Novel Thermal Diffusion Temperature Engineering Leading to High Thermoelectric Performance in Bi$_2$Te$_3$-Based Flexible Thin-Films

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Flexible Bi$_2$Te$_3$-based thermoelectric devices can function as power generators for powering wearable electronics or chip-sensors for internet-of-things. However, the unsatisfied performance of n-type Bi$_2$Te$_3$ flexible thin films significantly limits their wide application. In this study, a novel thermal diffusion method is employed to fabricate n-type Te-embedded Bi$_2$Te$_3$ flexible thin films on flexible polyimide substrates, where Te embeddings can be achieved by tuning the thermal diffusion temperature and correspondingly result in an energy filtering effect at the Bi$_2$Te$_3$/Te interfaces. The energy filtering effect can lead to a high Seebeck coefficient $\approx 160 \mu$V K$^{-1}$ as well as high carrier mobility of $\approx 200$ cm$^2$ V$^{-1}$ s$^{-1}$ at room-temperature. Consequently, an ultrahigh room-temperature power factor of 14.65 $\mu$W cm$^{-1}$ K$^{-2}$ can be observed in the Te-embedded Bi$_2$Te$_3$ flexible thin films prepared at the diffusion temperature of 623 K. A thermoelectric sensor is also assembled through integrating the n-type Bi$_2$Te$_3$ flexible thin films with p-type Sb$_2$Te$_3$ counterparts, which can fast reflect finger-touch status and demonstrate the applicability of as-prepared Te-embedded Bi$_2$Te$_3$ flexible thin films. This study indicates that the thermal diffusion method is an effective way to fabricate high-performance and applicable flexible Te-embedded Bi$_2$Te$_3$-based thin films.

1. Introduction

Thermoelectrics, as an emission-free power generation technology,[1–3] can provide eco-friendly power supply for the portable and wearable electronic devices, such as chip-sensors,[4] electrocardiographic systems,[5] and implantable electronic devices.[6] The conversion efficiency of a thermoelectric device is determined by the performance of composing thermoelectric materials, which is evaluated by dimensionless figure-of-merit $ZT (ZT = S^2\sigma T/\kappa)$, where $S$, $\sigma$, $S^2\sigma$, $T$, and $\kappa$ are the Seebeck coefficient, electrical conductivity, power factor, absolute temperature, and thermal conductivity, respectively.[7,8] Regardless of high-performance thermoelectric materials, such as SnSe,[9] Cu$_2$Se,[10] CoSb$_3$,[11] GeTe,[12] and Mg$_2$Sb$_2$,[13] traditional Bi$_2$Te$_3$-based thermoelectric materials are still widely studied because of their superior performance at room-temperature.[14–16]

With potentially high flexibility, thermoelectric thin films are more suitable for wearable electronics compared to the bulk form.[17–19] However, the unsatisfied performance of n-type Bi$_2$Te$_3$ flexible thin films significantly limits their wide application. In this study, a novel thermal diffusion method is employed to fabricate n-type Te-embedded Bi$_2$Te$_3$ flexible thin films on flexible polyimide substrates, where Te embeddings can be achieved by tuning the thermal diffusion temperature and correspondingly result in an energy filtering effect at the Bi$_2$Te$_3$/Te interfaces. The energy filtering effect can lead to a high Seebeck coefficient $\approx 160 \mu$V K$^{-1}$ as well as high carrier mobility of $\approx 200$ cm$^2$ V$^{-1}$ s$^{-1}$ at room-temperature. Consequently, an ultrahigh room-temperature power factor of 14.65 $\mu$W cm$^{-1}$ K$^{-2}$ can be observed in the Te-embedded Bi$_2$Te$_3$ flexible thin films prepared at the diffusion temperature of 623 K. A thermoelectric sensor is also assembled through integrating the n-type Bi$_2$Te$_3$ flexible thin films with p-type Sb$_2$Te$_3$ counterparts, which can fast reflect finger-touch status and demonstrate the applicability of as-prepared Te-embedded Bi$_2$Te$_3$ flexible thin films. This study indicates that the thermal diffusion method is an effective way to fabricate high-performance and applicable flexible Te-embedded Bi$_2$Te$_3$-based thin films.
for wearable electronic design. Particularly, n-type Bi₄Te₃-based and p-type Bi₅.₃Sb₁.₆Te₃-based thermoelectric materials have attracted extensive attentions [15–20]. Various material engineering strategies, such as defect-engineering [21,22], texturing engineering [23–26] and nanoengineering [24–26] have been used to boost thermoelectric performance of Bi₄Te₃-based flexible thin films. Till now, significant enhancements have been observed in p-type Bi₅.₃Sb₁.₆Te₃-based thermoelectric thin films. Varghese et al. [28] used a scalable screen-printing method with its p-type counterparts (the 𝜎 of 14.65 μV cm⁻¹ K⁻¹ at 420 K). To further develop the performance, high-performance n-type Bi₄Te₃-based thermoelectric thin films need further development due to the low 𝜎 (14 μV cm⁻¹ K⁻¹ [28–29]) comparing with its p-type counterparts (the 𝜎 of p-type Bi₅.₃Sb₁.₆Te₃-based thermoelectric thin films can approach as high as ≈20 μV cm⁻¹ K⁻¹ [26]).

