Magnetoresistance and magnetocaloric properties involving strong metamagnetic behavior in Fe-doped Ni45(Co1-xFex)5Mn36.6In13.4 alloys

L. Chen, F. X. Hu, J. Wang, L. F. Bao, J. R. Sun et al.

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## ADVERTISEMENT



## Magnetoresistance and magnetocaloric properties involving strong metamagnetic behavior in Fe-doped Ni<sub>45</sub>(Co<sub>1-x</sub>Fe<sub>x</sub>)<sub>5</sub>Mn<sub>36.6</sub>In<sub>13.4</sub> alloys

L. Chen,<sup>1,2</sup> F. X. Hu,<sup>2,a)</sup> J. Wang,<sup>2</sup> L. F. Bao,<sup>2</sup> J. R. Sun,<sup>2</sup> B. G. Shen,<sup>2</sup> J. H. Yin,<sup>1</sup> and L. Q. Pan<sup>1</sup>

<sup>1</sup>Department of Physics, University of Science and Technology Beijing, Beijing 100083,

People's Republic of China

<sup>2</sup>State Key Laboratory of Magnetism, Institute of Physics, Chinese Academy of Sciences, Beijing 100190, People's Republic of China

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Here, we report the co-substitution of Fe and Co for Ni atoms on metamagnetic behavior, martensitic transformation, and transport and magnetocaloric properties in Ni<sub>45</sub>(Co<sub>1-x</sub>Fe<sub>x</sub>)<sub>5</sub>Mn<sub>36.6</sub>In<sub>13.4</sub> ( $x = 0 \sim 0.05$ ) alloys. It is found that the introduction of Fe atoms stabilizes martensitic phase and shifts martensitic temperature (T<sub>M</sub>) to higher temperature. Meanwhile, the Curie temperature T<sub>C</sub> of parent phase notably decreases. Upon Fe doping, the low magnetization of martensitic phase keeps nearly unchanged while the magnetization of parent phase slightly decreases. As a result, the Fe-doped samples maintain strong metamagnetic behavior and show great MR and MCE in an extended temperature range around room temperature. The hysteresis loss is reduced upon Fe-doping, which leads to an enhancement of effective RC by 15%. © 2012 American Institute of Physics. [http://dx.doi.org/10.1063/1.4732525]

Since the discovery of huge shape memory effect in metamagnetic Heusler alloys  $Ni_{45}Co_5Mn_{50-x}In_x$  (x = 13.3, 13.4, and 13.5),  $^{1,2}$  lots of attention has been attracted to these compositions. For the Mn-rich metamagnetic alloys, the introduction of Co atoms can work as a "ferromagnetic activator" and tune the antiferromagnetic (AFM) coupling between Mn-Mn atoms into ferromagnetic (FM) coupling through modifying atomic occupations.<sup>3,4</sup> The incorporation of Co enhances the ferromagnetic properties of parent phase and enlarges the change of Zeeman energy across martensitic transformation, thus an extremely large stress can be generated by magnetic field.<sup>1</sup> Along with field-induced metamagnetic behaviors, large magnetocaloric effect (MCE)<sup>5,6</sup> and magnetoresistance (MR)<sup>7,8</sup> were also observed in the materials. To exploit these functions in a wide temperature range, one wishes to tune martensitic transformation temperature  $(T_M)$  while keep strong metamagentic properties unchanged.

People made lots of effort, but the obtained results are not perfect sometimes. The common way to tune T<sub>M</sub> is to adjust valence electron concentration (e/a) through changing the compositions or introducing other elements.<sup>9,10</sup> However, in most cases, metamagnetic behavior becomes weaker as the T<sub>M</sub> is successfully tuned to the target temperatures. Ni<sub>45</sub>Co<sub>5</sub>  $Mn_{50-x}In_x$  alloys exhibit strong metamagnetic behavior in a very narrow range of In concentration.<sup>1</sup> Recent reports<sup>11,12</sup> revealed that an effective way to tune T<sub>M</sub> and maintain the strong metamagnetic behavior is to modify atom positions through structure relaxations by post-annealing at low temperatures. The change of compositions not only affects T<sub>M</sub> but also alters the local atomic environments and Mn-Mn couplings. Co doing can work as a "ferromagnetic activator" through modulating atomic occupations and enhance the ferromagnetic properties of parent phase in many Mn-rich NiMnCoZ (Z = In, Sb, Sn, Ga) alloys.<sup>3,4</sup> However, the act of Fe atom does opposite though it is a close neighbor of Co in periodic table and shows similar number of valence electrons and atomic radius. One tried to improve mechanical properties through introducing Fe elements.<sup>13</sup> However, in most cases, the incorporation of Fe atoms weakens the FM properties due to the introduced AFM Fe-Mn coupling. In one's mind, Fe element is not a favorable choice to optimize functions (such as shape memory effect, MCE, MR) and adjust transition temperatures. It has been experimentally observed<sup>14</sup> that the substitution of Fe for Ni in stoichiometric Ni<sub>2</sub>MnGa causes a sharp decrease of magnetization. However, a recent investigation<sup>14</sup> indicated that with the substitution of Fe for Ni in Mn-rich NiMnGa ribbons, the magnetization increases greatly rather than decreases, implying that the Mn-Mn AFM coupling has been changed into FM one. This fact indicates that Fe atom plays a similar role to that of Co atoms in the Mn-rich alloys. However, the magnetization of austenite for NiFeMnGa is notably lower than that of NiCoMnGa due to the existence of Fe-Mn AFM coupling.

