

Article

A Flexible Organic Thermoelectric Generator with Optimized Interconnects Based on Doped Single-Walled Carbon Nanotube Clays

Yunxi Cheng ¹, Zhijie Liu ¹, Lihui Cai ¹, Xinchang Kang ², Jingda Liu ¹, Jianglin Wang ², Zhichun Liu ¹ and Limei Shen ^{1,*}

¹ School of Energy and Power Engineering, Huazhong University of Science and Technology, Wuhan 430074, China; chengyunxi70@gmail.com (Y.C.); zhijie1006@outlook.com (Z.L.); lihuicaihust@163.com (L.C.); u202311237@hust.edu.cn (J.L.); zcliu@hust.edu.cn (Z.L.)

² College of Life Science and Technology, Huazhong University of Science and Technology, Wuhan 430074, China; kxc214365@163.com (X.K.); jwang520@hust.edu.cn (J.W.)

* Correspondence: ep_shenlimei@hust.edu.cn; Tel.: +86-18674008783

Abstract

Organic thermoelectric generators (OTEGs) are promising for wearable low-grade heat harvesting, but their device-level output is often limited by interconnect-induced losses. This study investigates flexible OTEGs based on doped single-walled carbon nanotube (SWCNT) thermoelectric clays and optimizes their interconnect structure. P-type and n-type SWCNT clays were prepared by solution processing using TCNQ and TPP as dopants, respectively, and assembled into a five-pair flexible OTEG. The optimized p-type and n-type clays exhibited Seebeck coefficients of 40.81 and $-22.42 \mu\text{V K}^{-1}$, respectively. The initial OTEG, in which p-type thermoelectric clay was used as the interconnect, delivered a maximum output power of 16.47 nW at $\Delta T = 21 \text{ K}$. Replacing this thermoelectric-clay interconnect with a compliant Cu-foil/silver-paste interconnect reduced the internal resistance from approximately 372 Ω to 2 Ω , whereas the open-circuit voltage at $\Delta T = 21 \text{ K}$ increased only modestly from 4.95 to 5.08 mV. Under identical controlled temperature-gradient and load-scanning conditions, the optimized OTEG delivered 3.08 μW at $\Delta T = 21 \text{ K}$, corresponding to a power density of 356.36 nW cm^{-2} . Mechanical and wrist-worn tests further indicated the flexibility and practical voltage response of the optimized device. These results demonstrate that interconnect optimization is critical for improving SWCNT-clay-based flexible OTEGs.

Keywords: organic thermoelectric generator; low-grade heat harvesting; single-walled carbon nanotubes; thermoelectric clay; flexible interconnects; wearable electronics

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1. Introduction

With the development of wearable electronics, flexible sensing, health monitoring, and distributed Internet-of-Things nodes, in situ energy-harvesting technologies designed for human skin, textiles, curved surfaces, and low-grade heat sources have attracted increasing attention. Thermoelectric generators (TEGs) can directly convert the temperature difference between the hot and cold sides into electrical energy through the Seebeck effect. Owing to their advantages of no moving parts, silent operation, compact structure, and ease of miniaturized integration, TEGs are considered promising

candidates for micro-temperature-difference energy harvesting and self-powered electronic systems [1]. In wearable scenarios, the continuous but limited temperature difference between the human body and the ambient environment can provide auxiliary power for low-power sensors, electronic skins, and health-monitoring nodes [2]. However, body-heat harvesting is usually constrained by small temperature differences, unstable hot-side contact, limited cold-side heat dissipation, and device deformation during wearing. Therefore, improving wearable TEG performance requires not only the optimization of thermoelectric materials, but also the coupled design of materials, structures, interfaces, and operating conditions [3,4].

Conventional high-performance thermoelectric materials, such as Bi_2Te_3 , PbTe , and SnSe , generally exhibit high thermoelectric figures of merit, but their rigidity, brittleness, complex processing requirements, and poor compatibility with flexible substrates restrict their direct use in wearable micro-generators. In contrast, organic thermoelectric materials and their composites are lightweight, flexible, solution-processable, and intrinsically low in thermal conductivity, making them suitable for flexible organic thermoelectric generators (OTEGs) attached to skin, textiles, or curved surfaces [5]. Conducting polymers, organic small molecules, and carbon-based composites have been widely explored as organic thermoelectric materials. Previous studies have shown that their thermoelectric performance can be improved through molecular design, energy-level regulation, interfacial construction, and carrier-concentration optimization [6–8]. Nevertheless, for OTEGs, material properties alone cannot determine actual device output, because electrode interconnection, thermal contact, encapsulation, and device configuration also strongly affect the conversion from material performance to device performance.

Single-walled carbon nanotubes (SWCNTs) are attractive matrices for flexible organic thermoelectric materials owing to their high electrical conductivity, mechanical flexibility, environmental stability, and solution processability. However, pristine SWCNTs usually exhibit a limited Seebeck coefficient and relatively high thermal conductivity, requiring molecular doping, composite regulation, and structural design to improve their thermoelectric performance. Organic small molecules have been shown to regulate the thermoelectric transport behavior of SWCNT systems through charge transfer, energy-level matching, and carrier-concentration modulation, providing an effective route for tuning p-type and n-type thermoelectric properties [9,10].

Beyond material-level regulation, device morphology is also crucial for flexible thermoelectric performance. SWCNT-based thermoelectric materials have been processed into films, fibers, yarns, textiles, and reconfigurable structures, demonstrating the importance of heat-flow direction, spatial configuration, mechanical deformability, and conformal contact in wearable thermoelectric energy harvesting [11–14]. Meanwhile, air-stable n-type carbon nanotube materials, polymer/CNT composite yarns, and fibrous thermoelectric structures have further improved the p/n matching and structural adaptability required for series-connected flexible OTEGs [15,16]. Early OTEG studies based on organic/carbon composites, flexible films, and multi-thermocouple series structures have demonstrated the feasibility of flexible organic thermoelectric power generation, but their device-level output remains limited by material matching, internal resistance, interfacial contact, and the establishment of an effective temperature difference [17–19].

Recently, shape-deformable and reconfigurable carbon-nanotube-based thermoelectric materials have provided new opportunities for flexible OTEG design. Carbon nanotube thermoelectric clays and thermoelectric doughs combine thermoelectric functionality, macroscopic plasticity, and three-dimensional formability, making them promising for constructing flexible thermoelectric legs conformable to complex surfaces [20,21]. However, compared with conventional film- or fiber-based devices, the effects of

interconnect design, thermal contact, and small temperature differences on the device-level output of thermoelectric-clay-based OTEGs remain insufficiently understood.

Metal-free CNT-based thermoelectric generators have also been reported in which CNT-based materials function as both thermoelectric elements and electrical interconnections. Choi et al. selectively formed p-type and n-type regions along continuous CNT yarns while retaining undoped CNT sections exhibiting p-type behavior as electrodes or interconnections [22]. Zapata-Arteaga et al. fabricated monolithically integrated CNT-film modules from a common CNT ink, with CNT-based regions serving as p-type and n-type thermoelectric components as well as electrode components [23]. These studies demonstrate the feasibility of using CNT-based thermoelectric materials as electrical interconnections. However, the electrical performance of such configurations depends strongly on the continuity, geometry, conductivity, and interfacial contact of the interconnect.

Overall, organic and carbon-based flexible thermoelectric materials have made significant progress in molecular doping, fiber integration, textile structures, and wearable devices in recent years [24–27]. However, two key issues remain. First, many studies still emphasize material power factors or individual thermoelectric-leg performance, while the relationship between device-level output and realistic wearable conditions remains insufficiently understood. Second, electrode interconnections, interfacial contacts, and flexible encapsulation may introduce additional electrical resistance, thermal resistance, or parasitic thermoelectric effects, leading to measured Seebeck coefficients and output powers lower than their ideal values. Therefore, reducing internal device losses and improving effective temperature-difference utilization while maintaining flexibility and plasticity are critical for advancing OTEGs toward practical applications.

