



OPEN Facile Y-type Micro $\text{Ag}_2\text{Se}/\text{MgAgSb}$ flexible thermoelectric device based on lift-off technology

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The development of thermoelectric devices provides an ideal solution for self-sustaining Internet of Things (IoT) applications. However, existing thermoelectric devices face issues such as low integration levels and poor stability. To address these challenges, this paper presents a novel method for fabricating flexible thermoelectric devices using $\text{Ag}_2\text{Se}/\text{MgAgSb}$, which combines MEMS lithographic lift-off techniques to achieve precise control over the dimensions of the thermoelectric functional layer while also reducing fabrication costs. The optimal cell dimensions were identified through simulations. The device achieved an open circuit voltage of 0.49 mV at a temperature difference of 50 K. Additionally, the output power density of the $\text{Ag}_2\text{Se}/\text{MgAgSb}$ flexible Y-type thermoelectric device reached $3.39 \mu\text{W}/\text{m}^2$ at a temperature difference of 5 K. The device also demonstrated excellent flexibility; even after being bent 800 times around a glass rod with a diameter of 4 mm, its conductivity decreased by only 12%. Furthermore, it maintained stability under high humidity conditions. This work offers a novel strategy for the miniaturization and large-area controllable fabrication of flexible and reliable thermoelectric devices, as well as an enhancement in their reliability.

Keywords Flexible thermoelectric detectors, MEMS, Lift-off, Ag_2Se , MgAgSb

With the rise and development of the Internet of Things (IoT), traditional battery power supply technologies are insufficient to ensure stable long-term operation of smart devices, necessitating urgent exploration of new energy supply methods^{1,2}. Thermoelectric devices present a promising solution, as they can directly convert temperature differences into electrical potential without the need for specific conditions like solar panels or triboelectric nanogenerators. This capability positions thermoelectric technology as an ideal candidate for self-powered applications^{3,4}.

The integration of IoT devices demands the miniaturization of energy supply methods. By leveraging MEMS technology, it is possible to achieve significant reductions in device size^{5,6}. The structure of thermoelectric devices plays a critical role in determining their output performance. Generally, thermoelectric devices can be categorized into vertical (π -type)^{2,7,8}, planar^{9,10} and annular^{11,12} structures based on the arrangement of thermoelectric materials and electrodes. However, these conventional structures often require thick layers of thermoelectric material, which limits their compatibility with micro-devices. And at the micro-scale, contact resistance can greatly affect the performance and overall reliability of flexible thermoelectric devices. This phenomenon is intrinsically linked to a variety of factors such as material properties, surface morphology, contact situation and environmental conditions. Y-type devices have been explored to address this challenge, offering enhanced temperature acquisition capabilities¹³.

Despite advancements, methods such as chemical vapor deposition for fabricating thermoelectric functional layers typically result in low density, slow deposition rates, and high costs^{14,15}. In contrast, films produced by magnetron sputtering exhibit high quality, uniformity, rapid deposition rates, and scalability for large-area applications^{16,17}. In addition, in order to pattern the functional layer, etching technology is often used in the existing research^{18,19}. Existing research often employs etching technology for patterning functional layers; however, despite developments in ion etching^{20,21}, laser etching^{22,23} and other processes^{24,25}, etching processes tend to be slow and lack precision, making large-area fabrication problematic. Lift-off technology, which utilizes photoresist to remove excess materials, presents a cost-effective and straightforward alternative to etching, yet its application in thermoelectric devices remains underexplored^{26,27}. Therefore, there is a pressing need to investigate efficient fabrication techniques for Y-type devices using magnetron sputtering and lift-off methods.

In this paper, a fabrication method of Y-type flexible thermoelectric device based on MEMS controllable peeling technology is proposed. The thermoelectric functional layer is the $\text{Ag}_2\text{Se}/\text{MgAgSb}$ material studied by

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our group in the early stage²⁸. The materials have good thermoelectric properties and shows certain potential in the application of the Internet of things. The Y-type flexible thermoelectric device was successfully fabricated and systematically tested. The magnetron sputtering process was employed for preparing the thermoelectric functional layer, with its patterning achieved via peeling technology. This process is characterized by simplicity, low cost, and the ability for large-area fabrication. The designed Y-type thermoelectric device enhances the efficiency of thermoelectric temperature difference collection and can be customized according to specific requirements. The preparation method proposed herein offers an ideal solution for the miniaturization and large-area application of flexible thermoelectric devices.

Experiment Materials

PDMS was sourced from Suzhou Ruicai Semiconductor Co., while 3-inch single silicon wafers were obtained from Suzhou Research Materials Micro-Nano Technology Co., Ltd. Photosensitive polyimide photoresist (PI JAPB-101), RDP-2100P photoresist, Tetramethylammonium hydroxide (TMAH), and acetone were procured from Nantong Jingai Microelectronics Technology Co., Ltd. Ag₂Se and MgAgSb targets were purchased from Beijing Zhongnuo New Material Technology Co., Ltd. Hydrochloric acid (HCl) was acquired from Aladdin Biochemical Technology Co.

Design and optimize

The equivalent circuit of FTEG as a power supply with load resistance is shown in the Fig. 1. The matching of the internal resistance of the device and the load resistance is particularly important.