On the other hand, quinary Heusler alloys were rarely reported due to complex atomic occupations and inter-atom couplings. In this Letter, we introduced Fe atoms in the functional material  $Ni_{45}Co_5Mn_{36.6}In_{13.4}$  and investigated the effect of Fe-doping on atomic occupations, martensitic transformation, and magnetic and functional properties. It was found that a small amount of Fe-doping can increase  $T_M$ , lower  $T_C$  of parent phase, while maintain the strong metamagnetic behavior and great magnetoresistance and magnetocaloric properties. The hysteresis loss can be reduced upon Fe-doping, resulting in an enhancement of effective refrigeration capacity (RC).

Quinary alloys  $Ni_{45}(Co_{1-x}Fe_x)_5Mn_{36.6}In_{13.4}$  (x = 0, 0.02, and 0.05) were prepared by arc-melting technique. The obtained ingots were each wrapped with Ta foil and homogenized in a sealed quartz tube at 1173 K for 24 h, then

<sup>&</sup>lt;sup>a)</sup>Author to whom correspondence should be addressed. Electronic mail: fxhu@iphy.ac.cn.

quenched in ice water. We checked the compositions by energy-dispersive x-ray spectroscopy (EDX) and inductively coupled plasma-atomic emission spectrometry (ICP-AES) and found the actual compositions agree with the nominal ones. X-ray diffraction (XRD) measurements were performed using CuK $\alpha$  radiation to identify structure. Magnetic and transport measurements were performed using a superconducting quantum interference device (SQUID) equipped with a probe for four-point electrical resistance (*R*) measurements.

Because the martensitic transformation temperature is close to room temperature, XRD patterns (Fig. 1) detected the coexistence of body-centred tetragonal (bct) martensitic structure (space group: I4/mmm) and body-centred cubic (bcc) austenitic structure (space group: Fm $\bar{3}$ m) for all samples. With increasing Fe concentration, the lattice changes regularly. Although the parameter (a) of austenitic structure keeps nearly unchanged (noting Fe has a similar atomic radius compared to that of Co), but the parameter ratio c/a of bct martensitic structure monotonously decreases with Fe doping (inset of Fig. 1). The reduction ratio is about 0.83% as Fe content increases from x = 0 to x = 0.05, indicating the symmetry of bct martensitic structure enhances.

Figure 2(a) displays the temperature dependence of zerofield-cooled (ZFC) and field-cooled (FC) magnetization (M-T curves) measured under 0.01 T. The method of measuring ZFC-FC magnetization and electrical resistance can be found in Ref. 15. The start and finish transition temperatures for the martensitic and austenitic state (M<sub>s</sub>, M<sub>f</sub> and A<sub>s</sub>, A<sub>f</sub>, respectively) were defined following the rule in Ref. 1 and indicated in Fig. 2(a). Curie temperature of the parent phase,  $T_C$ , is defined as the temperature at which the largest slope occurs on heating. These transition temperatures for the sample with Fe content x = 0 are  $M_s = 279 \text{ K}$ ,  $M_f = 269 \text{ K}$ ,  $A_s = 278 \text{ K}$ ,  $A_f = 289 \text{ K}, T_C = 378 \text{ K}, \text{ and } M_s = 287 \text{ K}, M_f = 281 \text{ K}, A_s$  $= 290 \text{ K}, A_{f} = 295 \text{ K}, T_{C} = 372 \text{ K}$  for x = 0.02, and  $M_{s}$  $= 291 \text{ K}, \text{ M}_{f} = 285 \text{ K}, \text{ A}_{s} = 292 \text{ K}, \text{ A}_{f} = 298 \text{ K}, \text{ T}_{C} = 359 \text{ K}$ for x = 0.05, respectively. One can find the T<sub>C</sub> of parent phase obviously shifts to lower temperature while the martensitic temperature M<sub>s</sub> shifts to higher temperature with Fe doping. This fact suggests that a higher Fe content stabilizes martensitic phase and paramagnetic parent phase. Inset of Fig. 2(a) schematically shows the dependence of  $M_s$  and  $T_c$ 



