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The effect of the substitution of Cu for Ni on magnetoresistance and magnetocaloric properties of $Ni_{50}Mn_{34}In_{16}$

I. Dincer*, E. Yüzüak, Y. Elerman

Department of Engineering Physics, Faculty of Engineering, Ankara University, 06100 Besevler, Ankara, Turkey

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ABSTRACT

The effect of the substitution of Cu for Ni on the magnetoresistance behaviour and the magnetocaloric properties of the $Ni_{50}Mn_{34}In_{16}$ alloy has been investigated. The $(Ni–Cu)_{50}Mn_{34}In_{16}$ alloys crystallize in the cubic $L2_1$ structure in austenite phase. While the M_s temperature is about 160 K for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy, the martensitic transition is not observed for $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ alloy. To estimate the magnetic entropy change of the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy, the magnetization measurements as function of magnetic field are performed by continuous and noncontinuous heating methods. The $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy shows the magnetostructural transition whose irreversible ratio is about 50% at 160 K. The magnetocaloric effect strongly depends on the sample history due to the occurrence of the irreversible magnetostructural transition. For the magnetic field change of 2 T, giant magnetoresistance value is about −68% that is rather big among the similar bulk alloys.

1. Introduction

Magnetic refrigeration based on magnetocaloric effect (MCE) has attracted much attention due to its superiority over the gas refrigeration on energy savings and environmental concerns. A study of MCE in various magnetic solids is gaining worldwide attention. Many materials with first-order phase transition (magnetostructural phase transition) have been discovered to exhibit giant MCE. One of the typical MCE materials is the Ni–Mn-based Heusler alloys which undergo a structural transformation (martensitic transformation) from the austenite phase to the martensite phase on cooling and a reverse process on heating [1–4]. The characteristic temperatures of the martensitic transformation are the martensite start temperature M_s, the martensite finish temperature M_f, the austenite start temperature A_s and the austenite finish temperature A_f.

Giant magnetoresistance (GMR) effect first discovered in magnetic multilayers due to the spin-dependent scattering of conduction electrons [5] is an important property in magnetoresistive read-head and sensors technology. In addition to the magnetic multilayers, GMR has also been found in some bulk intermetallic materials such as Laves phase alloys and $Gd_5Ge_2Si_2$-type alloys. The GMR effect in these alloys is closely associated with a first order field-induced transition. Like these alloys, the Ni–Mn based Heusler alloys exhibit the GMR effect that occurs around the structural transformation temperatures from the austenite phase to the martensite phase on cooling and the reverse process on heating [6–8]. So the observation of the GMR and MCE makes the Ni–Mn based Heusler alloys potential technological material for the development of magnetic actuators, sensors and for magnetic refrigeration technology as magnetic coolant.

The full Heusler $Ni_{50}Mn_{25}In_{25}$ alloy is a ferromagnetic material with a Curie point at 290 K [9]. The $Ni_{50}Mn_{34}In_{16}$ alloy is derived from $Ni_{50}Mn_{25}In_{25}$ by substituting excess Mn at the In site. The $Ni_{50}Mn_{34}In_{16}$ alloy orders ferromagnetically below $T_C = 305$ K and undergoes martensitic transition from the high temperature cubic phase (austenite) to the low temperature monoclinic phase (martensite) around martensitic transformation temperatures [2,10]. The martensitic transformation temperatures are $M_s = 210$ K, $M_f = 175$ K, $A_s = 200$ K, and $A_f = 230$ K. For this alloy, the maximum value of the magnetic entropy change is about 12 J kg^{-1} K^{-1} in a magnetic field of 4 T at about 205 K [2]. It is also observed that the GMR is about −64% at 5 T around the martensitic transition temperatures for the bulk $Ni_{50}Mn_{34}In_{16}$ alloy [6].

The valence electron concentration (e/a) dependence of M_s in Ni–Mn–Z (Z:Al, Ga, In, Sn and Sb) is linear, but with different slopes for each Z species [11]. Therefore, it should be possible to manipulate M_s not only by varying e/a but also by holding e/a constant and replacing one Z species with another or Ni with another 3d transition metal (such as Cr, Fe, Co, Cu) [12–22]. In this manner, one may have the possibility of shifting and adjusting favourable features occurring around the martensitic transformation of a

* Corresponding author. Tel.: +90 312 203 3452; fax: +90 312 212 7343.
E-mail address: idincer@eng.ankara.edu.tr (I. Dincer).