The load voltage of the device (V_L) can be expressed as :

$$V_L = \frac{R_L V_0}{R_{teg} + R_L} \quad (1)$$

$$V_0 = n (S_n + S_p) \Delta T \quad (2)$$

Where V_0 is the open circuit voltage of the device, R_{teg} is the internal resistance of the device, R_L is the load resistance of the device, S_n is the Seebeck coefficient of n-type materials, and S_p is the Seebeck coefficient of p-type materials. The output power of the device (P_L) can be expressed as :

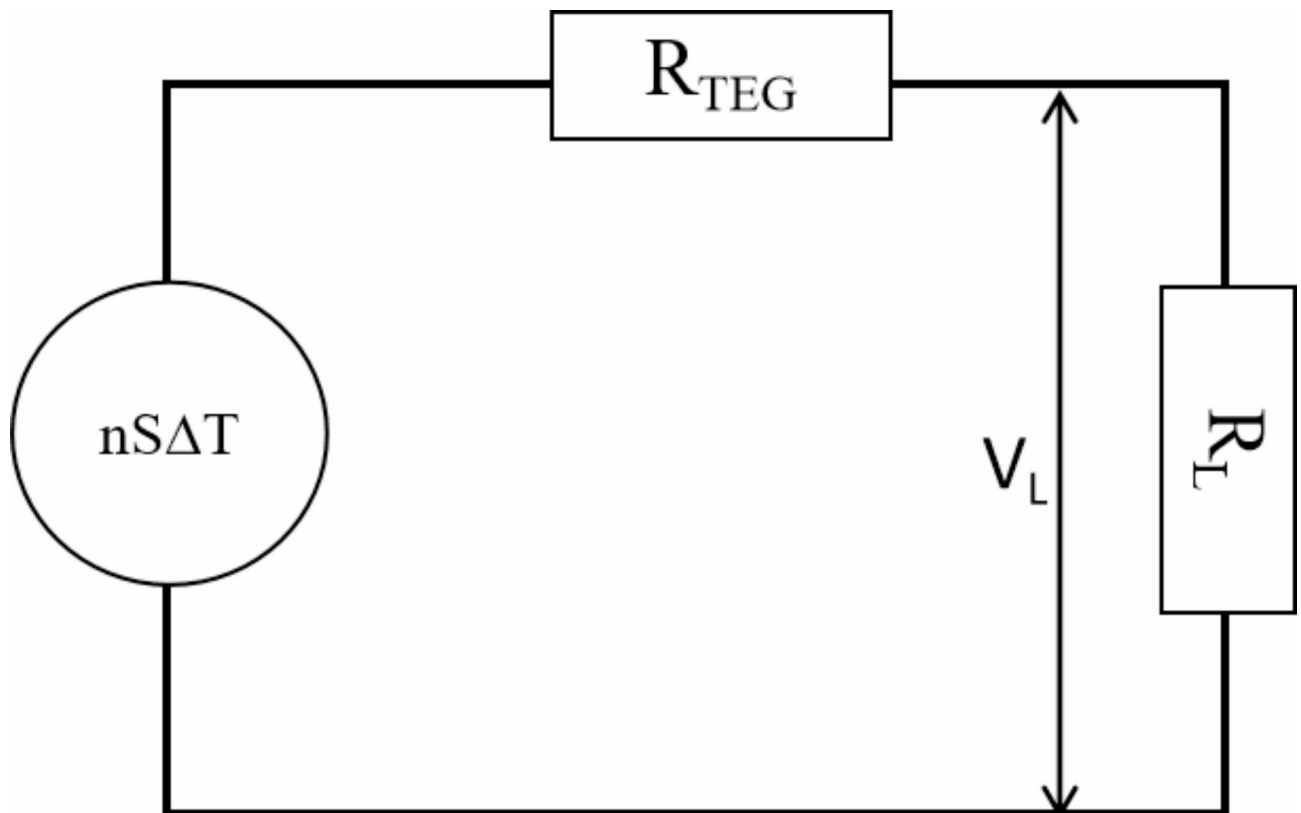


Fig. 1. Equivalent circuit diagram of FTEG as a power supply with load resistor.

$$P_L = V_L I_L = \frac{V_L^2}{R_L} = \frac{\left[\frac{V_0 R_L}{(R_{teg} + R_L)} \right]^2}{R_L} \quad (3)$$

Where I_L is the load current. When the internal resistance of the device is the same as the load resistance, the output power of the device is the largest, and the maximum output power (P_{max}) can be expressed as :

$$P_{Lmax} = \frac{(V_0)^2}{4R_{teg}} \quad (4)$$

The internal resistance of the device is affected by the resistance of the thermoelectric unit. The resistance of the thermoelectric unit is related to its size and can be expressed as

$$R_n = \rho_n \frac{l}{S} \quad (5)$$

$$R_p = \rho_p \frac{l}{S} \quad (6)$$

Where l is the length of the thermoelectric unit, S is the surface area of the thermoelectric device, ρ_n is the resistivity of the n-type materials, and ρ_p is the resistivity of the p-type materials. Therefore, the maximum output power of the device can be expressed as

$$P_{Lmax} = \frac{(V_0)^2}{4R_{teg}} = \frac{[n(S_n + S_p) \Delta T]^2}{4n(\rho_n \frac{l}{S} + \rho_p \frac{l}{S})} \quad (7)$$

Where n is the number of n/p thermoelectric unit groups. It can be seen from this formula that the output power of the device is affected by the length and width of the thermoelectric unit. Therefore, the Comsol software is used to simulate the length and width of the thermoelectric unit. The Fig. 2 shows the heat transfer simulation results of FTEG.

In a fixed space, increasing the size of the thermoelectric unit leads to an enhancement in the output power of the device. However, a simultaneous increase in both length and width results in higher resistance within the thermoelectric unit, which can decrease the overall integration of the device. Therefore, it is essential to consider the output power per unit weight of the device to determine its optimal dimensions. In the Fig. 3, analysis of the data reveals that when the length of the thermoelectric unit is set to 800 μm , the output power per unit weight reaches its maximum. Similarly, a width of 600 μm also yields the highest output power per unit weight. Consequently, the designed dimensions for the thermoelectric unit are 800 μm in length and 600 μm in width. The optimized design maximizes the output power per unit weight of the device, highlighting the importance of balancing size with resistance to achieve efficient energy conversion in the FTEG system.