FIG. 1. X-ray diffraction patterns of  $Ni_{45}(Co_{1-x}Fe_x)_5Mn_{36.6}In_{13.4}$  (x = 0, 0.02, and 0.05) alloys. Inset shows lattice parameter and ratio c/a of bct martensitic structure as a function of Fe concentration. Solid line guides eyes.



FIG. 2. (a) Temperature dependence of ZFC and FC magnetization (M-T curves) measured under 0.01 T, and (b) the comparison of M-T curves measured under 5 T and 0.01 T for Ni<sub>45</sub>(Co<sub>1-x</sub>Fe<sub>x</sub>)<sub>5</sub>Mn<sub>36.6</sub>In<sub>13.4</sub> (x = 0, 0.02, and 0.05) alloys. Inset of (a) displays the dependence of martensitic transformation temperature M<sub>s</sub> and Curie temperature T<sub>C</sub> of parent phase on Fe content. Arrows indicate heating and cooling path.

on Fe content. One can notice that both  $M_s$  and  $T_C$  are sensitive to Fe concentration. A small amount of Fe-doping (x = 0.02) can cause a big shift (8 K) of  $M_s$  from 279 K to 287 K, and a shift (6 K) of the  $T_C$  from 378 K to 372 K. Further increasing Fe doping to x = 0.05 does not shift  $M_s$  too much (4 K from 287 to 291 K) but notably lowers  $T_C$  from 372K at x = 0.02 to 359 K at x = 0.05. More important is that the martensitic phase keeps low magnetization and the difference of magnetization across martensitic transformation remains large, predicting strong metamagnetic behavior remains.

Valence electron concentration e/a is a critical factor that influences the characteristics of Heusler alloys. A general rule based on NiMnZ (Z = Sn, In, Al, Ga) ternary alloys shows that M<sub>s</sub> sharply decreases while T<sub>C</sub> of parent phase keeps nearly unchanged with decreasing e/a.<sup>9,10</sup> The decreasing slope of  $M_s$  with e/a varies depending on the specific Z elements in NiMnZ.<sup>10</sup> According to formula Ni<sub>45</sub>(Co<sub>1-x</sub>  $Fe_x)_5Mn_{36.6}In_{13.4}$ , it is easy to calculate the average e/a noting valence electron number of the elements: Ni: 3d<sup>8</sup>4s<sup>2</sup>-10, Co:  $3d^{7}4s^{2}-9$ , Fe:  $3d^{6}4s^{2}-8$ , Mn:  $3d^{5}4s^{2}-7$ , and In:  $5S^{2}5P^{1}-3$ . The obtained e/a is 7.9140, 7.9130, 7.9115 for x = 0, 0.02, and 0.05, respectively. We notice the change of e/a is very small due to the small amount of Fe-doping and the close number of valence electrons between Fe and Co. The maximal change of e/a is only 0.03% (x = 0.05). With the slight reduction of e/a with Fe doping, martensitic temperature T<sub>M</sub> does not decrease but shows a notable increase, meanwhile Curie temperature  $T_C$  of parent phase notably decreases. Such fact conflicts with the general dependence of  $T_M$  and  $T_C$ on e/a.<sup>9,10</sup> So we conclude that the little change of e/a due to Fe doping does not dominate the altering of  $T_M$  and  $T_C$  in present systems. However, the fact that the average e/a changes little does not mean electronic structure is unchanged with Fe doping. The comprehensive effect from the change of electronic structure and atomic occupations, altering of structure symmetry, and introduced Fe-Mn AFM coupling should be responsible for the shift of  $M_s$  and  $T_C$  and the change of magnetic properties. Further detailed investigations are still required for exact reasons.

