4-25-2020

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Recommended Citation

Koshkid'ko, Y., Pandey, S., Cwik, J., Dubenko, I., Aryal, A., Granovsky, A., Szymanski, D., Stadler, S., Lähderanta, E., & Ali, N. (2020). Relaxation phenomena in adiabatic temperature changes near magnetostructural transitions in Heusler alloys. *Journal of Alloys and Compounds, 821* https://doi.org/10.1016/j.jallcom.2019.153402

Journal of Alloys and Compounds 821 (2020) 153402

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Relaxation phenomena in adiabatic temperature changes near magnetostructural transitions in Heusler alloys

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ARTICLE INFO

Article history:
Received 21 June 2019
Received in revised form
6 December 2019
Accepted 13 December 2019
Available online 14 December 2019

Keywords:
Magnetocaloric effect
Adiabatic temperature change
Heusleralloys
Relaxation processes
Magnetostructural transitions
Latent heat

ABSTRACT

The relaxation processes of the adiabatic temperature changes (ΔT_{ad}) at the phase transitions in $Ni_{45}Mn_{43}CoSn_{11}$, $Ni_{50}Mn_{36.5}In_{13.5}$, and $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$ Heusler alloys with different magnetic structures have been studied using a direct extraction method in magnetic fields up to 14 T. It has been found that ΔT_{ad} exhibits short relaxation times (less than 10^{-1} (s)) in the vicinity of the second order phase transitions at the Curie temperatures. The relaxation times of the first order martensitic transitions strongly depend on the latent heat of the transition and can be characterized by a logarithmic law.

1. Introduction

The changes in magnetization in a magnetic material induced by external magnetic fields (or other external factors) are described as a multistage process of establishing thermodynamic equilibrium, which is characterized by a relaxation time. The relaxation time can change from 10^{-14}(s) in response to femtosecond laser pulses [1], to 10^2-10^3(s) in soft magnetic materials due to eddy-currents, magnetic aftereffects, and magnetic aging [2,3], to more than 10^5(s) in spin glasses.

The most interesting and the least understood processes related to magnetic relaxation are those involving induced phase transitions, where the structure and magnetic ordering change with the application of a magnetic field. The magnetostructural martensitic transitions observed in metamagnetic Heusler alloys in the Ni–Mn-X (X = In, Sn, Ga) family [4–6] are typical examples of such transitions. These alloys exhibit a number of unique and practically important physical properties, such as giant magnetocaloric effect, magnetic shape memory effects, giant magnetoesistance, giant anomalous Hall effects, and others (see, for example, [4]). Some of these alloys, when doped with a fourth element, show large magnetocaloric effects [5,6].

Isothermal and athermal transformations are considered as the primary mechanisms that drive martensitic transitions (MTs). The former results in a gradual increase in the proportion of the martensite (austenite) phase at a constant temperature, and are accompanied by a release (absorption) of heat. The latter is the athermal MT, where there is a gradual increase in the martensite fraction with decreasing temperature in the range spanning the martensitic transformation.

Most MTs are driven thermally. In alloys of the Ni–Mn-X family (X = In, Sn, Ga), the martensitic transitions occur in a definite temperature range, but have all the features of an isothermal transition [7,8], i.e., accompanied by a release (absorption) of the latent heat of the transition.

