Fabrication and thermoelectric properties of Yb-doped ZrNiSn half-Heusler alloys

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Half-Heusler (HH) alloys constitute an important class of materials that exhibit promising potential in high-temperature thermoelectric (TE) power generation. In this work, we synthesized Zr$_{1-x}$Yb$_x$NiSn ($x=0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) HH alloys using a time-efficient levitation melting and spark plasma sintering procedure. X-ray diffraction showed that the samples were predominantly single phased, and that the lattice constant increased systematically with increasing Yb doping ratio. The doping effects of Yb on the thermoelectric properties were studied. It was found that Yb doping consistently decreased the electrical and thermal conductivities. On the other hand, the effects of Yb doping on the Seebeck coefficient were found to be non-monotonic. The magnitude of the Seebeck coefficient (n-type) was increased upon Yb doping up to $x=0.02$, above which Yb doping introduced notable p-type conduction. As a result, the room-temperature Seebeck coefficient of the $x=0.10$ sample became positive although the magnitude was not high. The thermoelectric figure of merit, $ZT$, reached a maximum of $\sim 0.38$ at $900$ K for the $x=0.01$ sample. Selective doping on the Ni and Sn sites are necessary to further optimize the TE performance of Zr$_{1-x}$Yb$_x$NiSn alloys.

Keywords: half-Heusler alloy; thermoelectric power; levitation melting; spark plasma sintering; Yb doping

1. Introduction

In recent years the global energy crisis has resulted in a pressing need for the development of high-performance thermoelectric (TE) materials for direct thermal-to-electrical power generation. The performance of a TE material is gauged by the dimensionless figure of merit, $ZT = \alpha^2 \sigma T / \kappa$, where $\alpha$ is the Seebeck coefficient, $\sigma$ the electrical conductivity, $\kappa$ the thermal conductivity and $T$ the absolute temperature. In the simplest case, $\kappa = \kappa_L + \kappa_c$, where $\kappa_L$ and $\kappa_c$ are the lattice and carrier thermal conductivities, respectively. The power factor, $\alpha^2 \sigma$, which is intimately related to the electronic band structure, is in general optimized via doping to maximize the $ZT$ [1,2].

Half-Heusler (HH) ternary intermetallic compounds are formulated as XYZ, where X and Y are two different transition metal elements, and Z is an $sp$ element such as Sn, Sb or Bi. HH compounds are of considerable interest for TE power generation above room temperature. HH alloys adopt an MgAgAs-type structure (space group $F43m$), consisting

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of three interpenetrating face centered cubic (fcc) sublattices. The crystallographic sites (0, 0, 0) and (1/4, 1/4, 1/4) are occupied by two different transition metal elements X and Y, respectively, the (1/2, 1/2, 1/2) site is occupied by a \( sp \) element Z, whereas the site (3/4, 3/4, 3/4) is vacant [3]. The band structure of HH alloys is profoundly affected by the valence electron count (VEC): the compounds are narrow band gap semiconductors when \( \text{VEC} = 8 \) or 18, while the deviation from \( \text{VEC} = 18 \) may lead to metallic conduction [4].

From the thermoelectrics point of view, the group of HH alloys most intensively investigated is XNiSn (X = Ti, Zr, Hf). The XNiSn alloys are narrow band gap (0.1–0.2 eV) semiconductors [5,6]. The narrow band gaps give rise to a large effective mass that in turn leads to a large Seebeck coefficient [7]. A large Seebeck coefficient \( \sim 100 \mu \text{V K}^{-1} \) and a high electrical conductivity \( \sim 10^3–10^4 \text{ Scm}^{-1} \) have been attained [8,9]. In addition, XNiSn alloys have high melting points of 1400–1600 K [10] and excellent chemical stability with negligible sublimation near their melting points.

The TE performance of these XNiSn alloys can be optimized for practical TE power generation via selectively doping the three filled sublattices [11]. For example, doping on the Sn site tunes carrier concentration [12–14], while substituting the Ti and Ni sites promotes the mass fluctuations and strain fields that help reduce the lattice conductivity [15–18]. The \( ZT \) of n-type Hf\(_{0.6}\)Zr\(_{0.4}\)NiSn\(_{0.98}\)Sb\(_{0.02}\) is \( \sim 1 \) at 1000 K, which is the best and reproducible TE performance that n-type HH TE materials have attained so far [12].