Although carbon-nanotube-based thermoelectric clays and doughs and Cu-foil interconnects have been reported previously, their integration into flexible OTEGs still involves substantial device-level losses. The contribution of this study therefore lies not in introducing a new thermoelectric-clay material or interconnect material, but in quantitatively evaluating interconnect-induced losses in a five-pair SWCNT-clay OTEG. TCNQ- and TPP-doped SWCNT thermoelectric clays were used as the p-type and n-type legs, respectively. An initial device using p-type thermoelectric clay as the interconnect and an optimized device using mechanically compliant Cu-foil/silver-paste interconnects were fabricated with identical thermoelectric-leg compositions and geometries. The two devices were evaluated under the same controlled temperature differences and load-scanning procedure, enabling the effects of the interconnect structure on open-circuit voltage, internal resistance, and load-matched power to be distinguished. The results show that the Cu-foil/silver-paste assembly primarily improves device output by substantially reducing internal resistance, while mechanical deformation and wrist-worn tests further assess its compatibility with flexible and wearable operation.

2. Materials and Methods

2.1. Materials and Preparation of SWCNT Thermoelectric Clays

Single-walled carbon nanotube powder (SWCNTs, 10 g, >75 wt%) was supplied by Suzhou Tanfeng Graphene Technology Co., Ltd. (Suzhou, China). Triton X-100 (100 mL, analytical grade) and anhydrous ethanol (500 mL, 99%) were purchased from BioWo Biochemical Reagent Co., Ltd. (Fuzhou, China). 7,7,8,8-Tetracyanoquinodimethane (TCNQ, 5 g, 98%) and triphenylphosphine (TPP, 25 g, 99%) were purchased from Sigma-Aldrich (St. Louis, MO, USA) and used as p-type and n-type dopants, respectively. All chemicals were used as received without further purification. The main experimental instruments included a centrifuge (SN-LSC-3, Shanghai Shangyi Instrument Equipment Co., Ltd., Shanghai, China), an electronic balance, a vortex mixer (KM-XWY-2, Comet Instruments,

Ningbo, China), and a magnetic stirrer (SN-MS-H280D, Shanghai Shangyi Instrument Equipment Co., Ltd., Shanghai, China).

SWCNTs were used as the matrix material for preparing thermoelectric clays. Although SWCNTs possess high electrical conductivity, their Seebeck coefficient is generally low and their thermal conductivity is relatively high, resulting in limited thermoelectric performance. Therefore, organic small-molecule dopants were introduced to regulate the thermoelectric properties of SWCNTs. Triton X-100 was used as the dispersant to inhibit the aggregation of SWCNT bundles, while solution processing was employed to promote the interaction between the dopants and SWCNTs.

To prepare the undoped SWCNT clay, the mass ratio between SWCNTs and Triton X-100 was first defined as follows:

$$F = W_s : W_t \quad (1)$$

where W_s and W_t are the masses of SWCNTs and Triton X-100, respectively. SWCNT powder and Triton X-100 were mixed in a centrifuge at 2000 rpm for 10 min to obtain undoped SWCNT clay. By adjusting the value of F , SWCNT clays with different mechanical properties could be obtained. Based on previous studies and experimental verification, the SWCNT clay exhibited favorable mechanical properties when $F = 1:5$. The obtained SWCNT thermoelectric clay was reconfigurable and plastic, and could mechanically self-heal after damage through kneading, as shown in Figure 1.



Figure 1. Mechanical reconstruction of SWCNT thermoelectric clay. The arrows indicate the sequence from integrated clay to damaged fragments, followed by manual kneading and reconstruction into an integrated form.

For p-type doping, the mass fraction of TCNQ, F_p , and the mass fraction of SWCNTs, F_s , were defined as follows:

$$F_p = \frac{W_p}{W_p + W_t + W_s} \quad (2)$$

$$F_s = \frac{W_s}{W_s + W_t + W_c} \quad (3)$$

where W_p , W_t , W_s , and W_c are the masses of TCNQ, Triton X-100, SWCNTs, and anhydrous ethanol, respectively. F_s was used to describe the mass fraction of SWCNTs in the SWCNT/Triton X-100/ethanol dispersion before ethanol evaporation. Since ethanol was removed during the subsequent heating process, F_s was used only to control the dispersion state during preparation rather than to describe the final composition of the thermoelectric clay.

During the p-type doping process, TCNQ powder was first dissolved in Triton X-100, and anhydrous ethanol was added to facilitate the dissolution of TCNQ. The mixture was then placed on a vortex mixer for 5 min to ensure sufficient mixing. Subsequently, the

mixture was stirred at 80 °C using a temperature-controlled magnetic stirrer to accelerate the volatilization of anhydrous ethanol. After ethanol volatilization, the resulting mixture was combined with SWCNT powder and mixed in a centrifuge at 2000 rpm for another 10 min, yielding TCNQ-doped p-type SWCNT thermoelectric clay. During the preparation process, the mass fraction of SWCNTs was maintained at approximately 3.5% to ensure homogeneous dispersion of SWCNTs within the clay. By adjusting F_p , SWCNT thermoelectric clays with different TCNQ doping ratios were prepared. The SWCNT clay with 5 wt% TCNQ was subsequently selected as the p-type thermoelectric material for OTEG fabrication.

For n-type doping, the preparation procedure was similar to that used for p-type doping, except that TPP was used as the n-type dopant. The mass fraction of TPP, F_N , was defined as follows:

$$F_N = \frac{W_N}{W_N + W_t + W_s} \quad (4)$$

where W_N , W_t , and W_s are the masses of TPP, Triton X-100, and SWCNTs, respectively. TPP was mixed with Triton X-100 and anhydrous ethanol, followed by vortex mixing, magnetic stirring at 80 °C, ethanol volatilization, and subsequent mixing with SWCNT powder at 2000 rpm for 10 min. By varying F_N , TPP-doped SWCNT thermoelectric clays with different doping ratios were prepared. The SWCNT clay with 1 wt% TPP was subsequently selected as the n-type thermoelectric material for OTEG fabrication.

2.2. Fabrication of OTEGs

The p-type and n-type thermoelectric (TE) clays prepared in the previous section were selected as the materials for constructing the thermoelectric legs of the organic thermoelectric generators (OTEGs). The p-type thermoelectric legs were fabricated using the p-type TE clay with a 5 wt% TCNQ doping ratio, while the n-type thermoelectric legs were fabricated using the n-type TE clay with a 1 wt% TPP doping ratio.

For the fabrication of the initial OTEG, both p-type and n-type TE clays were molded into dimensions of 8 mm × 8 mm × 4 mm (length × width × height) using customized molds, as shown in Figure 2a. The p-type TE clay was subsequently used to connect the p-type and n-type thermoelectric legs, forming a p–n thermoelectric pair. This fabrication process was repeated to obtain five p–n thermoelectric pairs for device assembly.

The projected area of the ten thermoelectric legs was 6.40 cm², calculated from ten legs with a footprint of 8 mm × 8 mm each. For power-density calculation, the effective active-region area was defined as the projected thermoelectric-leg array area, including the thermoelectric legs and the inter-leg spacing but excluding the surrounding neoprene frame, polyurethane encapsulation, and external leads, as illustrated in Figure 2d. Based on the device geometry, this effective active-region area was approximately 8.64 cm².