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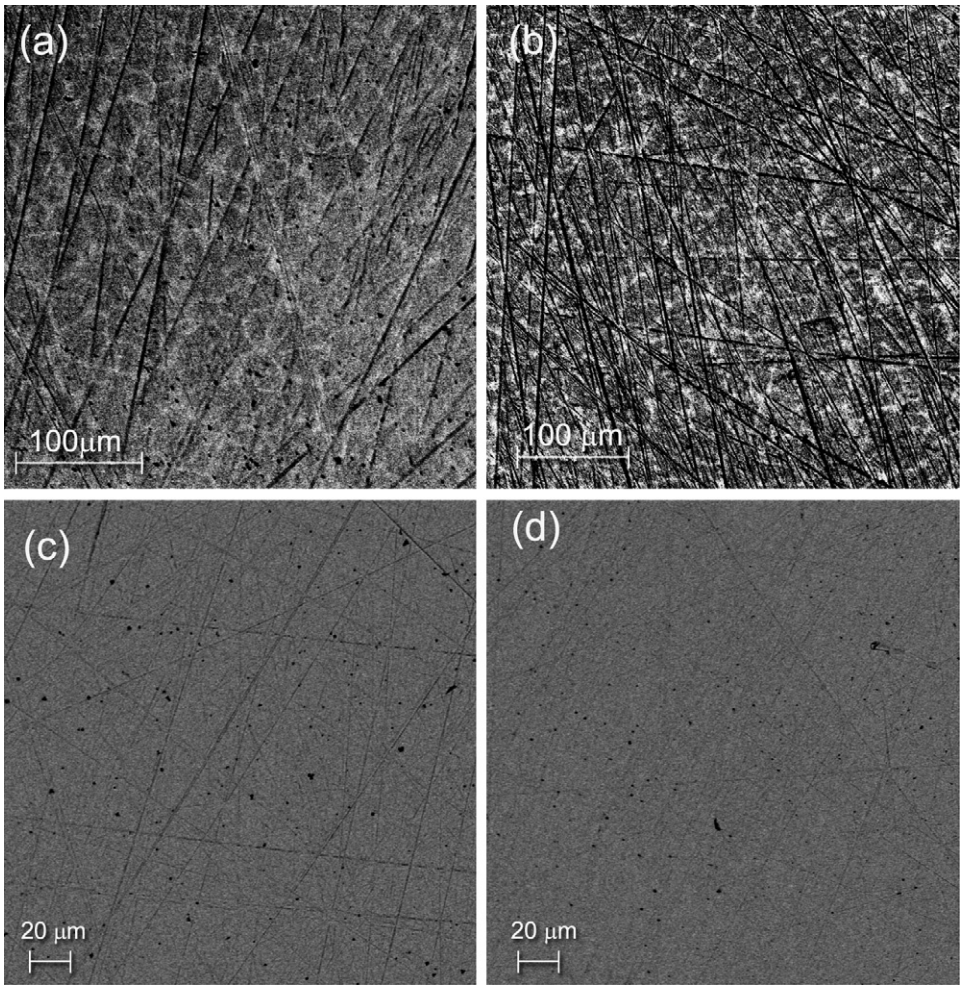

Fig. 1. The BSE pictures of $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$: (a) and (b) before annealing, (c) and (d) after annealing.

particular alloy to higher or lower temperatures. In view of technical interest, it would be desirable to shift the transition temperature to around room temperature. Since $Ni_{50}Mn_{34}In_{16}$ (e/a = 7.87) shows a field induced reverse martensitic transformation at round M_s and associated with it, a magnetocaloric effect and magnetoresistance properties, we substitute 2 and 4% Cu (e/a = 11) for Ni (e/a = 10) in $Ni_{50}Mn_{34}In_{16}$. With increasing of e/a value, these amounts of Cu may be expected to shift M_s to around room temperature.

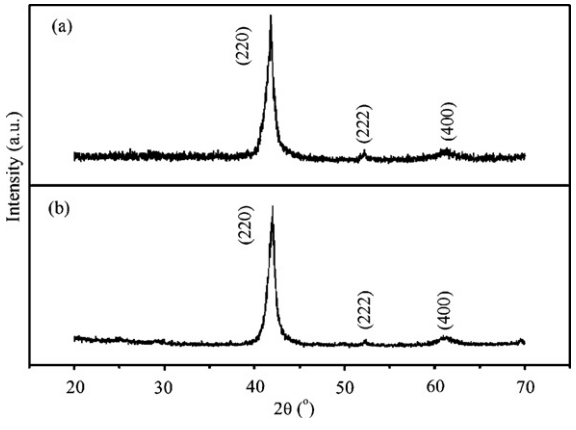

Fig. 2. The X-ray diffraction patterns of the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ (a) and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ (b) alloys at the room temperature.

