corresponding infrared thermal images.

under the impact stress of the falling weight without fracture. After removing the weight, the fiber recovered to its initial length within 3 s under electrical stimulation (3 mA). Infrared imaging confirmed a uniform temperature distribution along the fiber during recovery. The fiber effectively damped the impact of a high-speed object, suggesting its potential use as an electromechanical device similar like an air arrest cable.

3 Conclusions

In summary, a shape memory composite fiber was fabricated via a simple and scalable wet-spinning process, and synergistic enhancement effect between the CNT and the UPy-PU matrix was elucidated. Strong interfacial interactions led to the polymer chains wrapping around the CNT skeleton, forming a robust composite architecture that enhanced the mechanical strength. Moreover, the CNTs promoted the alignment of polymer chains and increased the physical crosslinking density, improving the actuation performance of the fiber. The PCF exhibited notable actuation characteristics, including large actuation strain (76%), actuation stress (15 MPa), high work capacity ($1339 \text{ J} \cdot \text{kg}^{-1}$), and rapid response ($< 1 \text{ s}$). Additionally, the fiber exhibited designable and programmable actuation behavior. Its weavable and integrable characteristics facilitated the design of suitable actuators for different target objects, while the cold-drawing programmability of the fiber enabled predefining its various deformations under specific stimuli, making

it adaptable to diverse operational environments. Combining large actuation strain, high actuation stress, strong work capacity, and designable integration capability, this fiber-based actuator was highly promising for use in wearable electronics, soft robotics, and smart electromechanical systems.

4 Experimental section

4.1 Materials

Multi-walled CNT powder (purchased from Jiangsu Cnano Technology Co., Ltd.), nitric acid, N,N-dimethylacetamide, N,N-dimethylformamide, polycaprolactone diol (average $M_w \approx 3000$), 4,4-methylenebis (cyclohexylisocyanate), dibutyltin dilaurate, 2-acetylbutanolide, guanidine carbonate, triethylamine, ethanol, and deionized water were used as received.

4.2 Preparation of UPy-PU

A mixture of 2-acetylbutyrolactone (2.2 mL, 20 mmol) and guanidine carbonate (1.5 g, 20 mmol) in anhydrous ethanol (20 mL) was refluxed for 12 h under catalysis by triethylamine (5.5 mL, 40 mmol). The reaction was continued until the mixture turned clear, followed by precipitation as a pale yellow solid. The precipitate was washed with ethanol and dried under vacuum to afford a white solid (2-ureido-4-pyrimidinone).

Polycaprolactone diol (7.5 g) was dissolved in anhydrous N,N-

dimethylacetamide (20 mL) with the addition of a few drops of dibutyltin dilaurate as catalyst for pre-activation. After stirring for 1 h, 4,4-methylenebis (cyclohexylisocyanate) (1.5 g) was added to form a prepolymer. UPy (0.3 g) was then introduced and the mixture was stirred for over 6 h until complete dissolution of solids was achieved. All preceding steps were conducted at 85 °C under an argon atmosphere. The temperature was subsequently lowered to 70 °C, and the chain extender ethylenediamine (dissolved in 10 mL of N,N-dimethylformamide) was added (0.1 g). Following 8 h of reaction, the process reached completion. The product was completely dried in a vacuum oven to obtain the UPy-PU solid (M_n = 46.1 kDa, M_w = 20.9 kDa, and polydispersity index (PDI) = 2.2; Figs. S15 and S16 in the ESM).

4.3 Preparation of PCFs

Acid-treated CNTs were pre-dispersed in the organic solvent N,N-dimethylacetamide (5 mL) via probe ultrasonication. After achieving a homogeneous dispersion (Fig. S17 in the ESM), UPy-PU (0.6 g) was added and stirred until fully dissolved. Subsequently, the remaining CNT powder was introduced, and the mixture was stirred thoroughly for 12 h to obtain the spinning solution. The resulting spinning solution was then transferred to a syringe and extruded at a rate of 10 mL·h⁻¹ through a 21-gauge needle (inner diameter: 0.51 mm) into a coagulation bath (deionized water). The fiber was retained in the bath for 5 min, after which it was collected and subjected to free-hanging drying in an oven at 70 °C.