FTEG fabrication

The overall flow chart of flexible thermoelectric devices prepared using the MEMS process is illustrated in Fig. 4. The fabrication process begins with the preparation of a 3-inch single-sided silicon wafer, followed by organic cleaning. Next, 300 nm of aluminum (Al) is sputtered onto the silicon wafer, serving as a metal sacrificial layer that can react with dilute hydrochloric acid.

Subsequently, photosensitive polyimide photoresist is spin-coated onto the Al sacrificial layer. The coating process involves low-speed rotation at 500 rpm for 10 s, followed by high-speed rotation at 3000 rpm for 45 s, achieving a film thickness of 6 μm . The coated wafer is then exposed under a UV lithography machine with an exposure dose of 200 mJ. TMAH (2.38%) developer is used to develop the pattern, resulting in the formation of the side electrode pattern.

The next step involves polyimide imidization, starting at a low temperature of 140 °C and gradually increasing to 320 °C, with intermediate steps at 180 °C and 250 °C—each held for 30 min. The final temperature of 320 °C is maintained for 3 h to achieve complete PI imidization.

Following this, a peeling pattern is created on the PI layer by spin-coating RDP-2100P photoresist. The spin-coating parameters are set to low-speed rotation at 500 rpm for 5 s and high-speed rotation at 3000 rpm for 60 s, resulting in a thickness of 6 μm . An exposure dose of 120 mJ is applied, and TMAH (1:6 with deionized water) is used for development over 70 s to form the thermoelectric functional layer pattern.

Next, the thermoelectric functional layer is prepared. DC sputtering is performed for Ag_2Se with a power of 80 W, a pressure of 2.0 Pa, and a duration of 1 h, yielding a layer thickness of 1.2 μm . The thickness ratio of the Ag_2Se layer to the photoresist layer exceeds 1:3, suitable for the lift-off process. After sputtering, the excess RDP-2100P photoresist and Ag_2Se are removed using acetone, retaining only the Ag_2Se in the desired pattern. The layer is then annealed in a vacuum at 300 °C for 30 min. The lithography and development processes are repeated for the photoresist before sputtering MgAgSb under similar conditions—80 W power, 3.7 Pa pressure, and 1 h duration, achieving a thickness of 1.2 μm . After sputtering, the excess photoresist and

MgAgSb are stripped, followed by annealing at 350 °C for 30 min in a vacuum^{29,30}.

In the seventh step, electrodes are prepared by spin-coating RDP-2100P photoresist corresponding to the PI pattern, following the same spin-coating and development process as before. A copper (Cu) metal seed layer with a thickness of 200 nm is sputtered to prepare for electroplating. Electroplating is conducted symmetrically

