

A small-bundle single-wall carbon nanotube electrothermal film for smart windows

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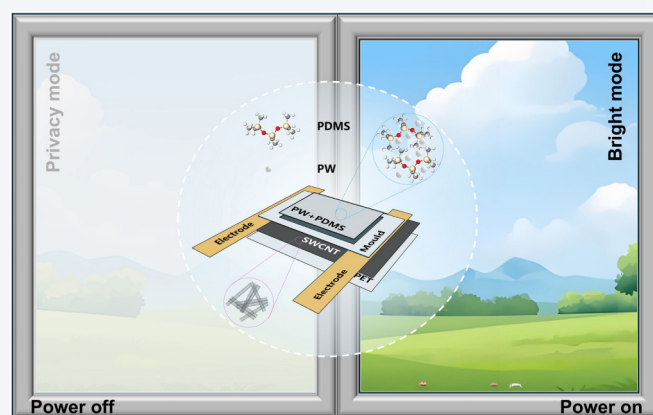
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ABSTRACT: The incorporation of electrothermal films into smart windows has been of intense research interest in developing highly conductive transparent films. However, achieving a high optical transparency and exceptional electrothermal performance simultaneously remains a great challenge. We reported a transparent conductive single-wall carbon nanotube (SWCNT) film comprised of highly-crystalline and long SWCNTs in small bundles. The SWCNT film with an average bundle diameter of 7.1 nm had a low sheet resistance of $26 \Omega/\square$ at a transmittance of 82%, and had a constant temperature of 102 °C at a low applied voltage of 20 V. Integrating this transparent heating film with thermochromic materials enabled the fabrication of a flexible smart window with thermally triggered dynamic transparency. The device has a broad reversible visible light transparency range ($\sim 78\%$) and excellent cycling stability.

KEYWORDS: single-wall carbon nanotube, small-bundle, film, electrothermal property



1 Introduction

A transparent heater is a cutting-edge interdisciplinary product that integrates electrical heating with high optical transparency. Because of its inherent advantages including superior energy efficiency, precise controllability, rapid response kinetics, and reliable operational safety [1], this technology has attracted considerable research interest and demonstrated enormous possible applications in fields requiring both thermal management and visual accessibility. Typical applications include smart windows, anti-icing/deicing systems, anti-fogging devices, and advanced display panels [2–5]. With the rapid evolution and widespread commercialization of curved and flexible electronics, the development of flexible transparent electric heating films with an excellent heating/optical performance has emerged as a critical

research frontier. We reported an advance that not only addresses the technical problems of traditional rigid transparent heaters in adapting to non-planar device structures but also opens up new ways to expand the technology.

Traditional materials and fabrication processes for transparent heaters often fail to achieve a good balance among their performance metrics. Indium tin oxide (ITO), despite being the dominant electrothermal material due to its excellent optical transparency and electrical conductivity, suffers from inherent brittleness and high cost, which severely limits its application in flexible devices. In contrast, metal nanowires or grids offering flexibility are prone to oxidation, have a rough surface, and are structurally unstable at high temperatures [6–8]. Therefore, fabricating electrothermal films that simultaneously have a high transparency, superior electrical conductivity, good stability, and excellent flexibility remains a major problem restricting the practical advance and large-scale commercialization of flexible transparent heating technology.

Transparent heaters based on nanocarbon materials such as carbon nanotubes, carbon nanofibers, graphene, and their hybrids offer a promising solution [9–11]. Because of their simple

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fabrication and excellent electrical/thermal conductivity, these materials have considerable application potential in wearable electronics [12–14]. Among them, carbon nanotube (CNT) films have an excellent transparency coupled with an ultrahigh conductivity and are believed to be an ideal candidate for fabricating next-generation transparent heaters. Hong et al. [15] developed a CNT-based electrothermal film with a high transmittance (> 80%) and good heating performance, which could reach 160 °C at 80 V, demonstrating its potential for rapid de-icing. However, the high operation voltage results in a high energy consumption and potential safety risks. Therefore, achieving excellent electrothermal performance while maintaining high optical transparency has become an urgent and pivotal prerequisite to advancing flexible transparent heating technology.

We addressed this issue by synthesizing single-wall CNT (SWCNT) films containing small nanotube bundles that are highly crystalline and have a long-length by a floating catalyst chemical vapor deposition (FCCVD) method. The long-length and high-crystallinity of the SWCNTs contribute to forming a highly connected two-dimensional conductive network, whereas the small-bundles produce a good balance between the electrical conductivity and transparency of the films. As a result, the SWCNT film obtained has an optical transmittance of 82% with a low sheet resistance of 26 $\Omega/\square$, and achieved a steady-state temperature of 102 °C at a low applied voltage of 20 V. Its excellent electrical and electrothermal properties remain stable under repeated bending cycles, thereby confirming its excellent mechanical durability. To validate its practical use, a flexible smart window was fabricated by integrating a large-area flexible SWCNT film with paraffin wax/polydimethylsiloxane (PW/PDMS). This smart window can be reversibly switched between opaque and transparent over a wide range of thermal stimulations with a maximum transmittance variation of ~ 78%. Our results show that SWCNT films with a high conductivity, excellent flexibility, and superior transparency are ideal transparent heaters that can be used in flexible electronics, electrothermal deicing, and smart windows.