To date, numerous methods have been adopted to prepare high-performance n-type Bi₄Te₃-based flexible thin films. Bi₄Te₃ nanowire-based flexible thin films prepared by solution phase printing can approach the 𝜎 of ≈1.10 μV cm⁻¹ K⁻¹ at 400 K [30]. Hybridizing Bi₄Te₃ with graphene oxide and form flexible thin films by vacuum filtration and annealing also approach the 𝜎 of ≈1.08 μV cm⁻¹ K⁻¹ at 297 K. [31] Se-doping in Bi₄Te₃ achieved by mechanical alloying in combination with dispenser printing to form flexible thin films can achieve the 𝜎 of ≈2.65 μV cm⁻¹ K⁻¹ at ≈297 K. [19] Through hybridizing Bi₄Te₃ with polyimide via dispenser printing, Jung et al. [32] successfully prepared a Bi₄Te₃ flexible thin film with the high 𝜎 of ≈3.43 μV cm⁻¹ K⁻¹ at ≈297 K. Hybridizing carbon nanotubes with Bi₄Te₃ and form flexible thin films by in situ solution method successfully enhanced the 𝜎 to ≈7.4 μV cm⁻¹ K⁻¹ at ≈297 K. [33] Additionally, a glass fabric-based Bi₄Te₃ flexible film prepared by screen printing has approached the 𝜎 of ≈13.32 μV cm⁻¹ K⁻¹ at ≈297 K. [34] Most of these methods are suffering from poor crystal growth, which has limited the carrier transport properties and correspondingly limited the thermoelectric performance of as-prepared Bi₄Te₃-based flexible thin films.

In this study, to render sufficient energy for Bi₄Te₃ crystal growth, we use an advanced thermal diffusion method to fabricate n-type Bi₄Te₃ flexible thin films on flexible polyimide (PI) substrates (Figure 1a). Our prepared Bi₄Te₃ flexible thin films (use the film prepared under the diffusion temperature of 623 K as an example) also possess high stability after bending test under various bending radius and bending cycles as evidenced by the minor change of resistance (ΔR/R₀) presented in Figure 1b,c, respectively. Through tuning the diffusion temperature (Tdiff), Te-embedding can form and lead to Bi₄Te₃/Te heterostructures (Figure 1d). It can induce energy filtering effect at the Bi₄Te₃/Te interfaces due to an energy offset (ΔE) between the Te and Bi₄Te₃ conduction band minimums as shown in Figure 1e, which is calculated by density functional theory (DFT) and in turn lead to a high Seebeck coefficient ≈160 μV K⁻¹ as well as high carrier mobility of ≈200 cm² V⁻¹ s⁻¹. Correspondingly a high maximum 𝜎 of 14.65 μV cm⁻¹ K⁻¹ can be achieved in the Te-embedded Bi₄Te₃ flexible thin film prepared under the diffusion temperature of 623 K, which is comparable with other Bi₄Te₃-based flexible thin films (Figure 1f). Applicability of as-prepared n-type Bi₄Te₃ flexible thin films are further demonstrated by an assembled thermoelectric sensor, which can fast reflect the finger touch status.