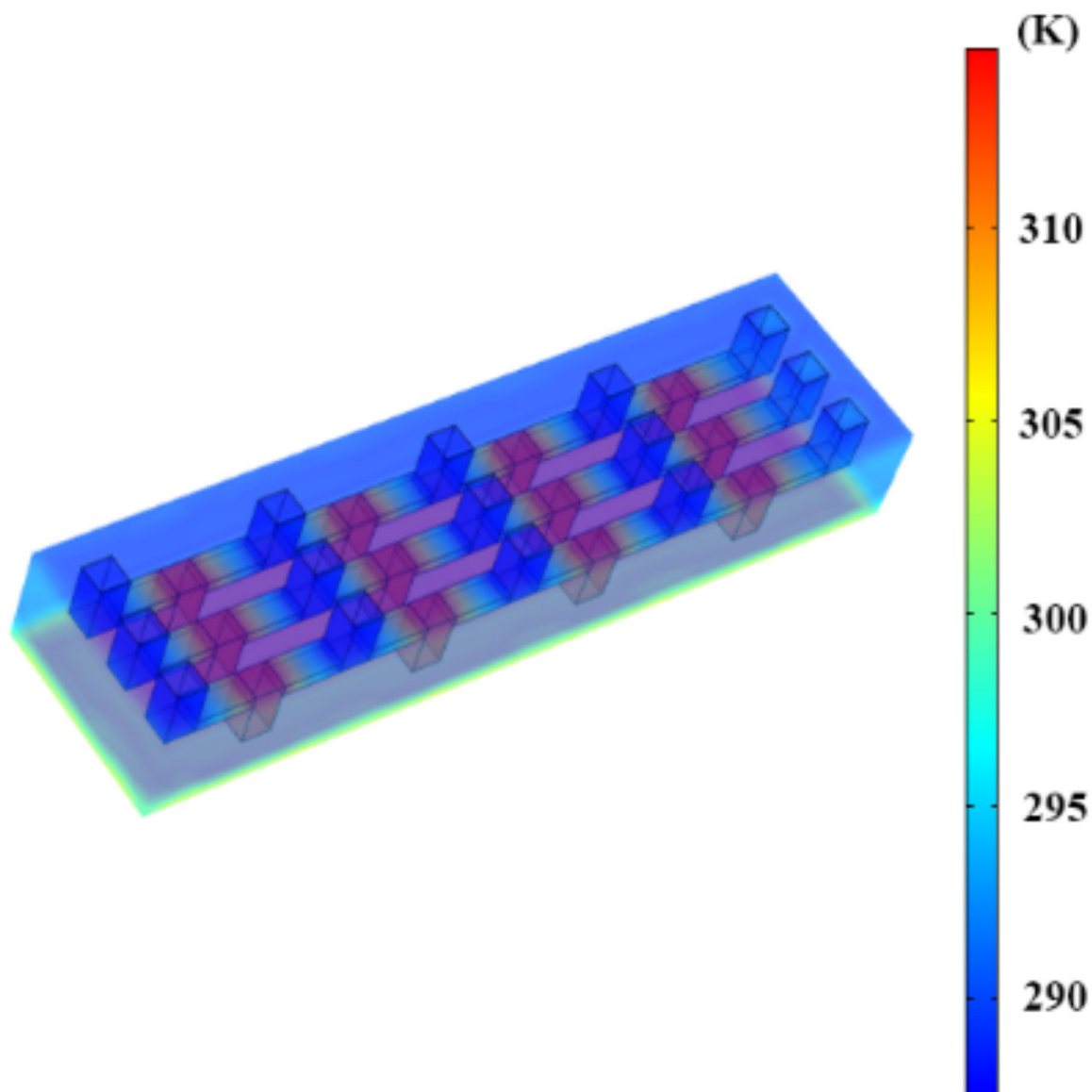


Fig. 2. FTEG heat transfer simulation.

on the 3-inch silicon wafer for four cycles at a current of 0.8 A for 5 s each cycle. Following electroplating, a lift-off process is carried out to remove excess photoresist and Cu, leaving only the patterned Cu metal intact. The process for preparing the other side electrode is identical, involving the selection of a different mask and following the same lithography and electroplating procedures.

Finally, PDMS encapsulation is performed. The PDMS is mixed with a curing agent at a 10:1 ratio, thoroughly stirred, and vacuum degassed. The mixed PDMS is then dropped onto the thermoelectric functional layer and spun at 1000 rpm for 30 s, resulting in a thickness of 100 μm . This is cured in a vacuum drying box at a constant temperature of 60 $^{\circ}\text{C}$ for one hour.

The last step involves device lift-off. The prepared silicon wafers are immersed in dilute hydrochloric acid with a molar ratio of 1:4, which corrodes the Al sacrificial layer while the PI and PDMS remain unaffected, thereby protecting the electrode and thermoelectric functional layer.

FTEG characterization

The surface and cross-section of the thermoelectric functional layer were characterized using electron microscopy. To evaluate the output performance of the flexible thermoelectric generator (FTEG), a dedicated test platform was constructed. The hot end of the FTEG is regulated by a heated stage, while the cold end is managed by a commercial Peltier device. The temperature control module (TCM-1030) ensures precise regulation of the Peltier temperature, achieving an accuracy of 0.01 $^{\circ}\text{C}$. A sliding rheostat serves as the load resistance for the FTEG. A digital multimeter (GDM-9061) is used to record the load voltage of the device at various resistances

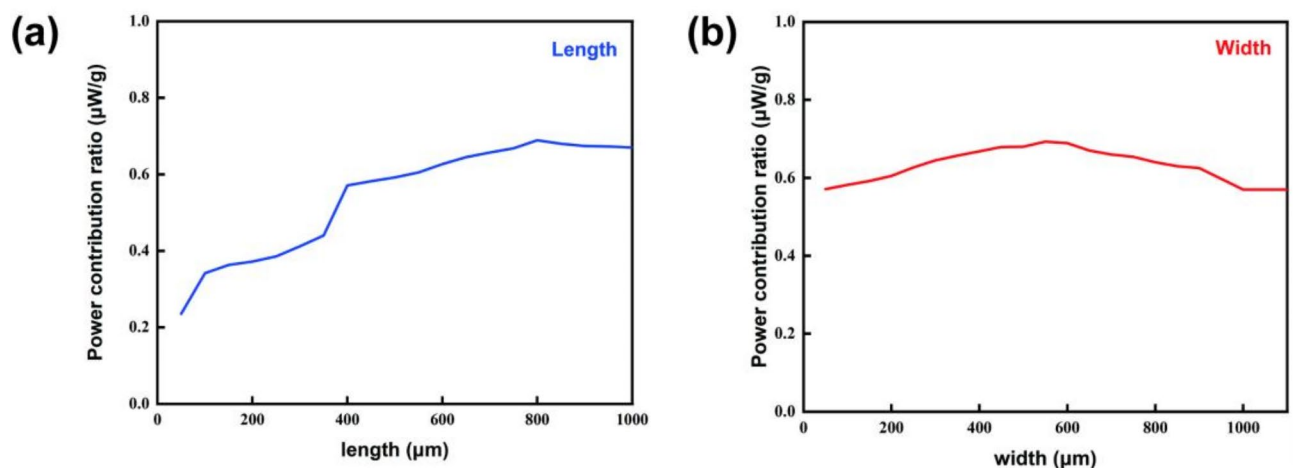


Fig. 3. Dependence of FTEG power output per unit weight on the (a) width and (b) length of the thermoelectric cell.