To help one intuitively understanding the magnetic structure and atomic occupations upon Fe-doping, Fig. 3 schematically displays a rough schema of the magnetic structure and atomic site of cubic austenite phase for Mn-rich Ni<sub>50</sub>Mn<sub>25+x</sub> In<sub>25-x</sub> and Fe/Co co-doped Ni<sub>50-v</sub>(Fe,Co)<sub>v</sub>Mn<sub>25+x</sub>In<sub>25-x</sub>. It has been demonstrated<sup>16</sup> that in Mn-rich alloys the extra "Mn" occupying In sites prefers AFM coupling with the nearest Mn atoms in both the austenite and martensite phases (Fig. 3(a)). Co atoms work as a "ferromagnetic activator" and tune the Mn-Mn AFM into FM coupling in Mn-rich Heusler alloys,<sup>3,4</sup> where magnetic moment mainly confines on Mn site while Co and Ni contributes little to the total moment.<sup>17</sup> The arrows in Fig. 3 indicate the direction of magnetic moment. The small arrow at Co(Ni) site indicates small moment. We suppose the introduced Fe atoms substituting Co randomly occupy Ni sites. Co atoms tune Mn-Mn coupling from AFM to FM as general, while Fe atoms play a similar role to that in Mn-rich NiFeMnGa ribbons<sup>14</sup> and assist the tuning process but the Fe-Mn coupling is still AFM (Fig. 3(b)). As a result, the magnetization of parent phase remains large and the metamagnetic behavior remains strong upon Fe doping. However, due to the Fe-Mn AFM coupling, T<sub>C</sub> of austenite phase decreases and the saturated magnetization reduces as the amount of Fe doping is large.

Fig. 2(b) presents the comparison of M-T curves measured under 5T and 0.01T. One can notice that a small amount of Fe-doping (x = 0.02) does not cause obvious change of saturated magnetization for both martensitic and parent phases though it can notably shift Ms and TC. Further increasing Fe content to x = 0.05 causes a slight drop of magnetization of parent phase but the one of martensitic phase still keeps unchanged. The difference of magnetization ( $\Delta M$ ) across martensitic transformation is as large as 101, 102, 87 emu/g for samples x = 0, 0.02, and 0.05, respectively. The resulted change of Zeeman energy,  $\mu_0 \Delta M \cdot H$ , pushes M<sub>s</sub> to lower temperatures at a nearly same rate of 5.8, 5.6, and 5.8 K/T, and the M<sub>s</sub> under 5 T locates at 250, 259, and 262 K for samples x = 0, 0.02, and 0.05, respectively. These results clearly demonstrate that the Fe-doped samples still retain strong metamagnetic properties. Hysteresis is a natural character of first order transition. The gap of hysteresis measures



FIG. 3. Magnetic structure sketch and atomic site of the top view of cubic austenite phase for Mn-rich Ni<sub>50</sub>Mn<sub>25+x</sub>In<sub>25-x</sub> and Fe/Co co-doped Ni<sub>50-y</sub>(Fe,Co)<sub>y</sub>Mn<sub>25+x</sub>In<sub>25-x</sub>. Arrows indicate the moment direction. Mn(In) indicates the In site is occupied by Mn. Co(Ni)/Fe(Ni) indicates the Ni site is occupied by Co/Fe.

frictions at phase boundaries during martensitic transformation.<sup>18</sup> Thermal hysteresis appears about 9.4, 8.8, and 7.0 K under 0.01 T and becomes 14.6, 12.0, and 9.2 K under 5 T for samples x = 0, 0.02, and 0.05, respectively. One can notice the hysteresis gap is getting smaller with Fe-doping. The smaller hysteresis indicates that less energy is enough to overcome the friction losses of phase boundary motion. Magnetic field enlarges the hysteresis, indicating the frictions enhance along magnetization.

Figures 4(a) and 4(b) typically shows the representative magnetization isotherms measured around martensitic transition for samples x = 0 and 0.05, respectively. One can notice that the field-induced metamagnetic transition becomes notably steeper with Fe doping but the hysteresis loss (enclosed area in a field cycle) is getting smaller. The maximal hysteresis loss is 153 and 110 J/kg for samples x = 0 and 0.05, respectively. It has been reported that many intrinsic and extrinsic factors affect the size of hysteresis loss, among which the change of electronic band structure is one of the important factors that affect hysteresis.<sup>19,20</sup> Upon Fe doping, the altering of electronic structure not only affects phase transitions and magnetic properties but also plays a possible role in affecting hysteresis loss. Figs. 4(c) and 4(d) shows the magnetic entropy change  $\Delta S$  calculated by using Maxwell equation for the corresponding samples. The effective  $\Delta S$ (not the spike but the high plateau value<sup>21,22</sup>) is 19.5 (~269 K) and 18.7 J/kgK (~281 K) under a field change of 0-5 T for samples x = 0 and 0.05, respectively. One can notice the effective  $\Delta S$  keeps nearly unchanged upon Fedoping. Refrigerant capacity (RC) is an important parameter of judging materials' magnetocaloric attribute. The direct RC evaluated by integrating the area (shadow area in Figs. 4(c) and 4(d)) under  $\Delta S$  curve from T<sub>1</sub> to T<sub>2</sub> (T<sub>2</sub>-T<sub>1</sub>: halfmaximum width) is 351 J/kg, 338 J/kgJ/kg, and the effective  $RC_{effe}$  after deducting the maximal hysteresis loss<sup>23</sup> is 198 J/ kg (~269 K) and 228 J/kg (~281 K) J/kg for samples x = 0and 0.05, respectively, for a field change of 0-5 T. One can notice the effective  $RC_{effe}$  of Fe-doped sample (x = 0.05)