2 Experimental

2.1 Preparation of SWCNT films

An injection FCCVD method (Fig. S1 in the Electronic Supplementary Material (ESM)) was used for the synthesis of SWCNTs [16, 17]. Briefly, a corundum tube reactor with an inner diameter of 46 mm was inserted into a vertical tubular furnace. A gas inlet and catalyst precursor injection port were installed at the top of the reaction furnace. The optimized mass ratio of toluene/thiophene/ferrocene was 10:0.045:0.3. The mixture was delivered into the reaction zone by hydrogen carrier gas, and the growth temperature was 1200 °C. The as-synthesized SWCNTs were transported by the gas flow and deposited onto the surface of a microporous membrane upon filtration, forming a continuous SWCNT film. The obtained film was then placed above a glass container filled with nitric acid at room temperature for 15 min to achieve HNO₃ doping.

2.2 Fabrication of an active light transmitting smart window by integrating the SWCNT film with thermochromic PW/PDMS

PW and PDMS with different weight ratios were melted and

thoroughly mixed at 65 °C under vacuum to remove any bubbles. The PDMS was prepared by combining the base agent and curing agent at a weight ratio of 10:1.

Strip-shaped copper foil electrodes were attached to both sides of the SWCNT film using silver glue to minimize the contact resistance. A rubber frame was then placed on the sample surface, and the prepared PW/PDMS liquid mixture (65 °C) was poured into the frame. Finally, the sample was cured in a drying oven at 65 °C for 6 h (Fig. S2 in the ESM).

2.3 Characterization of the samples

The morphology and structure of the samples were characterized using a FEI Verios G4 scanning electron microscopy (SEM), a transmission electron microscopy (TEM, JEM-F200), and Raman spectroscopy (WITec Alpha 300R) with a 633 nm laser. A polarizing optical microscopy (POM, Leica DM2700P) was used to observe the crystalline PW morphology. A stable direct current (DC) power (Keithley 2461 source meter) was directly connected to the SWCNT film to test its electrothermal properties. The phase change temperatures and enthalpies were determined using a differential scanning calorimeter (DSC, TA DSC25) under a nitrogen atmosphere at a heating/cooling rate of 10 °C/min. The sheet resistance was measured by using a four-point probe resistivity system (RTS-9).

3 Results and discussion

3.1 Characterization of the SWCNT films

Typical SEM images of the SWCNT films synthesized under carrier gas flow rates of 5000 and 6500 sccm are shown in Fig. 1(a) and Fig. S3 in the ESM, which show a porous network structure composed of randomly entangled SWCNT bundles. TEM images of these two samples (Figs. 1(b) and 1(c)) clearly show different bundle sizes, i.e., by simply regulating the carrier gas flow rate during SWCNT growth, the bundle diameter in the resulting SWCNT films is effectively controlled. The average bundle diameters of samples synthesized with 5000 and 6500 sccm carrier gas flows were statistically analyzed to be 14.9 and 7.1 nm, respectively, by measuring 254 individual bundles under TEM (Fig. 1(d)). Both of the samples have excellent crystallinity, and their G-band to D-band intensity ratios (I_G/I_D) reached 171 and 168, respectively (Fig. 1(e)).

The different bundle diameters are caused by the inherent tendency of SWCNTs to diffuse and coalesce by Brownian motion during the FCCVD process. The kinetics of this coalescence process is governed by the collision rate of aerosol particles which depends on the mobility diameter (D_M), temperature (T), and carrier gas viscosity (η), as described by Eq. (1) [18, 19]

$$\frac{dN}{dt} = -K(D_M, T, \eta) N^2 \quad (1)$$

where the collision rate of aerosol particles is determined by both the number concentration (N) and the coagulation coefficient (K). When the hydrogen carrier gas flow rate increased from 5000 to 6500 sccm, the concentrations of the catalyst, growth promoter, and carbon source in the reactor decreased accordingly, resulting in a lower number of available nucleation sites for SWCNT growth. Simultaneously, the increased flow rate decreased the residence time of the reactants in the high-temperature zone and hence, the growth time of SWCNTs. As a result, the total yield of SWCNT

