Segregation of NiTe$_2$ and NbTe$_2$ in $p$-Type Thermoelectric Bi$_{0.5}$Sb$_{1.5}$Te$_3$ Alloys for Carrier Energy Filtering Effect by Melt Spinning

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Abstract: The formation of secondary phases of NiTe$_2$ and NbTe$_2$ in $p$-type Bi$_{0.5}$Sb$_{1.5}$Te$_3$ thermoelectric alloys was investigated through in situ phase separation by using the melt spinning process. Adding stoichiometric Ni, Nb, and Te in a solid-state synthesis process of Bi$_{0.5}$Sb$_{1.5}$Te$_3$, followed by rapid solidification by melt spinning, successfully segregated NiTe$_2$ and NbTe$_2$ in the Bi$_{0.5}$Sb$_{1.5}$Te$_3$ matrix. Since heterointerfaces of Bi$_{0.5}$Sb$_{1.5}$Te$_3$ with NiTe$_2$ and NbTe$_2$ form potential barriers of 0.26 and 0.08 eV, respectively, a low energy carrier filtering effect can be expected; higher Seebeck coefficients and power factors were achieved for Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_{0.01}$ (250 µV/K and 3.15 mW/mK$^2$), compared to those of Bi$_{0.5}$Sb$_{1.5}$Te$_3$ (240 µV/K and 2.69 mW/mK$^2$). However, there was no power factor increase for NbTe$_2$ segregated samples. The decrease in thermal conductivity was seen due to the possible additional phonon scattering by the phase segregations. Consequently, $zT$ at room temperature was enhanced to 0.98 and 0.94 for Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_{0.01}$ and Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_{0.01}$, respectively, compared to 0.79 for Bi$_{0.5}$Sb$_{1.5}$Te$_3$. The carrier filtering effect induced by NiTe$_2$ segregations with an interface potential barrier of 0.26 eV effectively increased the Seebeck coefficient and power factor, thus improving the $zT$ of $p$-type Bi$_{0.5}$Sb$_{1.5}$Te$_3$, while the interface potential barrier of 0.08 eV of NbTe$_2$ segregation appeared to be too small to induce an effective carrier filtering effect.

Keywords: Bi$_{0.5}$Sb$_{1.5}$Te$_3$; heterointerface; phase separation; carrier energy filtering; secondary phase

1. Introduction

Research on renewable energy generation has attracted considerable attention because traditional energy resources have become more limited. In addition, approximately 60% of the total energy is lost as waste heat during energy generation and consumption. Therefore, thermoelectric power generation has several advantages as a power generation technology to use energy more efficiently [1,2]. Thermoelectric materials can directly convert the temperature gradient into electrical energy, and they have attracted considerable attention in recent decades. In particular, bismuth telluride (Bi$_3$Te$_3$)-based alloys exhibit high thermoelectric performance at near-room temperature [1,2]. They can be applied either to power generation from low grade waste heat, or in thermoelectric solid-state cooling to replace traditional refrigeration [1,2]. The thermoelectric efficiency is determined in terms of a thermoelectric dimensionless figure of merit, expressed as $zT = σ·S^2·T/κ$, where $σ$ is the electrical conductivity, $S$ is the Seebeck coefficient, $T$ is the absolute temperature, and $κ$ is the thermal conductivity [3].

Many approaches have been adopted to enhance the $zT$ of thermoelectric materials. To improve electrical transport properties, approaches such as band convergence, carrier
energy filtering, and resonance doping have been applied [4–6]. In addition, a variety of defect structures is introduced to the material to reduce the $\kappa$ by intensifying phonon scattering [7–9]. For example, nanograin-composite, dense dislocations formed at the grain boundary, and substitutional doping have been known as effective approaches to reduce $\kappa$ by intensifying phonon scattering in (Bi$_2$Te$_3$)-based alloys [8–12]. Among these approaches, introducing nanoparticles in the thermoelectric materials can improve $zT$ by increasing the electrical transport properties and reduction in $\kappa$. The reduction in $\kappa$ can be attained by additional phonon scattering by the embedded nanoparticles [13], and the increase in the electrical transport properties can be attributed to the carrier energy filtering effect caused by the nanoparticles. The carrier filtering effect is a strong energy dependence of the carrier relaxation time caused by band bending at the interface between metallic inclusions and thermoelectric materials [14–16].