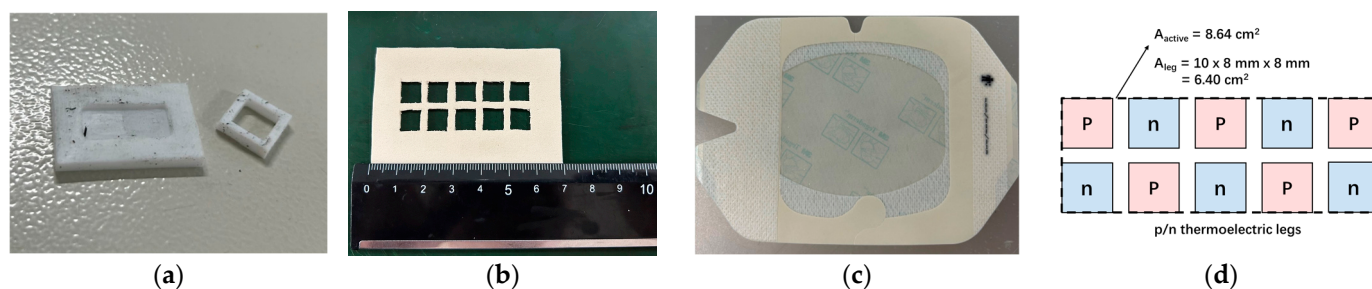


Figure 2. (a) molds for preparing p–n thermoelectric pairs; (b) Neoprene frame; (c) Polyurethane film; (d) schematic illustration of the effective active-region area used for power-density calculation.

Neoprene was selected as the flexible supporting frame material due to its flexibility and mechanical compliance. The dimensions of the neoprene frame were 70 mm × 45 mm × 0.5 mm (length × width × height), as illustrated in Figure 2b. The prefabricated p–n thermoelectric pairs were inserted into the neoprene frame and subsequently encapsulated using a polyurethane film, as shown in Figure 2c. Copper leads were then connected to the device for electrical output. The fabricated OTEG exhibited a flexible structure suitable for wearable applications. The front and back views of the fabricated initial OTEG are shown in Figure 3a and b, respectively.

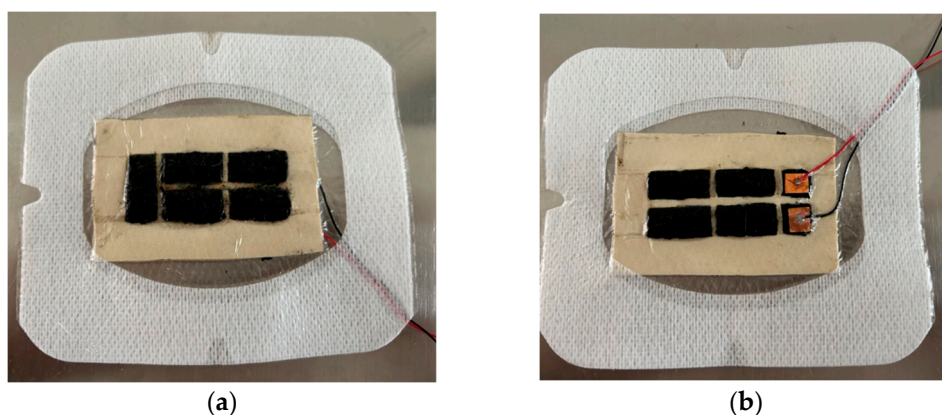


Figure 3. Front and back views of the initial OTEG: (a) front view; (b) back view.

To improve the electrical interconnection structure of the OTEG, the original thermoelectric clay interconnect was further optimized. In the optimized OTEG, the p-type thermoelectric clay originally used as the connecting electrode between adjacent p-type and n-type thermoelectric legs was replaced with copper foil. This modification retained the flexibility of the OTEG while improving the electrical connection between thermoelectric legs.

The copper foil was arranged with a slightly bent structure toward the neoprene frame between adjacent thermoelectric legs to enhance its flexibility during device deformation. In addition, conductive silver paste was applied between the copper foil and the thermoelectric legs to ensure reliable electrical contact and to prevent detachment of the copper foil during bending deformation of the OTEG. The front and back views of the optimized OTEG are shown in Figure 4a and b, respectively.

To further clarify the structural difference between the initial and optimized OTEGs, cross-sectional schematics of the two devices are provided in Figure 4c,d. In the initial OTEG, the p-type thermoelectric clay served as the interconnect between adjacent thermoelectric legs. In contrast, in the optimized OTEG, the thermoelectric-clay interconnect was replaced by a Cu-foil interconnect with conductive silver paste.



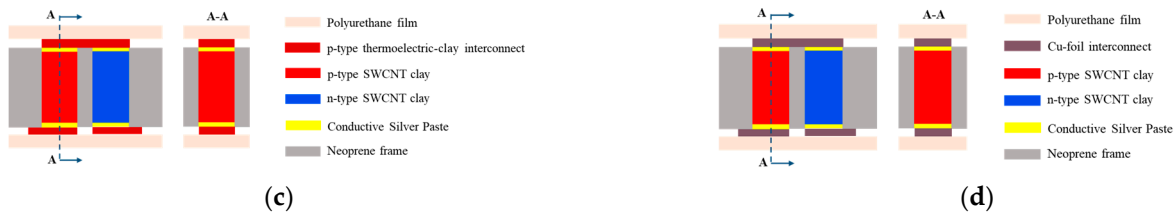


Figure 4. Photographs of the optimized OTEG and cross-sectional schematics of the initial and optimized OTEGs: (a) front view of the optimized OTEG; (b) back view of the optimized OTEG; (c) cross-sectional schematic of the initial OTEG with p-type thermoelectric-clay interconnects; (d) cross-sectional schematic of the optimized OTEG with Cu-foil interconnects and conductive silver paste. In (c,d), the blue dotted lines labeled A–A indicate the section planes, and the blue arrows indicate the viewing directions of the corresponding cross-sectional views.

2.3. Thermoelectric and Output Characterizations

The Seebeck coefficients of the SWCNT thermoelectric clays were measured using a customized vertical temperature-gradient test platform. As shown in Figure 5a, the upper part of the platform was an aluminum plate embedded with several heating rods, which were powered by a direct current (DC) power supply and served as the hot side. The lower part of the platform was an aluminum plate connected to a water-cooled plate and a high–low temperature circulator, serving as the cold side. Thermocouples were placed in grooves near the hot and cold surfaces to measure the temperatures of both sides, and the temperature data were monitored using a temperature recorder. The generated thermoelectric voltage was measured using a digital multimeter. The structure and working principle of the Seebeck coefficient test platform are illustrated in Figure 5b.

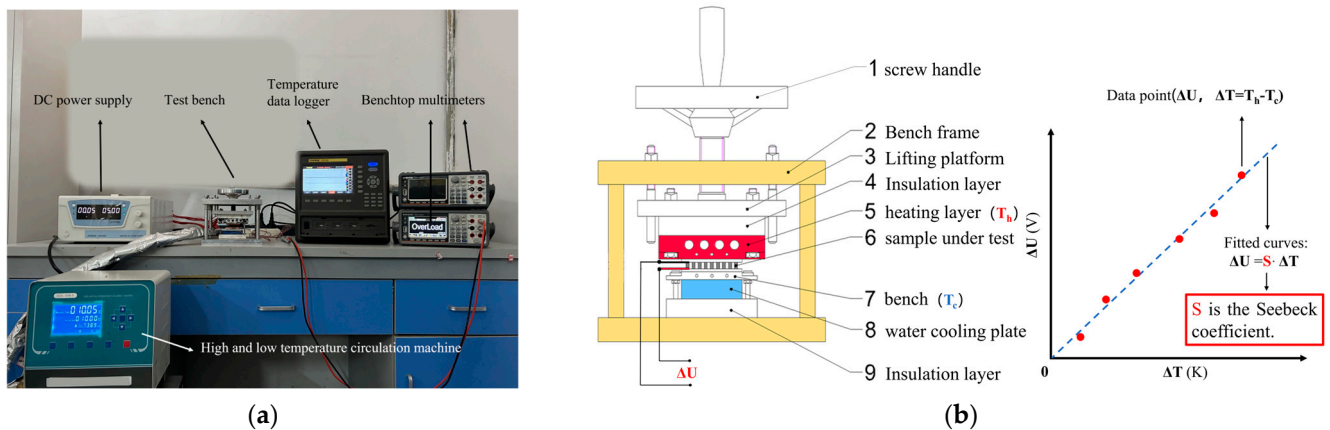


Figure 5. (a) SWCNT Clay Seebeck Coefficient Test Bench; (b) Test bench structure and schematic diagram.