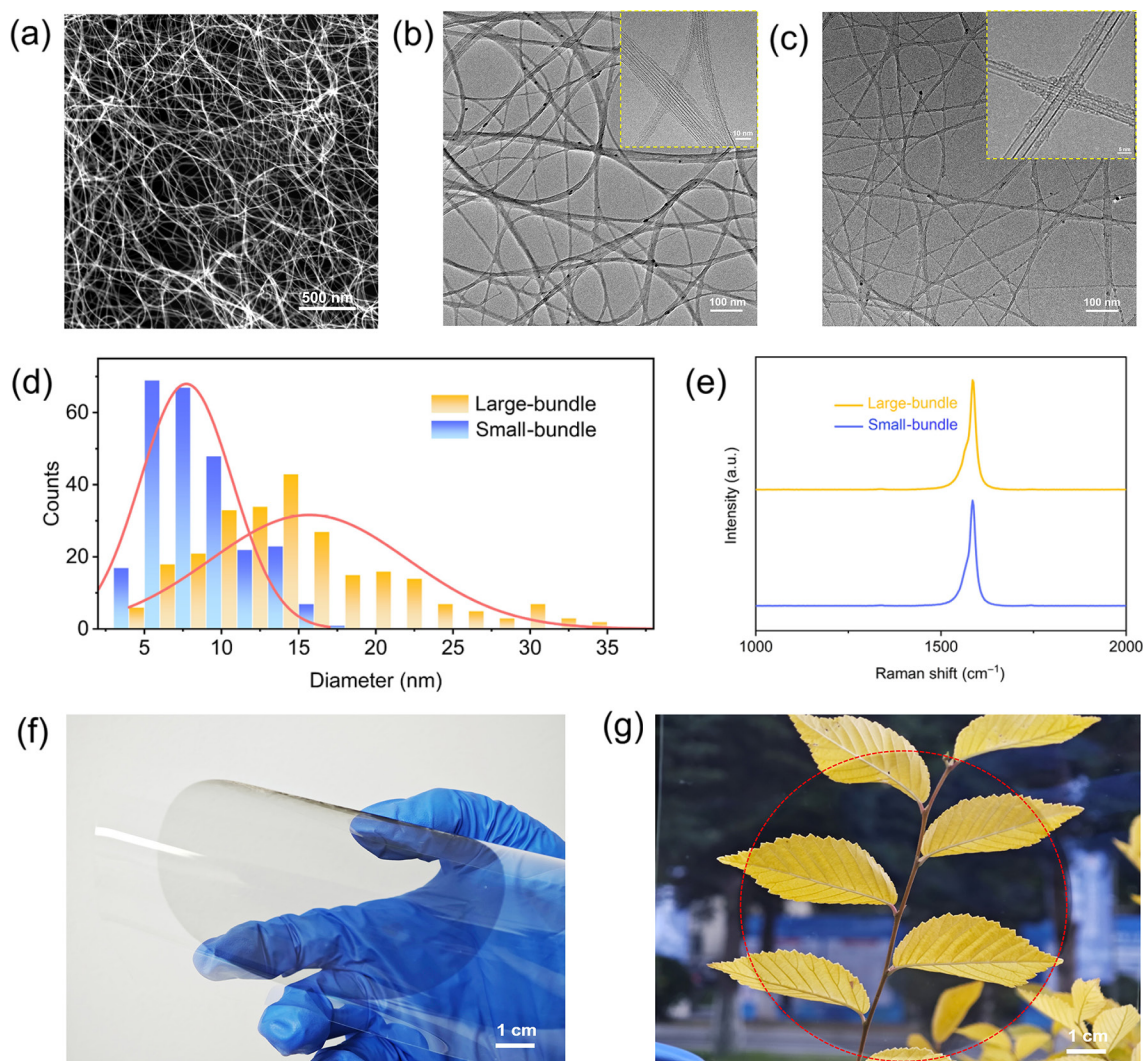


Figure 1 (a) Typical SEM image of the small-bundle SWCNT film. ((b) and (c)) Typical TEM images of (b) the large-bundle and (c) small-bundle SWCNT films. (d) Statistical bundle diameter distributions of large-bundle and small-bundle SWCNT films based on TEM observation and measurements. (e) Raman spectra of the small-bundle and large-bundle SWCNT films. ((f) and (g)) Optical images of a small-bundle SWCNT film transferred onto a PET film, demonstrating its good flexibility and high transparency.

decreased, which effectively suppressed the van der Waals interaction-driven agglomeration [20]. Therefore, SWCNT films are predominantly composed of smaller bundles or even individual nanotubes under high gas flow conditions. Figures 1(f) and 1(g) show the successful dry transfer of the SWCNT films onto a polyethylene terephthalate (PET) substrate by simple imprinting. The transferred films maintain a high optical transparency, good mechanical flexibility, and controllable size (Fig. S4(a) in the ESM). Typically, small-bundle SWCNT films with an optical transmittance of $\sim 82\%$ (Fig. S4(b) in the ESM) were used for the subsequent experiments in this study.