In this work, we present an experimental study on the off-stoichiometric Heusler alloys $(Ni–Cu)_{50}Mn_{34}In_{16}$. We have elucidated detailed magnetoresistance and magnetocaloric properties of these alloys through X-ray powder diffraction, magnetization and resistance measurements.

2. Experimental

The $Ni_{50-x}Cu_xMn_{34}In_{16}$ (x = 2 and 4) alloys of about 2 g were prepared by arc melting of high-purity elements under argon atmosphere. The alloys were then encapsulated under argon atmosphere in quartz glass and annealed at 1073 K for 2 h to ensure homogeneity. Afterwards, they quenched in ice water. The compositions of the alloys were determined by energy dispersive X-ray (EDX) analysis with using the EVO 40 scanning electron microscope-SEM. The X-ray diffraction experiments with Cu Kα radiation were performed at the room temperature. The magnetization and resistivity measurements were made in a physical properties measurement system PPMS with a magnetic field up to 7 T in the temperature range from 5 to 350 K. Since the M(T) measurements in the different modes give us more information about magnetic properties of the alloys, the temperature dependence of magnetization were measured in zero-field-cooled (ZFC), field-cooled (FC) and field-heated (FH) modes. The electrical resistance measurements were performed by the standard four probe technique.

3. Results and discussion

3.1. Structural properties

We performed SEM analysis of the $Ni_{50-x}Cu_xMn_{34}In_{16}$ (x = 2 and 4) alloys before and after of the annealing to see better the effect of the annealing process. Back scattering electron-BSE, SEM

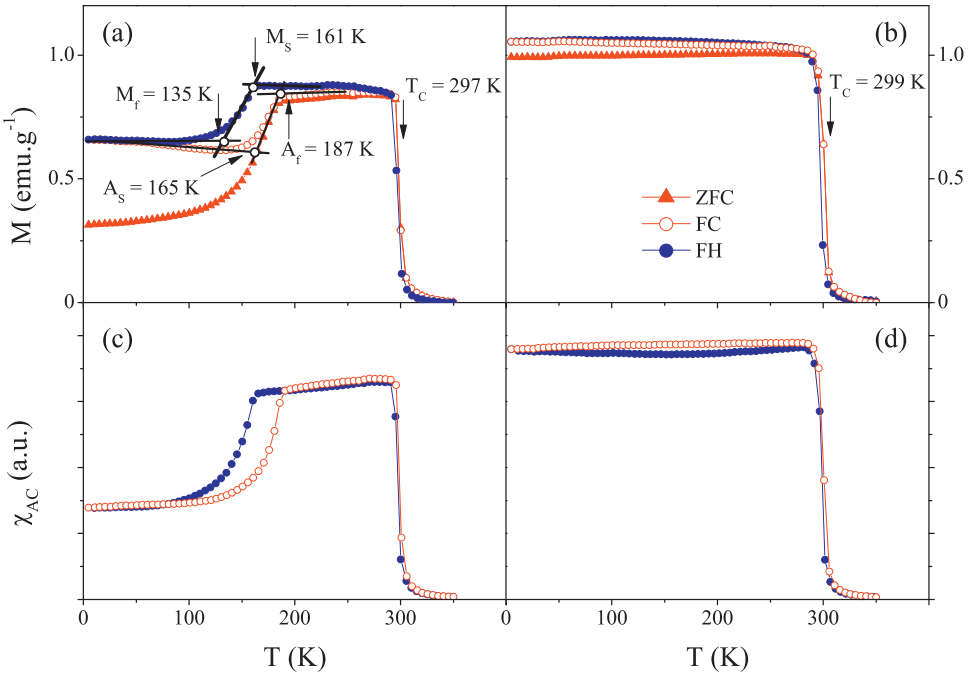

Fig. 3. The M(T) curves in an applied external field of 5 mT for $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ (a) and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ (b). The temperature dependence of AC susceptibility of $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ (c) and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ (d).

