



## A novel n-type hybrid composite with a large Seebeck coefficient around room temperature based on poly(3,4-ethylene dioxydiphenylene) and titanium disulfide

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A great effort is given to make flexible thermoelectric modules to convert thermal energy to electric energy, recently. A thermoelectric module is fabricated by connecting several p- and n-type thermoelectric material couples. Although air stable p-type polymeric thermoelectric materials made progress in performance and stability, n-type polymeric materials are still confronted with significant challenges for achieving efficient and stable thermoelectric material.

An efficient n-type thermoelectric material was simply produced in this study by mixing the  $\text{TiS}_2$  with PEDOT:PSSA dispersion and then ultra-sonicating the mixture in an ice bath. The highest power factor of  $1516 \mu\text{W m}^{-1} \text{K}^{-2}$  was achieved and the highest figure of merit,  $Z_T$  of 1.14 was estimated at 300 K by assuming  $0.4 \text{ W m}^{-1} \text{K}^{-1}$  of the thermal conductivity of the composite containing 20% of  $\text{TiS}_2$ . To the best of our knowledge, this power factor is the highest among the n-type polymeric thermoelectric materials reported in the literature.

Keywords: Thermoelectric, PEDOT:PSS,  $\text{TiS}_2$  composite, Seebeck coefficient.

### Introduction

Thermoelectric (TE) generators can directly produce electricity by harvesting thermal energy from solar heat, radioactive decay, body heat, thermal springs and industrial waste heat, etc.<sup>1,2</sup>. TE generators have many advantages such as simple in structure, small size, easy maintenance, quiet operation and no pollutants. The efficiency of a TE material is determined by the dimensionless thermoelectric figure of merit ( $Z_T$ ) defined as;  $Z_T = S^2\sigma T/\kappa$  where  $S$ ,  $\sigma$  and  $\kappa$  are Seebeck coefficient, electrical and thermal conductivity of the material, respectively, at the absolute working temperature,  $T$ . The higher  $Z_T$  means the higher efficiency. The greatest difficulty to enhance  $Z_T$  is that the three parameters are interdependent, i.e.  $\kappa$  increases but  $S$  decreases with increase of  $\sigma$  of the material. A value of  $Z_T \geq 1$  is required to make an efficient commercial TE device. The state-of-the-art inorganic TE materials which are alloys such as bismuth-telluride and lead-telluride cannot be used widely for large-scale applications because of their toxicity, brittleness and scarcity in nature.

On the other hand the conductive polymers which are promising candidates as a TE material<sup>3,4</sup> have benefits such as abundance, non-toxicity, flexibility, low cost of processing, low thermal conductivity and suitability for printing or roll-up processing, which are expected to make up for the shortages of inorganic TE materials. Recent research has been focused on the development of new polymeric materials with a high power factor ( $\text{PF} = S^2\sigma$ ) instead of  $Z_T$  since the magnitude of thermal conductivities of the polymers are intrinsically low, and close to each other and do not change significantly with inorganic additives. Conductive polymers can operate properly around room temperature while their use at higher temperatures is hindered due to their low stability. Therefore the values of  $\sigma$  and  $S$  of conjugated polymers need to be significantly enhanced to meet the requirements in such applications. To achieve more efficiency, the TE generator modules must include pairs of p- and n-type material. However conventional n-type organic or polymeric semiconductors are far behind their p-type counterparts<sup>5</sup>. The reported PF of the polymers has reached to  $469 \mu\text{W m}^{-1} \text{K}^{-2}$  for p-type<sup>6</sup> and to  $28 \mu\text{W m}^{-1} \text{K}^{-2}$  for n-type<sup>7</sup> so far.

