Enhancement of Thermoelectric Properties of Bismuth Telluride Composite with Gold Nano-Particles Inclusions Using Electrochemical Co-Deposition

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In order to enhance the figure of merit ZT, gold nano-particles-bismuth telluride composites have been synthesized using the electrochemical co-deposition and a significant improvement of thermoelectric properties has been achieved. The composite with 5 wt% of 5 nm-diameter gold nano-particles shows highest absolute Seebeck coefficient (~380 μV/K), low thermal conductivity (~0.5 W/m·K) and high figure of merit ZT (~0.62) at room temperature. This ZT value is approximately forty times better than that of the as-deposited pure bismuth telluride film synthesized using the electrochemical deposition with the similar condition.

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Among thermoelectric materials, bismuth telluride (Bi2Te3) always keep the attraction due to its highest performances for thermoelectric energy conversion applications near room temperature.1-5 The efficiency of thermoelectric material can be evaluated by the figure of merit ZT. The figure of merit ZT of thermoelectric materials is defined as $\alpha^2T/\rho k$, where $\alpha$ is the Seebeck coefficient, $\rho$ is the electrical resistivity and $k$ is the thermal conductivity at the temperature $T$. There have been reports to synthesize pristine Bi2Te3 films using the electrochemical deposition with high figure of merit ZT. However, the ZT values of electrodeposited Bi2Te3 films are generally lower than those of the bulk nature. The maximum ZT value of approximately 0.1 is obtained in electrodeposited Bi2Te3 by Manzano et al.6 Improved ZT value of roughly 0.2 is found in the electrodeposited Bi2Te3 film by optimizing the ratio of off-on cycles in the pulsed electrodeposition technique, suggesting that deposition conditions can improve the performances of electrodeposited Bi2Te3.7 Nano-inclusion is another promising approach to obtain thermoelectric materials with high ZT values. Recent studies on thermoelectric materials with nanostructures have provided strong experimental evidences that it is possible to improve performances of $\alpha$, $\rho$, and $k$, and thus high ZT values are achieved.8-22 However, the detailed mechanisms governing these improvements remain challenges to be further researched. These mechanisms usually depend on the size of nanostructures and the manufacturing method to prepare the relevant nanostructures. One of general mechanisms utilizes a quantum effect for the control of electron transport based on low dimensional nanostructures.23,24 Another mechanism utilizes phonon scattering due to nanostructures such as nanocomposites with nano-particles in the matrix of the bulk host materials.3,13,16,18 When nano-particles with diameters of approximately several nanometers are embedded in the host thermoelectric materials, it is expected that not only thermoelectric properties but also thermal conductivity is changed due to the phonon-edge scattering behavior.24,25 Although the nanocomposite structure has been proved to improve performances of Bi2Te3, the usage of the electrochemical co-deposition to synthesize Bi2Te3 nanocomposite is rare.26-27 Non-metallic nano-particles are usually candidates for electrochemically deposited Bi2Te3 nanocomposites. In the research of Wang et al., nano-silicon carbide (SiC) particles are included in Bi2Te3 to form the electrodeposited SiC-Bi2Te3 nanocomposite film.28 However, the improvement of figure of merit ZT is not provided. The enhancement of the electrical conductivity property is observed in multi-walled carbon nanotubes-Bi2Te3 composite film formed by the electrochemical deposition method.27 However, the improvements of Seebeck coefficient and thermal conductivity are not fully indicated in the research. Other methods including solvothermal deposition28 and spark plasma sintering29 have been widely used to synthesize metallic nano-particles-Bi2Te3 composites. However, these methods have limitations due to process complexity, expensive equipment and toxic precursors. Gold (Au) and silver (Ag) nano-particles-Bi2Te3 composites synthesized using the chemical solution-based bottom-up method and subsequent spark plasma sintering at low temperature have been proposed in Refs. 30, 31. Both Au and Ag nano-particles-Bi2Te3 composites show significant improvements of figure of merit ZT at 450K. However, the Au and Ag nano-particles in these composites are large with the particle diameters of approximately 20 nm due to limitations of synthesis methods. In the research of Sie et al., copper (Cu) nano-particles are embedded in Bi2Te3 matrix using Cu-based acetate decomposition and spark plasma sintering method. Similarly, by embedding Cu nano-particles with a diameter of 50–100 nm in the Bi2Te3 matrix, higher Seebeck coefficient and lower thermal conductivity than those of pure Bi2Te3 can be observed.32

In this work, we proposed a new approach of electrochemical co-deposition to synthesize Au nano-particles bismuth telluride composite. In the electrochemical deposition method, Au is known as one of the best seed layers for growing Bi2Te3. Therefore, Au nano-particles are promising candidates to be co-deposited in the Bi2Te3 matrix due to their good adhesion with Bi2Te3 and an inert nature in the electrolyte. Moreover, Au as a nanodot in Bi2Te3 matrix is chosen because its work function (Φ) is approximately 5.31–5.47 eV.30 This value is suitable for the electron affinity (χ ~ 4.5 eV) and the work function of Bi2Te3 (Φ ~ 5.3 eV).30 As a result, in this work, Au nano-particles with a diameter of approximately 5 nm are successfully embedded in the Bi2Te3 matrix to form the nanocomposite with a high ZT value.