During the measurement, the SWCNT thermoelectric clay was placed between the hot and cold plates. After the temperature difference between the two sides became stable, the corresponding voltage and temperature data were recorded to evaluate the thermoelectric properties of the SWCNT thermoelectric clay.

The electrical conductivity of the SWCNT thermoelectric clays was measured using a digital bridge method. During the measurement, the two electrodes of the bridge were connected to the two ends of the clay sample, and the resistance of the sample was recorded after the reading became stable. The electrical conductivity was then calculated from the measured resistance and the sample geometry. Each sample was measured three times under the same conditions, and the average value was used for further analysis. The

detailed electrical-conductivity measurement setup is shown in Figure S1, and the main instruments used for thermoelectric and electrical characterizations are listed in Table S1.

The thermal conductivity of the SWCNT thermoelectric clays was measured using a DSC2500 differential scanning calorimeter (TA Instruments, New Castle, DE, USA). The thermal conductivity values were obtained according to the instrument testing procedure based on the thermal response of the samples. These values were used together with the measured Seebeck coefficient and electrical conductivity to calculate the thermoelectric power factor and figure of merit. The corresponding thermal-conductivity measurement setup is provided in Figure S2.

The output performance of the fabricated OTEG was characterized using a customized test platform, as shown in Figure 6. During the measurement, the temperatures of the hot and cold sides of the OTEG were monitored using the temperature recorder. After the temperature difference became stable, an external resistance box was connected as the load resistance. The load resistance was gradually increased from $0\ \Omega$ to higher values, and the corresponding voltage and current were recorded using a multimeter under each resistance condition.

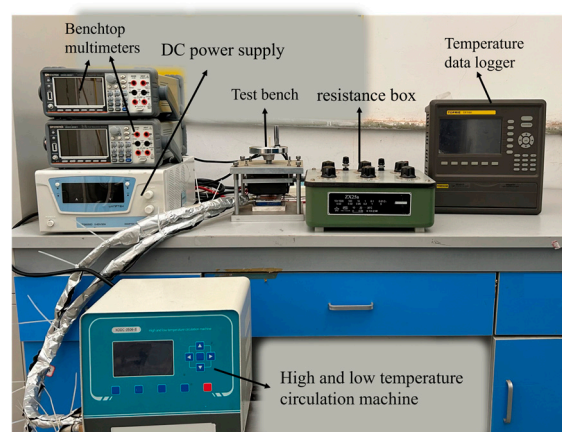


Figure 6. OTEG Output Performance Test Bench.

To investigate the output characteristics of the OTEG under different temperature differences, the output power of the DC power supply was adjusted to regulate the heating power of the heating rods. After the temperature difference across the OTEG became stable again, the electrical output of the OTEG was measured under the new temperature condition. The output performance of the OTEG was measured under temperature differences of 2.5 K, 6 K, 10 K, 16 K, and 21 K.

For both the initial and optimized OTEGs, the output characteristics were measured using the same test platform, the same controlled temperature differences, and the same load-resistance scanning procedure. The output voltage U and current I were recorded under each load-resistance condition, and the output power was calculated as $P = UI$. The open-circuit voltage V_{oc} and short-circuit current I_{sc} were obtained from the intercepts of the voltage–current characteristics. The internal resistance R_{in} was calculated as $R_{in} = V_{oc}/I_{sc}$. The maximum output power P_{max} was determined from the load-matched output-power curve.

2.4. Mechanical Reliability Tests

Due to the size limitation of the mechanical fatigue-testing platform, a simplified device containing two thermoelectric pairs was fabricated for mechanical reliability evaluation. The simplified device employed the same SWCNT thermoelectric clays, Cu-foil interconnects, conductive silver-paste contacts, and flexible supporting structure as the

optimized OTEG. Therefore, it was used to evaluate the mechanical stability of the key thermoelectric-leg/Cu-interconnect structure. The detailed configuration of the mechanical-reliability test platform is shown in Figure S3.

Rolling, bending, and stretching tests were performed to evaluate the deformation adaptability of the simplified device. During the rolling test, the device was wound around cylindrical shafts with different radii to investigate the resistance variation under rolling deformation and after recovery. The rolling radii were set as 15, 12.5, 10, and 7.5 mm. During the bending and stretching tests, the device was mechanically deformed using the testing platform, and the electrical resistance was measured in both the deformed and recovered states.

Cyclic deformation tests were further conducted to evaluate fatigue reliability. During the bending-cycle test, the two ends of the device were fixed using clamps, and repeated bending deformation was applied by controlling the reciprocating motion of the sliding block. The device was subjected to 1000 bending cycles. During the rolling-cycle fatigue test, the shaft radius was sequentially changed from 15 mm to 12.5 mm, 10 mm, and 7.5 mm, with 1000 rolling cycles applied at each radius. Therefore, a total of 4000 rolling cycles were applied.

After selected deformation states and deformation cycles, the electrical resistance and device-level Seebeck coefficient were measured to evaluate the stability of the thermoelectric-leg/Cu-interconnect structure. The resistance was normalized as R/R_0 , where R_0 represents the initial resistance of the simplified device before mechanical testing.

2.5. Wearable Tests

To evaluate the practical wearable performance of the optimized OTEG, the device was tested under conditions simulating actual wearing on the skin surface. The optimized OTEG was worn on the wrist under an ambient room temperature of 22 °C and placed below an air-conditioning outlet. The physical wearing state and the corresponding infrared thermal image were recorded to observe the temperature distribution between the skin-contacting side and the air-exposed side of the device. The open-circuit voltage of the optimized OTEG under wrist-worn conditions was measured using a handheld multimeter (UT61E+, Uni-Trend Technology Co., Ltd., Dongguan, China).

The optimized OTEG was further tested under indoor and outdoor conditions. The indoor air temperature was 16 °C, and the outdoor air temperature was 15 °C. During the test, the device was worn on the wrist while the wearer remained stationary and while walking at a speed of approximately 1 m/s. The open-circuit voltage of the OTEG was recorded using a handheld multimeter to evaluate the output response of the device under different environments and motion states.

3. Results

3.1. Thermoelectric Properties of SWCNT Thermoelectric Clays

The morphology and thermoelectric properties of the undoped SWCNT clay were first characterized to establish a baseline for subsequent molecular doping. As shown in Figure 7a, the scanning electron microscope (SEM) image of the undoped SWCNT clay indicates that the SWCNT bundles were distributed without obvious large-scale aggregation, suggesting that Triton X-100 effectively contributed to the dispersion of SWCNTs in the clay matrix. The Seebeck coefficient measurement of the undoped SWCNT clay is shown in Figure 7b. The undoped SWCNT clay exhibited p-type thermoelectric behavior, with a Seebeck coefficient of 7.45 $\mu\text{V/K}$. The corresponding basic physical properties are summarized in Figure 7c.