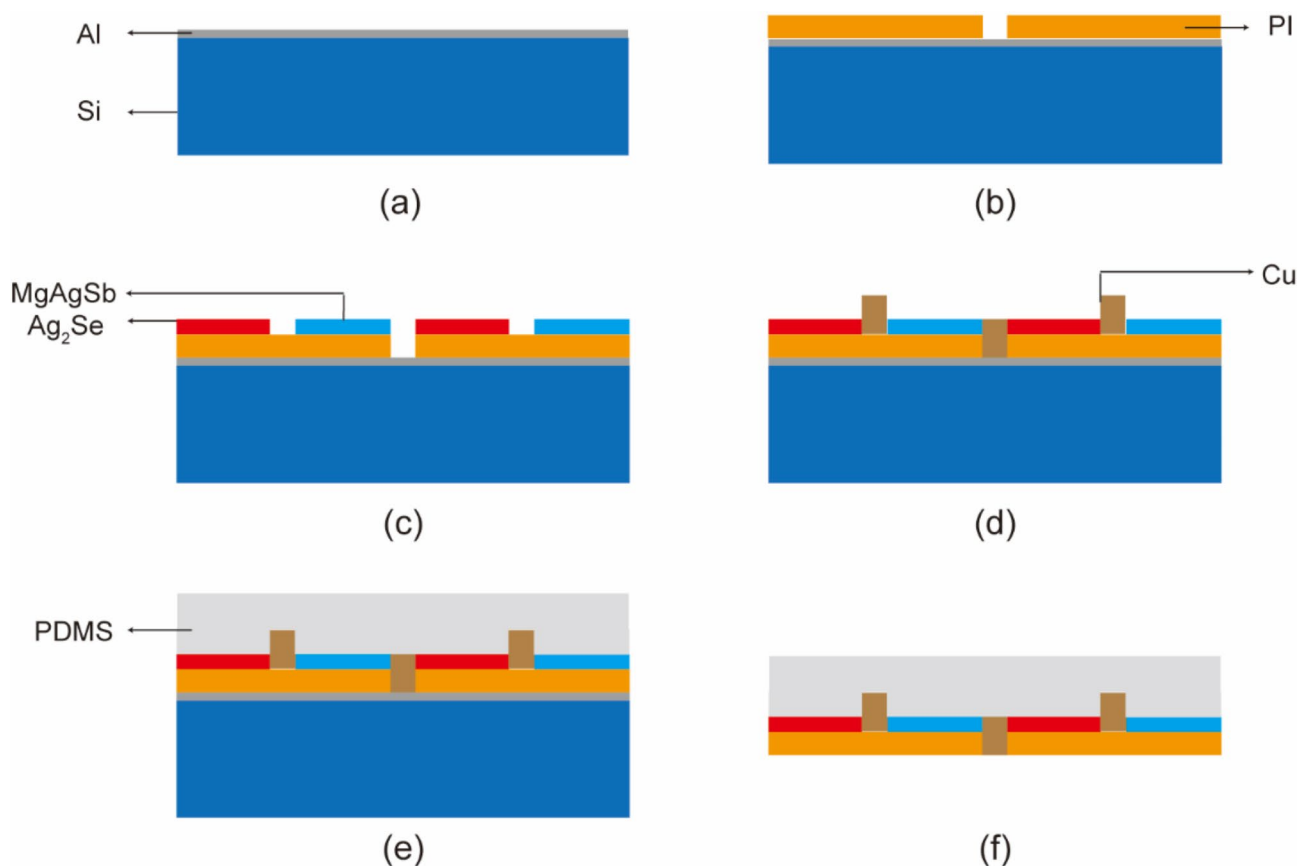


Fig. 4. MEMS preparation process for flexible Y-type thermoelectric devices.

and temperatures. The output power of the FTEG is subsequently calculated using the measured voltage and load resistance.

Result and discussion

The lift-off technology has excellent pattern control. The prepared flexible thermoelectric devices are shown in the Fig. 5 a. As shown the Fig. 5b, c, the surface of Ag₂Se and MgAgSb. The diagram shows that Ag₂Se and MgAgSb are in good condition and match the design dimensions. In the Fig. 5d, it can be seen from the cross section of the device that the thicknesses of Ag₂Se and MgAgSb are 1.2 μm. Lift-off technology enables precise

