Martensitic transformation and magnetocaloric properties of NiCoMnSn magnetic shape memory alloys

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ABSTRACT

We report on the magnetic, magnetocaloric properties, and hysteresis of Ni45Co5Mn38Sn12 and Ni45Co5Mn38.5Sn11.5 Heusler polycrystalline alloys under several magnetic field values. Both alloys have a moderate magnetization difference (≈70 emu/gr) between austenite and martensite phase with a moderate thermal hysteresis (≈8 K) around the martensitic transition. Magnetic field dependent measurements reveal that the application of a magnetic field stabilizes the austenite phase, which has higher magnetization. Both alloys have substantial inverse magnetic entropy changes and adiabatic temperature changes. In addition, we investigate the accessibility of the calculated entropy change that is quantified by means of the minimum magnetic field required to start and to complete the magnetostructural phase transition.

1. Introduction

The caloric effect originates from the thermal response of a material as a result of an external change in field and is characterized either by the adiabatic temperature change or by the isothermal entropy change that occurs upon application or removal of an external field, that might be magnetic field, applied pressure/force, electric field [1]. Since the energy consumption and energy need in countries across the world is continuously increasing, the demand of environmental-friendly refrigeration techniques is also increasing [2]. Thus materials displaying large caloric effect are receiving much attention, that can be classified as magnetocaloric, barocaloric, electrocaloric and elastocaloric effect, when driving external field magnetic field, pressure, electric field and force, respectively [3].

Among these caloric effects, the most studied one is the magnetocaloric effect (MCE) [3,4]. The key issue of materials having a giant magnetocaloric effect is the occurrence of a first-order phase transition, namely magneto-structural phase transition. In addition, magnetic materials that show large changes in magnetic properties, those with first-order phase transitions around room temperature, are of significant interest because of their potential applications in spintronics and magneto mechanical devices, such as actuators and transducers [5]. However, the first-order phase transitions generate dissipation, thus hysteresis that represents a drawback for the reproducible entropy change and reduces the materials’ refrigerant capacity [6,7]. Previous studies stated hysteresis reduces the effective MCE of a material by both limiting the extent of the phase transition and resulting in dissipative energy loss [7,8]. Thereby, the applied magnetic field should be strong enough to overcome the thermal hysteresis of the material for a reversible transition since MCE strongly depends on the width of thermal hysteresis [9,10]. In addition, the conventional S(T) diagram is the representation in a thermal equilibrium and it does not consider metastability and hysteresis of the materials with first-order transition [10]. In order to minimize the effect of hysteresis, it is possible to use a thermodynamic cycle within minor loops of the hysteresis, which preserves a certain fraction of both phases and enhances the reversibility [11]. Nanoindentation was recently purposed to reduce the nucleation barrier, thereby hysteresis which is a microstructural approach [12].