In this study, the formation of secondary phases of NiTe$_2$ and NbTe$_2$ in $p$-type Bi$_{0.5}$Sb$_{1.5}$Te$_3$ thermoelectric alloys related with the carrier filtering effect was investigated through in situ phase segregation by using the melt spinning process. Metallic NiTe$_2$ and NbTe$_2$ were chosen for the successful segregation in Bi$_{0.5}$Sb$_{1.5}$Te$_3$ alloys, using the melt spinning process, which was observed in the preliminary experiment. Adding excess stoichiometric composition of NiTe$_2$ and NbTe$_2$ in the melt-process of Bi$_{0.5}$Sb$_{1.5}$Te$_3$ synthesis successfully segregated NiTe$_2$ and NbTe$_2$ phases through the rapid solidification process by melt spinning. As heterointerfaces of semiconducting Bi$_2$Sb$_2$Te$_3$ with metallic NiTe$_2$ and NbTe$_2$, a possible carrier energy filtering effect can be expected. In addition, the segregation of NiTe$_2$ and NbTe$_2$ would intensify the phonon scattering, possibly reducing $\kappa$.

2. Materials and Methods

Ingots of Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ ($x = 0, 0.003, 0.01, 0.1$) and Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0, 0.005, 0.01, 0.1$) were prepared by conventional melting and quenching techniques using high-purity elemental Bi (99.999%, shots, 5N Plus, Montreal, QC, Canada), Sb (99.999%, shots, 5N Plus), Te (99.999%, pieces, 5N Plus), Nb (99.99%, powder, Sigma Aldrich), St. Louis, MO, USA), and Ni (99.9%, powder, Sigma Aldrich), as starting materials. Stoichiometric mixtures were loaded into a quartz tube (15 mm in diameter). The tube was vacuum-sealed under $10^{-4}$ torr, and the contents were melted in a box furnace at 1423 K for 20 h, and then quenched to room temperature. The ingots were ground using a high-energy ball mill (8000D, SPEX, Metuchen, NJ, USA) for 5 min. Subsequently, melt spinning of the prepared ingots (except for Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_{0.1}$ and Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_{0.1}$) was performed, under a copper wheel rotation of 3600 rpm, and the molten solution was shot at 0.03 MPa under Ar. The ribbons were grounded in an agate mortar. Then, disk-shaped polycrystalline bulk samples (10 mm in diameter and 10 mm thickness) were prepared by spark plasma sintering (SPS) at 430 °C for 5 min, under 50 MPa in vacuum.

X-ray diffraction (XRD, D8 Discover, Bruker, Billerica, MA, USA) was performed for phase analysis. The temperature-dependent $S$ and $\sigma$ parameters were measured within a temperature range from room temperature to 480 K on the samples, perpendicular to the direction of the applied pressure (ZEM-3 Advanced-RIKO, Yokohama, Japan). The nominal temperature gradients were given as 10, 20, and 30 K. The experimental error margin for $S$ and $\sigma$ are estimated as ±5% and ±3%, respectively. The carrier concentrations were determined by van der Pauw measurements under a magnetic field of 0.5 T (AHT-55T5, Ecopia, Anyang, Korea) in the same direction; the input current can be regulated between 1 nA and 20 mA. The van der Pauw method typically provides the carrier concentration with relative error within 5%. The $\kappa$ values of the samples were calculated from the expression $\kappa = \rho_c \cdot C_p \cdot \lambda$, considering their theoretical density ($\rho_c$), heat capacity ($C_p$), and thermal diffusivity ($\lambda$) measured across the same direction (perpendicular to the pressing direction), so the $zT$ can be determined. The $C_p$ values from 300 K to 400 K were measured by using a physical property measurement system (PPMS, Quantum Design, San Diego, CA, USA), and 121.6 J mol$^{-1}$ K$^{-1}$ were used, which were estimated from Dulong–Petit fitting. The densities of all samples were measured as being higher than 98.5%, from
the sintered samples of cylinder shape and 10 mm diameter with ~12 mm height, by the Archimedes method, and the \( \rho_s \) for Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) of 6.88 g/cm\(^3\) was used for samples with small addition (\( x \leq 0.01 \)). The \( \lambda \) were measured using a laser flash installation (Laser Flash Analysis, LFA 467, Netzsch, Selb, Germany). The experimental error margin for \( \lambda \) is estimated as \( \pm 3\% \).

