Structure, Magnetocaloric Effect and Critical Behaviour in Ni$_{50}$Mn$_{30}$(Sn,In)$_{20}$ Heusler Alloys

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Abstract
A detailed investigation of structure, critical behaviour and magnetocaloric properties of Ni$_{50}$Mn$_{30}$Sn$_{20}$ (Sn$_{20}$) and Ni$_{50}$Mn$_{30}$In$_{20}$ (In$_{20}$) alloys has been investigated by means of X-ray diffraction and magnetic measurements. Ni$_{50}$Mn$_{30}$Sn$_{20}$ alloy shows a cubic austenite L$_{21}$ structure and undergoes a second order magnetic transition at a Curie temperature of $T^{A}_{c1}$(Sn$_{20}$) = 333 K. However, the Ni$_{50}$Mn$_{30}$In$_{20}$ alloy exhibits a mixture of cubic L$_{21}$ and B$_2$ austenite structures having Curie temperatures of $T^{A}_{c2}$(In$_{20}$) = 285 K and $T^{*}_{c}$(In$_{20}$) = 330 K, respectively. The modified Arrott plots, Kouvel-Fisher curves and critical isotherm analysis have been used to estimate the critical exponents ($\beta$, $\gamma$ and $\delta$) around the Curie temperature. For Sn$_{20}$ alloy, the reliable exponents are consistent with the mean field model, revealing long-range ferromagnetic interactions. Nevertheless, the critical exponents of In$_{20}$ alloy around 330 K cannot be arranged into any of the universality classes of well-known classical standard models. The maximum entropy change under 5 T of Sn$_{20}$ ($\Delta S^{\text{max}}_M = 2.43 \frac{J}{kgK}$) is slightly higher than that of In$_{20}$ ($\Delta S^{\text{max}}_M = 2.05 \frac{J}{kgK}$). The experimental results of entropy changes are in good agreement with those calculated using Landau theory.

Keywords NiMn-based alloys · Magnetic transition · Critical behaviour · Magnetocaloric effect · Landau theory

1 Introduction

Heusler alloys have attracted considerable attention due to their several technological applications such as sensors, spintronic devices and magnetic refrigeration [1–3]. Several experimental [4, 5] and theoretical [6, 7] studies have been carried out to investigate the properties of Heusler alloys. Off-stoichiometric NiMn-based ferromagnetic alloys exhibit an exchange bias phenomena (EB) [8], magnetoresistance [9, 10], magnetocaloric effect (MCE) [11, 12], magnetic super-elasticity [13] and piezoresistance [14]. It has been reported that Ni$_{50}$Mn$_{50-x}$Sn$_x$ series crystallize in a cubic austenite L$_2$ structure, space group $Fm\bar{3}m$, for $x \geq 15$ and a martensite structure for $x < 15$ [15, 16]. An extended annealing of Ni$_{50}$Mn$_{39}$Sn$_{11}$ and Ni$_{50}$Mn$_{37}$Sn$_{13}$ alloys at 773 K involves a phase decomposition of the eutectoid-type structure into Ni$_{54}$Mn$_{45}$Sn$_{1}$ and Ni$_{50}$Mn$_{10}$Sn$_{20}$. Wherein, the Curie temperature was about 350 K [17]. The standard enthalpy of formation, $\Delta f^\circ H$, of Ni$_{50.6}$Mn$_{29.6}$Sn$_{19.8}$ alloy was $-21.0$ kJ/mol of atom [18]. A linear shift of the reverse and forward martensitic transition temperatures in Ni$_{46.4}$Mn$_{32.8}$Sn$_{20.8}$ alloy occurs by high magnetic field application up to 14 T [19]. Moreover, a spin glass behaviour was found in Ni$_{50}$Mn$_{30}$Sn$_{20}$ alloy [20]. For Ni-Mn-In alloys, the structural transition was observed in Mn-rich composition where both the austenite and martensite phases exhibit a ferromagnetic order [21, 22]. Krenke et al. reported that Ni$_{50}$Mn$_{50-x}$In$_x$ alloys with 0.16 $\leq x \leq 0.05$ show a first-order structural transformation, and the Curie temperature decreases from 310 K for $x = 20$ to 290 K for $x = 25$ [23].
Furthermore, the martensitic transition temperature MT is influenced by change in elemental composition through the change of valence electron concentration per atom (e/a).