Among the most promising materials, Heusler type Ni-Co-Mn-X (X = In, Sn, Sb) magnetic shape memory alloys (MSMAs) are attractive candidates for both fundamental research and application. These alloys exhibit diverse properties ranging from memory effect, magnetocaloric effect, magnetoresistance, magnetothermal conductivity and direct conversion of heat to electricity [2,13–15]. The Ni-Co-Mn-Sn alloy system reported here is a candidate magnetic shape memory alloy that does not require cost inhibitive elements in its manufacture and reveals...
both the one and two-way magnetic shape memory effect [16]. Among Ni-Co-Mn-Sn family, Ni_{45}Co_{5}Mn_{38}Sn_{12} was reported to exhibit low thermal hysteresis (6 K) while also exhibiting one of the highest magnetization changes across the martensitic transformation (ΔM = 100 AM^{2}kg^{-1}) among the Ni-Co-Mn-Sn alloy family [17]. However, this large magnetization change, and MT occurs well above room temperature (∼400 K) [17], this is the major drawback of this composition, since from the practical point of view, the material should exhibit the structural transition around room temperature. In the present study, the magnetic and martensitic transformation behaviors of these alloys were investigated. These alloys show martensitic phase transformation closer to room temperature and large magnetization change (ΔM) accompanied with a moderate hysteresis. In the literature, generally the studies on magnetocaloric properties of magnetic shape memories (MSMAs) focus on the magnetic field-induced temperature change or indirectly measured entropy change. However, these are not enough to define the performance of MSMA refrigerant. In order to well indicate the performance of MSMA refrigerant, we may consider other parameters such as, the minimum magnetic field required to induce martensitic transition, the magnetic field to complete transition, and martensitic characteristics (transformation range, hysteresis). With this respect, we report here the temperature- and magnetic field-dependent martensitic transformation of representative polycrystalline magnetic shape memory alloys (MSMAs), Ni_{45}Co_{5}Mn_{38.5}Sn_{11.5} and Ni_{45}Co_{5}Mn_{38}Sn_{12} alloys. Fig. 1(a)-(b) illustrates the DSC curve for the Sn11.5 and Sn12 alloys, respectively. The large endothermic and exothermic peaks are due to reverse and forward transformations arising from the latent heat of the martensitic transformation. In addition to these peaks, there exists another peak in that is from the ferromagnetic/paramagnetic transition in the austenite phase around 360 K that is attributed to the Curie transition of austenite phase (T_{C}). The temperatures for the forward and reverse transformation are denoted as A_{t} (austenite start), A_{f} (austenite finish), M_{s} (martensite start), and M_{f} (martensite finish), A_{t} = 359 K, M_{s} = 341 K, M_{f} = 314 K for Sn11.5 and A_{t} = 263 K, M_{s} = 284 K, M_{f} = 250 K for Sn12 alloy, determined by double tangent method as shown in Fig. 1. The thermal hysteresis of Sn11.5 computed as A_{t}-M_{f} = 15 K and is shown to be symmetric i.e. A_{t}-M_{f} = A_{f}-M_{s} = 15 K which have less symmetric thermal hysteresis loop. The entropy change of the complete transition (ΔS_{T}) of both alloys were determined from DSC measurement as 33.8 J kg^{-1} K^{-1} and 21.5 J kg^{-1} K^{-1} for Sn11.5 and Sn12, respectively, derived from enthalpy of the samples.

### 2. Experimental details

The polycrystalline Ni_{45}Co_{5}Mn_{38.5}Sn_{11.5} and Ni_{45}Co_{5}Mn_{38}Sn_{12} alloys were arc-melted on a water-cooled copper hearth from high-purity elements with the appropriate amounts of Ni, Mn, Co, and Sn under argon protection, after purging the chamber several times under vacuum. After fabrication, these alloys were then sealed in quartz tubes under Ar and annealed at 1173 K for 24 h followed by water quenching [14,18]. To identify the level of homogeneity, wavelength-dispersive X-ray spectroscopy (WDS) was performed on 3 different grains within a single homogenized sample. Samples studied with the microprobe were encased in an epoxy mold, mechanically polished, and finished with colloidal silica and a felt cloth. The martensite transformation temperatures were measured using differential scanning calorimeter (DSC) at a rate of 10 °C/min for both heating and cooling. All thermo-magnetization curves were measured by field cooling (FC) and field heating (FH) protocols with a Quantum Design MPMS3 SQUID VSM. Employing a similar experimental sequence proposed in Ref. [20], magnetic field dependent magnetization data were obtained by discontinuous cooling to construct entropy change diagrams. Before each M-H measurement, the sample was first heated to austenite then decreased to the desired temperature. Next the field was applied from 0 to 7 T while magnetization was measured. At each step, the magnetization was measured isothermally.

### 3. Results and discussion

#### 3.1. Microstructure of NiCoMnSn

Table 1 lists the measured compositions (in at. %) of each alloy over 3 different locations and their standard deviations. The compositions (in at. %) of the prepared samples were identified and according to WDS, were close to nominal as Ni_{45.5}Co_{5.4}Mn_{38.9}Sn_{7.2} = 0.4 Sn_{12.3} ± 0.1 and Ni_{45.6}Co_{5.0}Mn_{38.0}Sn_{7.0} = 0.4 Sn_{12.7} ± 0.1. Hereafter these alloys will be abbreviated as Sn11.5 and Sn12 for simplicity.