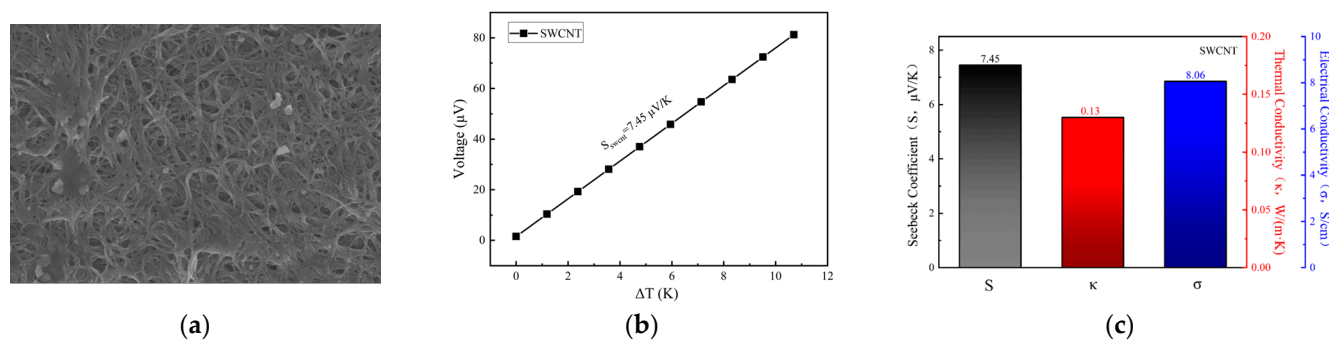


Figure 7. Characterization of the undoped SWCNT clay: (a) SEM image; (b) Seebeck coefficient measurement result at a cold-side temperature of 290 K; (c) basic physical properties of the undoped SWCNT clay.

To enhance the p-type thermoelectric performance of the SWCNT clay, TCNQ was introduced as a p-type dopant. SWCNT thermoelectric clays with different TCNQ doping ratios were prepared by adjusting the TCNQ mass fraction. As shown in Figure 8a, all TCNQ-doped SWCNT clays exhibited positive Seebeck coefficients over the investigated doping range, confirming the preservation of p-type thermoelectric behavior.

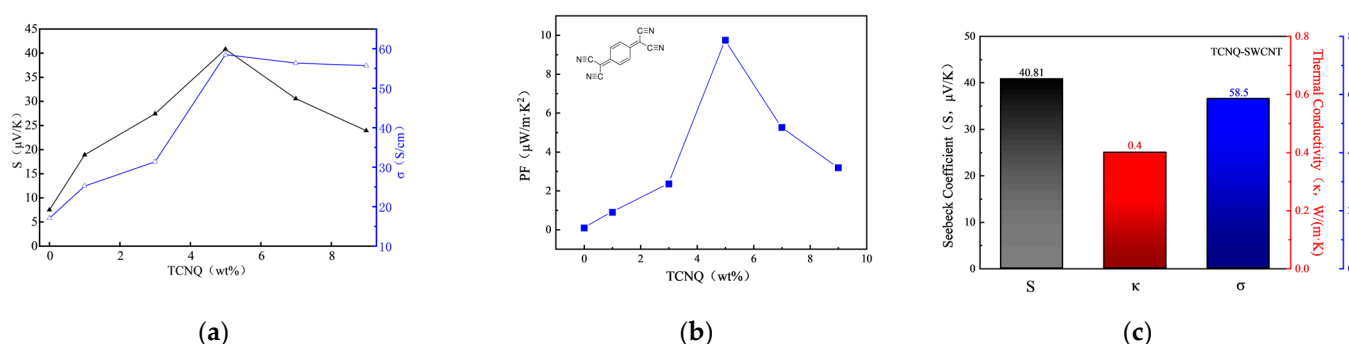


Figure 8. Thermoelectric properties of TCNQ-doped SWCNT thermoelectric clays: (a) Seebeck coefficients and electrical conductivity of SWCNT thermoelectric clays with different TCNQ doping ratios; (b) power factor as a function of TCNQ doping ratio; and (c) thermoelectric parameters of the optimized 5 wt% TCNQ-doped SWCNT clay.

With increasing TCNQ content, the Seebeck coefficient initially increased and then decreased, reaching its maximum at a TCNQ doping ratio of 5 wt%. Meanwhile, the electrical conductivity increased with increasing TCNQ content (Figure 8a). The combined variations in the Seebeck coefficient and electrical conductivity resulted in a doping-dependent change in the power factor. As shown in Figure 8b, the maximum power factor of $9.74 \mu W/(m \cdot K^2)$ was obtained for the 5 wt% TCNQ-doped SWCNT clay, indicating the optimal balance between the Seebeck coefficient and electrical conductivity.

Thermal conductivity was measured only for the optimized compositions selected for device fabrication, namely the 5 wt% TCNQ-doped p-type clay and the 1 wt% TPP-doped n-type clay. As summarized in Figure 8c, the optimized p-type clay exhibited a Seebeck coefficient of $40.81 \mu V/K$, an electrical conductivity of $58.5 S/cm$, and a thermal conductivity of $0.4 W/(m \cdot K)$, corresponding to a ZT value of 0.0071 at 293.15 K. Therefore, the 5 wt% TCNQ-doped SWCNT clay was selected as the p-type thermoelectric material for OTEG fabrication.

TPP was further introduced as an n-type dopant to regulate the carrier polarity of the SWCNT thermoelectric clay. As shown in Figure 9a, the TPP-doped SWCNT clay retained

p-type behavior at a TPP doping ratio of 0.5 wt%, whereas its carrier polarity was converted to n-type at TPP doping ratios of 1, 1.5, and 2 wt%.

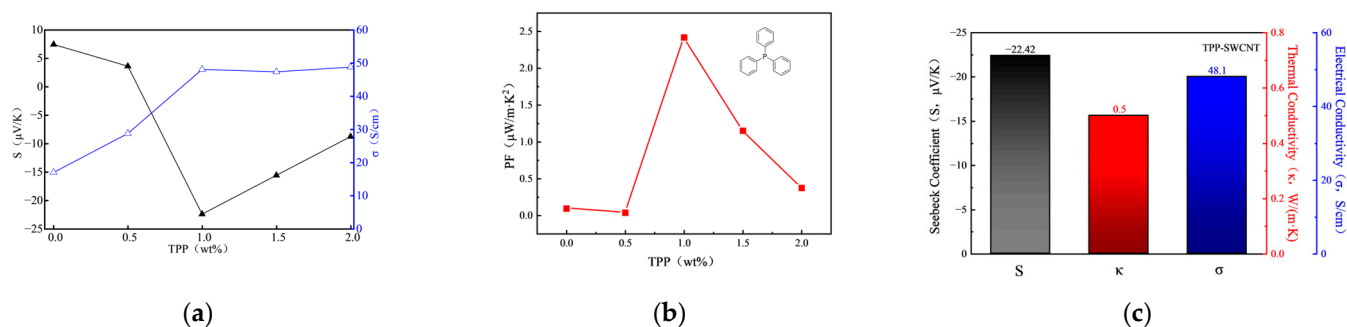


Figure 9. Thermoelectric properties of TPP-doped SWCNT thermoelectric clays: (a) Seebeck coefficients and electrical conductivity of SWCNT thermoelectric clays with different TPP doping ratios; (b) power factor as a function of TPP doping ratio; and (c) thermoelectric parameters of the optimized 1 wt% TPP-doped SWCNT clay.

The Seebeck coefficient and electrical conductivity exhibited different trends with increasing TPP content (Figure 9a), reflecting the coupled effects of TPP doping on the thermoelectric transport properties of the SWCNT clay. Among the investigated compositions, the 1 wt% TPP-doped SWCNT clay exhibited the largest Seebeck-coefficient magnitude, with a Seebeck coefficient of $-22.42 \mu\text{V/K}$ and an electrical conductivity of 48.1 S/cm . Consequently, the maximum power factor of $2.42 \mu\text{W}/(\text{m}\cdot\text{K}^2)$ was achieved at 1 wt% TPP (Figure 9b), demonstrating the best thermoelectric performance among the investigated n-type samples.