Experimental

The depositions of the pure Bi2Te3 film and Au nano-particles-Bi2Te3 composite films are performed using an electrochemical method at room temperature under a stirring speed of 300 rpm. The electrolyte solution for the pure Bi2Te3 deposition consists of 4 mM Bi³⁺, 3.6 mM HTeO₂⁺ and 1 M HNO₃ solutions. Meanwhile, the compositions of 4 mM Bi³⁺, 3.6 mM HTeO₂⁺, 1 M HNO₃, and 5 nm-diameter gold nano-particles dispersed in PVP (Polyvinylpyrrolidone) are prepared for the electrolyte of the composite. The solution is prepared from the following steps. At first, both Bi₂O₃ and TeO₂ are dissolved in nitric acid to obtain the electrolyte with a pH = 0. Subsequently, various volumes of gold nano-particles dispersed in PVP solution are added to the electrolyte.

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The deposition is conducted on the 300 μm-thick silicon substrate coated with Cr-Au seed thin films with thicknesses of 10 nm and 150 nm, respectively. A three-electrode electrochemical deposition system is utilized as follows; the Cr-Au coated silicon substrate is used as a working electrode, a Bi₂Te₃ rod is used as a counter electrode to stabilize the electrolyte concentration, and Ag-AgCl with a 3 M KCl (Potassium Chloride) solution is used as a reference electrode. According to our previous work, a DC voltage of 20 mV for the working electrode is found to be suitable to synthesize a pure Bi₂Te₃. Therefore, this condition is used for the syntheses of composites in this work.

To evaluate the Seebeck coefficient and electrical resistivity, the deposited films are transferred into epoxy resin substrates to avoid the short-circuiting behavior via the seed substrates. The in-plane Seebeck coefficient of the deposited films is measured at room temperature where the temperature difference is imposed along in-plane direction of the films, and the Seebeck coefficient is obtained from the generated voltage at two points. A four terminal method is used to measure the electrical resistivity. Energy Dispersive X-ray spectroscopy (EDX) is used to evaluate the compositions of the deposited films. Structural identification of the deposited films is performed with an X-Ray Diffractometer (XRD) using Cu Kα radiation (λ = 1.5418 Å, 40 kV, 40 mA, step size 0.02°, 2 s/step, sample rotation 60 rpm), Transmission Electron Microscopy (TEM) and SAED (Selected Area Electron Diffraction) patterns are used to evaluate the nanostructures of the samples. The thermal conductivity is measured using a cross-plane thermal conductivity measurement system based on a differential 3ω method. A thin Cr-Au pattern is deposited on the top of the sample consisting of 300 μm-thick Si substrate, 200 nm-thick Cr-Au seed layer, 2 μm-thick electrochemically deposited thermoelectric films and 200 nm-thick SiO₂ film, as shown in Fig. 1. The Cr-Au pattern works as both a heater and a thermometer. An alternating current with the frequency ω is applied through the metallic pattern. Due to the Joule heating behavior, the current induces the temperature oscillation ΔT(ω). Using the differential 3ω method, the thermal conductivity of the thin film can be measured by subtracting temperature oscillations of the sample consisting of the thin thermoelectric film and the sample without the thin thermoelectric film.

**Results and Discussion**

Figure 2 shows the effect of the inclusion of the Au nano-particles on the surface morphology of as-deposited films. The pure Bi₂Te₃ sample exhibits a standing plate-like shape with large grains sizes of approximately 1 μm, as shown in Fig. 2a. When the Au nano-particles are embedded, the surface grain size decreases down to ~500 nm and the morphology changes to a granular structure (Fig. 2b). Obviously, the inclusion of Au nano-particles has a significant effect on the crystal morphology of the composite material. In addition, the deposited films are analyzed by TEM (Transmission Electron Microscopy). Figures 3a and 3b show the SAED (Selected Area Electron Microscopy) and the HRTEM (High Resolution Transmission Electron Microscopy) patterns of cross section of the pure Bi₂Te₃ film, respectively. The diffraction spots of SAED pattern and the TEM image show that this film has polycrystalline structures with relatively large grains. In contrast, the SAED pattern of the composite reveals that the sample basically consists of randomly-oriented fine grain structures, as shown in Fig. 3c. According to the HRTEM image shown in Fig. 3d, the embedded gold nano-particles with diameters of approximately 5 nm are observed in the film.