Hybridization of conductive polymers with inorganic TE particles offers a simple way to prepare flexible p- and n-type polymeric TE materials having low thermal conductivity. Recently, there have been notable studies on composite thermoelectric materials<sup>3</sup>. It was proposed that the PF can be enhanced in the hybrid materials through mechanisms like energy filtering<sup>8</sup> or modulation doping<sup>9</sup>. It is required to choose the appropriate particle sizes to benefit from the mean free paths of electron and phonon since the particle network can also conduct heat together with the electric conduction. Bi<sub>2</sub>Te<sub>3</sub> ( $Z_T = 0.97$ ) is inorganic TE material generally recognized to exhibit the highest  $Z_T$  near room temperature. Related to the interest in possible quantized effects on the S some nano- and micro-sized particles of Bi<sub>2</sub>Te<sub>3</sub> have been hybridized with conducting polymers<sup>10</sup>. The smallest particles are reported to enhance the S whereas maintain or decrease the  $\sigma$ , depending on the mixing condition. In the energy filtering scheme, low energy carriers are scattered at the inorganic/organic interfaces, and consequently enhancing the S<sup>9</sup>. It is demonstrated that the careful control and utilization of the interface can be a useful route to make enhancement of the thermoelectric properties in the hybrid materials. This emphasize the importance of controlling (or tuning) the interface for electric conduction. It appears that an easy and convenient pathway to prepare stable n-type polymeric TE materials is to introduce n-type inorganic nanoparticles into the polymer.

Titanium disulfide (TiS<sub>2</sub>) is a layered compound which exhibits n-type conductivity<sup>11</sup> Imai and co-workers have reported a large S value for TiS<sub>2</sub> ( $S \approx -251 \mu\text{V/K}$ ) at 300 K; and somewhat low and metallic-like conductivity ( $\sigma \approx 588 \text{ S/cm}$ ) at 300 K<sup>12</sup>. Thus, TiS<sub>2</sub> has considerably high PF  $\sim 3700 \mu\text{W m}^{-1} \text{ K}^{-2}$  which is close to that of the current Bi<sub>2</sub>Te<sub>3</sub> materials (PF  $\sim 4000 \mu\text{W m}^{-1} \text{ K}^{-2}$ ). Despite the large PF, the  $Z_T$  of TiS<sub>2</sub> is 0.16 at 300 K, because of considerably large thermal conductivity,  $6.8 \text{ W/m}^{-1} \text{ K}^{-1}$ . Wan and co-workers synthesized n-type flexible thermoelectric materials via electrochemical intercalation to fabricate a hybrid superlattice of alternating inorganic TiS<sub>2</sub> monolayers and organic cations<sup>13</sup>. n-Type carriers for energy transport were provided by externally injected electrons into the inorganic layers of TiS<sub>2</sub> and then stabilized by organic cations. The electrical conductivity of  $790 \text{ S cm}^{-1}$  and PF of  $450 \mu\text{W m}^{-1} \text{ K}^{-2}$  and  $Z_T$  of 0.28 at 373 K were obtained for a hybrid superlattice of TiS<sub>2</sub>/[(hexylammonium)<sub>x</sub> (H<sub>2</sub>O)<sub>y</sub> (DMSO)<sub>z</sub>], with an in-plane lat-

tice  $\kappa$  of  $0.12 \pm 0.03 \text{ W m}^{-1} \text{ K}^{-2}$ , which is smaller fifty times of magnitude than those of the single-layer and bulk TiS<sub>2</sub>. Later, Wan and co-workers were able to achieve a remarkably high PF of  $904 \mu\text{W m}^{-1} \text{ K}^{-2}$  which is very high among the n-type flexible hybrid thermoelectric materials by electrochemically intercalating two kinds of organic materials with different boiling points into the van der Waals gap between the inorganic layers of TiS<sub>2</sub><sup>14</sup>.

Tian and co-workers presented exfoliation-and-reassembly approach to produce a flexible n-type TiS<sub>2</sub>/organic hybrid film for low-temperature TE applications<sup>15</sup>. The obtained film shows a superlattice structure with alternative layers of TiS<sub>2</sub> and organic molecules. Charge transfer occurs when TiS<sub>2</sub> and organic molecules form intercalation complexes, which gives rise to a high  $\sigma$  but a low S. Annealing resulted in a PF of  $210 \mu\text{W m}^{-1} \text{ K}^{-2}$  at room temperature.

The highest PF of  $\sim 1500 \mu\text{W m}^{-1} \text{ K}^{-2}$  was reported for the polymer based n-type materials by Zhou and co-workers in 2017<sup>16</sup>. They prepared nanocomposites by drop casting the solution of polyethyleneimine in ethyl alcohol onto the n-type as-grown single walled carbon nanotube continuous networks. The produced composites films were flexible and stable in air for 3 months.