As far as making a practical TE power generation module is concerned, both n- and p-type materials are needed, better with similar composition and mechanical properties. In contrast to the promising TE properties of n-type HH alloys, p-type HH compounds are rarely reported, and the reported TE performance was not satisfactory [19–22]. For example, \( ZT \) values of \( \sim 0.5 \) and \( \sim 0.29 \) were attained in the p-type Zr\(_{0.5}\)Hf\(_{0.5}\)CoSb\(_{0.8}\)Sn\(_{0.2}\) [23] and ErNiSn\(_{0.01}\)Sb\(_{0.99}\) [19], respectively. It is thus highly desirable to control the type and the number of charge carriers in XNiSn by doping on the X, Ni or Sn sites [7,24,25].

In this vein, Katsuyama et al. [24] and Hlil et al. [26] proposed that Y doping in ZrNiSn was equivalent to doping with acceptors since the number of valence electrons of Y (4\( d^{15} s^2 \)) is one less than that of Zr (4\( d^2 s^2 \)), so heavy Y doping might increase the hole concentration and even turn ZrNiSn into p-type. Zhu et al. [27] recently showed that Y doping in Hf\(_{0.6}\)Zr\(_{0.4}\)NiSn\(_{0.98}\)Sb\(_{0.02}\) reduced thermal conductivity due to the reduced carrier thermal conductivity, and increased the Seebeck coefficient due to the decreased carrier concentration.

Another promising class of dopants is the rare earth lanthanide elements [28]. Lanthanide elements are characterized by 4\( f \) electrons. It is established that rare earth-containing compounds often have strong hybridization between the 4\( f \) electrons and the conduction band of the parent compounds, giving rise to enhanced electronic density of states near the Fermi energy and thus higher Seebeck coefficient. Moreover, heat is mostly carried by short-wavelength phonons at temperatures where HH TE materials operate, and hence the rare earth doping will introduce short-range disorder and strain fields to effectively scatter heat-carrying phonons. Nonetheless, the electrical conductivity, Seebeck coefficient and thermal conductivity are inter-related in a given material, the detailed effects of rare earth doping on the TE properties are material specific and subject to exploratory experimental investigations.

In order to verify whether and to what extent the rare earth lanthanide doping can change the carrier concentration and even the type of the primary charge carrier in ZrNiSn, in this work we investigated the effects of Yb (4\( f^{14} s^2 \)) doping at the Zr (4\( d^2 s^2 \)) sites on the TE properties of ZrNiSn.
2. Experimental procedures

One general concern of HH alloy synthesis is the homogeneity of samples, and, as such, most synthesis procedures involve a long annealing time to help homogenize the sample. To ensure sample homogeneity, avoid crucible-related cross-contamination and have a shorter synthesis time, we adopted a combined levitation melting and spark plasma sintering (SPS) procedure recently developed by our group [12]. Yb-doped Zr$_{1-x}$Yb$_{x}$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) HH alloys were prepared by levitation melting of stoichiometric amounts of Zr (99.99%), Ni (99.999%), Sn (99.999%) and Yb (99.999%) in an argon atmosphere for 2 min, and the melt was quenched in a water-cooled copper crucible. The ingot was melted twice to ensure homogeneity. The ingots were pulverized through a ball milling (BM) process at 200 rpm for 4 h. The powders were then pelletized to $\sim 99\%$ theoretical density using a SPS procedure (SPS-1050, Sumitomo Coal Mining Co.) at 1173 K for 10 min under 65 MPa.