Depending on the nature of the magnetic transition, the magnetocaloric effect can be categorised. For the second-order magnetic transition, the resulted negative entropy change around the Curie temperature of the austenite structure, \( T_A^{\text{c}} \), produces a direct MCE. However, an inverse magnetocaloric effect, IMCE, is engendered with a positive value of the entropy change around the martensitic transformation, MT. The existence of a universal behaviour in materials can be investigated through the critical exponents (\( \beta \), \( \gamma \) and \( \delta \)) associated with the phase transitions. By using several models [24] such as the mean field model associated with long-range mean field theory where \( \beta = 0.5 \) and \( \gamma = 1 \), short-range interactions as specified by the Heisenberg model with \( \beta = 0.365 \) and \( \gamma = 1.336 \), 3D-ising model with \( \beta = 0.325 \) and \( \gamma = 1.241 \), and tricritical mean field model with \( \beta = 0.25 \) and \( \gamma = 1 \), the critical exponents \( \beta \), \( \gamma \) and \( \delta \) can be estimated from the isothermal magnetization curves, \( M(H) \). The exponent \( \beta \), which is linked to the evolution of the spontaneous magnetization with respect to the temperature (\( M_s \propto (T - T_c)^\beta \)), describes the growth of the ordered moment below \( T_c \); \( \gamma \) is related to the temperature dependence of the initial magnetic susceptibility (\( \chi_0^{-1} \propto (T - T_c)^\gamma \)). It defines the divergence of \( \chi_0 \) at \( T_c \), and \( \delta \) is associated to the critical isothermal magnetization. It describes the curvature of the isothermal magnetization curves \( M(H) \) at \( T_c \). The mechanisms of magnetic phase transitions are to some extent vague due to the influence of many parameters such as the preparation method, the alloy composition and the thermal history. Consequently, the aim of the present work is to investigate the magnetocaloric effect and the critical behaviour near the Curie temperature in the melt spun Ni_{50}Mn_{30}(Sn, In)_{20} ribbons.

2 Experimental Details

Polycrystalline alloys with nominal compositions of Ni_{50}Mn_{30}Sn_{20} and Ni_{50}Mn_{30}In_{20}, henceforth called Sn20 and In20, respectively, have been prepared by arc melting. 

![Fig. 1 Rietveld refinement of the XRD pattern of Sn20 alloy](image1)

![Fig. 2 Rietveld refinement of the XRD pattern of In20 alloy](image2)
high-purity elements Ni, Mn, Sn and In (99.9%) under an argon atmosphere. The ingots were re-melted five times to ensure homogeneity. Sn20 and In20 alloy ribbons have been elaborated by melt spinning onto a surface of copper wheel rotating at a linear speed of 40 m/s under argon atmosphere. According to the EDX analysis, the ribbons compositions are Ni49.6Mn28.9Sn21.5 and Ni54.9Mn26.6In18.5 for Sn20 and In20, respectively. The crystal structure was checked by X-ray diffraction (XRD) on a Bruker D8 Advance diffractometer using Cu-Kα radiation (λCu = 0.154056 nm) in a θ − 2θ Bragg Brentano geometry. The XRD patterns were computer refined using the MAUD program which is based on the Rietveld method [25]. Magnetization measurements were carried out in a superconducting quantum interference device (SQUID) magnetometer and a BS2 magnetometer developed at the Néel Institute under an applied magnetic field of 0.05 T. The critical exponents and the magnetic entropy changes around the Curie temperature have been determined from the isothermal magnetization measurements as a function of the applied magnetic field.

3 Results and Discussion

Figures 1 and 2 show the Rietveld refinement of the XRD patterns of Sn20 and In20 alloys, respectively. The XRD patterns exhibit thin and intense peak characteristic of a fully crystalline microstructure due to the fast crystallization and growth kinetics of the samples during the melt spinning process. One observes also that the peak intensities of Sn20 are slightly high than those of In20. The high order of L21 austenite in both alloys is confirmed by the (111), (311) and (331) reflections. In addition, Sn20 is a textured alloy as shown by (311) reflection intensity. Such preferential orientation might be due to fact that during the rapid solidification, the heat removal induces directional growth of the crystalline phase. The importance of (111) and (311) peak intensities compared to those obtained in the Ni50Mn30Sn20 Heusler alloy prepared by arc melting followed by annealing at 1000 °C for 24 h [20] confirms the effect of the preparation method on the structural properties. Usually, the non-stoichiometry of the alloy with a low content of Z (In, Sn) element gives rise to weak minor peaks of the ordered structure in Heusler alloys. While the Sn20 alloy shows a single austenite L21 structure (lattice parameter a = 0.5994(3) nm, space group Fm3m), the In20 alloy exhibits a mixture of an ordered L21 phase (volume fraction ~ 85%, and lattice parameter a = 0.6088(3) nm), and a partially ordered B2 phase (space group Pm3m, volume fraction ~ 15% and lattice parameter a = 0.3011(2) nm). The formation of B2 structure can be linked to the degree of atomic order.