3.2 Heating performance of the SWCNT film

The core of the electrothermal performance lies in the conversion of electrical energy to thermal energy. This process follows the principle of thermal equilibrium: When a material is electrified and heat is generated, its temperature rises until the rate of heat production balances the rate of heat dissipation [21]. Applying a DC voltage U causes the surface temperature of SWCNTs to gradually increase from T_0 (initial temperature) to a stable

saturation temperature T , indicating the establishment of thermal equilibrium. The relationship between the saturation temperature T and the voltage U can be expressed as [22]

$$T = T_0 + \frac{U^2}{RhA} \quad (2)$$

where A and h represent the working area and convective heat transfer coefficient of the material, respectively, and R is the sheet resistance of the film. It can be seen that a material with better electrical conductivity will reach a higher saturation temperature and correspond to better electrothermal conversion efficiency.

We first investigated the electrical properties of SWCNT films. As shown in Fig. 2(a), the sheet resistances of the small-bundled and large-bundle SWCNT films after nitric acid doping are 26 and $60.8 \Omega/\square$, respectively. In addition, we evaluated the uniformity of the ~ 8.3 cm-diameter film by dividing it into 44 identical regions and measuring their transmittance and sheet resistance. As shown in Fig. S5 in the ESM, the deviations for the transmittance and sheet resistance are only 0.3% and 1.0%, respectively, demonstrating a good uniformity of the SWCNT films. Furthermore, we adopted a