The pellets were directly used to measure the thermal conductivity, and then cut into rectangular bars for electrical conductivity and Seebeck coefficient measurements. These measurements were carried out from 300 K to 900 K, if not otherwise noted. The phase structure of the samples after BM and after SPS was checked by X-ray diffraction (XRD) on a Rigaku D/MAX-2550PC diffractometer using Cu K$_{\alpha}$ radiation ($\lambda = 1.5406$ Å). The electrical conductivity and the Seebeck coefficient were measured on a custom-designed, computer-assisted apparatus using a DC four-probe method and a differential voltage/temperature technique, respectively [29]. The thermal conductivity, $\kappa$, was calculated by using $\kappa = D\rho C_p$, where $\rho$ is the sample density measured by an Archimedes’s method. The thermal diffusivity, $D$, and specific heat, $C_p$, were measured by a laser flash method on a Netzsch LFA457 with a Pyroceram standard [30]. The uncertainties for $D$ and $C_p$ measurements were $\pm 3\%$ and $\pm 5\%$, respectively.

3. Results and discussion

The XRD patterns of the powders of levitation melted Zr$_{1-x}$Yb$_{x}$NiSn alloys are shown in Figure 1a. All major peaks could be indexed to the ZrNiSn phase with MgAgAs-type structure (JCPDS No.23-1281), while some weak peaks from a small amount of Sn were detected. After the SPS process, all samples were single phased except the $x = 0.1$ sample, which showed a trace of impurity phase (Figure 1b). As shown in the inset of Figure 1b, the lattice constant increases with the increase of the nominal Yb doping ratio, and the variation agrees fairly well with Vegard’s law. In view of the fact that the ionic radius of Yb (0.86 Å) is larger than that of Zr (0.79Å), this is direct evidence of Yb doping into the lattice and indicates that the actual Yb doping ratio is proportional to the nominal one. These observations are also consistent with our previous work [27], showing that the combined levitation melting and SPS procedure is effective in preparing high-quality HH alloy samples in a time-efficient manner.

The temperature dependences of electrical conductivity, $\sigma$, and Seebeck coefficient, $\alpha$, of Zr$_{1-x}$Yb$_{x}$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) alloys are shown in Figures 2a and b, respectively. The $\sigma$ of all samples increases with increasing temperature, showing semiconducting behavior. We note that Yb doping consistently decreases the magnitude of $\sigma$ over the entire temperature range studied. On the other hand, Yb doping impacts the magnitude and the temperature dependence of $\alpha$ in a non-monotonic fashion. The magnitude of $\alpha$ first increases with increasing Yb content up to $x = 0.02$ with a maximum $\alpha \sim -210$ µVK$^{-1}$, above which it starts decreasing. In particular, the sign of $\alpha$ is changed
to positive (p-type) at room temperature for the $x = 0.10$ sample (see inset of Figure 2b), suggesting that heavy Yb doping establishes p-type conduction in the material although the magnitude of $\alpha$ is not yet sufficient for a high p-type performance. The trend of this n-type to p-type changeover is apparent for the $x = 0.04, 0.06$ and 0.10 samples near room temperature: the value of $\alpha$ changes from $-61 \mu V K^{-1}$ to $14 \mu V K^{-1}$. In view of the electronic configurations of Yb ($4f^{14}6s^2$), Y ($4d^15s^2$) and Zr ($4d^25s^2$), the sign change of $\alpha$ suggests that Yb doping introduces acceptors into the material in the same way that Y doping does [24,27]. In this context, the decrease of $\sigma$ upon Yb doping can be thus ascribed to the decreased carrier concentration due to the charge (electron–hole) compensation effect. The temperature dependence of $\alpha$ for all samples is typical of a degenerate semiconductor: the magnitude of $\alpha$ first increases with increasing temperature until reaching a maximum (inflection point), and then decreases due to the bipolar effect (Figure 2b). Leaving the magnitude of $\alpha$ aside, the maximum of $\alpha$ systematically shifts toward higher temperature with increasing Yb content, indicating that the Yb doping alters the band structure of ZrNiSn.