The highest PF value for the p-type composite materials up to the present was reported by our group last year<sup>17</sup>. We achieved the largest S of  $7000 \text{ mV K}^{-1}$  and the highest PF of  $2646 \mu\text{W m}^{-1} \text{ K}^{-2}$  for the composite containing 20% of Cu<sub>1.8</sub> in PEDOT:PSSA.

In this study, we aimed to prepare a flexible n-type polymer based hybrid TE composite material by mixing n-type inorganic TiS<sub>2</sub> with PEDOT:PSSA. We achieved highest PF among the n-type polymeric composites reported up to the present in the literature.

## Experimental

3,4-Ethylene dioxythiophene (EDOT), poly(4-styrene sulfonic acid) (PSSA), hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), copper nitrate hydrate (Cu(NO<sub>3</sub>)<sub>2</sub>·H<sub>2</sub>O) and TiS<sub>2</sub> were purchased as analytical reagent grade from Sigma-Aldrich Co. and used without further purification.

A known amount of EDOT was added into a known amount of PSSA (30%) solution in small amount deionized water and the mixture was stirred vigorously for 1 h at room temperature. Then, a known amount of H<sub>2</sub>O<sub>2</sub> was added drop

by drop into the mixture as oxidation agent. The mole ratios of the reagents were kept constant throughout the study. Finally, a small piece of crystal  $\text{Cu}(\text{NO}_3)_2 \cdot 8\text{H}_2\text{O}$  was put into the prepared mixture. After the reaction mixture was stirred vigorously for 24 h at room temperature, PEDOT:PSSA was obtained as a dark blue solution. The composites were prepared by mixing of PEDOT:PSSA with  $\text{TiS}_2$  and then ultra-sonication.

The glass substrates were cleaned in the concentrated sulfuric acid solution then successively transferred to water, pure water and acetone. The films of PEDOT:PSSA and/or composites were prepared by drop casting on the cleaned glass substrate. The composites were prepared in various proportions of PEDOT:PSSA and  $\text{TiS}_2$  powder by either non-sonicating (just mixing) or sonicating with a ultra-sonicator homogenizer. Thin films on the glass substrates were then dried at  $60^\circ\text{C}$  for 1 h after drying at ambient conditions for 1 day.

The values of  $\sigma$  and  $S$  of the samples were obtained using FPP470 model four point probe and 001 model Seebeck coefficient measuring device produced by Entek-Turkey, respectively. X-Ray diffraction (XRD) analysis of the powder samples was conducted with a diffractometer (D/max-2200/PC, Rigaku Corp., Tokyo, Japan) equipped with a high-intensity  $\text{Cu K}\alpha$  radiation ( $\lambda = 105418 \text{ \AA}$ ). The FTIR measurements were carried out on a Thermo Fisher Scientific Nicolet IS10 FTIR-ATR spectrometer. The binding energy of 2p electrons of sulfur atoms on the composite surface was examined using Thermo Scientific K-Alpha X-ray Photoelectron spectrometer. The morphology of the samples were characterized using LS10 model of Zeiss EVO scanning electron microscope with an accelerating voltage of 7 kV.

## Results and discussion

The measured  $S$  and  $\sigma$  values of thin films of pristine PEDOT:PSSA and tablet of pristine  $\text{TiS}_2$  as well as thin film of non-sonicated composites are presented in Table 1. It can be seen that PEDOT:PSSA is p-type material with considerably large  $S$  but low conductivity. The PF of  $103 \mu\text{W m}^{-1} \text{K}^{-2}$  was found for pristine PEDOT:PSSA. However,  $\text{TiS}_2$  is an n-type material with large  $S$  ( $-250 \text{ mV K}^{-1}$  and high conductivity ( $580 \text{ S cm}^{-1}$ ) in agreement with the reported values in the literature<sup>11,12</sup>. In the composites, the values of  $S$  (with positive sign) and  $\sigma$  increased and PF reached to  $461 \mu\text{W m}^{-1} \text{K}^{-2}$  at  $\text{TiS}_2$  concentration of 5%. The composites main-

**Table 1.** Thermoelectric parameters of thin film samples prepared from non-sonicated  $\text{TiS}_2$ /PEDOT:PSSA composites and tablet sample prepared from  $\text{TiS}_2$  as received