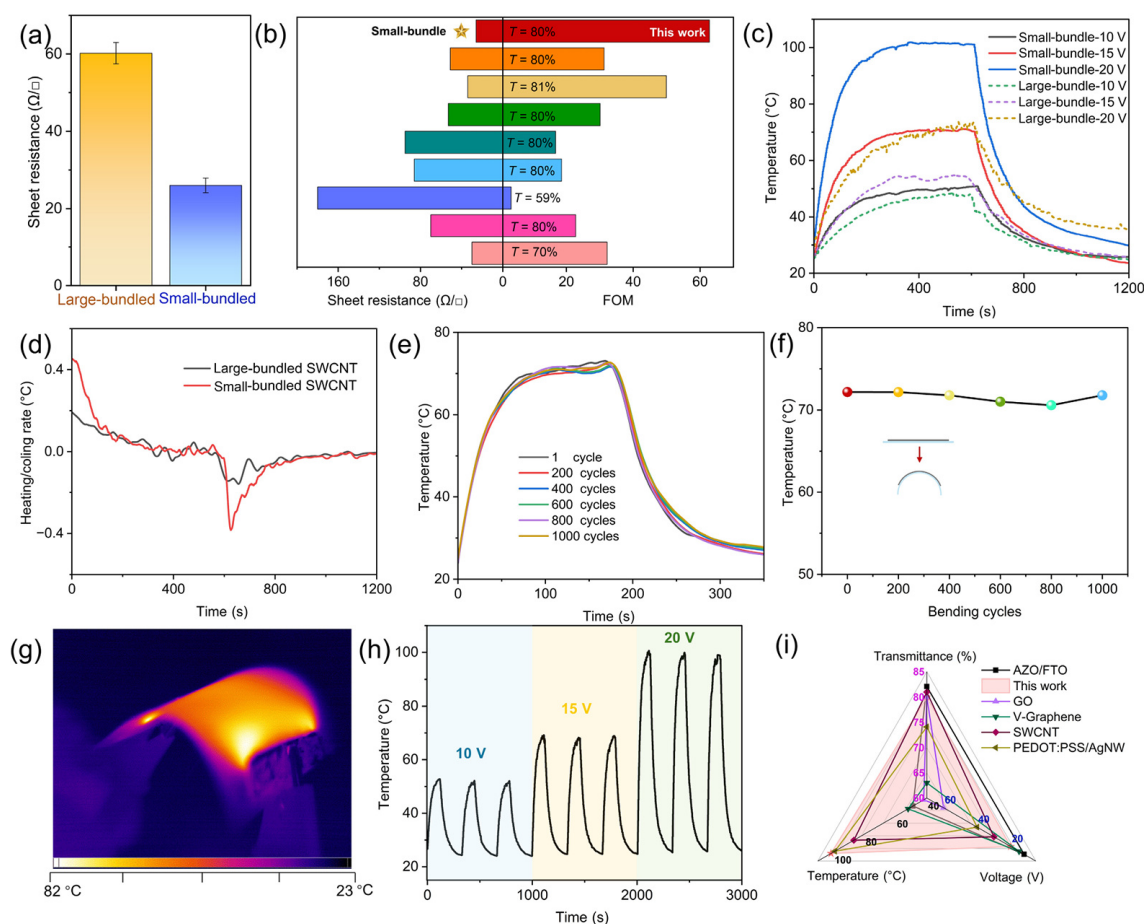


Figure 2 (a) Sheet resistance of small- and large-bundle SWCNT films. (b) FoM comparison of our small-bundle SWCNT film with those of reported CNT films. (c) Electrothermal response of small- and large-bundle SWCNT films at applied voltages of 10, 15, and 20 V. (d) Heating rates of small- and large-bundle SWCNT films at 15 V. (e) Electrothermal curves and (f) steady-state temperatures of the small-bundle SWCNT film under different bending cycles at 15 V, and (g) the corresponding infrared (IR) images recorded after 1000 bending cycles. (h) Temperature evolution of the small-bundle SWCNT film during a step-wise voltage increase from 10 to 15 and 20 V. (i) Radar chart comparing the electrothermal performance of our small-bundle SWCNT film with those of previously reported materials.

figure of merit (FoM) to comprehensively evaluate the performance of these SWCNT films (Note S1 in the ESM) [18, 23]. The small-bundle SWCNT film has a record high FoM value of 62 (Fig. 2(b)) [24–30], which is 2.3 and 1.26 times that of the large-bundle one and the highest value reported for SWCNT films [31], respectively.

The electrothermal heating performance of the SWCNT films was also investigated. All tested SWCNT films were circular with a diameter of 50 mm. Under an applied voltage of 10 V, the temperature of the small-bundle SWCNT film rapidly increased to ~ 50 °C (Fig. 2(c)), about 4 °C higher than that of the large-bundle SWCNT film. When the applied voltage was increased to 20 V, the temperatures of the small-bundle and large-bundle SWCNT films reached 102 and 70 °C, respectively. Overall, the large-bundle SWCNT film had an inferior performance in both the final temperature and heating rate than the small-bundle SWCNT film at voltages of 10, 15, and 20 V (Figs. 2(c) and 2(d), and Fig. S6 in the ESM). To mimic potential deformation scenarios under practical operation conditions, we conducted 1000 bending cycles for the small-bundle SWCNT film. It was found that the electrical properties of the SWCNT film remained stable throughout the process, demonstrating excellent flexibility and cyclic durability that far surpass those of commercial silver nanowires and indium tin oxide (Fig. S7 in the ESM). The temperature–time curves and real-time heating performance of the deformed film were also recorded

throughout the cycling (Figs. 2(e)–2(g)), and the small-bundle SWCNT film heater rapidly achieved the target temperature where it showed good stability with temperature fluctuations within ± 2 °C (Fig. 2(h)). A radar plot comparing the multifunctional properties of our small-bundle SWCNT film with those of other reported flexible transparent conducting films is shown in Fig. 2(i) [32–36]. Our small-bundle SWCNT film had a high optical transparency of ~ 82%, a low driving voltage of 20 V, and a steady-state temperature of 102 °C. These performance metrics are far superior to those of other reported transparent electric heating materials, making it an ideal candidate for fabricating flexible thermal management devices.

3.3 Effect of bundle size on the electrothermal performance of SWCNT films

The superior electrical performance of the small-bundle SWCNT films primarily stems from the structure of their conductive network, a process governed by the fundamental principles of percolation theory. As illustrated in the diagrams of Figs. 3(a) and 3(b), for the same transmittance, which corresponds to the same nanotube loading, individual or small-bundle SWCNTs form a denser conductive network than large-bundle nanotubes, resulting in a substantial reduction in the percolation threshold of the network. In other words, a network composed of small-bundle

SWCNTs provides more good conductive paths per unit area. To quantitatively assess these conductive paths in the thin films, the number density of conductive channels was computed for large-bundle and small-bundle films at a fixed transmittance ($\sim 95\%$). TEM images (Figs. 3(c) and 3(d)) were binarized using the Otsu thresholding method, which maximizes inter-class variance between foreground and background pixels to locate and identify carbon nanotube regions. A statistical analysis based on this (Figs. 3(e) and 3(f)) shows that the SWCNT area fraction is 9.16% in the large-bundle film and 16.81% in the small-bundle one. The small-bundle system thus has a greater areal nanotube density, producing more efficient conductive paths and producing a higher electrical conductivity at the same transmittance. According to the above analysis, individual-dominant SWCNT films showed the best transparent conductive performance. However, the growth rate of such SWCNT films is usually very slow (Table S1 in the ESM) [17, 37]. Therefore, we prepared small-bundle SWCNT films with a high efficiency in this study, even though their optoelectrical performance decreased slightly.