Figure 4a shows the relationship between the weight percentage of embedded Au nano-particles and the Seebeck coefficient of the composites. It is shown that the Seebeck coefficient is significantly enhanced by the inclusion of the nano-particles while the electrical resistivity increases with the enhancement of the concentration of the nano-particles. From the results, the composite sample with 5 wt% of Au nano-particles is paid attention due to the highest power factor value, as shown in Table I. The average weight percentage of Au is evaluated using the Energy Dispersive X-ray spectroscopy (EDX) technique. Additionally, under the EDX and XRD analyses, oxidized Au nano-particles are not observed in the composite, as shown in Fig. 5. In detail, the composite with 5 wt% of Au nano-particles shows...
Figure 4. (a) Dependence of the weight percentage of the gold nano-particles on the Seebeck coefficient; (b) Annealing temperature dependences on the Seebeck coefficients of the composite with 5 wt% of the gold nano-particles and pure Bi$_2$Te$_3$; (c) Annealing temperature dependence on the Seebeck coefficient and the electrical resistivity of the composite with 5 wt% of the gold nano-particles.

A high Seebeck coefficient of approximately $-380 \mu$V/K, which is approximately seven times larger than that of the pure Bi$_2$Te$_3$, as shown in Fig. 4a. This is the highest Seebeck coefficient value of bismuth telluride synthesized using an electrochemical deposition to date.

According to previous studies, the band bending behavior at the interfaces of thermoelectric semiconductors and metals can significantly enhance the Seebeck coefficients of thermoelectric materials. The nano-particles are included in the mother phase of the semiconductor, the charge transfers between the metallic nano-particles and the mother phases are formed. The charge transfer behavior results in band bending at the metal-semiconductor interface, characterized by an electrical potential variation. This behavior forms the energy-dependent barrier that acts as a filter to scatter low-energy electrons. Only high-energy electrons can pass over the energy-dependent barrier while low-energy electrons are scattered at the interface’s potential barrier. If the work function (\( \Phi \)) of metal is higher than that of Bi$_2$Te$_3$, the potential barrier of approximately 0.1 eV has been proved to be optimal to improve the Seebeck coefficient of Bi$_2$Te$_3$. Because \( \Phi \) of Au and Bi$_2$Te$_3$ are approximately 5.31–5.47 eV and 5.3 eV, respectively, the gold nano-particles are among ideal materials to improve the Seebeck coefficient of Bi$_2$Te$_3$ in the form of composites.

The band bending can be roughly estimated as following:

\[
V_b = \Phi_M - \Phi_{Bi_2Te_3},
\]

if the work function of the metal (\( \Phi_M \)) is larger than that of Bi$_2$Te$_3$ (\( \Phi_{Bi_2Te_3} \)), or

\[
V_b = \chi - \Phi_M,
\]

if the work function of the metal is smaller than that of Bi$_2$Te$_3$, where \( \chi \) is electron affinity (~0.45 eV).

However, the measured electrical resistivity of the composite is larger than that of the pure Bi$_2$Te$_3$. The high density of grain boundary defects in composite structures causes the carriers scattering behavior, resulting in the increase of the electrical resistivity. At high density of Au nano-particles embedded in the composite, a distance between particles becomes close to each other. These behavior leads to the increase of scattered electrons, resulting in the increase of electrical resistivity of composites with the high concentration of Au nano-particles. As a result, the composite with 2 wt% of Au nano-particles possesses a slightly improved Seebeck coefficient. The composite with a high Au nano particle concentration (~10 wt%) exhibits a high electrical resistivity.