micrographs of these alloys are shown in Fig. 1. When the microstructure is examined using back scattering electron-BSE imaging it is more evident that there is a compositional gradient between light and dark areas matrix. As shown in Fig. 1, there is no compositional gradient for both alloys after annealing process. The average compositions of the $Ni_{50-x}Cu_xMn_{34}In_{16}$ (x = 2 and 4) alloys are calculated from EDX analysis are summarized in Table 1. The reported compositions are an average of multiple points on the alloys and no compositional heterogeneity is observed. The X-ray diffraction patterns of these alloys are shown in Fig. 2. The X-ray diffraction studies reveal that the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ alloys have a cubic $L2_1$ structure (space group *Fm*-3*m*) at the room temperature. Similarly, the $Ni_{50}Mn_{34}In_{16}$ alloy has also the cubic $L2_1$ structure (space group *Fm*-3*m*) at the room temperature in austenite phase [2,10].

3.2. Magnetic properties

Fig. 3(a) and (b) shows the M(T) curves in 5 mT taken on a zero-field-cooled (ZFC), field-cooled (FC) and field-heated (FH) sequences for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ and $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ alloys, respectively. The temperature dependence of the AC susceptibility curves of the both alloys in the temperature range between 5 and 350 K are shown in Fig. 3(c)

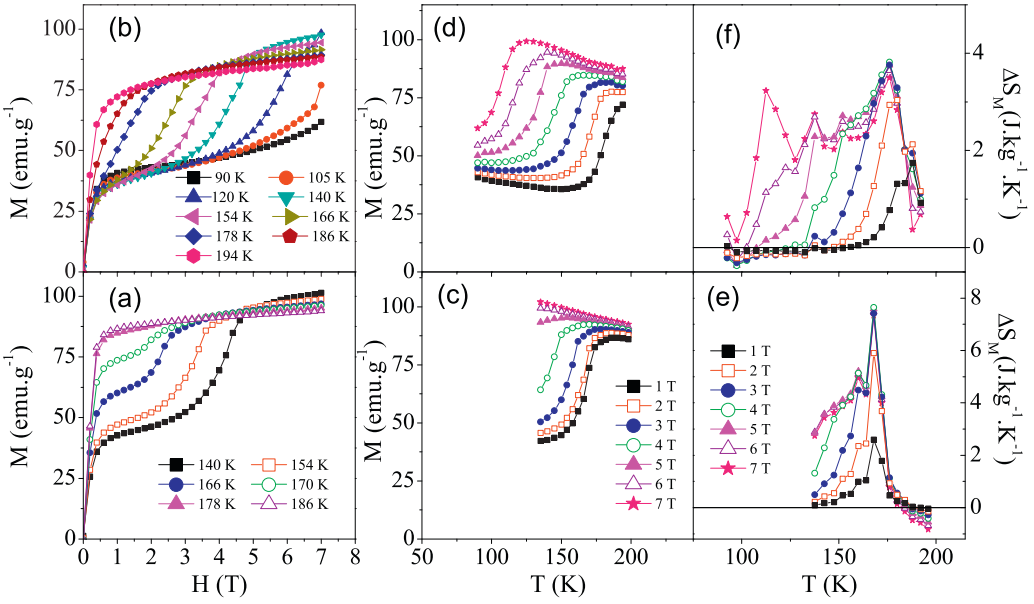

Fig. 4. The M(H) curves of $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ at the different temperatures: (a) for continuous and (b) for noncontinuous heating method. The M(T) curves of the same alloy obtained from the the M(H) curves: (c) for continuous and (d) for noncontinuous heating method. The temperature and magnetic field dependence of the magnetic entropy change by Maxwell relation for the same alloy: (e) for continuous and (f) for noncontinuous heating method.

Table 1
Compositions of the $Ni_{50-x}Cu_xMn_{34}In_{16}$ alloys determined by EDX analysis.

x (nominal)	Ni	Cu	Mn	In
2	47.5(9)	2.6(2)	34.0(7)	15.9(6)
4	45.5(9)	4.6(2)	33.8(7)	16.2(6)

and (d). With decreasing the temperature, the sharp decrease in M(T) and χ_{AC}(T) around 160 K and the thermal hysteresis between FC and FH curves can be attributed to the martensitic transition occurring in the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy. On the other hand, the $Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$ alloy does not exhibit the martensitic transition since there is no thermal hysteresis between FC and FH curves for this alloy. The behaviour of M(T) for this alloy is that of a typical ferromagnetic material. The characteristic temperatures of the martensitic transformation for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy are defined as the intersections of extrapolations from linear regions of the data [23]. The characteristic temperatures of the martensitic transition for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ (e/a = 7.89) alloy decrease with increasing Cu concentrations as compared with $Ni_{50}Mn_{34}In_{16}$ (e/a = 7.86), while the valence electron concentrations per atom e/a increase [12]. All the transition temperatures are gathered in Table 2 for these alloys.