3.4 Fabrication of a SWCNT-PW/PDMS electrothermal-driven transmittance-tunable smart window

To construct a smart window, a thermochromic composite layer capable of undergoing a phase transition triggered by thermal stimulation is essential. A layer of PW and PDMS was used to adjust the light-transmittance, where PW functions to change the light transmittance by a thermally-driven solid-liquid phase transition (Figs. S8(a) and S8(b) in the ESM), while PDMS serves to encapsulate the PW while maintaining a high light transmittance (Fig. S8(c) in the ESM).

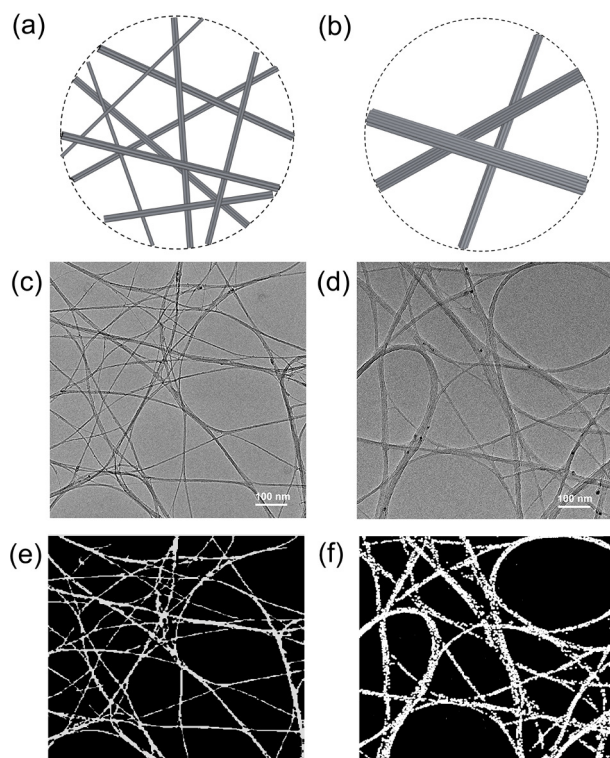


Figure 3 Comparison of SWCNT films with different bundle sizes at the same transmittance. ((a) and (b)) Schematics, ((c) and (d)) typical TEM images, and ((e) and (f)) corresponding binarized images for the small- and large-bundle samples.

The PW/PDMS composite films had an excellent repeatability in changing the light transmittance without detectable PW leakage (Fig. S8 in the ESM). To achieve low-voltage-driven operation and wide-range of light transmittance for the integrated device, a series of PW/PDMS composite films with PW mass fractions of 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.% were fabricated. Their surface and cross-sectional morphologies were characterized by SEM (Figs. 4(a)–4(d) and Fig. S9 in the ESM). For films with PW mass fractions of 5 wt.%, 10 wt.%, and 15 wt.%, the PW was fully and homogeneously dispersed in the PDMS matrix. However, when its content increased to 20 wt.%, many micropores were observed in the material and it failed to be optically transparent even at temperatures above the melting point of PW (Fig. S10 in the ESM). The composite film with 15 wt.% PW was therefore selected for the fabrication of the smart window.

We investigated the dynamic microstructural changes of the composite during heating using POM. As shown in Fig. 4(e), this process consists of three stages including opacity (PW crystals dispersed in PDMS matrix scattered incident light due to a refractive index mismatch), transparency (PW crystals melted and became amorphous above 65°C , thereby increasing the light transmittance), and opacity (PW recrystallized upon cooling). This reversible microstructural transformation enables stable and reversible changes in light transmittance (Fig. S11 in the ESM) [38–40]. Heating-cooling cycle experiments (Figs. 4(f) and 4(g)) confirmed the excellent structural recovery of the PW/PDMS composite. Raman spectra showed negligible changes in pre- and post-cycling, consistent with the reversible PW melting-crystallization behavior observed by POM. DSC analysis showed that the material undergoes a phase transition at 50°C (Fig. S12(a) in the ESM). This temperature of the PW/PDMS composite can be changed to 58°C by using long-chain PW (Fig. S12(b) in the ESM), which facilitates the fabrication of flexible smart window materials with different operating temperatures, thus meeting the practical requirements of diverse applications.

We designed a smart window device with the detailed structure schematically shown in Fig. 5(a). Upon applying a voltage, the SWCNT film was heated through electrothermal effect temperature, when the heat was transferred to the adjacent PW/PDMS thermochromic layer. This process enabled the device to actively and dynamically change its transmittance, meeting the requirements for light management in practical applications (Fig. 5(b)). The smart window was constructed by integrating a small-bundle SWCNT film (Fig. S13 in the ESM) and the PW/PDMS thermochromic layer and had a working area of $13\text{ cm} \times 13\text{ cm}$ (Fig. 5(c)). The window showed reversible switching between opaque and transparent states under electrothermal stimulations (Figs. 5(c) and 5(d)). Figure 5(e) shows the cross-sectional SEM image of the composite laminated structure, revealing a dense and uniform thermochromic layer conformally covering the SWCNT film. Furthermore, the cross-sectional SEM image of the composite (Fig. S14 in the ESM) after 1000 repeated bending exhibits negligible morphological changes, indicating a strong interfacial adhesion. In addition, the PW/PDMS layer also functions in suppressing the HNO_3 de-doping of the SWCNT film (Fig. S15 in the ESM). The window had a visible light transmittance of only 0.17% without an applied voltage, whereas it increased to 78% when a voltage of 15 V was applied (Fig. 5(f)). This demonstrates that the optical transparency of this material has a high degree of active controllability. We also evaluated the cycling stability of the window, and as shown in Fig. 5(g) and Fig. S16 in