### Table I. Thermoelectric properties of composites with various concentrations of gold nano-particles.

| Composite (2 wt% of gold nano-particles) | Composite (5 wt% of gold nano-particles) | Composite (10 wt% of gold nano-particles) |
|----------------------------------------|----------------------------------------|----------------------------------------|
| Seebeck coefficient (±20 \( \mu \)V/K) | $-120$ | $-380$ | $-386$ |
| Electrical resistivity (±10 \( \mu \)Ω·m) | 60 | 140 | 180 |
| Power factor (\( \mu \)W/mK$^2$) | 240 | 1031 | 802 |
resistivity while the Seebeck coefficient is not larger than that of the composite consisting of 5 wt% of Au nano-particles. The power factors of the composites with 2 wt% and 10 wt% of Au nano-particles are lower than that of the composite with 5 wt% of Au nano-particles, as shown in Table I. Therefore, this work only focuses on the composite consisting of 5 wt% of Au nano-particles. Additionally, this paper reports the annealing effects on the Seebeck coefficient and electrical resistivity of the composite. The composite films are annealed at various temperatures in N2 ambience for 2 hours. The heat ramp rate during the annealing process is 2°C/min. As a result, different effects of the annealing process on the pure bismuth telluride and the bismuth telluride composite have been found. While the annealing process improves the Seebeck coefficient of the pure Bi2Te3 sample, the composite remains the value of Seebeck coefficient during the annealing treatment. Additionally, the highest annealing temperature to maintain the effect of gold nano-particles on the Seebeck coefficient is found to be less than 150°C, as shown in Fig. 4b. If the annealing temperature is ≥150°C, the Seebeck coefficient dramatically reduces. This behavior is explained due to the reason of the eutectic reaction of Bi2Te3 and Au at the high temperatures.36 The annealing temperature dependences on the Seebeck coefficient and electrical resistivity for the composite consisting of 5 wt% of Au nano-particles are shown in Fig. 4c. Although the observed Seebeck coefficient declines (∼−50 μV/K) at annealing temperatures ≥150°C, the electrical resistivity remains a high value of approximately 110 μΩ·m. This behavior suggests that the grain boundary defects of the composite material remain the effects on the electrical resistivity.

Thermal conductivities of Bi2Te3 and composites are measured using the differential 3ω method. The temperature oscillations induced by the applied current are varied with the thermoelectric materials, as shown in Fig. 6a. As a result, the thermal conductivities of the pure Bi2Te3 and the composite consisting of 5 wt% of Au nano-particles are 1.14 and 0.53 W/m·K, respectively, as shown in Fig. 6b. The mechanism of the reduction of the thermal conductivity caused by the inclusion of nano-particles is relevant to the phonon scattering effects in thermoelectric materials.37–40 In general thermoelectric materials with low electrical conductivities, the total thermal conductivity is mainly contributed by the lattice thermal conductivity, which is given by

\[ k_L = \frac{1}{3}C_v V_l I. \]  

where \( k_L \) is the lattice thermal conductivity, \( C_v \) is the heat capacity, \( V_l \) is the phonon velocity and \( l \) is the mean free path of phonons. When embedding the nano-particles in the matrix of the host materials, the phonons in the composite material are scattered by the nano-particles. Significant decreases of the velocity and the mean free path of phonons significantly reduce the lattice thermal conductivity of the composite. The in-plane thermal conductivities are obtained from cross-plane thermal conductivities using the theoretical model proposed by Kudo et al.41 According to this model, the in-plane thermal conductivities of the pristine Bi2Te3 and composite with 5 wt% of Au nano-particles are approximately 0.95 and 0.5 W/m·K, respectively. As a result, the figure of merit \( ZT \) of the composite with 5 wt% of gold nano-particles is calculated to be approximately 0.62. Meanwhile, the as-deposited pure Bi2Te3 specimen synthesized with the same condition as the composite exhibits approximately 0.016 of figure of merit \( ZT \). With the inclusion of 5 wt% of Au nano-particles, the figure of merit \( ZT \) of the composite is approximately forty times better than that of the pure Bi2Te3. The comparison between thermoelectric properties...
of the pure bismuth telluride and Au nano-particles bismuth telluride composite is summarized in the Table II.

**Table II. Comparison of pure Bi$_2$Te$_3$ samples and gold nano-particles composite.**

|                       | Bi$_2$Te$_3$ (Similar synthesis condition with the composite) | Composite with 5 wt% of gold nano-particles |
|-----------------------|-------------------------------------------------------------|--------------------------------------------|
| Seebeck coefficient   | $\pm 20 \mu$V/K                                             | $-50$                                      |
| Electrical resistivity | $\leq 10 \Omega \cdot$m                                       | $50$                                      |
| In-plane thermal conductivity | 0.95 (W/m·K)                               | $140$                                      |
| Figure of merit ZT (at 300K) | 0.016                                                | $0.62$                                      |

Conclusions

In conclusions, this paper demonstrates a novel idea of improving the thermoelectric properties by the inclusion of gold nano-particles in nanocomposite materials approached by the electrochemical codeposition. The Seebeck coefficient of the bismuth telluride composite is achieved as much as seven times larger than that of the pure bismuth telluride. Additionally, the thermal conductivity of the bismuth telluride composite is 0.5 W/m·K, which is approximately half of the pure bismuth telluride. Although the electrical resistivity increases by the factor of roughly four, the figure of merit ZT of the bismuth telluride composite is significantly optimized (~0.62). This value is approximately forty times better than that of the as-deposited pure bismuth telluride film (~0.016).

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Figure 6. (a) Temperature oscillation and (b) thermal conductivity of pure Bi$_2$Te$_3$ and composite with 5 wt% of gold nano-particles measured using 30ω method. The thermal conductivities are plotted against measured modulation frequency.
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