$\text{TiS}_2$ % in the composite <sup>a</sup>	Seebeck coefficient ( $\mu\text{V/C}$ )	Electric conductivity $\sigma$ ( $\text{S/cm}$ ) <sup>b</sup>	Power factor PF ( $\mu\text{W/mK}^2$ )	$Z_T$ estimated at 300 K <sup>b</sup>
0	1087	0.87	103	0.08
5	1200	3.2	461	0.34
10	220	18	87	0.065
15	1000	0.7	70	0.05
20	-2400	0.53	305	0.23
25	-467	3	65	0.05
30	-630	3	119	0.09
100 (tablet)	-250	580	3625	0.16

<sup>a</sup>weight percent. <sup>b</sup> $\kappa \sim 0.4 \text{ W m}^{-1} \text{K}^{-1}$  for pristine PEDOT:PSSA and its composites however  $\kappa \sim 6.8 \text{ W m}^{-1} \text{K}^{-1}$  for  $\text{TiS}_2$  tablet were used in the calculation of estimated  $Z_T$  at 300 K.

tained the p-type conductivity up to  $\text{TiS}_2$  concentration reached to 20% by weight. The sign of  $S$  changed to negative at this concentration indicating the conversion of n-type conductivity.  $S$  of  $-2400 \text{ mV m}^{-1} \text{K}^{-2}$  was measured without a significant decrease in electrical conductivity. PF of  $305 \mu\text{W m}^{-1} \text{K}^{-2}$  was calculated at this concentration. The change of the sign of the  $S$  is a common phenomenon with the change of the proportion of two components of the composites<sup>5</sup>. As  $\text{TiS}_2$  amount increase, the composites maintained the n-type conductivity although absolute values of the  $S$  decrease. Using another batch of the PEDOT:PSSA, the composite samples were ultra-sonicated for 30 min in an ice bath in order to homogenize them. The results of the thin films of the ultra-sonicated composites were given in Table 2. The  $S$  of  $+2400 \text{ mV K}^{-1}$  was measured for the pristine PEDOT:PSSA. Its  $Z_T$  value was estimated as 0.26 by assuming the  $\kappa$  is  $0.4 \text{ W m}^{-1} \text{K}^{-1}$  which is a maximum value of the measured  $\kappa$  values for PEDOT:PSSA in the literature<sup>3</sup>. The  $S$  of  $+2300 \text{ mV K}^{-1}$  was measured for the composite containing 5%  $\text{TiS}_2$ . The sign of  $S$  turns negative when the concentration of  $\text{TiS}_2$  becomes 10%. Then, both  $\sigma$  and absolute magnitude of  $S$  increase as the concentration of  $\text{TiS}_2$  increase. The inverse dependence between  $S$  and  $\sigma$  appears to be disrupted. The maximum  $S$  of  $-1080 \text{ mV K}^{-1}$  was measured when the  $\text{TiS}_2$  concentration reaches to 20%. The highest PF of  $1516 \mu\text{W m}^{-1} \text{K}^{-2}$  was calculated for the composite containing  $\text{TiS}_2$  in 20%. The highest  $Z_T$  of 1.14 was estimated for this composite by assuming  $\kappa$  equals to  $0.4 \text{ W}$

**Table 2.** Thermoelectric parameters of thin film samples prepared from TiS<sub>2</sub>/PEDOT:PSSA composites homogenized ultrasonically

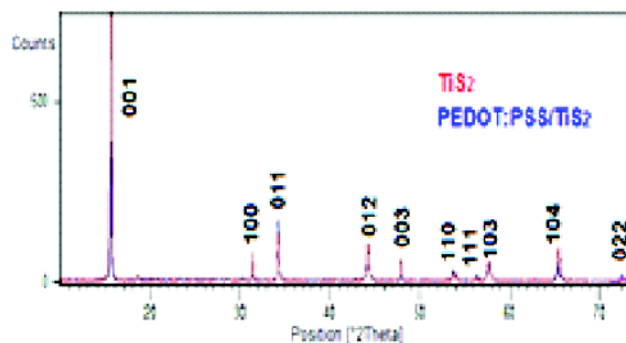
TiS <sub>2</sub> % in the composite <sup>a</sup>	Seebeck coefficient S (μV/C)	Electric conductivity σ (S/cm) <sup>b</sup>	Power factor PF (μW/mK <sup>2</sup> )	Z <sub>T</sub> estimated at 300 K <sup>b</sup>
0	2400	0.6	346	0.26
5	2300	0.7	370	0.28
10	-296	1.0	9	0.007
15	-312	1.5	15	0.011
20	-1080	13	1516	1.14
30	-380	16	231	0.17
40	-294	23	199	0.15
50	-286	28	229	0.17
60	-345	34	405	0.30
70	-280	4.3	34	0.025
80	-243	6	35	0.027