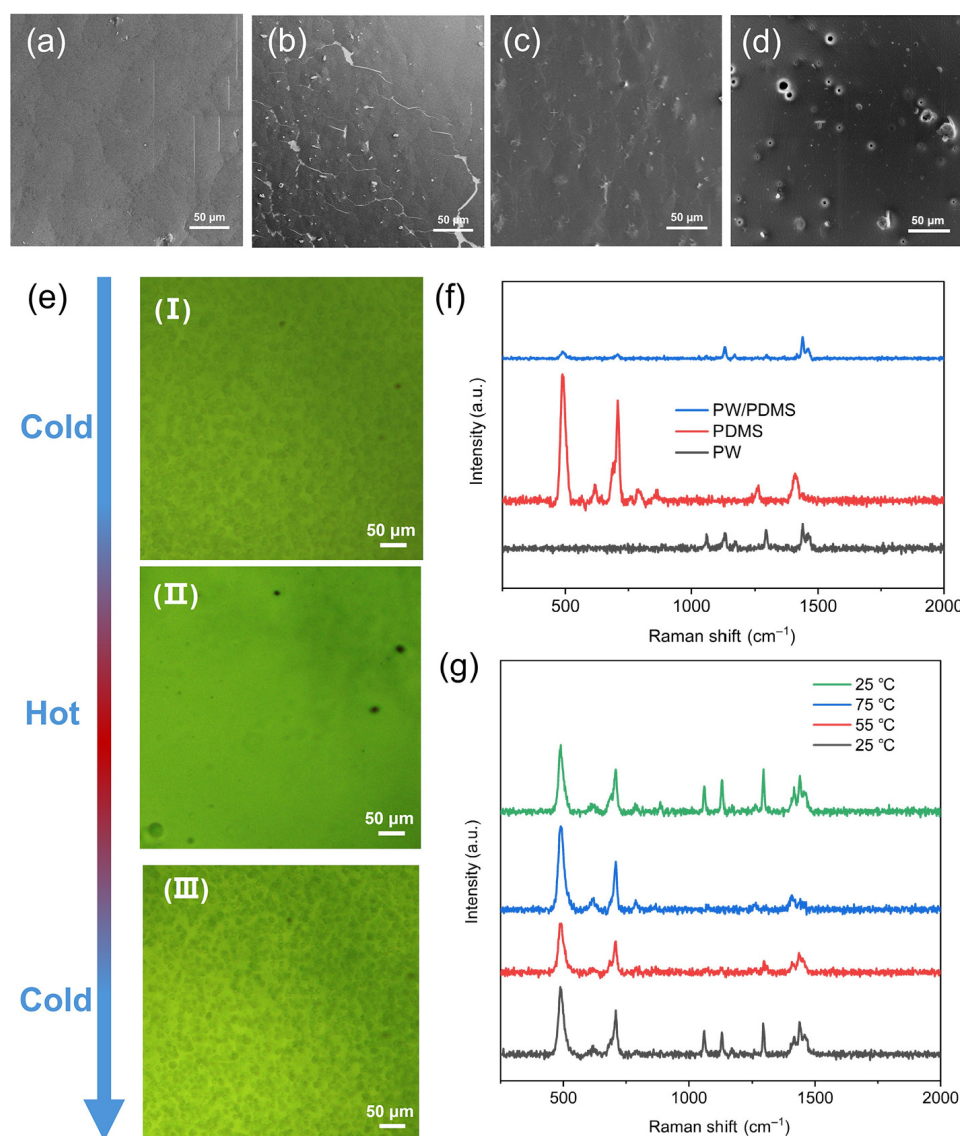


Figure 4 ((a)–(d)) SEM images of the PW/PDMS composite with PW mass contents of 5 wt.%, 10 wt.%, 15 wt.%, and 20 wt.%. (e) POM images of PW/PDMS obtained at (I) 25 °C, (II) 65 °C, and (III) upon cooling to 25 °C. (f) Raman spectra of pure PW, pure PDMS, and the PW/PDMS composite. (g) Raman spectra of the PW/PDMS composite recorded during a heating-cooling cycle.

the ESM, it showed exceptional stability with negligible performance degradation even after 105 switching cycles. After 105 cycles, the microstructure of the PW/PDMS composite remained stable, with PW maintaining a crystal state at room temperature and melting at high temperature within the PDMS matrix (Figs. S17(a)–S17(c) in the ESM). Meanwhile, no obvious defects were observed in the sample (Fig. S17(d) in the ESM). This multifunctional, flexible, and high-performance smart window has great promise for future use in architectural glazing, display panels, and intelligent transparent interfaces.