For the optimized 1 wt% TPP-doped SWCNT clay, the measured thermal conductivity was $0.5 \text{ W}/(\text{m}\cdot\text{K})$. As summarized in Figure 9c, its Seebeck coefficient, electrical conductivity, and thermal conductivity corresponded to a ZT value of 0.0014 at 293.15 K. Compared with the optimized p-type clay, the n-type clay exhibited a lower Seebeck-coefficient magnitude and ZT , indicating a p/n performance imbalance that may limit the overall output of the OTEG.

3.2. Output Performance of the Initial OTEG

Based on the optimized p-type and n-type SWCNT thermoelectric clays, an initial flexible OTEG containing five p–n thermoelectric pairs was fabricated and characterized under different temperature differences. The output performance curves of the initial OTEG are shown in Figure 10. As the temperature difference increased, both the output voltage and output power of the OTEG increased accordingly, indicating that the device could effectively convert the applied temperature gradient into electrical output.

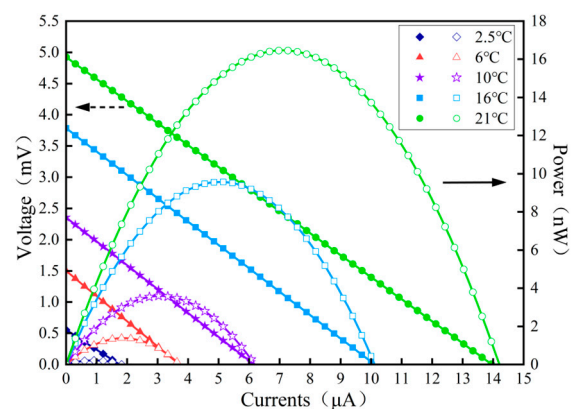


Figure 10. Output performance curves of the initial OTEG under different temperature differences. The left- and right-pointing arrows indicate that the corresponding datasets should be read against the left and right y-axes, respectively.

The output performance of the OTEG was measured at temperature differences of 2.5 K, 6 K, 10 K, 16 K, and 21 K. The corresponding maximum output powers were 0.22 nW, 1.38 nW, 3.59 nW, 9.57 nW, and 16.47 nW, respectively. At a temperature difference of 21 K, the maximum output power reached 16.47 nW, corresponding to a power density of 1.91 nW/cm² based on the effective active-region area of the thermoelectric-leg array, namely 8.64 cm², including the thermoelectric legs and the inter-leg spacing.

Based on the Seebeck coefficients of the p-type and n-type thermoelectric clays, $S_{\text{TCNQ}} = 40.81 \mu\text{V K}^{-1}$ and $S_{\text{TPP}} = -22.42 \mu\text{V K}^{-1}$, respectively, the ideal Seebeck coefficient of the five-pair OTEG was calculated to be $316.15 \mu\text{V K}^{-1}$. Linear fitting of the controlled $V_{\text{oc}}-\Delta T$ data yielded a device-level Seebeck coefficient of $235.74 \mu\text{V K}^{-1}$ for the initial OTEG. This difference establishes a device-level deviation from the ideal value but does not identify a single loss mechanism. Thermal-contact resistance, nonuniform temperature distribution, variations among the thermoelectric legs, p/n performance imbalance, and the non-ideal interconnect structure may jointly contribute to this discrepancy.

3.3. Output Performance Comparison Between the Initial and Optimized OTEGs

Figure 11 shows the output characteristics of the optimized OTEG under different temperature differences. Similar to the initial OTEG, both the output voltage and output power increased with increasing temperature difference. However, the optimized OTEG exhibited a much lower internal resistance and a higher load-matched output power under the same temperature-gradient conditions. This result indicates that replacing the thermoelectric-clay interconnect with the Cu interconnect effectively reduced interconnect-induced electrical loss and improved device-level power output.

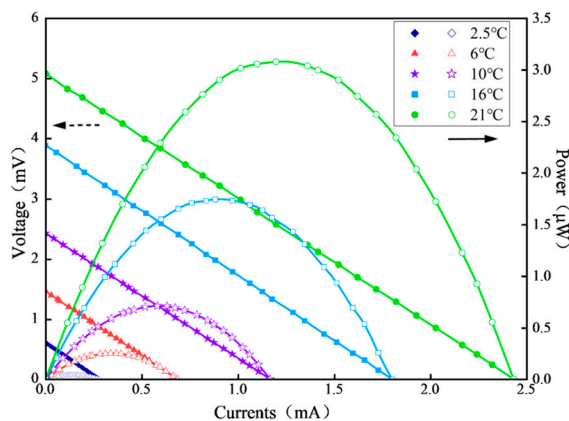


Figure 11. Output performance curves of the optimized OTEG under different temperature differences. The left- and right-pointing arrows indicate that the corresponding datasets should be read against the left and right y-axes, respectively.

At $\Delta T = 21$ K, the optimized OTEG exhibited an open-circuit voltage of 5.08 mV, a short-circuit current of 2.43 mA, and a maximum output power of 3.08 μW , with an internal resistance of approximately 2.09 Ω . In comparison, the initial OTEG delivered a maximum output power of only 16.47 nW under the same temperature difference. The detailed comparison between the initial and optimized OTEGs is summarized in Table 1.

Table 1. Comparison of the output performance of the initial and optimized OTEGs under controlled temperature differences.

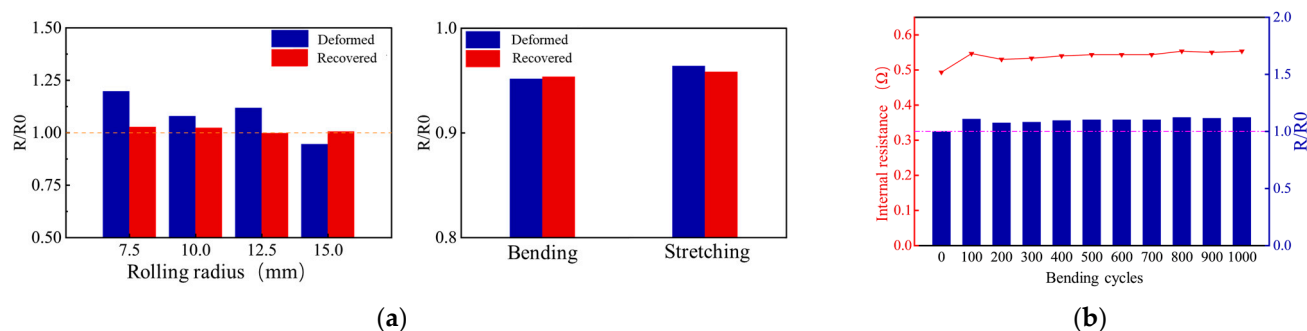
$\Delta T/K$	V_{oc}/mV	$I_{sc}/\mu A$	R_{in}/Ω	P_{max}/nW	Power Density/ $nW\ cm^{-2}$
2.5	0.59/0.60	1.49/277.79	394.60/2.17	0.22/41.84	0.03/4.84
6	1.41/1.46	3.90/685.07	362.34/2.13	1.38/249.25	0.16/28.85
10	2.36/2.43	6.09/1166.59	386.90/2.08	3.59/709.58	0.42/82.13
16	3.77/3.89	10.15/1799.72	371.56/2.16	9.57/1745.19	1.11/201.99
21	4.95/5.08	13.31/2432.87	371.91/2.09	16.47/3078.95	1.91/356.36

Values are shown as initial OTEG/optimized OTEG. V_{oc} , I_{sc} , and R_{in} were obtained from the voltage–current characteristics. P_{max} was obtained from the load-matched output-power curves. The power density was calculated based on the effective active-region area of $8.64\ cm^2$.