The temperature dependence of the magnetization, M(T), recorded at 0.05 T for both Sn20 and In20 alloys, is displayed in Fig. 3. The Curie temperature was inferred from the minimum in dM/dT versus temperature curve as shown by the inset in Fig. 3. For the Sn20 alloy, the ferromagnetic to paramagnetic transition $T^p_{c}(\text{Sn20})$ occurs at 333 K, whereas the
In20 alloy displays two magnetic transition temperatures at 285 K and 330 K that can be attributed to $L_2$ and $B_2$ structures, respectively. This result agrees well with the XRD one and confirms thus the biphasic character of In20 alloy. The magnetic transitions of both phases are different from those reported in the literature \[26–28\]. Those discrepancies might be ascribed to the composition change and/or the preparation conditions. For example, the obtained lower Curie temperature ($T_c \approx 222$ K) for the austenite phase, by R.C. Bhatt et al. \[20\], has been attributed to the deficiency of Sn atoms in the system.

The isothermal magnetization curves of In20 and Sn20 alloys, $M(H)$, around the Curie temperature are presented in Fig. 4 as a function of the applied magnetic field up to 6 T. For both alloys, the magnetization does not saturate even at 5 T. In the magnetic system, the MCE is related to the magnetic entropy change, $\Delta S_M$ which might be induced by an applied magnetic field that leads to the change in the magnetic order. The magnetic entropy change $\Delta S_M$ can be deduced from the isothermal magnetization, $M(H)$, curves via the following Maxwell relation:

$$\Delta S_M(T, H) = S_M(T, H_2) - S_M(T, H_1) = \int_{H_1}^{H_2} \left( \frac{\partial M}{\partial T} \right)_T \, dH$$ \hspace{1cm} (1)

where $H_1$ and $H_2$ are the applied magnetic fields with $H_1 < H_2$ and $\Delta H = H_2 - H_1$. $\Delta S_M$ can be calculated by integrating the $M(H)$ curves at small discrete magnetic field and temperature intervals, and can be approximated by the succeeding equation:

$$\Delta S_M(T, M) = \sum_i \frac{M_{i+1}(T_{i+1}, H_{i+1}) - M_i(T_{i+1}, H_{i+1})}{T_{i+1} - T_i} \Delta H$$ \hspace{1cm} (2)

with $M_i$ and $M_{i+1}$ are the experimental data of the magnetization at $T_i$ and $T_{i+1}$, respectively, under the magnetic field $H_i$. The variation of $\Delta S_M$ as a function of the temperature is shown in Fig. 5. The maximum magnetic entropy change, $\Delta S_{M}^{\text{max}}$, increases with increasing applied magnetic field and reaches 2.43 J/kg K for Sn20 and 2.05 J/kg K for In20. One notes that $\Delta S_{M}^{\text{max}}$ value of Sn20 alloy is ~16% higher than that obtained in Ni$_{50}$Mn$_{35}$Sn$_{15}$ alloy prepared in the same conditions \[29\].

The relative cooling power, $RCP$, associated with the magnetic entropy change is an important feature to evaluate the magnetocaloric efficacy of materials. The relative cooling power is given by the following equation:

$$RCP = -\int_{T_1}^{T_2} \Delta S_M(T) \, dT$$ \hspace{1cm} (3)

where $T_1$ and $T_2$ temperatures are defined by the full width at half maximum of $\Delta S_M(T)$ peak. For Sn20 and In20 ribbons, the calculated $RCP$ values are of about 172.88 J/kg and 240 J/kg, respectively, for an applied magnetic field of 5 T. The $RCP$ value of Sn20 is much higher than that obtained for the same composition by Bhatt et al. ($RCP = 54.6$ J/kg) under a magnetic field of 3 T \[20\].

Amaral et al. proposed a theoretical model based on the contribution of the magneto-elastic interactions of electrons \[30\] and on Landau theory in the critical region \[31\].
energy ($G$) is developed as a function of the magnetization ($M$):

$$G(M, T) = G_0 + \frac{a(T)}{2} M^2 + \frac{b(T)}{4} M^4 + \frac{c(T)}{6} M^6 + \cdots - H M$$

For the equilibrium condition, $\partial G/\partial M = 0$, the total magnetization in the critical region is given by the following relationship:

For Fig. 6, the superposition of experimental $-\Delta S_m(T)$ curves and calculated ones under applied fields up to 5 T, and the corresponding Landau’s coefficient $a(T)$, $b(T)$ and $c(T)$, respectively.

