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Giant reversible inverse magnetocaloric effects in $Ni_{50}Mn_{35}In_{15}$ Heusler alloys

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ABSTRACT

The magnetic properties and reversibility of the magnetocaloric effect of $Ni_{50}Mn_{35}In_{15}$ have been studied in the vicinity of the phase transition using magnetization and direct adiabatic temperature change (ΔT_{ad}) measurements in magnetic fields up to 14 T. The magnetostructural phase transitions (MSTs) between a martensitic phase (MP) with low magnetization (paramagnetic or antiferromagnetic) and a nearly ferromagnetic austenitic phase were detected from thermomagnetic curves, M(T,H), at the applied magnetic fields up to 5 T. The MST temperature was found to be nearly independent of magnetic field for H < 5 T, and shifted to lower temperature with the further increase of magnetic field to 14 T. A large and nearly reversible inverse magnetocaloric effect (MCE) with $\Delta T_{ad} \sim -11$ K for a magnetic field change of $\Delta H = 14$ T was observed in the vicinity of the MST. The irreversibility of ΔT_{ad} was found to be 1 K. A direct ΔT_{ad} of +7 K for $\Delta H = 14$ T was detected at the second order ferromagnetic-paramagnetic phase transition. The obtained results have been discussed in terms of the suppression of antiferromagnetic correlations with the application of a strong magnetic field, and a reversibility of the initial magnetic state of the MP with applied magnetic field when the MST coincides with T_C.

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1. Introduction

The off-stoichiometric Ni–Mn–In based Heusler alloys with compositions near $Ni_{50}Mn_{35}In_{15}$ undergo first order temperature-induced structural/martensitic transitions at $T = T_M$ near room temperature. The magnetic states of the high temperature austenitic phase (AP) and low temperature martensitic phase (MP) were found to be different in these compounds near T_M. Thus, the martensitic transition can be induced by external magnetic field and results in a jump-like variation in the magnetization and magneto-responsive properties, i.e., it undergoes a magnetostructural phase transition (MST). Magnetic transitions of paramagnetic (PM) MP to a PM austenitic phase, low-magnetization state (LM, paramagnetic or antiferromagnetic) MP to ferromagnetic AP, and ferromagnetic MP – paramagnetic AP types have been reported at MSTs in Refs. [1–4]. Normal and inverse magnetocaloric effects (MCEs), exchange bias, temperature- and field-induced phase transitions, giant magnetoresistance, and giant Hall effects have been observed in systems based on $Ni_{50}Mn_{50-x}In_x$ in the vicinity of $x = 15$ [5–7]. Consequently, these materials are attractive candidates for multifunctional devices, where magnetic and structural characteristics may be manipulated by applied magnetic fields. Since, the magnetocaloric effect results from magnetization changes, remarkable MCE values and time dependency of adiabatic

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temperature changes have already been reported for these alloys [1,5–14] and, in Refs. [15], respectively. An important MCE parameter that characterizes the applicability of materials for magnetic refrigeration is the adiabatic temperature change (ΔT_{ad}). However, for practical applications besides high values of ΔT_{ad} its reversibility in cycling magnetic fields is of primary importance.

The ΔT_{ad} of alloys based on $Ni_{50}Mn_{50-x}In_x$ with x ~ 15 have been studied in the vicinity of the phase transitions using direct measurements in Refs. [4–13]. The largest measured changes were $\Delta T_{ad} = -2$ K and 2 K near the martensitic (first order) and ferromagnetic (second order, at T_C) transitions for $\Delta H = 1.8$ T, respectively [7,11]. Our preliminary results showed that these values can be significantly enhanced up to ~11 K near the MST in strong magnetic fields (up to 14 T) for the first magnetization cycle [12,13]. A large ΔT_{ad} of −6.2 K ($\Delta H = 1.9$ T) was reported in Ref. [16] for a $Ni_{45.2}Mn_{36.7}In_{13}Co_5$ alloy at the MST. However, the initial value of $\Delta T_{ad} = -6.2$ K observed for the first magnetization cycle was found to drop to about −2 K for the second and subsequent cycles. Thus, the irreversibility of ΔT_{ad} was found to be about 70%. The ΔT_{ad} of −12.8 K was measured for $Ni_{45}Co_5Mn_{36.7}In_{13.3}$ for the first cycle of a pulsed magnetic field ($\Delta H = 15$ T) in Ref. [16] but the reversibility was not demonstrated.

In the manuscript, we report the dependence of the MST temperature of $Ni_{50}Mn_{35}In_{15}$ on magnetic field, and the associated reversibility of ΔT_{ad}. A nearly reversible ΔT_{ad} of about −10 K (1 K less for the subsequent cycles) was observed in the vicinity of the MST. The observed behavior has been attributed to specific features of the magnetic states of both the MP and AP close to the MST connected with strong antiferromagnetic correlations and the proximity to long-range ferromagnetic order.