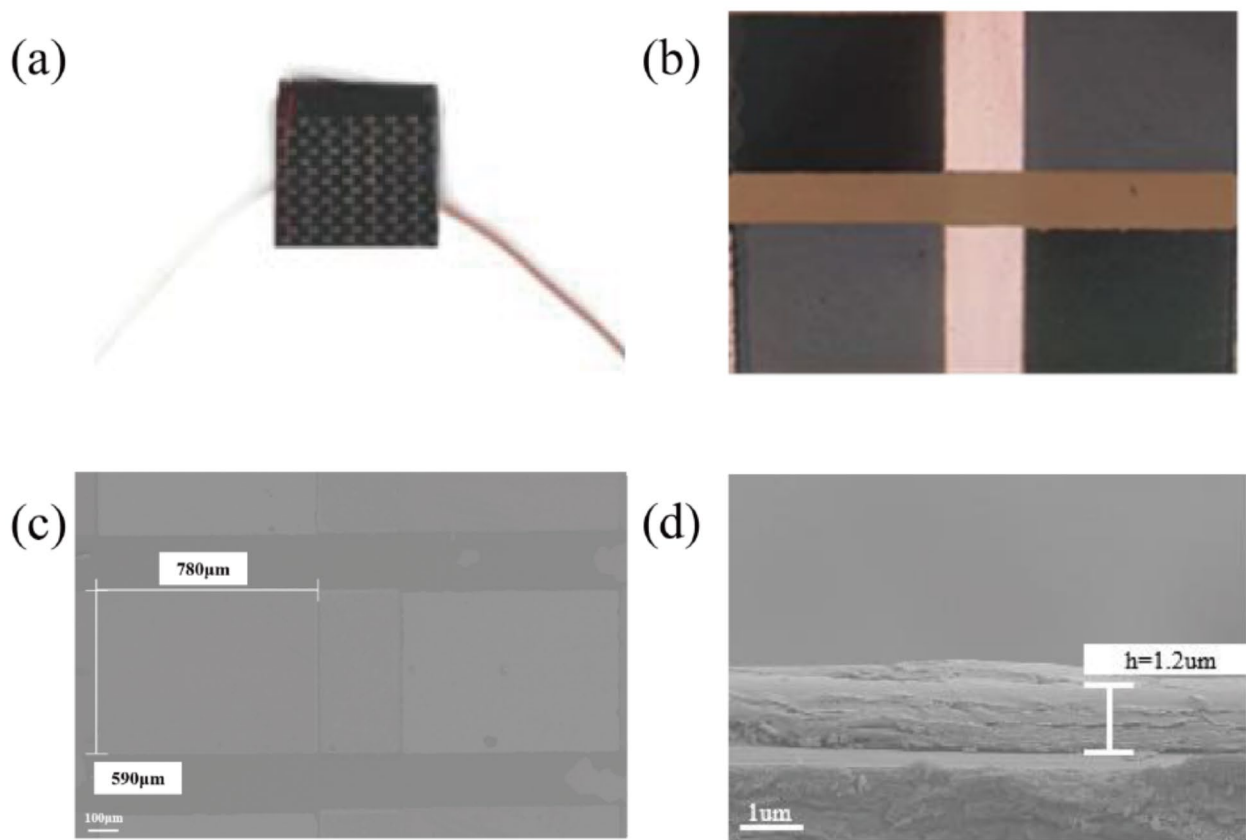


Fig. 5. (a) Physical appearance of the thermoelectric device after lift-off, (b) Confocal microscopy image of the two thermoelectric units, (c) SEM image of the surface, (d) SEM image of the cross-section.

and accurate pattern lift-off of thermoelectric functional layers. During the annealing process, the grains of the material will be rearranged, and the internal stress will be released, which leads to the decrease of the thickness of the material. The mapping of the SEM image can be seen in Fig. 6, where Cu is used as an electrode on both sides of the film, and the Ag element is contained in a group of adjacent units because it is contained in both compounds, but the mapping colour of the Ag element in the Ag_2Se unit is heavier because of the difference in the Ag at% ratio between Ag_2Se and MgAgSb . The rest of the elements are uniformly distributed in their respective regions.

Figure 7a shows the open-circuit voltage test results of the device. It can be seen from Fig. 7b that the open circuit voltage of the device maintains a good linear relationship with the temperature. This indicates that the output voltage of the device follows the formula

$$\Delta V = S \times \Delta T \quad (8)$$

Where S represents the Seebeck coefficient, V denotes the voltage, and T signifies the temperature, thereby demonstrating the device's capacity to generate voltage in response to temperature difference. These results confirm its effective performance in converting temperature difference into electrical energy. When the ΔT is 50 °C, the open circuit voltage of the device is 0.46 mV. Figure 7c is the test diagram of the output power density of the device. When the load resistance is the same as the internal resistance of the device, the output power of the device is the largest. Therefore, the internal resistance of the actual device is 28.9 K Ω . It is larger than the expected internal resistance. The reason may be due to the ohmic contact between the metal electrode and the thermoelectric material layer and the uncontrollable oxidation of the thermoelectric material layer. The dependence of the output power density of the device on temperature is shown in the figure. The output power of the device is calculated by the output voltage and load resistance of the device. When the temperature difference is 5 K, the maximum output power density of the device is 3.39 $\mu\text{W}/\text{m}^2$. Due to the relationship of $P = V^2/R$, and then according to Eq. (8), the output power is quadratically related to the temperature difference, which is shown in Fig. 7d as a function of the temperature difference. It shows that the output power of the device also increases nonlinearly according to the increase of temperature difference.

In addition, as a flexible thermoelectric device, the flexibility of the device should also be considered. As shown in the figure, the device is bent on a glass rod with a radius of 4 mm. After 800 bending cycles, the conductivity of the device decreased to 88% of the original, which may be due to the fracture of the ohmic contact between

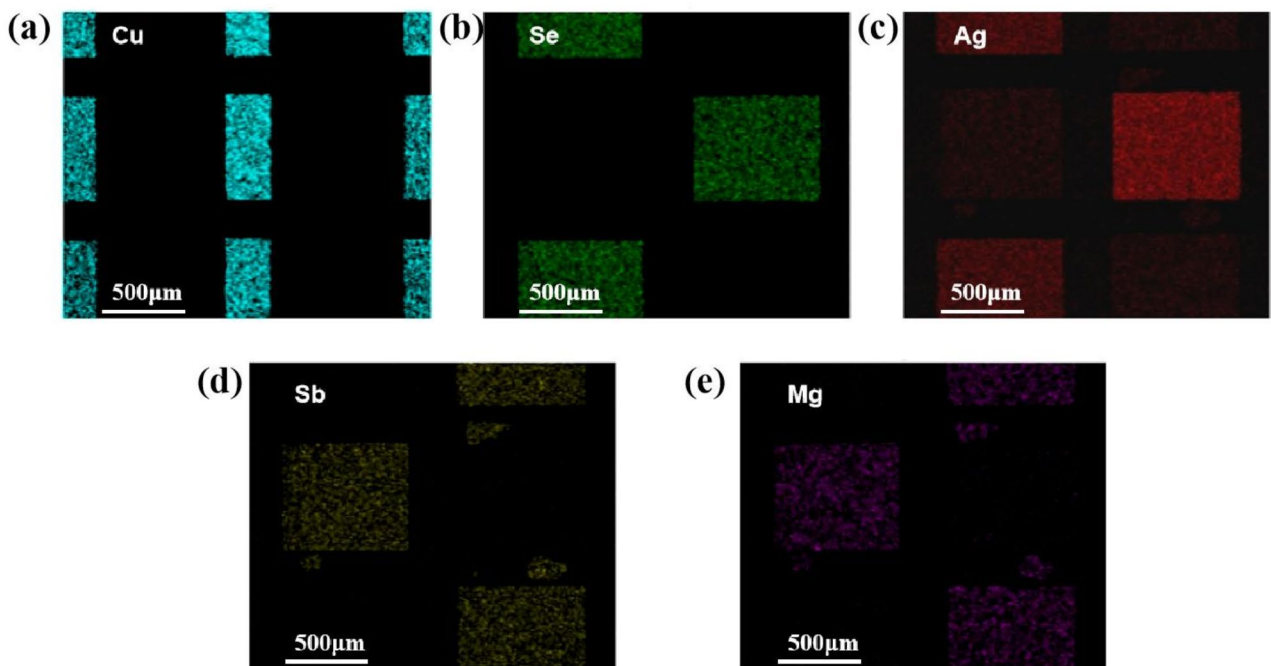


Fig. 6. Mapping of the SEM image (a)Cu, (b)Se, (c)Ag, (d)Sb, (e)Mg.