3. Results

Figure 1a,b shows XRD patterns of the melt-spun ribbons of Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) (\( x = 0.1 \)) and Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NbTe\(_2\))\(_x\) (\( x = 0.1 \)). Figure 1c,d shows XRD patterns of the SPS-sintered samples of Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NiTe\(_2\))\(_x\) (\( x = 0, 0.003, 0.01, \) and 0.1) and Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NbTe\(_2\))\(_x\) (\( x = 0, 0.005, 0.01, \) and 0.1). For all samples, the diffraction peaks of the major phase were related to the Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) structure (JCPDS #00-49-1713). The XRD patterns of Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NiTe\(_2\))\(_x\) and Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NbTe\(_2\))\(_x\) with \( x = 0.1 \) showed additional peaks of the segregated phases of NiTe\(_2\) (JCPDS #01-089-2642) and NbTe\(_2\) (JCPDS #00-021-0605), respectively. Thus, it was confirmed that in situ phase segregation is possible after applying a rapid solidification process. For the samples doped with small fractions (\( x \leq 0.01 \)), no secondary phase was observed. However, it can be expected that the same phase segregation of NiTe\(_2\), and NbTe\(_2\) occurred while the amount of the segregations was too small to be detected by XRD. Figure 1e,f shows the lattice parameters calculated from the XRD patterns for Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NiTe\(_2\))\(_x\) (\( x = 0, 0.003, \) and 0.01) and Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\)(NbTe\(_2\))\(_x\) (\( x = 0, 0.005, \) and 0.1) with no changes in lattice parameters.

Figure 2 represents a schematic of energy band diagrams at heterointerfaces formed between Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) and the secondary phases, based on the concept of carrier energy filtering [14,15]. The main concept, which involves a Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) matrix with the precipitates, is illustrated in Figure 2. Carrier transfer induces band bending away from the heterointerfaces between the BiSbTe matrix and precipitates. The work function, electron affinity, and bandgap data for NiTe\(_2\), NbTe\(_2\), and Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) are shown in Table 1. The electron affinity of Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) is 4.50 eV and the bandgap is 0.2 eV [15]. The work functions of NiTe\(_2\) and NbTe\(_2\) are 4.44 eV and 4.62 eV, respectively [17,18]. Barrier potentials may form near the interface; the presence of the electrostatic potential causes energy-dependent scattering of carriers at the heterointerfaces for NiTe\(_2\) and NbTe\(_2\), with Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) forming potential barriers of approximately 0.26 and 0.08 eV, respectively, as depicted in Figure 2. When hole carriers pass near the surfaces of secondary phases, low energy carriers are scattered, inducing the carrier filtering effect [14,15]. However, the energy blocking effect near the interfaces between NbTe\(_2\) would be smaller due to their low barrier height of 0.08 eV (Figure 2b).

Figure 3 shows the \( \sigma \) and \( S \) as a function of temperature of the samples after melt spinning with a small addition (\( x \leq 0.01 \)). Bi\(_{0.5}\)Sb\(_{1.5}\)Te\(_3\) exhibited an \( \sigma \) of 465 S/cm at room temperature. As shown in Figure 3a, no change in \( \sigma \) was observed for \( x = 0.003 \); however, the \( x = 0.01 \) showed an increased \( \sigma \) (~500 S/cm at room temperature). In Figure 3b, the \( S \) values, as a function of temperature, are shown, while there is no distinct change in the error range. However, as shown in inset of Figure 3b, the power factor (\( \sigma S^2 \)) was increased from 2.68 to 3.15 mW/mK\(^2\) for \( x = 0.01 \) as both the \( \sigma \) and \( S \) increased. In Figure 3c, after the addition of a small amount of NbTe\(_2\), there is no significant change in the \( \sigma \). As in Figure 3d, the \( S \) shows no distinct change in error range by NbTe\(_2\) addition. Therefore, power factors (\( \sigma S^2 \)) also show no increase with the addition of NbTe\(_2\) at all compositions (inset of Figure 3d).
Figure 1. X-ray diffraction patterns for (a,b) melt-spun ribbons of (a) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ ($x = 0.1$) and (d) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0.1$)(c) and (c,d) SPS-sintered pellet of Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ ($x = 0, 0.003, 0.01, and 0.1$) and (d) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0, 0.005, 0.01, and 0.1$). Lattice parameters calculated for (e) SPS-sintered Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ ($x = 0, 0.003, and 0.01$) samples and (f) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0, 0.005, and 0.01$).