2. Results and Discussion

To understand the crystal structure of the as-deposited Bi₄Te₃-based flexible thin films, we first performed XRD analyses and their XRD patterns are shown in Figure 2a. As can be seen, when the thermal diffusion temperature is 523 K, the Bi-Te reaction is insufficient and large amount of Bi (JCPDS 44-1246) can be observed as well as the formed Bi₄Te₃ (JCPDS 15-0863). With increasing the thermal diffusion temperature to 548 K, the as-prepared thin film is mainly composed of Bi₄Te₃ phase. Further increasing the temperature to above 573 K, Te precipitates can be observed. When the temperature is above 648 K, almost pure phase Bi₄Te₃ can be observed. To clarify the crystal structure of Bi₄Te₃-based thin film prepared by our thermal diffusion method, the enlarged (102) peak was plotted in Figure 2b, where the appearance of Te at the deposition temperature of 573–623 K can be clearly observed. Unobvious Te is observed in the XRD pattern, indicating that there is fewer Te phase above 648 K. Figure 2c schematically shows the thermal diffusion reaction process under different diffusion temperatures. With high saturation vapor pressure and diffusion energy [37,38], Te atoms can easily sublimate and diffuse into the Bi precursor film and form Bi₄Te₃ under a high diffusion temperature and high gas pressure. At 523 K, poor Te diffusion leads to poor formation of Bi₄Te₃. With increasing the diffusion temperature to 548 K, Te sublimate and react with Bi to form Bi₄Te₃ more sufficiently. With further increasing the diffusion temperature to 573 K, Te fully sublimate with insufficient Bi₄Te₃ formation due to Te over-sublimation. When the diffusion temperature is as high as 648 K, the Bi precursor film and sublimated Te can fully react to form Bi₄Te₃. To render a more detailed semiquantitative analysis of the Te content, the chemical compositions of the Bi₄Te₃ thin films prepared at different diffusion temperatures are summarized in Table S1 (Supporting Information). As can be seen, due to insufficient Te diffusion at 523 K, the film is in lack of Te. With the diffusion temperature increased to over 548 K, the Te content increased. Optical images of the Te precursor films after thermal diffusion reaction are shown in Figure S1 (Supporting Information) which demonstrates Te remains under the diffusion temperature of 548 K and fully sublimate at 573 K. Figure 2d–f shows the scanning electron microscope (SEM) images of as-prepared Bi₄Te₃ flexible thin films which are prepared at the diffusion temperature of 573, 623, and 648 K, respectively. The average grain sizes of the Bi₄Te₃-based films prepared at 573, 623, and 648 K are ≈210, ≈285, and ≈310 nm, respectively (detailed size distribution was shown in Figure S2a–c, Supporting Information). The Bi₄Te₃-based flexible thin films are growing denser with increasing diffusion temperature. The increased grain size and density with increasing diffusion temperature should be attributed to faster grain growth rate and element diffusion rate at higher temperature.