Figure 3a shows the temperature dependence of the thermal conductivity, $\kappa$, of the Zr$_{1-x}$Yb$_x$NiSn alloys. In general, the $\kappa$ is the sum of the carrier thermal conductivity, $\kappa_c$, and the lattice thermal conductivity, $\kappa_L$, i.e. $\kappa = \kappa_L + \kappa_c$. The $\kappa_c$ can be estimated by the Wiedemann–Franz relationship, $\kappa_c = L \sigma T$ with the Lorentz number

![Figure 1. XRD patterns of the Zr$_{1-x}$Yb$_x$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) compounds after BM (a) and after SPS (b). The calculated lattice constants after SPS are shown in (b) as a function of the nominal Yb doping ratio.](image1)

![Figure 2. Temperature dependences of electrical conductivity $\sigma$ (a) and Seebeck coefficient $\alpha$ (b) for Zr$_{1-x}$Yb$_x$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) compounds. The room temperature Seebeck coefficient $\alpha$ as a function of the nominal Yb-doping ratio is shown in the inset of (b).](image2)
Figure 3. Temperature dependences of thermal conductivity $k$ (a) and the electrical thermal conductivity $k_e$ and lattice thermal conductivity $k_L$ (b) for Zr$_{1-x}$Yb$_x$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) compounds. Temperature dependence of specific heat $C_p$ is presented in the inset of (a).

$L = 2.0 \times 10^{-8}$ V$^2$ K$^{-2}$. The calculated $k_L$ and $k_e$ are shown in Figure 3b. We note that (i) the $k_L$ significantly decreases while the $k_e$ only slightly decreases with increasing Yb doping ratio, and (ii) the $k_L$ predominates the $k$ in all compounds. It is also noted that Yb doping effectively reduce $k$ and $k_L$ in the vicinity of room temperature (Figure 4) but the extent of reduction quickly gets marginal at elevated temperatures. To elucidate the origin of this observation, we inspect the temperature dependence of specific heat, $C_p$ (inset of Figure 3a) from 300 K to 900 K. It is plausible to assume that $C_p$ is predominated by the lattice specific heat above room temperature. Furthermore, fitting the observed $C_p$ by the Debye formula of lattice specific heat, $C_p = 9R(T/\theta_D)^3 \int_0^{\theta_D/T} \exp(x)x^4/(\exp(x) - 1)^2dx$, $x = \hbar \omega/k_BT$, $\omega$ is the frequency of phonons, yields the Debye temperature, $\theta_D$, where $C_p$ is the molar specific heat. As shown in the inset of Figure 4, $\theta_D$ systematically decreases with increasing Yb doping ratio. We note that the derived $\theta_D$ value is somewhat higher than previously reported [31], but this will not affect the following discussion as we are only concerned with the trend of $\theta_D$ on doping. In a simple kinetic model, the $k_L$ is proportional to the product of $C_p$, the velocity of sound, $v_s$, and the phonon mean free path, $l_{ph}$. The value of $\theta_D$ is a rough gauge of the velocity of sound $v_s$. In this vein, the decrease of $k_L$ can be ascribed to enhanced phonon scattering and thus a decreased $l_{ph}$ by the dopant induced mass fluctuation mechanism.
Figure 5. Temperature dependences of power factor (a) and figure of merit $ZT$ (b) for Zr$_{1-x}$Yb$_x$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06$ and $0.10$) compounds.

Finally, the temperature dependences of the power factor and figure of merit, $ZT$, of Zr$_{1-x}$Yb$_x$NiSn alloys are shown in Figures 5a and b, respectively. Due to the degraded power factor upon Yb doping, there is not much improvement in the $ZT$ values of Yb-doped samples as compared with the pristine ZrNiSn. Nonetheless, further improvement of $ZT$ may be attained by selectively doping the Ni and Sn sites to increase the power factor, and/or via the ‘nanocompositing’ approach recently reported by Yan et al. [32].

4. Conclusions

In this paper, we have reported the thermoelectric properties of Zr$_{1-x}$Yb$_x$NiSn ($x = 0, 0.01, 0.02, 0.04, 0.06, 0.10$) compounds prepared by a time-efficient levitation melting and spark plasma sintering procedure. Yb doping appeared to introduce holes into the system, and the room temperature Seebeck coefficient changed into p-type at a nominal Yb doping content at $x = 0.10$ although the magnitude of Seebeck coefficient was not high. We did not see significant improvement of $ZT$ by Yb doping. Further efforts, e.g. doping on the Ni and Sn sites, will be carried out to further improve the $ZT$.

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