#### 3.2. Transformation characteristics of NiCoMnSn

Fig. 1(a)-(b) illustrates the thermo-magnetization curves M(T) of Sn11.5 and Sn12, respectively, at several magnetic fields was measured in the temperature range 200 K ≤ T ≤ 400 K. Fig. 2(a)-(d) show M(T) curves under 0.05 T for the Sn11.5 alloy and Sn12 alloy, respectively, while cooling the Sn11.5 alloy (Fig. 2(a)) magnetization increases abruptly at ≈365 K (T_{C}). Moreover, the magnetization abruptly decreases at ≈330 K during cooling and an abrupt increase at ≈335 K during heating, due to martensitic and reverse transformations, respectively. Similarly, while cooling the Sn12 alloy (Fig. 2(b)) magnetization abruptly increase at ≈370 K at T_{C}. In this case, martensitic and reverse transformations are observed at ≈265 K and ≈275 K during cooling and heating, respectively. It is clear that the martensitic transformation temperatures decrease as the Sn content increases. It is stated that the substitution of Co for Ni is very effective in increasing T_{C} and decreasing T_{M} (temperature where the martensitic transformation occurs) [13,21], however, in that case since Co content is almost the same, thus the T_{C} of both alloys is almost the same. The hysteresis between FC – FH curves across the martensitic transition confirms the first order nature of this transition, which is consistent with the result of the DSC measurements. The hysteresis determined from the 0.05 T magnetic field M(T) curves are 6 K and 8.5 K for Sn11.5 and Sn12 alloys, respectively. However, here exists a discrepancy between these DSC and M(T) data that may have resulted from different specimen sizes, slightly different compositions, or different heating and cooling rates. The M(T) curves under higher magnetic field reveals similar behavior to 0.05 T data, accompanied with the transition temperatures shifted to lower temperatures (Fig. 2(c)-(d)). Since an external magnetic field stabilizes the phase with higher magnetization (austenite phase in this study), the transition shifted to lower temperatures. The hysteresis of the both alloys is almost independent of magnetic field. The characteristic transition temperatures of Sn11.5 and Sn12 determined from the 50 mT magnetic field M(T) curve are given in Table 2. The martensitic transformation temperatures under magnetic field of 1 T, 3 T, 5 T, and 7 T, are plotted in Fig. 2(e)-(f) for Sn11.5 alloy and Sn12 alloy, respectively. Fig. 2(e)-(f) illustrate the martensitic

### Table 1

| Composition of the phases determined with wavelength dispersive spectroscopy (WDS) in the Ni_{45}Co_{5}Mn_{38}Sn_{12} alloy (x = 11.5 and 12). |
|---------------------------------|----------------|----------------|----------------|----------------|
| Sample (nominal at. %)          | Ni (at. %)     | Co (at. %)     | Mn (at. %)     | Sn (at. %)     |
| Ni_{45}Co_{5}Mn_{38}Sn_{11.5}   | 45.5 ± 0.5     | 5.0 ± 0.1      | 37.2 ± 0.4     | 12.3 ± 0.1     |
| Ni_{45}Co_{5}Mn_{38}Sn_{12}     | 45.2 ± 0.5     | 5.1 ± 0.1      | 37.0 ± 0.4     | 12.7 ± 0.1     |
transformation temperatures decrease with application of magnetic field. The characteristic temperatures, $M_f$ and $M_s$, linearly decrease with the field at a rate $dM_f/dH = -1.93 \, \text{K T}^{-1}$, $dM_s/dH = -1.60 \, \text{K T}^{-1}$ for the Sn11.5 and $dM_f/dH = -3.44 \, \text{K T}^{-1}$, $dM_s/dH = -3.15 \, \text{K T}^{-1}$ for the Sn12. The decrease of the transformation temperatures is line with the literature but lower than Co rich samples such as (∼5.7 K T$^{-1}$ for Ni$_45$Co$_{4.9}$Mn$_{37.2}$Sn$_{12.9}$), since it is stated that this ratio is related with Co content [13]. We observed transformation ranges ($M_s - M_f$ and $A_f - A_s$) are around 15 K and 17 K for the Sn11.5 and Sn12, respectively. In order to further investigate the performance of these samples, we would like introduce some other martensitic transformation characteristics determined using thermo-magnetization results under 0.05 T given in Table 2, which will be used in later in text. The temperature where the martensitic transformation occurs is defined as $T_M = (M_s + A_f)/2$. $\Delta T_{bys}$ is defined as thermal hysteresis of transformation which is equal to $[(A_f + A_s)-(M_s + M_f)]/2$. Temperature interval to complete the transformation is defined as $\Delta T_{comp} = (A_f - M_f)$ [6,7].