4 Conclusions

Small-bundle SWCNT films synthesized by FCCVD had a superior electrothermal performance to the large-bundle SWCNT counterpart. Specifically, a small-bundle SWCNT film with a transmittance of 82% could be operated at temperatures up to 102 °C under an applied voltage of 20 V. The excellent electrical and electrothermal performance of the small-bundle SWCNT film

stems from its unique microstructure, which enables a higher areal nanotube density and more efficient conductive paths at the same transmittance. A small-bundle SWCNT film with high transmittance and flexibility and good electrothermal performance was integrated with a PW/PDMS functional layer to fabricate a large-area flexible smart window. The window had an excellent electrochromic performance including an impressive transmittance range from 0.17% to 78% and exceptional cyclic stability. This study overcomes the trade-off between transparency and conductivity in transparent electrothermal films, and has broad applications.

Electronic Supplementary Material: Supplementary material (the preparation method of SWCNT films, optical photographs, uniformity, performance comparison and table, and calculation methods; the preparation method of composite structures, SEM, DSC, POM, and *I*–*V* curves) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908790>.

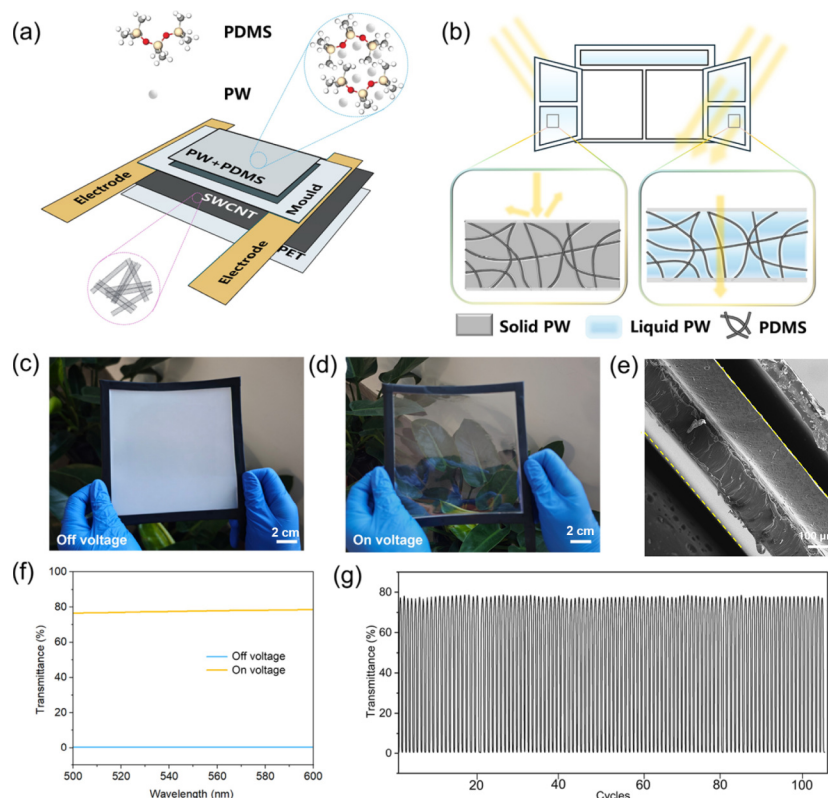


Figure 5 (a) Schematic showing the structure of the SWCNT-PW/PDMS smart window. (b) Diagram depicting the changes in light intensity during the phase transition of the smart window by turning the power on/off. ((c) and (d)) Optical photographs of the large-area smart window (c) before and (d) after the transparency change induced by a voltage. (e) Cross-sectional SEM image and (f) transmittance of the SWCNT-PW/PDMS composite. (g) Stability of the smart window during cyclic 20 V on–off tests.

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

W.-T. D.: Writing – original draft, writing – review & editing, methodology, investigation, data curation. Y. M.: Methodology, writing – original draft, funding acquisition, data curation. H. Y.: Software, methodology. A.-P. W.: Investigation. R.-S. M.: Software, methodology. K. L.: Software, methodology. P.-X. H.: Supervision, conceptualization, funding acquisition, writing – review & editing. C. L.: Supervision, conceptualization, funding acquisition, writing –

review & editing. All the authors have approved the final manuscript.

Use of AI statement

None.

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