To further characterize the formation of Te-embeddings, Figure 3a presents the XPS spectrum of Bi₄Te₃ thin film prepared at the diffusion temperature of 623 K. The binding energy corresponding to Bi and Te are clearly detected. Figure 3b shows the observed peaks at 583.18 and 572.88 eV, which can be mainly ascribed to the binding energy of Te 3d₃/₂ and Te 3d₅/₂, deriving from
Figure 1. a) Optical images of as-prepared Bi$_2$Te$_3$ flexible thin film under (a1) flat and (a2) bending statuses, respectively. b) The $\Delta R/R_0$ of the Bi$_2$Te$_3$ flexible thin film prepared under the diffusion temperature of 623 K as a function of bending radius at the bending cycles of 800 times. c) The $\Delta R/R_0$ of the Bi$_2$Te$_3$ flexible thin film prepared under the diffusion temperature of 623 K as a function bending cycles at the bending radius of 8 mm. d) Schematic structure of Bi$_2$Te$_3$ and Te. e) DFT calculated electronic band structures of Te and Bi$_2$Te$_3$, respectively. f) Comparison of the maximum room-temperature $S^2\sigma$ between our Bi$_2$Te$_3$-based flexible thin films and other state-of-art n-type Bi$_2$Te$_3$-based flexible thin films prepared by different methods, including thermal evaporation (2011),\textsuperscript{[35]} mechanically alloyed (MA) and dispenser printing (2012),\textsuperscript{[19]} screen print (2014),\textsuperscript{[34]} disperser printing (2017),\textsuperscript{[32]} solution phase printing (2019),\textsuperscript{[30]} vacuum filtration and annealing (2019),\textsuperscript{[31]} in situ solution (2019),\textsuperscript{[33]} magnetron sputtering (2020),\textsuperscript{[36]} and this work.
Te$^{2-}$ of Bi$_2$Te$_3$, respectively.$^{[36]}$ The deviation between the detected Te spectrum and the $\text{Te}^{2-}$ standard spectrum of Bi$_2$Te$_3$ is induced by Te$^{0}$ (blue area), which proves the existence of Te. The binding energy detected at 162.48 and 157.08 eV should be attributed to the Bi 4f$_{7/2}$ and Bi 4f$_{5/2}$ (Figure 3c), respectively, which is consistent with that of Bi$_2$Te$_3$. $^{[39]}$ Raman spectroscopy ranging from 90 to 210 cm$^{-1}$ is also employed to study the composition changes of the Bi$_2$Te$_3$ thin films prepared at the diffusion temperature range of 573–648 K as shown in Figure 3d. The peaks located at 103.0 and 134.3 cm$^{-1}$ are attributed to the E$_{2g}$ and A$_{1g}$ modes, respectively, which are in agreement with the rhombohedral structure of Bi$_2$Te$_3$. $^{[40]}$ The E$_{2g}$ (121.6 cm$^{-1}$) and A$_{1g}$ (140.2 cm$^{-1}$) peaks are assigned to the phonon mode of Te phase. $^{[40]}$ With increasing diffusion temperature, the E$_{2g}$ and A$_{1g}$ peaks gradually disappeared at the diffusion temperature of 648 K indicating disappeared Te. Figure 3e–i shows the low- and high-magnification SEM images with corresponding energy-dispersive spectrometer (EDS) maps and line scan spectra of the Bi$_2$Te$_3$ film prepared at the diffusion temperature of 623 K. As can be seen, the observed Te precipitate in the size of $\approx$400 nm.

To further characterize the crystal structure of as-prepared Bi$_2$Te$_3$ thin films, TEM image of a typical sample prepared...
under the diffusion temperature of 623 K is shown in Figure 4a, which clearly shows two typical grains of Te and Bi$_2$Te$_3$, respectively. Figure 4b shows the high-resolution TEM (HRTEM) image of the right part of Figure 4a, which can well-superimpose with the crystal structure of Te along $[\bar{2}21]$ zone axis. Corresponding HRTEM image of the left part of Figure 4a is presented in Figure 4c and can well-superimpose with the crystal structure of Bi$_2$Te$_3$ along [01$\bar{1}$0]. This indicates the left and right part of Figure 4a are highly crystallized Te and Bi$_2$Te$_3$, respectively. Figure 4d–f shows corresponding EDS maps. Te can be observed across the whole area. Bi can only be characterized on the left part. EDS line scan further clarified this point as shown in Figure 4g. This suggests the left part of Figure 4a is composed of both Bi and Te and should be Bi$_2$Te$_3$, and the right part of Figure 4a is composed of only Te (should be Te), which are consistent with the HRTEM analyses. In addition, Te-rich Bi$_2$Te$_3$ with dense precipitates can make the grain boundaries and interfaces dense, introduce additional dislocations and point defects, and correspondingly strengthen phonon scattering leading to improved thermoelectric performance. [20]
Room-temperature thermoelectric performance of as-prepared Bi$_2$Te$_3$ thin films is shown in Figure 5. Figure 5a shows the $\sigma$ of the Bi$_2$Te$_3$ thin films as a function of diffusion temperature, which increases with increasing the diffusion temperature. The $\sigma$ value increases with increasing the diffusion temperature, which approaches as high as 567.69 S cm$^{-1}$ at the diffusion temperature of 648 K. To understand the evolution of $\sigma$ with increasing diffusion temperature, Figure 5b presents the $n_e$-dependent $\mu$ of as-prepared Bi$_2$Te$_3$-based thin films in comparison with the single parabolic band (SPB) model calculated values. The calculation process of the SPB model is shown in Equations (S1)–(S5) of the Supporting Information. As can be seen, the $\mu$ dramatically increases while the $n_e$ stays around 2–4 x 10$^{19}$ cm$^{-3}$, which indicates the increasing $\mu$ is not induced by the reduced $n_e$. The $\mu$ approaches the maximum of ≈200 cm$^2$ V$^{-1}$ s$^{-1}$ at the diffusion temperature of ≈623 K. Figure 5c displays the calculated deformation potential coefficient ($E_{\text{def}}$) as a function of diffusion temperature. The $E_{\text{def}}$ decreases with increasing temperature and also minimizes at the diffusion temperature of ≈623 K, which is consistent with the maximized $\mu$ at the same diffusion temperature. This should be attributed to the Bi$_2$Te$_3$/Te heterostructure, which induced energy filtering effect and correspondingly increased carrier energy and $\mu$.$^{43,42}$