The isothermal M(H) curves of the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy are measured by two different methods: continuous and noncontinuous heating methods. Fig. 4(a) and (b) shows the M(H) data taken on continuous and noncontinuous heating methods at the different temperatures for this alloy [12]. For clarity, only some selected M–H curves are shown in Fig. 4(a) and (b). After zero field cooling from 330 K to 50 K, the temperature goes up to desirable temperature. After that at this temperature, the sample is magnetized from 0 to 7 T and subsequently demagnetized from 7 to 0 T. After the cycle is completed, temperature is increased up to next temperature and the M(H) measurement was repeated. This method is called as "continuous heating" method. In the second method, prior to measuring M(H) curves at a constant temperature, the sample is initially zero field cooled down to 50 K to ensure a complete martensite state and then zero magnetic field heated to the desirable temperature. At this temperature, the magnetic field dependence of magnetization is measured from 0 to 7 T, and subsequently demagnetized from 7 to 0 T. After the magnetization measurement is completed, the sample is initially zero field cooled down to 70 K and then zero magnetic field heated to the next temperature. This sequence is termed as noncontinuous heating method. To show the influence magnetic field and temperature on the magnetostructural transition, we performed the continuous and noncontinuous heating methods. Fig. 4(c) and (d) shows the M(T) curves at the different magnetic fields obtained from the M(H) data. Since there is a big difference between the M(T) curves obtained from continuous and noncontinuous heating M(H) curves, the magnetostructural transition is not purely magnetoelastic.

The temperature dependence of the spontaneous magnetization-I_S obtained by the each M(H) curve (from noncontinuous method) for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy is shown in Fig. 5. The concentration dependence of I_S of the field-induced parent-P phase for the $Ni_{50}Mn_{50-x}In_x$ alloys can be explained by the model in which the magnetic moment of the excess Mn at the

Table 2
The valence electron concentrations per atom e/a, and the characteristic temperatures of the martensitic transition (M_s, M_f, A_s and A_f). The data of the $Ni_{50}Mn_{34}In_{16}$ alloy was taken from Ref. 2.

Alloy	e/a	M_s^M (K)	M_f^M (K)	A_s^M (K)	A_f^M (K)	T_C (K)
$Ni_{50}Mn_{34}In_{16}$	7.86	210	175	200	230	305
$Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$	7.89	135	161	165	187	299
$Ni_{45.5}Cu_{4.6}Mn_{33.8}In_{16.1}$	7.91					301

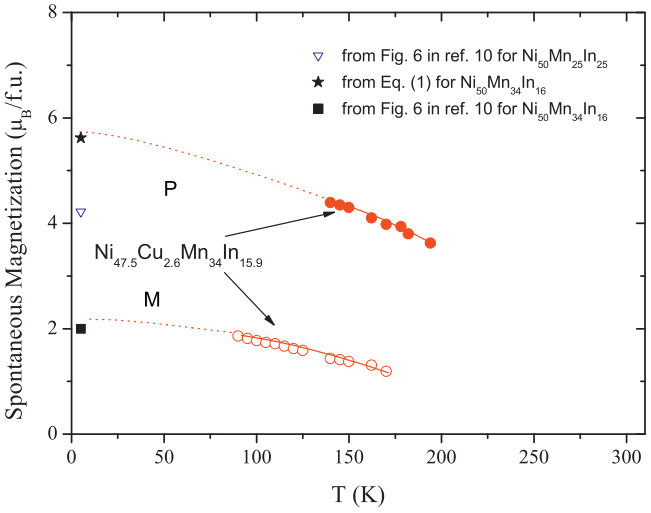

Fig. 5. The spontaneous magnetization as a function of temperature obtained from noncontinuous M(H) curves for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy.