2. Experimental details

The $Ni_{50}Mn_{35}In_{15}$ (nominal composition) compounds were fabricated by conventional arc-melting in a high-purity argon atmosphere using 4N purity elements and were subsequently annealed in high vacuum (~10^{-4} Torr) for 24 h at 850 °C. The phase purities of the compounds were confirmed by x-ray powder diffraction at room temperature using CuKα radiation. Thermomagnetic curves M(H,T) have been acquired using a superconducting quantum interference device (SQUID) magnetometer (by Quantum Design, USA) and by a Quantum Design PPMS-9T platform in fields up to H = 5 T and 9 T, respectively, in a temperature interval of 5–400 K. Magnetization (M) was measured in the temperature range (4.2–300) K by the vibrating magnetometer method. As the source of the magnetic field, an Oxford Instruments superconducting solenoid was used, producing magnetic fields up to 14 T. Direct measurements of ΔT_{AD} under applied magnetic fields up to 14 T were done using a Bitters electromagnet and employing the "extraction/insertion" method (see for details of this set-up in Ref. [13]). The time of "extraction/insertion" from maximum to zero magnetic fields regions was 2.5 s. Direct measurements of ΔT_{AD} at $\Delta H = 1.8$ T have been done using an adiabatic magnetocalorimeter (MagEqMMS 801) in a temperature range of 250 K–350 K. The external magnetic fields were ramped at a rate of up to 2.0 T/s during the measurements. The $Ni_{50}Mn_{35}In_{15}$ samples were heated to 380 K prior to the measurements. The temperature dependences of the magnetization, M(T), at applied magnetic field (H) were carried out during heating after the samples were cooled from 380 K to 10 K at zero magnetic field (ZFC), and during a field-cooling cycle (FCC).

3. Results and discussion

A mixture of high temperature cubic AP and low temperature MP was observed in the room-temperature x-ray diffraction patterns. The MP and AP were identified as orthorhombic and cubic, respectively (see Fig. 1). The co-existence of AP and MP crystal phases results from the austenitic and martensitic phase temperature hysteresis originating from the temperature-induced, first order structural transition.

The behavior of the magnetization curves was found to be different at low and high magnetic fields. The inverse (at T_A) and direct (at T_M) martensitic transformations, and the FM-PM (at T_C), are clearly identified from the jump-like changes in magnetization and inflection points of the M(T) curves, respectively, at magnetic fields H > 2 T (see Fig. 2). However, at low magnetic fields (see for H = 0.005 and 0.01 T in Fig. 2), no features characteristic of the FM-PM transition were observed. The obtained maximum values of magnetization of about 0.3 emu/g (on heating) and 1.2 emu/g (on cooling) of the austenitic phase in the vicinity of the martensitic transition (MT) are comparable to that of 0.6 emu/g observed above the structural phase transition from a paramagnetic martensitic state to a paramagnetic austenitic state in $Ni_{50}Mn_{36.5}In_{13.5}$ Heusler alloys [14] at the same magnetic field of 0.01 T. Taking into account the small value of magnetization of the AP of about 1.25/0.6 emu/g at H = 0.01 T (see Fig. 2), one can conclude that the AP phase remains in the paramagnetic (or "quasiferromagnetic") state at low magnetic fields for all temperatures above T_M, and therefore the hypothetical T_C of the AP is slightly below T_M. The martensitic transformation temperature, T_A and T_M, are nearly constant at magnetic fields less than 5 T, and decreases by about 28 K at H = 14 T (see Fig. 3). In our case the MST is unconventional, namely, at low magnetic fields (less than 2 T), the MST results in a transition from a low magnetization MP to paramagnetic AP, which state is very close to long-range ferromagnetic order, the subsequent increase in applied magnetic field promotes the FM ordering of AP, and therefore the decrease in the martensitic transition temperatures. Apparently, in such a case when the MST occurs between two "paramagnetic" phases one can expect the reversibility for MCE, like in the case of MCE in paramagnetic salts at low temperatures. The MST temperature can be described as a quadratic function of applied magnetic field (see Fig. 3).

An inverse ΔT_{AD} of about −1 K (for $\Delta H = 1.8$ T) and −11 K (for $\Delta H = 14$ T), and the ΔT_{AD} of 1 K (for $\Delta H = 1.8$ T) and about 7 K (for $\Delta H = 14$ T) were observed at the first order MST, and at the second order magnetic transition at T_C, respectively (see Fig. 4). Thus, the application of a strong magnetic field ($\Delta H = 14$ T) results in a giant

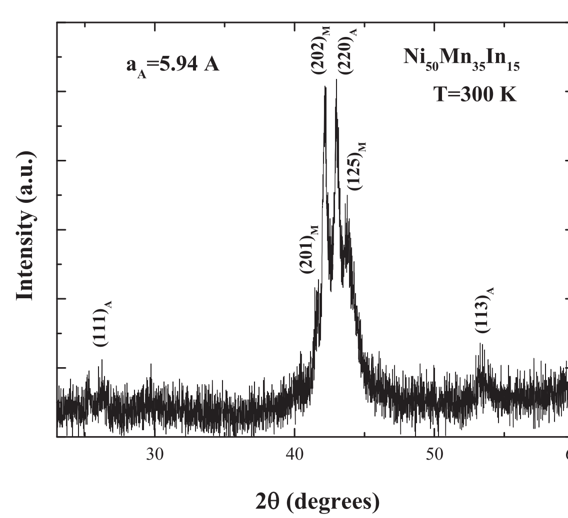

Fig. 1. Powder XRD patterns of $Ni_{50}Mn_{35}In_{15}$ at room temperature.

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