Figure 5d shows the $n_e$-dependent $|S|$ of as-prepared n-type Bi$_2$Te$_3$-based thin films in comparison with the SPB model calculated values. With increasing diffusion temperature, the $|S|$ increased from ≈140 $\mu$V K$^{-1}$ at the diffusion temperature of 523 K to ≈165 $\mu$V K$^{-1}$ at the diffusion temperature of 623 K. The high $|S|$ at 623 K is attributed to the strong energy filtering effect at the Bi$_2$Te$_3$/Te interfaces with the maximum element Te content. Further increasing the diffusion temperature to 648 K oppositely reduces the $|S|$ due to sufficiently reacted Te. The minor change of $S$ with the diffusion temperature lower than 623 K can be mainly attributed to the change of $n_e$ regardless of the slightly varying effective mass ($m^*$) ranging from 0.7 to 0.93 $m_0$. Figure 5e shows the $n_e$-dependent $S^2\sigma$ of as-prepared n-type Bi$_2$Te$_3$-based thin films in comparison with the SPB model calculated values, where the $S^2\sigma$ approaches as high as 14.58 $\mu$W cm$^{-1}$ K$^{-2}$ at the diffusion temperature of 623 K. This should be mainly ascribed to the energy filtering effect boosted $\mu$ while the $n_e$ is close to the optimized level. Figure 5f presents the repeatedly measured $\sigma$, $|S|$, and $S^2\sigma$ of Bi$_2$Te$_3$ flexible thin film prepared under the diffusion temperature of 623 K. As can be seen, thermoelectric performance of as-prepared Bi$_2$Te$_3$ flexible thin film remains closely consistent during the repeating measurement process, which indicates high stability of the film. The
evaluation of repeatability of $\sigma$, $S$, and $S^2\sigma$ of Bi$_2$Te$_3$ flexible thin film is further shown in Figure S3 (Supporting Information) also indicating high repeatability and stability in their thermoelectric performance. To demonstrate the applicability of our n-type Te-embedded Bi$_2$Te$_3$ flexible thin films, we have assembled a demonstrating sensor with corresponding p-type Sb$_2$Te$_3$ legs (thermoelectric performance is shown in Table S2, Supporting Information) as shown in Figure 5g. Touching the electrode with finger can induce a temperature difference between the center and the surrounding area as simulated in Figure 5h and correspondingly induce electrical signals. Continuous response of the thermoelectric sensor is measured for 7 cycles. The time interval between peak and valley of each pulse is $\approx 1$ s, which includes device response time and finger heat transfer time. The output signals of the thermoelectric sensor show a fast on-off response as shown in Figure 5i, where the state of finger touching is “on,” and the state of finger moving away is “off.”