In site couples ferromagnetically with that of Mn at the Mn site. The magnetic moments of Ni (μ_{Ni}), Mn (μ_{Mn}) and In (μ_{In}) in the $L2_1$-type Ni_2MnIn alloy have been theoretically reported to be 0.28 μ_B, 3.72 μ_B and −0.07 μ_B, respectively [10]. The magnetic moments of Cu (μ_{Cu}), Mn (μ_{Mn}) and In (μ_{In}) in the $L2_1$-type Cu_2MnIn alloy have been experimentally reported to be 0.11 μ_B, 4.22 μ_B and −0.02 μ_B, respectively [24]. The magnetic moments of Mn may change, even if the Cu content varies. The spontaneous magnetization value of $Ni_{50}Mn_{34}In_{16}$ alloy is around 5.57 μ_B/f.u. obtained from the Eq. (1) in Ref. [10]. The spontaneous magnetization value of the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy is about 5.8 μ_B near 5 K obtained from Fig. 5. According to the results of the study [10], the spontaneous magnetization value of the $Ni_{50}Mn_{25}In_{25}$ alloy is 4.12 μ_B (as seen in Fig. 6 in Ref. [10]). The value of I_S for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy is bigger than that of the $Ni_{50}Mn_{25}In_{25}$ alloy. This result shows that the ferromagnetic coupling occurs between the Mn atoms at the Mn site and the In site.

3.3. Magnetocaloric properties

Due to the first-order magneticstructural transition and considerable difference in the magnetization of the martensitic and $L2_1$ states at the transition temperature, substantial magnetocaloric effects can be expected. The magnetic entropy change ΔS_M around the martensitic transition temperatures can be estimated from magnetization measurements by the Maxwell relation [25]:

$$\Delta S_M \int_0^H \left(\frac{\partial M}{\partial T}\right)_H dH \quad (1)$$

from which the magnetocaloric effect can be estimated by numerical integration using the data in Fig. 4(a) and (b). The resulting ΔS_M around the martensitic transition temperatures is plotted in Fig. 4(e) and (f). The sign of ΔS_M is positive for all the temperatures, indicating that an inverse magnetocaloric effect is present, i.e., the sample cools when a magnetic field is applied adiabatically as in the $Ni_{50}Mn_{35}Sn_{15}$ and $Ni_{50}Mn_{37}Sn_{13}$ alloys [3]. While the maximum value of ΔS_M for the $Ni_{50}Mn_{34}In_{16}$ alloy is about 12 J kg^{-1} K^{-1} in a magnetic field change of 5 T at about 205 K, the maximum value of ΔS_M for the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy is about 7.8 J kg^{-1} K^{-1} and 3.8 J kg^{-1} K^{-1} in a magnetic field change of 5 T at about 167 K and 176 K for the continuous and noncontinuous heating methods, respectively. Since the M(H) curves measured with continuous and noncontinuous heating method at the same temperature are not