Linear fitting of the controlled V_{oc} - ΔT data yielded device-level Seebeck coefficients of 235.74 and $242.27\ \mu V\ K^{-1}$ for the initial and optimized OTEGs, respectively. Both values remained below the ideal value of $316.15\ \mu V\ K^{-1}$ calculated from the individual thermoelectric legs. Thus, replacing the p-type clay interconnect produced only a modest increase in open-circuit voltage and did not fully account for the discrepancy from the ideal Seebeck coefficient. Treating the devices as Thevenin sources, $P_{max} = V_{oc}^2/(4R_{in})$ predicts maximum powers of 16.47 nW and $3.09\ \mu W$ at $\Delta T = 21\ K$, in close agreement with the measured values of 16.47 nW and $3.08\ \mu W$. The approximately 187-fold power enhancement was therefore dominated by the reduction in internal resistance rather than by the small change in open-circuit voltage.

3.4. Mechanical Reliability of the SWCNT Thermoelectric Clay/Cu-Interconnect Structure

The static deformation and recovery behavior was first evaluated, as shown in Figure 12a. During rolling deformation, the normalized resistance R/R_0 increased slightly when the rolling radius decreased, indicating that more severe deformation temporarily affected the contact state and conductive pathways. However, after the rolling deformation was released, the resistance returned to a value close to its initial level under all tested rolling radii. Similarly, only limited resistance variation was observed during bending and stretching deformation, and the resistance also recovered after the deformation was released. These results indicate that the deformation-induced resistance changes under rolling, bending, and stretching conditions were largely reversible.



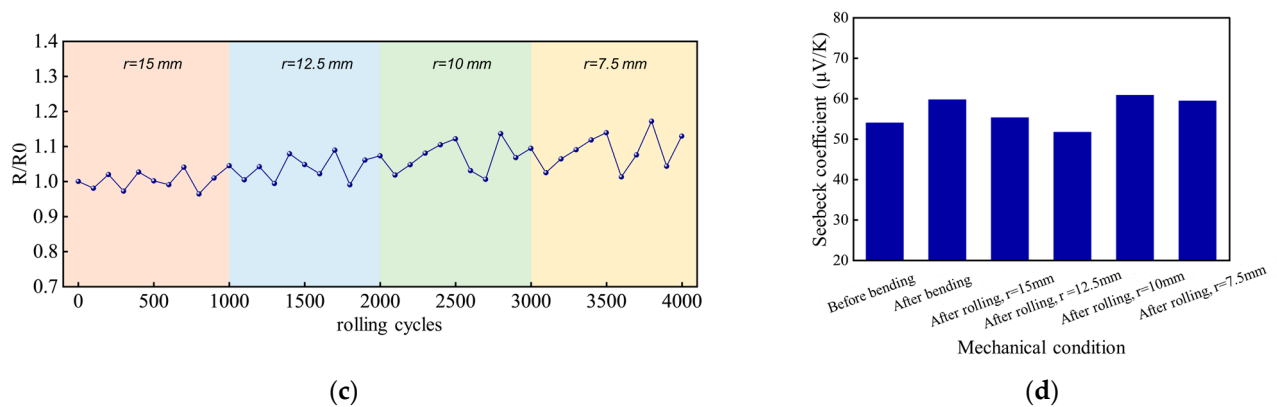


Figure 12. Mechanical reliability of the SWCNT thermoelectric clay/Cu-interconnect structure based on a simplified two-pair device: (a) normalized resistance during deformation and after recovery under rolling deformation with different rolling radii and under bending/stretching deformation; (b) internal resistance and normalized resistance during 1000 repeated bending–release cycles using a reciprocating fixture, during which no single fixed minimum bending radius was imposed or recorded; (c) normalized resistance during cumulative rolling cycles at rolling radii of 15, 12.5, 10, and 7.5 mm, with 1000 cycles applied at each radius; (d) device-level Seebeck coefficient before deformation, after bending cycles, and after rolling cycles under different rolling radii. The dotted lines in (a,b) indicate the reference value $R/R_0 = 1$, corresponding to the initial resistance.

The bending-cycle stability was then investigated under repeated bending deformation. As shown in Figure 12b, the internal resistance changed only slightly during 1000 bending cycles, and the normalized resistance remained close to its initial value. This result confirms that the Cu-foil interconnect and conductive silver-paste contacts maintained stable electrical continuity during repeated bending cycles.

The rolling-cycle fatigue behavior was further evaluated using a cumulative rolling protocol. The rolling radius was sequentially changed from 15 mm to 12.5 mm, 10 mm, and 7.5 mm, and 1000 rolling cycles were applied at each radius. As shown in Figure 12c, after 4000 cumulative rolling cycles, R/R_0 showed only a moderate increase without abrupt resistance failure, indicating stable electrical conduction under repeated rolling deformation.

In addition to resistance stability, the device-level Seebeck coefficient was measured before and after mechanical tests. As shown in Figure 12d, the Seebeck coefficient showed only limited fluctuation after bending cycles and after rolling cycles under different rolling radii. This result indicates that the thermoelectric connection between the p-type and n-type legs was not significantly degraded by mechanical loading. Overall, the SWCNT thermoelectric clay/Cu-interconnect structure exhibited good mechanical reliability under rolling, bending, and stretching deformation, supporting its use in flexible and wearable OTEGs.

3.5. Wearable Performance of the Optimized OTEG

To evaluate the practical performance of the optimized OTEG under wearable conditions, the device was worn on the wrist and tested under ambient conditions. As shown in Figure 13a, the optimized OTEG conformed closely to the wrist surface. The corresponding infrared thermal image is shown in Figure 13b. Under an ambient room temperature of 22 °C and with the device placed below an air-conditioning outlet, a temperature difference of approximately 1–1.5 K was established between the skin-contacting side and the air-exposed side of the OTEG. The output voltage measured under this condition is shown in Figure 13c, with a voltage of 0.34 mV.

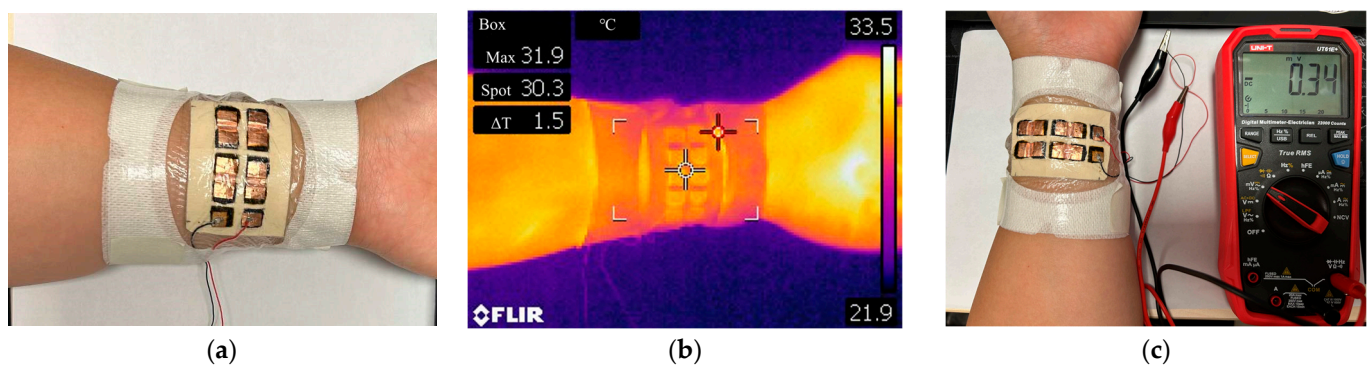


Figure 13. Wearable performance of the optimized OTEG on the wrist: (a) photograph of the wrist-worn optimized OTEG; (b) infrared thermal image of the wrist-worn optimized OTEG; (c) output voltage of the optimized OTEG during wrist wearing.