Fig. 3 (a)-(b) illustrate the magnetic field dependent magnetization curves at several temperatures in increasing and decreasing fields up to 7 T. Because of the fact that magnetization isothermal curves have been planned to pick up the full irreversible effect; we cooled the sample well above martensitic transition then decreased to desired temperature before starting every isothermal M(H) curve to avoid the presence of “ghost peaks” in the $\Delta S(T)$ curve [20,22]. In Fig. 3 (a) the magnetization curves show a gradual transition to from martensite to austenite alloy in the temperature interval of 318 K–335 K; the curves clearly
Martensitic transformation characteristics of Sn11.5 and Sn12 samples determined from the thermomagnetization results under 0.05 T illustrated in Fig. 2 (a) and (b).

<table>
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<th></th>
<th>(M_f (K))</th>
<th>(M_s (K))</th>
<th>(A_s (K))</th>
<th>(A_f (K))</th>
<th>(A_s M_f (K))</th>
<th>(dA_f/dH (K/T))</th>
<th>(dA_s/dH (K/T))</th>
<th>(\Delta T_{comp} (K))</th>
<th>(\Delta T_{comp} (K))</th>
<th>(T_m (K))</th>
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<td>344</td>
<td>6</td>
<td>−1.88</td>
<td>−1.64</td>
<td>6</td>
<td>23</td>
<td>341</td>
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<tr>
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<td>251</td>
<td>259.5</td>
<td>285</td>
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<td>−3.57</td>
<td>8.5</td>
<td>34</td>
<td>280.5</td>
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Table 2

The magnetic field induced transformation (MFIT) from martensite to austenite in Sn11.5 alloy [18]. The Sn11.5 alloy is not saturated even under the magnetic field of 7 T. The highest magnetic field hysteresis of \(\approx 2\) T is observed at each temperature. In the case of Sn12 alloy (Fig. 3(b)) almost fully reversible MFIT is achieved in a wider temperature interval of 229 K–268 K. Contrary to the Sn11.5 alloy, the Sn12 alloy almost saturated under 7 T. For the both alloys metamagnetic shape memory effect can be expected as result of MFIT. The magnetization curves show the martensite and austenite phases are magnetically soft in both alloys and the martensite phase has a significantly lower magnetization than the austenite phase in all magnetic field values.

Moreover, in a recent paper, a new methodology considering the magnetic field level requirements \(H_{req}\) and \(H_{comp}\) were developed [6,7,23]. \(H_{req}\) is defined as the minimum required magnetic field to induce the transformation and \(H_{comp}\) is defined the magnetic field to induce a complete structural transformation. In order to investigate whether 7 T is enough to induce the transformation and to complete structural transformation, we implemented this methodology to our data. These values calculated assuming that alloys were held at a constant temperature equal to the martensite finish temperature, \(M_f\). The magnetic field values were computed as \(H_{req} = (A_s - M_f)/(dA_s/dH)\) and \(H_{comp} = (A_f - M_f)/(dA_f/dH)\), where \(dA_s/dH\) is the austenite start temperature's sensitivity to external field and \(dA_f/dH\) is the austenite finish temperature's sensitivity to external field. The transformation temperatures and Clausius-Clapeyron (CC) slopes are given Table 2 for each alloys. Accordingly, \(H_{req}\) and \(H_{comp}\) values for Sn11.5 are 3.8 T and 14.0 T and for Sn12 sample 2.3 T and 9.5 T. Namely, 7 T is adequate to induce the transition however it is not adequate to complete the whole transition. These results indicate that the Sn11.5 and Sn12 alloys do not exhibit good reversibility with field cycling and that very large magnetic fields are needed to generate a reversible martensitic transformation.