<sup>a</sup>weight percent. <sup>b</sup> $\kappa \sim 0.4 \text{ W m}^{-1} \text{ K}^{-1}$  for pristine PEDOT:PSSA and its composites was used in the calculation of estimated Z<sub>T</sub> at 300 K.

m<sup>-1</sup> K<sup>-1</sup>. The estimated highest Z<sub>T</sub> (1.14) achieved in this study is higher than that of Bi<sub>2</sub>Te<sub>3</sub> which is the most efficient state-of-the art inorganic thermoelectric material at ambient conditions. The σ values of the composites continues to increase but S starts to decrease as TiS<sub>2</sub> concentration increase when the concentration of TiS<sub>2</sub> continues to increase. This suggests that the optimum concentration of TiS<sub>2</sub> should be 20% for the maximum performance. It is noteworthy to say that magnitudes of S and σ do not change significantly

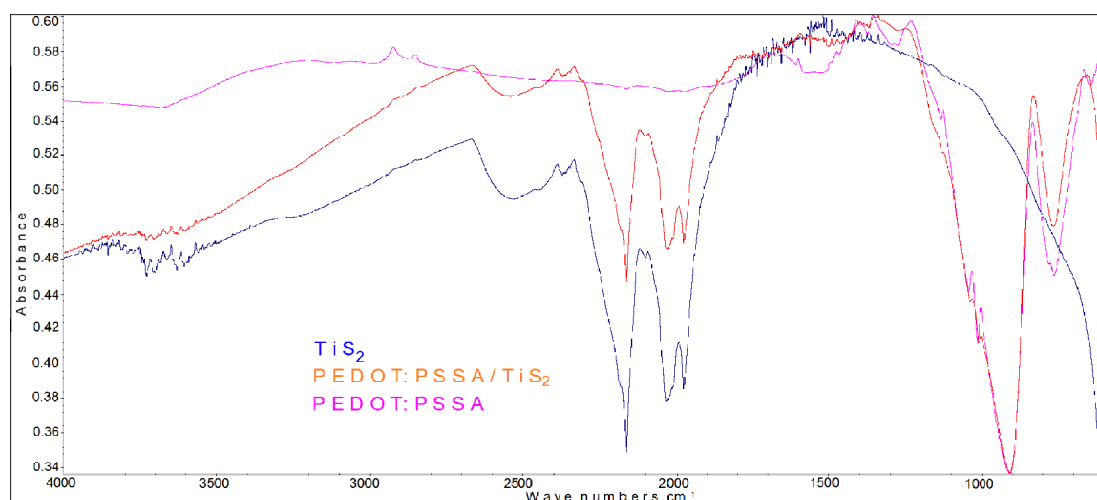
for one month.

X-Ray diffraction (XRD) spectra of the TiS<sub>2</sub> powder as-received is shown in Fig. 1 together its PEDOT:PSSA composite. The peaks at 15.64°, 31.49°, 34.26°, 44.18°, 47.96°, 53.85°, 57.74°, 65.54°, 72.10° for TiS<sub>2</sub> correspond to the planes 001, 011, 012, 003, 110, 111, 103, 104, 022 which agree with the previous reported results<sup>18,19</sup>. These diffraction peaks can be indexed in the hexagonal TiS<sub>2</sub> phase (Space Group, S.G. *P-3m1*). It can be seen in Fig. 1 that no diffraction peak is appeared belonging to the PEDOT:PSSA in the composite. It can be seen also that TiS<sub>2</sub> does not include any impurity in crystalline state.



**Fig. 1.** X-Ray powder diffraction patterns of TiS<sub>2</sub> as-received (the red line) and its composite with PEDOT:PSSA (blue line).