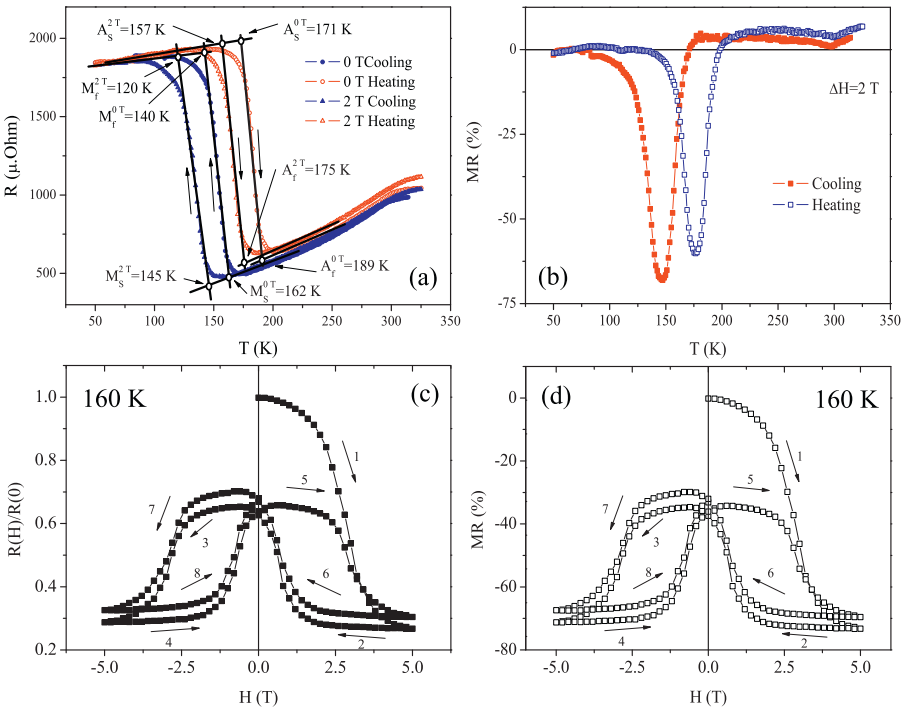

Fig. 6. For the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy, (a) the temperature dependence of the resistance in cooling and heating modes in an applied external field of 0 and 2 T. (b) The temperature dependence of the magnetoresistance in the magnetic field change of 2 T. (c) The normalized magnetic field dependence of the resistance (R(H)/R(0)) at 160 K. (d) The magnetic field dependence of the magnetoresistance at 160 K.

equal, the temperature dependence of ΔS_M is different for both method. This results show that the magnetostructural transition is not purely reversible (magnetoelastic). It is clear that the measurement method of M(H) data plays a significant role for the estimating of ΔS_M. However, one should be carefully for the estimating of ΔS_M from magnetization data for the alloys which have irreversible (magnetoplastic) or non-pure reversible transition.

3.4. Magnetoresistance properties

Fig. 6(a) shows the temperature dependence of electrical resistance – R(T) of the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloys between 50 and 325 K, both in the heating and cooling cycles, under zero magnetic field and 2 T. The austenite and martensite start and finish temperatures are also determined from the R(T) curves as seen in Fig. 6(a). Here, magnetoresistance (MR) has been calculated from the R(T) data collected at zero and 2 T:

$$\mathrm{MR}(\%) = \frac{\Delta R}{R} \times 100 = \frac{\mathrm{R(H)} - \mathrm{R(0)}}{\mathrm{R(0)}} \times 100 \qquad (2)$$

Fig. 6(b) shows the temperature dependence of MR(T) in the heating and cooling cycles for the magnetic field change of 2 T. While the $Ni_{50}Mn_{34}In_{16}$ alloy exhibits −64% of MR close to 220 K on the cooling branch for the magnetic field change of 5 T [6], the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy −68% of MR close to 145 K on the cooling branch for the magnetic field change of 2 T. The shift of M_s temperature is about 17 K for 2 T, which produces a giant negative MR in this alloy. Fig. 6(c) and (d) shows the normalized magnetic field dependence of the resistance (R(H)/R(0)) and MR at 160 K. For the R(H) measurement at 160 K, the temperature goes down to 50 K from 325 K under zero magnetic field. After that the temperature goes up to 160 K under zero magnetic field to perform the R(H) measurement. According to the R(H) data at 160 K, increasing magnetic field causes the martensite to austenite transition and hence a sharp reduction in resistance is observed. When the magnetic field decreases from 5 T to zero, the austenite to martensite transition is observed. But the zero magnetic field resistance values before and after the application of the magnetic field show a irreversibility and the irreversible ratio ($[R_1(0) - R_2(0)]/[R_1(0) - R_1(5\,T)]$) is about 50% at 160 K. The similar behaviour was also observed for the $Ni_{49.5}Mn_{35.5}In_{16}$ alloy as seen in Fig. 2(a) and (b) in Ref. [26]. The results of Fig. 3 also confirm that the magnetostructural transition is not purely magnetoelastic. Because of this reason, one should be very carefully to estimate the magnetic entropy change from the M(H) data for the materials which show structural phase transition.

4. Conclusions

The effects of varying the electron concentration by introducing small amounts of Cu in place of Ni on the MCE and MR properties of the $Ni_{50}Mn_{34}In_{16}$ alloy are studied in this work. Unlike our expectation, the decreasing of M_s temperature is observed on this study with increasing e/a. This result emphasizes that the e/a dependence of M_s is more complex in the Ni-based off-stoichiometric Heusler alloys [12]. The $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy shows the magnetostructural transition whose irreversible ratio is about 50% at 160 K. The peak position and value of the magnetic entropy change is closely related to the sample history due to the occurrence of the irreversible magnetostructural transition. The determination of the magnetic entropy change in alloys which show the magnetostructural transition has carefully been studied. The value of magnetoresistance associated with the martensitic transformation in the $Ni_{47.5}Cu_{2.6}Mn_{34.0}In_{15.9}$ alloy is about −68% that is rather big among the similar bulk alloys for the magnetic field change of 2 T.

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