Furthermore, we have estimated the field induced entropy change (\(\Delta S\)) around the martensitic transformation for both samples using the relationship [12],

\[
\Delta S_m(T_{c}, 0 \rightarrow H) = \frac{1}{\Delta T_m} \left[ \int_{0}^{H} M_{H_{1+}} dH - \int_{0}^{H} M_{H_0} dH \right]
\]

where, numerically from the M(H) isotherms for both increasing and decreasing magnetic field presented in Fig. 4 (a)-(b), for Sn11.5 alloy and Sn12 alloy, respectively. In order to depict the effect of increasing and decreasing the magnetic field, the entropy change was drawn conversely for decreasing field in presented in Fig. 4 (a)-(b). Both alloys have positive (i.e. inverse) entropy change and their maximums were found to be 21.6 J kg\(^{-1}\) K\(^{-1}\) and 15.8 J kg\(^{-1}\) K\(^{-1}\), for Sn11.5 and Sn12 alloy, respectively, under 7 T. Both alloys seem to have conventional entropy change close to the \(T_C\), which is expected to be much smaller than inverse entropy change, but it was out of the out of the (M(H) measurement temperature range. The fact that there is no sharp peak on \(\Delta S-T\) curve confirms that our \(\Delta S_m\) values are reliable [24]. The width of \(\Delta S_m - T\) curves (Fig. 4 (a)-(b)) increases with increasing magnetic field thereby leading to an increase in the refrigerant capacity. Noteworthy, the plateau covers a broad temperature interval from 338 K to 320 K for Sn11.5 and 274 K–244 K for Sn12 under 7 T, leads to an effective operating temperature window of 18 K and 30 K (determined from the full width half maximum of the \(\Delta S_m\) peak). This is quite beneficial to the magnetic refrigeration. The increment for the width of \(\Delta S_m - T\) is higher for Sn12 sample, since its characteristic temperature shift (e.g Sn12 \(dM_f/dH = −3.44\) K T\(^{-1}\)) with the applied magnetic field is higher than Sn11.5 sample \(dM_f/dH = −1.93\) K T\(^{-1}\)). From point of view reversibility of magnetocaloric effect which is related to the hysteresis of the transition and to sensitivity of transition temperatures to the applied magnetic field \(dH/dT\). Higher sensitivity of martensitic transformation temperatures with applied magnetic field will yield a greater level of reversibility on field cycling over a wider range of temperatures. The transition entropy change \(\Delta S_m\) is 33.8 J/kg K and 21.5 J/kg K, however calculated entropy change is 21.6 J kg\(^{-1}\) K\(^{-1}\) and 15.8 J kg\(^{-1}\) K\(^{-1}\) for Sn11.5 and Sn12 sample, respectively. In a further aspect, we may conclude that the shift of the transition temperature in magnetic fields is not enough to completely transform the samples in 7 T. The situation is also known as dilemma of inverse magnetocaloric materials. Namely, a large magnetization change deteriorates the achievable MCE, meanwhile a large \(\Delta M\) results higher shift of the transition temperatures [25]. This dilemma is consequence of the competition between the lattice and the magnetic contribution of the entropy change [25,26]. The \(\Delta S_m\) values of the present alloys (15.9 J/kg K for Sn11.5 and 11.2 J/kg K for Sn12 under 5 T) are lower than the Ni\(_{45.8}\)Fe\(_{4.2}\)Mn\(_{38.0}\)Sn\(_{12.0}\) (33.8 J/kg K under 5 T, \(\Delta T_{hys} = 17.5\) K, \(|A_s/\Delta H| = 2.4\) K T\(^{-1}\)) [27], however are higher than Ni\(_{45.8}\)Co\(_{4.2}\)Mn\(_{38.0}\)Sn\(_{12.0}\) (14.9 J/kg K under 5 T, \(\Delta T_{hys} = 8\) K, \(|A_s/\Delta H| = 8.1\) K T\(^{-1}\)) [24]. Albeit, from perspective of

![Fig. 3. M(H) curves for Sn11.5 (a) and Sn12 (b).](image-url)
reversible $\Delta S_m$, Ni$_{40}$Co$_{10}$Mn$_{40}$Sn$_{10}$ will show the best performance since it has highest sensitivity of martensitic transformation temperatures with applied magnetic field and lower hysteresis.