4.4 Characterization of actuation stress

The composite fibers were directly tested for both thermal and electrical actuation using a universal testing machine (Instron 5843). The heat source was provided by a hot air gun, and the power source was supplied by a Keithley 2400 digital sourcemeter. The fiber samples were clamped in place by the two grips of the testing machine, using a tensile mode with a predefined strain value. The machine was then switched to a constant strain stress relaxation mode, and the position of the grips was adjusted to eliminate any external forces applied by the apparatus. At this point, the fiber was in a naturally extended and fixed state. Subsequently, thermal or electrical stimuli were applied to trigger actuation, and the resulting actuation stress was measured over time.

4.5 Pre-programming of spatially deformable patterns

The spatial deformations of various shapes shown in the text represent the recovery process of fibers (temporary shapes) when exposed to external stimuli. The shapes were designed during the drying process, and the temporary shapes (deformations induced by external force) could be fixed via cold-drawing at room temperature. The composite films were prepared by casting the spinning solution into a mold. Shape recovery for all configurations was triggered using either a heat gun set at 70 °C or an electrical power supply.

4.6 Characterization and measurement

Nuclear magnetic resonance (NMR) hydrogen spectra were used to determine the peak spectra of UPy and UPy-PU. The molecular weight of UPy-PU was determined by gel permeation chromatography (GPC). The fiber morphology (surface and cross-section) was characterized by scanning electron microscopy (SEM). Thermal stability and guest–host material content were evaluated by thermogravimetric analysis (TGA) under an air atmosphere.

Dynamic mechanical analysis (DMA) was performed using a DMA 850 instrument in air.

Additionally, the T_g and ΔH_m of the fibers were measured by DSC under a nitrogen atmosphere at a heating rate of 10 °C·min⁻¹. The overall crystallinity of the samples was calculated based on their melting enthalpy.

$$\chi_c = \frac{\Delta H_m}{\Delta H_m^0} \times 100\% \quad (1)$$

where ΔH_m is the experimentally determined melting enthalpy, and ΔH_m^0 is the melting enthalpy for the fully crystalline polymer, taken here as 136 J·g⁻¹.

The orientation and crystalline structure of the fibers were determined using a small-angle instrument equipped with a Cu X-ray source ($\lambda = 1.542 \text{ \AA}$). The orientation parameters f was then calculated according to the Hermans–Stein orientation distribution function

$$f = \frac{1}{2} (3 \langle \cos^2 \theta \rangle - 1) \quad (2)$$

$$\langle \cos^2 \theta \rangle = \frac{\int_0^{\pi/2} I(\theta) \cos^2 \theta \sin \theta d\theta}{\int_0^{\pi/2} I(\theta) \sin \theta d\theta} \quad (3)$$

where $I(\theta)$ is the intensity and θ is the azimuthal angle.

The mechanical properties of the fibers were evaluated using a universal materials testing machine (Instron 5843), including the actuation stress of samples under different loads and pre-strains. Electrical performance was tested with a Keithley 2400 instrument, which also supplied the electrical power during electro-active actuation. Temperature changes during the electro-thermal shape memory process were recorded by an infrared camera (Guide PS600).

4.7 Statistical analysis

All fiber samples used for mechanical property and thermal/electrical actuation stress tests had a gauge length of 15 mm and a diameter of approximately 250 μm . For the weight-lifting experiments, the portions of the fiber beyond the gauge length were used for clamping and weight suspension. Each data point in the graphs represents the mean value and error range derived from three independent experimental measurements.

Electronic Supplementary Material: Supplementary material (SEM images, mechanical properties, thermal analysis, WAXD patterns, shape memory curves, actuator test data graphs, cyclic actuation, NMR, GPC, particle size distribution, and comparison table) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908858>.

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

M. L.: Experimental design, data curation, validation, writing manuscript. K. C.: Data analysis. D. Z.: Validation. Z. M. Y.: Investigation. Q. W.: Data curation. Z. F. J.: Optimization. Y. J. Z.: Project administration. Y. Y. S.: Project administration, writing manuscript. A. Y. C.: Project administration, funding acquisition, writing manuscript. All the authors have approved the final manuscript.

Use of AI statement

None.

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