the electrode and the thermoelectric functional layer. The environmental adaptability of the device was tested. In the glove box, the device was tested in high humidity environment. The resistance and output performance of the device were tested by using a humidifier to humidify and simulate different humidity environments. As shown in the Fig. 8b, the resistance of the device remains basically unchanged in high humidity environment. At 80 humidity, the conductivity of the device decreases, which may be due to the oxidation of the external electrode, resulting in the increase of the overall resistance of the device. In addition, the high temperature service characteristics of the device in the air environment were also studied. The resistance of the device in an air environment was also investigated as a function of temperature, and the conductivity of the device was tested by holding it at different temperatures for 30 min. As shown in Fig. 8c, the overall internal resistance of the device decreases with increasing temperature. the conductivity of Ag₂Se and MgAgSb is dependent on temperature, with the conductivity increasing with increasing temperature^{29,30}. An increase in the conductivity of the thermoelectric functional layer leads to a decrease in the overall resistance of the device. In summary, the FTEG shows strong performance and adaptability under a variety of conditions, maintaining conductivity under high humidity conditions, and resistance reduction at elevated temperatures will show higher efficiency.

Conclusion

This paper presents a method for fabricating flexible Y-type thermoelectric devices that leverage MEMS-controlled lift-off technology. The thermoelectric unit, composed of Ag₂Se/MgAgSb, features a functional layer created through magnetron sputtering, resulting in a dense film. This lift-off technique offers several advantages over traditional etching methods, such as ease of use, reduced fabrication costs, and the ability to produce large-area components. Through COMSOL simulations, the optimal dimensions for the thermoelectric unit were identified as 800 nm in length and 600 nm in width. The innovative Y-type structure effectively enhances the integration and collection of larger temperature gradients within flexible thermoelectric devices. At a temperature difference of 50 K, an output voltage of 0.49 mV was achieved, while an output power density of 3.39 $\mu\text{W}/\text{m}^2$ was obtained at a specified temperature difference of 5 K. Moreover, the device demonstrates impressive flexibility; even after being bent 800 times around a glass rod with a diameter of 4 mm, its conductivity decreases by only 12%. It also exhibits excellent stability under harsh environmental conditions. The successful integration of MEMS lift-off miniaturization and large-area applications in the field of thermoelectrics, paving the way for future advancements in this area.