For Fig. 7, modified Arrott plots for Sn20 and In20 alloys: isotherms of $M^{1/\beta}$ against $(H/M)^{1/\gamma}$ around $T_c$ with the mean field model.
\( H = a(T)M + b(T)M^3 + c(T)M^5 \)  

The Landau’s coefficient \( a(T), b(T) \) and \( c(T) \) are determined from the polynomial fit of the experimental \( M(H) \) data according to Eq. 5. The variation of \( a(T) \) shows a minimum around \( T^A_c \), and \( b(T^A_c) = 0 \) proves the second-order character of the magnetic transition. The derivative of the free energy with respect to the temperature gives a theoretical model of the magnetic entropy change:

\[
-\Delta S_M(T, H) = \frac{dG}{dT} \bigg|_H = \frac{a'(T)}{2} M^2 + \frac{b'(T)}{4} M^4 \\
+ \frac{c'(T)}{6} M^6
\]  

where \( a'(T), b'(T) \) and \( c'(T) \) are the derivative of Landau’s coefficients with respect to the temperature. The calculated \(-\Delta S_M\) curves are in a good agreement with the experimental results (Fig. 6). Thus, the calculated Landau’s coefficients are accurate.

In order to understand the interaction type of the magnetic moments and the universality class of Sn20 and In20 ribbons, the critical exponents have been analysed through different techniques such as the modified Arrott plots (MAP) [32], Kouvel-Fisher plots \((K-F)\) [33, 34] and critical isotherm (CI). Hence, the critical exponents \( \beta, \gamma \) and \( \delta \) can be determined using Equations 7–9:

\[
M_S(T) = M_0(-\varepsilon)^\beta; \quad \varepsilon < 0, \quad T < T_c
\]  

\[
\chi_0^{-1}(T) = \left( \frac{h_0}{M_0} \right)\varepsilon^\gamma; \quad \varepsilon > 0, \quad T > T_c
\]  

\[
M = DH^{1/6}; \quad \varepsilon = 0, \quad T = T_c
\]

Table 1 Critical exponent values of Sn20 and In20 alloys with those of the theoretical models

| Sample   | Technique | \( \beta \)       | \( \gamma \)       | \( \delta \)       | Ref.        |
|----------|-----------|-------------------|-------------------|-------------------|------------|
| Sn20     | MAPs      | 0.421 ± 0.076     | 1.078 ± 0.142     | 3.560 ± 0.0661    | This work  |
|          | K-F       | 0.421 ± 0.076     | 1.171 ± 0.118     | –                 |            |
|          | CI        | 0.349 ± 0.040     | 3.710 ± 0.007     | –                 |            |
| In20     | MAP       | 0.461 ± 0.032     | 1.537 ± 0.174     | –                 | This Work  |
|          | K-F       | 0.494 ± 0.045     | 1.170 ± 0.206     | –                 |            |
|          | CI        | 0.469 ± 0.050     | 3.439 ± 0.007     | –                 |            |
|          | MAP       | 0.434 ± 0.037     | 2.698 ± 0.453     | –                 |            |
|          | K-F       | 0.466 ± 0.050     | 0.737 ± 0.490     | –                 |            |
|          | CI        | 0.702 ± 0.043     | 2.645 ± 0.003     | –                 |            |
| Mean field model | 0.5 | 1.0 | 3.0 | [24] |
| 3D-Heisenberg model | 0.365 | 1.336 | 4.80 | |
| 3D-Ising model | 0.325 | 1.241 | 4.82 | |
| Tricritical mean field | 0.25 | 1.0 | 5.0 | |

Fig. 8 Relative slope (RS) as a function of temperature (RS = \( S(T)/S(T_c) \)) deduced from different models
Fig. 9 Temperature dependence of spontaneous magnetization $M_s(T, 0)$ and the inverse of initial susceptibility $\chi_0^{-1}(T, 0)$ around $T_c^A$.

Fig. 10 Kouvel-Fisher plots for the spontaneous magnetization and the inverse of initial susceptibility around $T_c^A$.

Fig. 11 Critical isotherms with the log-log plot shown in insets.

Fig. 12 Scaling plots indicating universal curves above and below Curie temperatures for Sn20 and In20 alloys.
structures, the mean field model is the best model that describes both In20 and Sn20 ribbons since RS are much closer to 1. By fitting the spontaneous magnetization $M_s(T, 0)$ and the inverse of the initial susceptibility $\chi^{-1}(T, 0)$ curves using Eqs. 7 and 8, respectively, the new values of $\beta$, $\gamma$ and $T^*_{Ac}$ (Table 1) can be determined (Fig. 9). One notes that the $\beta$ and $\gamma$ values are close to the mean field model with a long-range order of magnetic interactions, and the Curie temperatures are close to those obtained from the $M(T)$ curves.