Based on the measured open-circuit voltage and the temperature difference obtained from infrared imaging, the apparent device-level Seebeck coefficient under wrist-worn conditions was estimated to be in the range of 226.67–340 $\mu\text{V/K}$. Because this temperature difference was derived from infrared imaging and was only approximately 1–1.5 K, this value should be regarded as an apparent estimate under practical wearing conditions rather than a calibrated device-level Seebeck coefficient. Therefore, the wrist-worn measurement is used here to demonstrate the practical output response of the optimized OTEG under wearable small-temperature-difference conditions.

The optimized OTEG was further tested in indoor and outdoor environments to investigate its output response under different practical conditions. The indoor and outdoor air temperatures were approximately 16 °C and 15 °C, respectively. During the test, the device was worn on the wrist while the wearer remained stationary or walked at a speed of approximately 1 m/s. The output voltage curves under these conditions are shown in Figure 14.

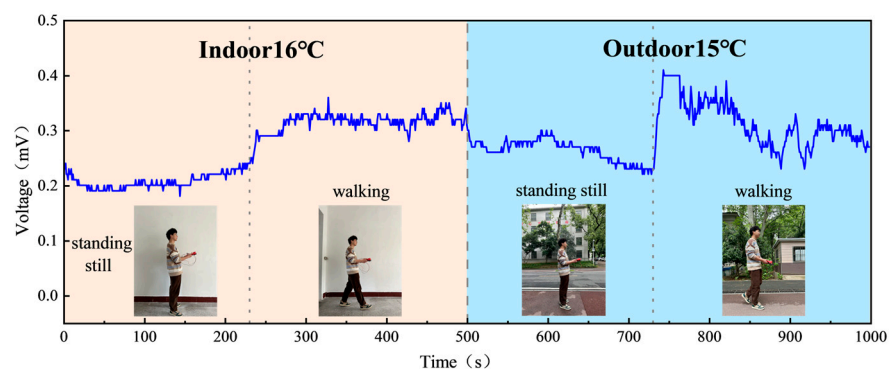


Figure 14. Output voltage of the optimized OTEG worn indoors and outdoors under stationary and walking states. The three vertical dotted lines indicate, from left to right, the transition from standing still to walking indoors, the transition from indoor to outdoor testing, and the transition from standing still to walking outdoors.

As shown in Figure 14, the output voltage of the optimized OTEG varied with both the environmental condition and the motion state of the wearer. Under the same activity condition, the output voltage was higher outdoors than indoors. Under the same environmental condition, walking produced a higher output voltage than the stationary state. These results indicate that the optimized OTEG could respond to changes in practical wearable conditions and maintain a measurable open-circuit voltage.

4. Discussion

The results demonstrate that doped SWCNT thermoelectric clays can serve as deformable p-type and n-type legs for flexible OTEGs. However, the substantially lower Seebeck-coefficient magnitude, power factor, and ZT of the n-type clay indicate that p/n performance matching remains a major material-level limitation. Improving the thermoelectric performance and environmental stability of n-type SWCNT clays is therefore important for further device optimization.

The controlled device comparison reveals that the properties of individual thermoelectric legs cannot be directly translated into device-level performance. Linear fitting yielded Seebeck coefficients of 235.74 and 242.27 $\mu\text{V K}^{-1}$ for the initial and optimized OTEGs, respectively, both below the ideal value of 316.15 $\mu\text{V K}^{-1}$ calculated from the individual legs. The small difference between the two measured values indicates that replacing the p-type clay interconnect did not substantially restore the open-circuit thermoelectric voltage. Although the clay interconnect may produce a counteracting thermoelectric contribution under a nonuniform temperature distribution, the controlled comparison suggests that this contribution was secondary. The remaining deviation from the ideal value may arise from imperfect thermal contact, nonuniform temperature distribution, p/n imbalance, and variations in the geometry or properties of individual legs.

By contrast, the interconnect had a dominant influence on internal resistance and load-matched power. At $\Delta T = 21$ K, replacing the clay interconnect with the Cu-foil/silver-paste assembly reduced the internal resistance from 371.91 to 2.09 Ω , whereas the open-circuit voltage increased only from 4.95 to 5.08 mV. The maximum powers predicted using $P_{\text{max}} = V_{\text{oc}}^2/(4R_{\text{in}})$ agreed closely with the measured values. Therefore, the approximately 187-fold power enhancement was primarily attributable to the reduction in series resistance rather than to the removal of a substantial counteracting thermoelectric voltage. The principal role of the Cu-foil/silver-paste assembly was thus to provide a compliant, low-resistance electrical interconnect.

Mechanical testing of the simplified two-pair device indicated that the optimized interconnect maintained electrical continuity during bending, rolling, and stretching. Its resistance remained relatively stable throughout 1000 bending cycles and a cumulative 4000 rolling cycles. Nevertheless, these results cannot fully establish the durability of the complete five-pair device under long-term wearing conditions, where repeated multidirectional deformation, perspiration, and variations in skin contact may introduce additional degradation.

The wrist-worn measurements provide a proof of concept under practical small-temperature-difference conditions. The observed voltage variations were influenced by skin contact, ambient temperature, cold-side heat dissipation, and body motion. Because these measurements involved a single wearer and an infrared-estimated temperature difference of only approximately 1–1.5 K, the resulting apparent Seebeck coefficient should not be interpreted as a calibrated intrinsic device parameter. A limitation of the present study is that statistical variation among independently fabricated samples and devices was not evaluated. Future work should therefore address n-type material performance, contact-resistance characterization, independent-device reproducibility, calibrated temperature measurements, thermal management, and long-term wearable reliability.

5. Conclusions

TCNQ- and TPP-doped SWCNT clays enabled the fabrication of p-type and n-type thermoelectric legs for a five-pair flexible OTEG. The principal contribution of this study is the controlled demonstration that interconnect resistance strongly affects device-level power output. At $\Delta T = 21$ K, replacing the p-type clay interconnect with a compliant Cu-

foil/silver-paste assembly reduced the internal resistance from 371.91 to 2.09 Ω , whereas the open-circuit voltage increased only from 4.95 to 5.08 mV. Consequently, the maximum output power increased approximately 187-fold, from 16.47 nW to 3.08 μ W, corresponding to a power density of 356.36 nW cm⁻². Mechanical tests on a simplified two-pair device indicated that the optimized interconnect maintained electrical continuity during repeated deformation, while wrist-worn measurements demonstrated measurable voltage generation under practical conditions. Further development requires improved p/n matching, rigorous contact-resistance and device-reproducibility analyses, calibrated wearable measurements, and long-term durability evaluation.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/en19153626/s1>, Figure S1: Electrical-conductivity measurement setup for SWCNT thermoelectric clays; Figure S2: Thermal-conductivity measurement setup based on DSC measurement; Figure S3: Customized bending, rolling, and cyclic-fatigue test platform; Table S1: Instruments used for thermoelectric and mechanical characterizations.

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Abbreviations

The following abbreviations are used in this manuscript:

TEG	Thermoelectric generator
OTEG	Organic thermoelectric generator
TE	Thermoelectric
SWCNT	Single-walled carbon nanotube
TCNQ	7,7,8,8-Tetracyanoquinodimethane
TPP	Triphenylphosphine
PF	Power factor
ZT	Thermoelectric figure of merit
SEM	Scanning electron microscopy

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