3. Conclusion

In this study, we have successfully prepared n-type Bi$_2$Te$_3$ flexible thin films on flexible PI substrates by a novel thermal diffusion reaction method. Reaction control can lead to the formation of Te-embeddings and Te/Bi$_2$Te$_3$ heterostructures in the Bi$_2$Te$_3$ thin films, which theoretically can induce energy filtering effect due to the $\Delta E_g$ between Bi$_2$Te$_3$ and Te valence bands. Experimentally, the
reaction condition and formation of Te embeddings can be engineered through adjusting the diffusion temperature. An excellent $S/\sigma$ of 14.58 $\mu$W cm$^{-1}$ K$^{-2}$ has been achieved at room temperature due to Te-embeddings induced energy filtering effect and corresponding ultrahigh $\mu$ of $\approx$ 200 cm$^2$ V$^{-1}$ s$^{-1}$. Our prepared n-type Te-embedded Bi$_2$Te$_3$ flexible thin film possesses comparable thermoelectric performance comparing with state-of-art Bi$_2$Te$_3$-based flexible thin films. This result well proves that tuning the diffusion temperature during the thermal diffusion process can effectively introduce embeddings and Te/Bi$_2$Te$_3$-heterostructures and boost the thermoelectric performance of Bi$_2$Te$_3$ flexible thin films. The assembled thermoelectric sensor with fast response characteristics further proved the applicability of as-prepared Te-embedded Bi$_2$Te$_3$ thin films prepared by our thermal diffusion method.

4. Experimental Section

Bi$_2$Te$_3$ Thin Film Preparation: The n-type Bi$_2$Te$_3$ thin films were deposited on a flexible PI substrate using a thermal diffusion reaction method. First, Bi and Te thin films were deposited separately on PI substrates by using radiofrequency magnetron sputtering technique. The deposition parameters of the thin film are summarized in the Supporting Information. Then, as-deposited Te and Bi thin films were tightly pressurized, under the external pressure of $5 \times 10^4$ Pa in a vacuum $(2 \times 10^{-3}$ Pa) chamber followed by a heating process. The diffusion temperature was increased to the target temperature within 30 min and dwelled for 2 h, the samples were cooled down to room temperature under vacuum. The thickness of the thin films is ranging $\approx$550 nm and the measured results by using have been added in Table S1 (Supporting Information).

Device Fabrication: First, Bi, Sb, and Te precursor films were deposited on the PI substrates. Subsequently, the flexible thermoelectric prototype sensor was fabricated using the thermal diffusion reaction method.

Characterization of the Bi$_2$Te$_3$ Films: The crystal structures as-prepared Bi$_2$Te$_3$ films were characterized by X-ray diffraction (XRD, D/max 2500 Rigaku Corporation, CuKα radiation). The surface morphology and cross-section microstructures were investigated by SEM (Zeiss supra 55). The compositions of Bi$_2$Te$_3$ films are analyzed by an EDS (Bruker Quantax 200). Samples for cross-sectional TEM analysis were fabricated by a dual-beam SEM-FIB (focused ion beam, Scios, FEI). The crystal structures of as-prepared Bi$_2$Te$_3$-based flexible thin films were also investigated by the transmission electron microscope (TEF, FEI, Titan3 Themis G2). The chemical bonding was investigated by Raman spectra measurement system (Horiba Jobin Yvon). X-ray photoelectron spectroscopy (XPS, Thermo Escalab 250XI) was used to study the valence states of the composing elements. Carrier concentration ($n_e$) and mobility ($\mu$) values were recorded from the Hall measurement system (HLS500PC, Nano metrics). The $\sigma$ and $S$ of Bi$_2$Te$_3$ film were simultaneously measured by the SBA458 (Nezsch). The error bars of the measured data were determined from the SBA458 (5% for $S$, 5% for $\sigma$, and 10% $S^2/\sigma$).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

D.-W.A., W.-D.L., and Y.-X.C. contributed equally to this work. D.-W.A.: Investigation, Conceptualization, Methodology, Writing – original draft. W.-D.L.: Investigation, Writing – review & editing, Y.-X.C.: Writing – review & editing. M.W.: Investigation, Conceptualization, Methodology. B.J.: Investigation, Data curation, Validation. F.L.: Data curation, Validation. X.-L.S.: Data curation, Validation. Z.-H.Z.: Resources, Writing – review & editing, Supervision, Funding acquisition. G.L.: Resources, Writing – review & editing. X.-Z.C.: Supervision, P.F.: Resources, Writing – review & editing, Funding acquisition. Z.-G.C.: Supervision, Writing – review & editing.

Data Availability Statement

Research data are not shared.

Keywords

Bi$_2$Te$_3$, thermal diffusion method, thermoelectrics, flexible thin films

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