Figure 2. Schematic of energy band diagrams of the samples at the heterointerfaces between Bi$_{0.5}$Sb$_{1.5}$Te$_3$ and the secondary phase: (a) NiTe$_2$ and (b) NbTe$_2$. 
Table 1. Work function (Φ) for NbTe2 and NiTe2, and electron affinity (χ) and band gap (Eg) for (Bi0.5Sb1.5)Te3.

<table>
<thead>
<tr>
<th>Phase Segregation</th>
<th>Properties</th>
<th>Work Function (Φ) or Electron Affinity (χ) and Band Gap (Eg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NbTe2</td>
<td>Metallic</td>
<td>Φ = 4.62 eV</td>
</tr>
<tr>
<td>NiTe2</td>
<td>Metallic</td>
<td>Φ = 4.44 eV</td>
</tr>
<tr>
<td>(Bi0.5Sb1.5)Te3</td>
<td>Semiconducting</td>
<td>E_g = 0.2 eV, χ = 4.50 eV</td>
</tr>
</tbody>
</table>

Figure 3. Temperature dependence of (a,c) the electrical conductivity and (b,d) the Seebeck coefficient for Bi0.5Sb1.5Te3(NiTe2)x (x = 0, 0.003, 0.01) and Bi0.5Sb1.5Te3(NbTe2)x (x = 0, 0.005, 0.01). Inset graphs in (b,d) exhibit the power factors of the samples.

Figure 4 shows the results from the Hall measurement (Hall carrier concentration, nH, and Hall mobility, μH) and the Pisarenko plots at room temperature [14,15,19]. In Figure 4a, no clear relationship between nH and μH is seen, while the nH does not change for NiTe2-added samples in the range of 9.5 × 10¹⁸–9.6 × 10¹⁸ cm⁻³. Figure 4b shows a Pisarenko plot (S as a function of nH) with the effective mass m* of 0.5 m₀, 0.55 m₀, and 0.6 m₀ shown as lines, based on Equation (1).

\[ S = \frac{8π²k_B²}{3e²h²} \left( \frac{π}{3m} \right)^{2/3} m^* T \]  

(1)

where \( h \) is the Planck constant, \( k_B \) is the Boltzmann constant, \( e \) is the elemental electric charge and \( n \) is the carrier concentration. The gradual increase in \( m^* \) is clearly shown as \( x \) increased for NiTe2-added samples (Figure 4b), although the noticeable decrease in \( n_H \) is not seen (Figure 4a). As \( n_H \) could vary for various reasons, the systematic increase in the \( m^* \) would suggest the presence of the carrier filtering effect due to the energy barrier of 0.26 eV (Figure 2a) as the density of interfaces between NiTe2 and the matrix increases; for instance, the simultaneous increase in \( σ \) and \( S \) in NiTe2-added samples (x = 0.01 compared to \( x = 0 \) of Bi0.5Sb1.5Te3(NiTe2)x).
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Figure 4c shows that the $n_H$ of Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0$, 0.005, 0.01) gradually decreased with $x$; the $n_H$ values of 9.5 × 10$^{18}$, 9.0 × 10$^{18}$, and 8.7 × 10$^{18}$ cm$^{-3}$ for $x = 0$, 0.005, and 0.01, respectively. In contrast, the, $\mu_H$ increased gradually with increasing $x$, leading to values of 314, 327, and 343 cm$^2$/Vs for $x = 0$, 0.005, and 0.01, respectively. However, no change in $m^*$ was shown. It seems that there is less carrier filtering influence on these samples due to the smaller barrier height (Figure 2b).