The Kouvel-Fisher plots of $M_s(T, 0)$ and $\chi^{-1}(T, 0)$ against the temperature (Fig. 10) show straight lines with slopes $1/\beta$ and $1/\gamma$, respectively. Moreover, the $\beta$ and $\gamma$ exponent values are interconnected to the third exponent $\delta$ via the Widom scaling relation ($\delta = 1 + (\gamma/\beta)$) [35]. The calculated $\delta$ values (Table 1) are comparable to those estimated from the CI curves (Fig. 11). The validity of the critical exponents and the Curie temperature values are confirmed by the scaling hypothesis [36] through the following equation:

$$ M(H, \varepsilon) = \varepsilon^\beta f_+ (H/\varepsilon^{\beta+\gamma}) $$

The regular analytic functions $f_+$ and $f_-$ are undertaken for $T > T^*_c$ and $T < T^*_c$, respectively. The $M_s(T, 0)$ as a function of $H/\varepsilon^{(\beta+\gamma)}$ are plotted in the vicinity of the Curie temperature (Fig. 12). The presence of two distinct branches below and above $T^*_c$ confirms the fact that the predicted critical exponents and Curie temperatures are reasonably accurate.

The modified Arrott plots (not shown here) of the disordered cubic $B_2$ structure around $T^*_{Ac}(In20) = 330$ K show curved lines rather than straight lines, and the linear fit at high field region has negative intercept. Accordingly, those models are unable to describe the system. The exponents’ values were obtained from the optimum fitting of $M_s(T, 0)$ and $\chi^{-1}(T, 0)$ curves using Eqs. (7) and (8), respectively, which were determined from the high field region of $M_s(T, 0)$ against $(\mu_0H/M)^{1/\gamma}$ plot. This process was repeated several times until the convergence. The nonlinear fit to the $M_s(T, 0)$ and $\chi^{-1}(T, 0)$ plots gives $\beta = 0.434 \pm 0.037$, $\gamma = 0.737 \pm 0.490$ and $T^*_c = (331.328 \pm 0.919)$ K values (Fig. 13). As shown in Fig. 14, the modified Arrott plot presents almost parallel isotherms.
near $T_c^*$ by using the unconventional exponents. The Kouvel-Fisher plots are presented in Fig. 15, and the convergence was reached with nearby values to those obtained from the modified Arrott plots with $\beta = 0.466 \pm 0.050$, $\gamma = 0.702 \pm 0.043$ and $T_c^* = (331.420 \pm 1.142)$ $K$. The validity of those unconventional critical exponents and $T_c^*$ values were confirmed by the scaling hypothesis as shown in Fig. 16. The third exponent $\delta$ was determined experimentally from the critical curve $M = f (H, T_c^*)$ (Fig. 17), and analytically by the Widom scaling relation. The experimental and calculated $\delta$ values were $\delta = 2.645 \pm 0.003$ and $\delta_{\text{cal}} = 2.698 \pm 0.453$, respectively. The critical exponents of Sn20 and In20 alloys as well as those of the different models are reported in Table 1. One observes that $\beta$ has smaller values indicating a faster growth of the ordered moment. The initial susceptibility has a softer divergence at $T_c^*(\text{Sn}20)$ and $T_c^*(\text{In}20)$, and sharper divergence at $T_c^*(\text{In}20)$. The $\delta$ values reflect the faster saturation of $M(H)$ curves at $T_c^*$, the less curvature and slower saturation at $T_c^*(\text{In}20)$.

4 Conclusion

Structure, magnetocaloric effect and critical behaviour of the Ni$_{29}$Mn$_{30}$(Sn,In)$_{20}$ alloys were studied. Sn20 ribbons exhibit a single cubic $L_2_1$ structure and undergo a second-order magnetic transition at 333 K. The presence of $L_2_1$ and $B_2$ structures in the In20 ribbons is confirmed by the existence of two Curie temperatures ($T_c^*(\text{In}20) = 285 K$ and $T_c^*(\text{In}20) = 330 K$). Magnetic entropy change, RCP and critical exponents’ values of Sn20 are higher than those of In20. The mean field model, with long-range ferromagnetic interactions, was the best model that describes the magnetic interactions around $T_c^*$. However, the critical exponents values around $T_c^*$ are different from the well-known universality class.

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