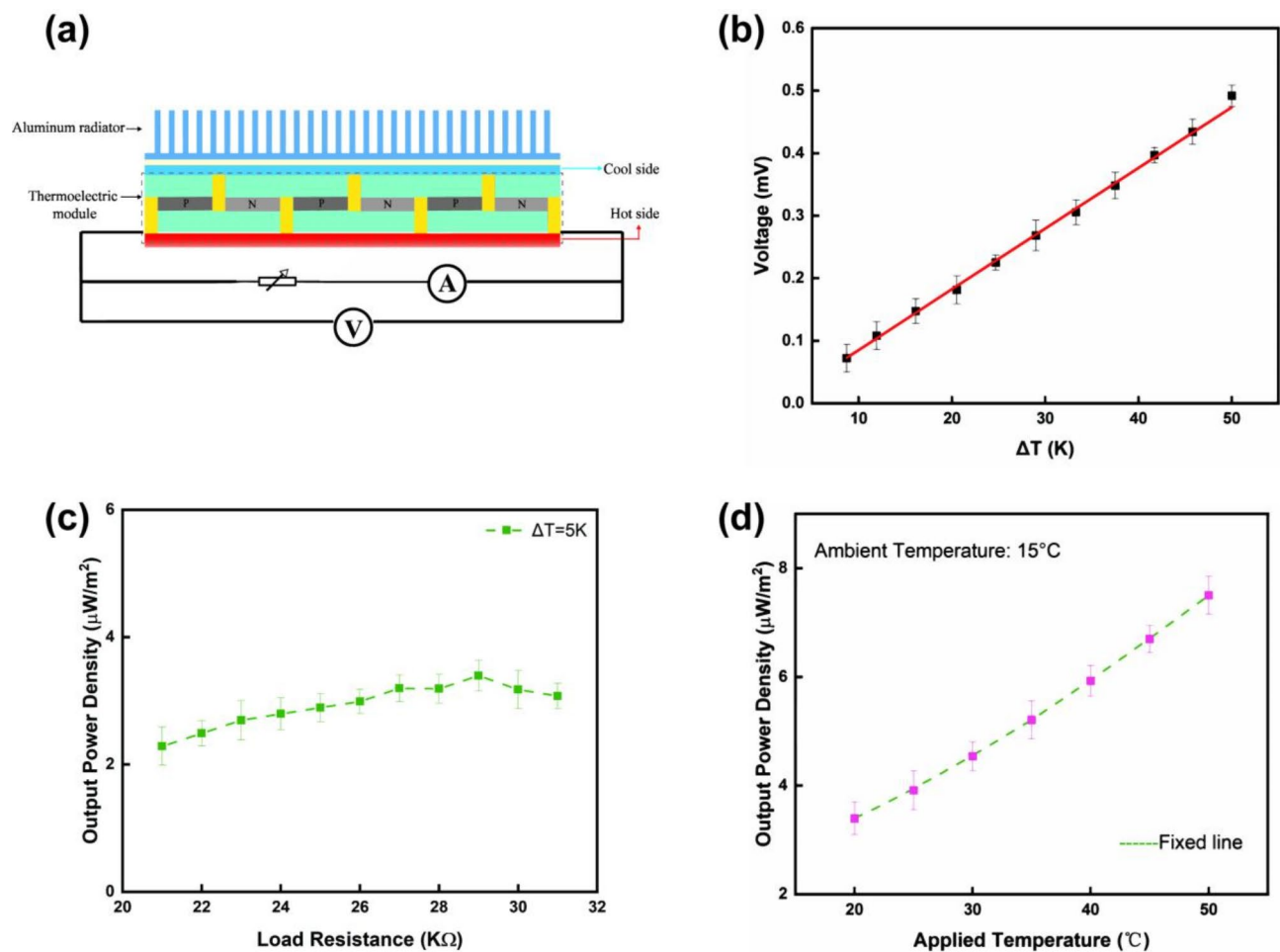


Fig. 7. (a) Output performance test system equivalent diagram, (b) FTEG open circuit voltage test results, (c) FTEG output power with load resistance test results and (d) FTEG output power variation with temperature test results.

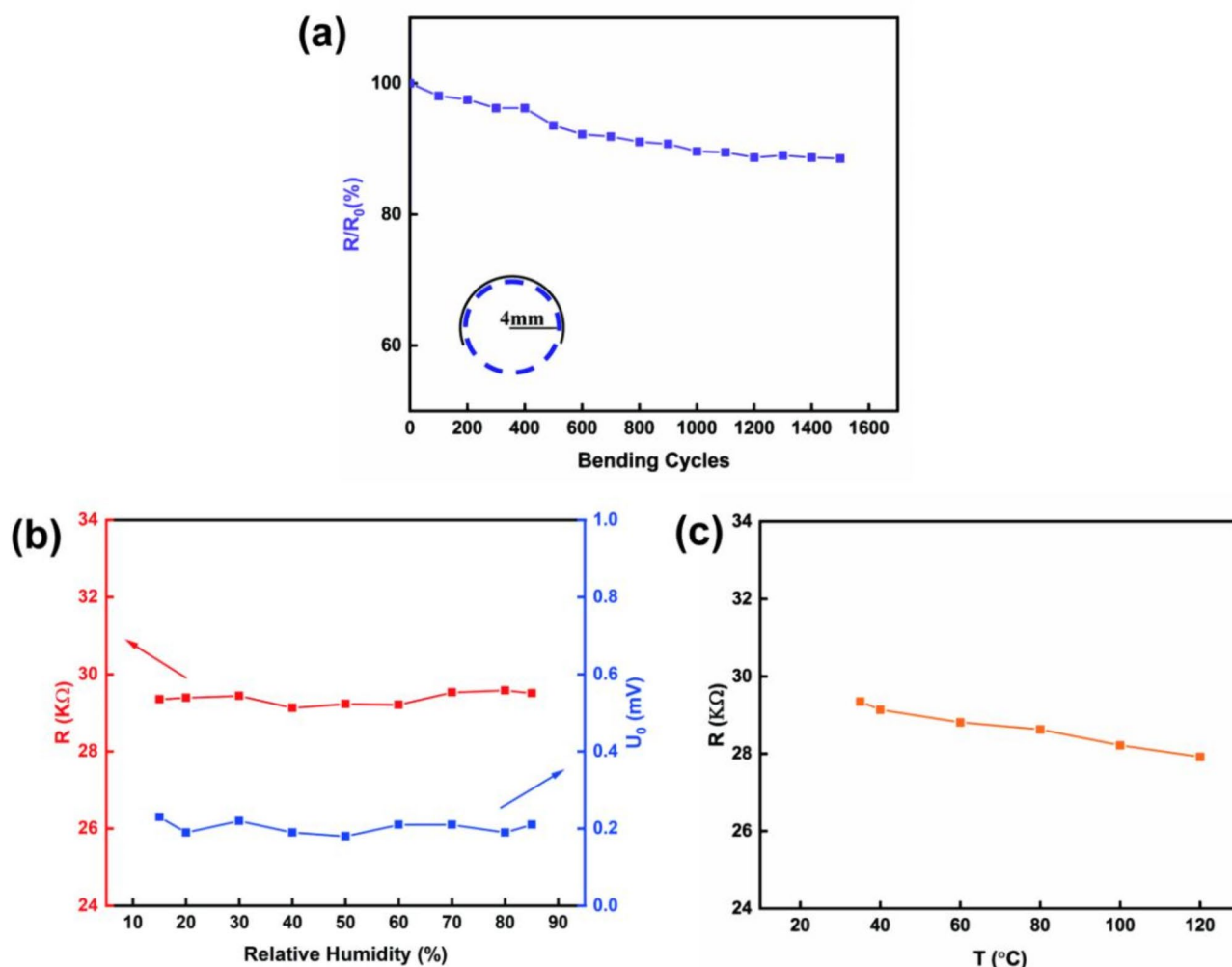


Fig. 8. (a) FTEG flexibility test results, (b) Changes in resistance and output voltage of FTEG at different humidity levels and (c) Changes in resistance of FTEG at different temperatures.

Data availability

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

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Author contributions

T. G. and S. W.: Conceptualization, Methodology, Software, Investigation, Formal Analysis, Writing - Original Draft; G. R. and G. R.: Data Curation, Writing - Original Draft; Z. W.: Visualization, Investigation; L. D. (Corresponding Author): Conceptualization, Funding Acquisition, Resources, Supervision, Writing - Review & Editing; X. C.: Conceptualization, Funding Acquisition, Resources.

Declarations

Competing interests

The authors declare no competing interests.

Additional information

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