Figure 5 shows the temperature dependent $x$ of all samples (the uncertainty of $x$ would be around ±3%), while the inset shows the lattice thermal conductivity ($\kappa_{latt}$), calculated by subtracting the electronic contribution ($\kappa_{elec}$) calculated by the Wiedemann–Franz law ($\kappa_{elec} = L\sigma T$), where $L$ is Lorenz number, which can be estimated with Equation (2) (where $L$ is in 10$^{-8}$ WΩK$^{-2}$ and $S$ in $\mu$V/K) [20],

$$L = 1.5 + exp\left(\frac{|S|}{116}\right)$$

(2)

Figure 5. Thermal conductivity for (a) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ ($x = 0$, 0.003, 0.01) and (b) Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ ($x = 0$, 0.005, 0.01). Insets shows the lattice thermal conductivity ($\kappa_{latt}$).
For Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ samples, a gradual decrease in $\kappa_{\text{latt}}$ (0.81, 0.78, and 0.75 W/mK for $x = 0$, 0.003, and 0.01) was seen at room temperature (inset of Figure 5a), while there seems to be no distinct change in $\kappa$ in Figure 5a. At high temperatures, the different influences from the bipolar conduction may affect the $\kappa_{\text{latt}}$. The second phases of NiTe$_2$ may scatter the phonons further to reduce room temperature $\kappa_{\text{latt}}$. For Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_{0.01}$, the $\kappa$ (and $\kappa_{\text{latt}}$) decreased to 0.92 W/mK (0.70 W/mK) at room temperature, compared to 1.03 W/mK (0.81 W/mK) of Bi$_{0.5}$Sb$_{1.5}$Te$_3$.

The estimated $zT$ values of the samples are shown in Figure 6. For Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ samples (Figure 6a, the uncertainty of $zT$ would be around ±17%), the $zT$ was enhanced from 0.79 to 0.82 and 0.98 for $x = 0.003$ and 0.01, respectively, as the power factor increased due to increases in $S$, power factor, and $m^*$ (Figures 3b and 4b). We speculate that the interface barrier height of 0.26 eV between Bi$_{0.5}$Sb$_{1.5}$Te$_3$ and NiTe$_2$ (Figure 2a) induced the carrier filtering effect, while there was slight systematic $\kappa_{\text{latt}}$ reduction as well (inset of Figure 5a). For Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ samples (Figure 6b), with the much smaller barrier height of 0.08 eV between Bi$_{0.5}$Sb$_{1.5}$Te$_3$ and NbTe$_2$ (Figure 2b), no increases in $S$, power factor, and $m^*$ (Figures 3d and 4d) were seen. However, the reduction in $\kappa_{\text{latt}}$ to 0.70 W/mK at room temperature (inset of Figure 5b) was achieved for $x = 0.01$. Therefore, the $zT$ was also enhanced slightly from 0.79 to 0.83 and 0.94 for $x = 0.005$ and 0.01; however, this was mainly due to decreased $\kappa$ (and $\kappa_{\text{latt}}$) for $x = 0.01$ (Figure 5b). For Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_x$ samples, the improved electronic transport properties, caused by the possible carrier filtering effect, enhanced the $zT$, while the reduced thermal conductivity enhanced the $zT$ in Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NbTe$_2$)$_x$ samples.

4. Conclusions

The electrical and thermal transport properties of Bi$_{0.5}$Sb$_{1.5}$Te$_3$ with the formation of secondary phases of NiTe$_2$ and NbTe$_2$ were analyzed in regard to the carrier filtering effect. The possible carrier filtering effect was shown with enhanced Seebeck coefficient, power factor and effective mass for the NiTe$_2$ added samples, where the heterointerfaces are expected to form the energy barrier of 0.26 eV with the Bi$_{0.5}$Sb$_{1.5}$Te$_3$ matrix. However, for the NbTe$_2$ added samples with the energy barrier of 0.08 eV, only slight enhancement of the power factor was seen without increase in effective mass, implying that there is only minor influence of the carrier filtering effect. While the additional phonon scattering by the segregation was anticipated, the influence on $\kappa$ was slight. Consequently, $zT$ the $zT$ was enhanced to 0.98 for Bi$_{0.5}$Sb$_{1.5}$Te$_3$(NiTe$_2$)$_{0.01}$ from 0.79 for Bi$_{0.5}$Sb$_{1.5}$Te$_3$ at room temperature, mainly due to the increases in $S$ and $m^*$ by the possible carrier filtering effect.

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**References**