FIG. 4. Magnetization isotherms for  $Ni_{45}(Co_{1-x}Fe_x)_5Mn_{36,6}In_{13.4}$  (a) x = 0and (b) x = 0.05, and the magnetic entropy change  $\Delta S$  as a function of temperature under different magnetic fields for (c) x = 0 and (d) x = 0.05. The shadow area indicates the direct RC evaluated by integrating the area under  $\Delta S$  curve from  $T_1$  to  $T_2$  ( $T_2$ - $T_1$ : half-maximum width). Arrows indicate field changing path.



FIG. 5. (a) Temperature dependent electrical resistance under 0 T (R<sub>0T</sub>) and the ZFC-FC resistance under 5 T (R<sub>5T</sub>) on heating and cooling processes, and (b) the deduced MR as a function of temperature for Ni<sub>45</sub>(Co<sub>1-x</sub>Fe<sub>x</sub>)<sub>5</sub> Mn<sub>36.6</sub>In<sub>13.4</sub> (x = 0, 0.02, and 0.05) alloys. Arrows indicate heating and cooling path.

enhances by  $\sim 15\%$  compared to its mother compound due to the reduction of hysteresis loss.

We also measured transport properties for all samples. The retained strong metamagnetic behavior upon Fe doping predicts large MR. Fig. 5(a) shows the temperature dependent electrical resistance under 0 T (R<sub>0T</sub>) and the ZFC-FC resistance under 5 T ( $R_{5T}$ ) on heating and cooling processes. Fig. 5(b) shows the deduced MR as a function of temperature. The gap of thermal hysteresis in  $R_{0T} \sim T$  curve measures 15.0 K, 12.7 K, and 9.1 K for samples x = 0, 0.02, and 0.05, respectively. One can notice the sample x = 0.05 still shows the smallest temperature hysteresis, which further confirms the fact that Fe-doping can reduce the hysteresis gap. As expected, electrical resistance R shows step change along with martensitic and reverse transformation. As the sample experiences concurrent martensitic and magnetic transformation, the increased interfacial scattering at the twin boundaries and spin scatterings result in an enhanced resistivity.<sup>3,7,8</sup> With T<sub>M</sub> shifting to a lower temperature under an external field, a negative MR  $\sim (R_{5T}-R_{0T})/R_{0T}$  was observed. The maximal MR relating to the field-driven metamagnetic transition under 5 T is about -76.5% (244 K), -73.0% (262 K), and -73.2% (266 K) for samples x = 0, 0.02, and 0.05, respectively. One can notice that the MR value keeps large while M<sub>S</sub> varies near room temperature with tuning Fe-doping. The temperature scope is about 50 K from  $\sim$ 240 K to  $\sim 290$  K where large MR ( $-70\% \sim -77\%$ ) appears.

In summary, we investigated the effect of Fe-doping on martensitic transformation and magnetocaloric and transport properties in  $Ni_{45}(Co_{1-x}Fe_x)_5Mn_{36.6}In_{13.4}$  alloys. It was found that a small amount of Fe doping can tune martensitic temperature while maintain the strong metamagnetic behavior, which is indicative of excellent functions at tunable temperatures, such as shape memory effect, MR, and MCE. With increasing Fe doing both thermal and magnetic hysteresis are

getting smaller. As a result, effective RC<sub>effe</sub> enhances by  $\sim 15\%$  as Fe doping is x = 0.05. Meanwhile, magnetoresistance associated the field-driven metamagnetic behavior remains a high magnitude of  $-70\% \sim -77\%$  under 5 T. The tunable T<sub>M</sub> and excellent functions make the Fe-doped NiCo-FeMnIn quinary alloys attractive for practical applications in functional devices.

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