Finally, we calculated the adiabatic temperature change ($\Delta T_{ad}$), it is reported that the general method to compute $\Delta T_{ad}$ which is $\Delta T_{ad} \approx -\frac{T}{C} \Delta S_m$ is not valid for martensitic transition since it is derived using a single free energy model [19]. However, for the first-order structural phase transformations (martensitic transition), one should take into account the change in free energy of one phase to another. Instead it is suggested that, $\Delta T_{ad}$ is calculated as follows [22],

$$\Delta T_{ad} = \frac{\Delta S_m - \Delta S_m^\text{martensite}}{\Delta S_m + \Delta S_m^\text{martensite}}$$

(2)

where $\Delta S_m = (dM_f/dH) dH$, $dM_f/dH$ is the magnetic field sensitivity of the $M_f$ temperature ($dA_f/dH = -1.64$ K T$^{-1}$ for Sn11.5 and $dA_f/dH = -3.57$ K T$^{-1}$ for Sn12), $\Delta S_m$ is the magnetic field induced entropy change given in Eq. (1), and $C_{\text{martensite}}$ is the specific heat capacity of the martensite phase measured at temperature $M_f$. This equation is valid for first-order systems and is limited to cases when the field-induced transition temperature shift is smaller than the transformation range as in that study. We assumed $C_m$ of martensite will be approximated to $\approx 500$ J kg$^{-1}$ K$^{-1}$ as for Ni$_{43}$Mn$_{42}$Co$_{4}$Sn$_{11}$ [6]. Therefore, the maximum adiabatic temperature changes of Sn11.5 and Sn12 are computed $-6.9$ K and $7.4$ K for 7 T as illustrated in Fig. 5. In order to compare them with literature, we computed $\Delta T_{ad} = 1.8$ K and $-2.3$ K, respectively, for 1.8 T. These values are higher than reported Ni$_{50}$Mn$_{30}$Ga$_{20} + 0.8$ K, Ni$_{45}$Co$_{5}$Mn$_{30}$Ga$_{20} - 1.45$ K, Ni$_{45}$Co$_{3}$Mn$_{31}$Ga$_{19} - 1.5$ K, and similar to Ni$_{41}$Co$_{9}$Mn$_{32}$Ga$_{16}$In$_{2} - 2.3$ K [22].

4. Conclusions

In conclusion, both alloys are paramagnetic at higher temperatures and while cooling both alloys become ferromagnetic in austenite at $T_C$. By further cooling, the alloys exhibit an austenite to martensite phase transformation. The temperature dependent magnetization results revealed austenite phase has higher magnetization. Since application of magnetic field stabilizes the austenite phase, martensitic transformation temperatures shifted to lower temperatures. Namely, the martensite start temperature $M_s$ linearly decreases with the field at a rate $dM_s/dH = -1.93$ K T$^{-1}$ and -3.44 K T$^{-1}$ for the Sn11.5 and Sn12 alloy, respectively. Larger $\Delta M/\Delta S$ value is found especially in Sn12 alloy, thus a significant magnetic-field-induced decrease of martensitic transformations temperatures is observed and a fully reversible magnetic-field-induced structural transformation (MFIT) can also achieved. Meanwhile, Sn11.5 has higher entropy change since it is first order transition occurs in narrower temperature interval with a smaller hysteresis loss. There exists very little difference in composition between both compounds, however, the transition temperatures are significantly different then each other. This concludes that even little chemical compositions differences lead to significant differences in magnetic and magnetocaloric properties. The reason of this striking effect might be
due to the difference in nucleation and elastic energy storage [6], since the larger transformation range was an indication of the difficulty for transformation phase front motion [6]. Energy dissipated at the martensitic transition diminishes the reversible magnetocaloric effect (MCE) which has a direct impact on the efficiency of magnetic refrigerant. The entropy change measured with DSC and magnetic measurement differs. This indicates the transformation entropy change is largely function of magnetic field, and entropy production from hysteresis has a remarkable detrimental effect on the entropy change of martensitic transformation (MT). Therefore, managing the hysteresis is one of the key issues while working on magnetocaloric effect material.

In this study, the adiabatic temperature changes were computed using a methodology developed earlier as approximately \(-1.8\, \text{K}\) and \(-2.3\, \text{K}\) for Sn11.5 and Sn12, respectively, during application of 1.8 T. The accessibility of magnetic field induced entropy change was investigated by computing the magnetic field levels needed to repeatedly generate the effect, \(H_{\text{req}}\) and \(H_{\text{comp}}\). It was shown that the applied magnetic field of 7 T was able to induce the transition however was not able complete the transition. Therefore, that very large magnetic field levels are required to generate a reversible magnetocaloric effect in these alloys, owed to the inadequacies of the magnetic field sensitivities of the martensitic transformation temperatures.

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