The FTIR spectra of PEDOT:PSSA, TiS<sub>2</sub> and their composite are given in the Fig. 2. The bands of the PEDOT:PSSA



**Fig. 2.** FTIR spectra of TiS<sub>2</sub> as-received (the blue line), PEDOT:PSSA (red line) and the composite (orange line).

are as follows: 1650, 1465, 1355, 1231, 1124, 832, 664  $\text{cm}^{-1}$ . The broad band centered at 1650  $\text{cm}^{-1}$  and 1465  $\text{cm}^{-1}$  were assigned as the C=C stretching of quinoid and benzenoid rings of PEDOT, respectively. The weak peak appearing at around 1355  $\text{cm}^{-1}$  was assigned to inter-ring stretching vibration of C-C of the PEDOT. The bands at around 1231, 1124  $\text{cm}^{-1}$  can be attributed to the C-O-C bending vibration of ethylene dioxy group of the EDOT. The bands at 832  $\text{cm}^{-1}$  and 664  $\text{cm}^{-1}$  are stretching vibrations of C-S-C group in the thiophene group<sup>20</sup>. It can be seen in the FTIR spectrum of the composite that the C=C stretching band of PEDOT was blue shifted from 1650 to 1780  $\text{cm}^{-1}$ . This can be attributed to the increase of effective  $\pi$ -electron delocalization due to linear conformation of the quinoid ring of PEDOT<sup>21</sup>. It can be speculated that PEDOT chains becomes more linear by intercalation into the van der Waals gap between the S-Ti-S layers of  $\text{TiS}_2$ .

We performed x-ray photoelectron spectroscopy (XPS) measurements for the composite (Fig. 3). The binding energy values were determined by the curve fitting method from the raw data. The peaks for the S2p spectra can be fitted by two component peaks corresponding to the sulfur atom in PEDOT (centered at 165.0 eV and 163.8 eV) and the three component peaks corresponding to the sulfur atom in PSS (centered at 169.9 eV and 169.1 eV and 168.1 eV)<sup>22</sup> and the two component peaks corresponding to the sulfur atom in

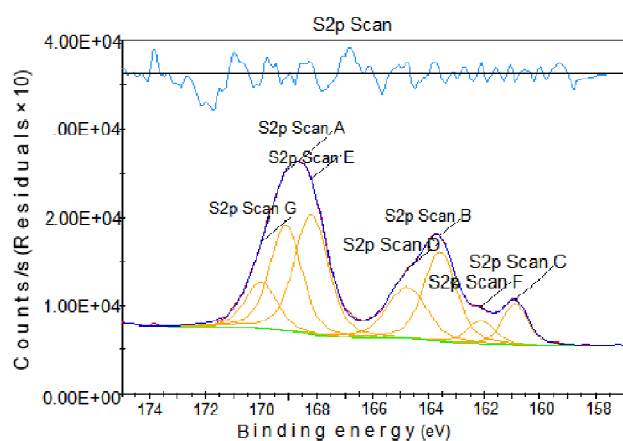


Fig. 3. XPS spectra of the composite of  $\text{TiS}_2$  and PEDOT:PSSA.

$\text{TiS}_2$  (162.2 eV and 160.8 eV). The two non-equivalent states with different binding energies of sulfur S2p spectra is shifted

toward higher energies compared to values given in the literature at 161.8 eV and 160.6 eV<sup>23</sup>. This shift may be caused by a change of the  $\text{TiS}_2$  bond angle due to intercalation of positively charged PEDOT molecules between the interlayered sections of  $\text{TiS}_2$ .

The layered structure of  $\text{TiS}_2$  as received can be seen in the Fig. 4. It can be stated that the deviations of S and  $\sigma$  values of the composites might be resulted from the anisotropic layered structure of  $\text{TiS}_2$ .



Fig. 4. SEM image of the  $\text{TiS}_2$  as-received.

## Conclusions

In summary, high performance and stable n-type polymer based thermoelectric materials can be simply prepared by introduction of  $\text{TiS}_2$  into the PEDOT:PSSA dispersions. The maximum PF of 1516  $\mu\text{W m}^{-1} \text{K}^{-2}$  was obtained for the composite containing 20% of  $\text{TiS}_2$ . To the best of our knowledge, this is the highest PF reported at room temperature so far in the literature. Also its estimated  $Z_T$  value is higher than that of  $\text{Bi}_2\text{Te}_3$  which has the highest performance TE material used in commercial modules. Therefore,  $\text{TiS}_2/\text{PEDOT:PSSA}$  composites can be used to fabricate safer, cheaper, flexible, stable and efficient polymeric TE modules. We are conducting further studies on fabricating a prototype TE module with p-type and n-type materials which we produced.

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