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https://doi.org/10.1016/j.jallcom.2019.153402
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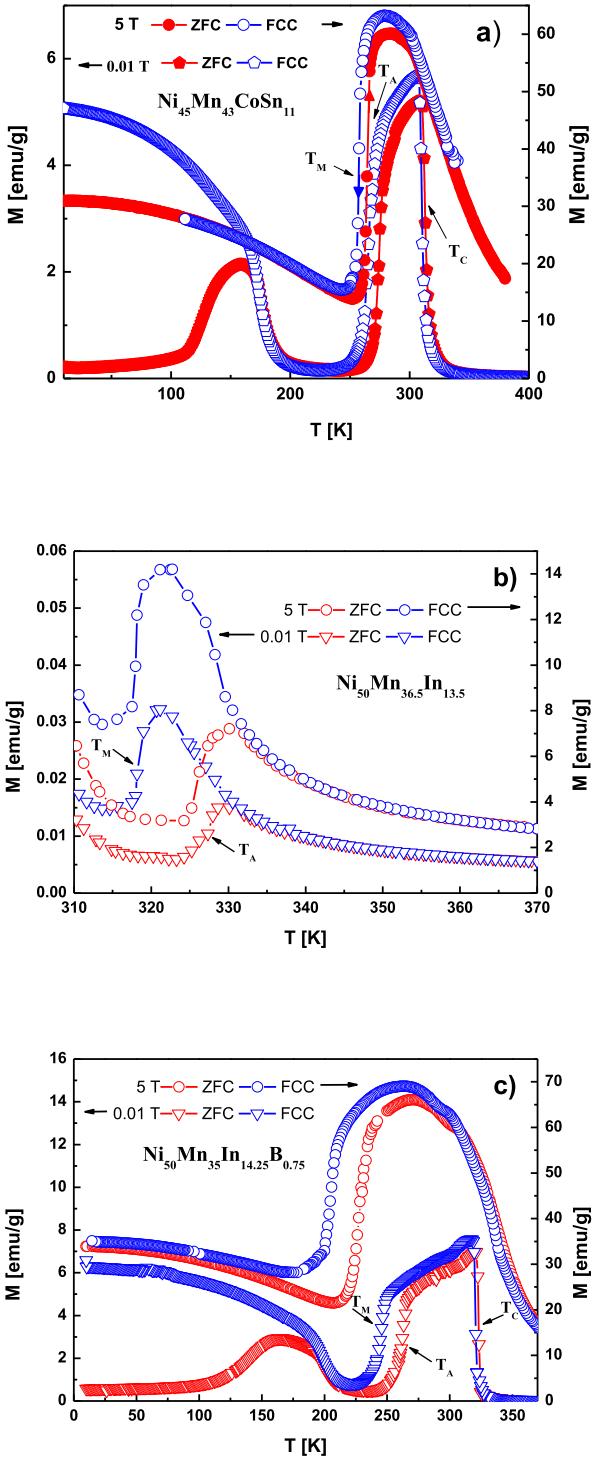

Fig. 1. ZFC and FC magnetization of the samples as a function of temperature obtained at magnetic fields $\mu_0 H = 0.01$ and 5 T for (a) $Ni_{45}Mn_{43}CoSn_{11}$, (b) $Ni_{50}Mn_{36.5}In_{13.5}$, and (c) $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$. Arrows indicate magnetic and martensitic transitions temperature.

In Refs. [9,10] it was shown that the relaxation time of the magnetization in a Ni–Mn–In–Co alloy under isothermal conditions was several minutes at 50 K, and could be described by a logarithmic law similar to that used to describe magnetic aftereffects. In Ref. [5] it was shown that the process of nucleation of the austenitic phase (AP), and the movement of the phase boundaries determine the duration of the martensitic transition (MT). In Ref. [6] the in-situ relaxation of the formation of the AP was observed in a Ni–Mn–In alloy as the applied magnetic field changed from 0 to 1.1T. A similar result was reported for Ni–Mn-Ga at magnetic fields up to 14 T [11]. It is possible that such relaxation phenomena can affect not only the dynamics of the magneto-structural transitions (MST), but also the associated magnetocaloric effects (MCE). It was shown in Ref. [12] that the MCE is nearly vanished entirely in Ni–Co–Mn-Ga when the frequencies of the alternating magnetic field of magnitude 1.8 T exceeded 20 Hz.

The isothermal entropy and adiabatic temperature changes induced by changes in magnetic field (ΔH) are two parameters that are generally used to characterize MCE. Studies of adiabatic temperature changes, ΔT_{ad}, are largely carried out either in insufficiently strong alternating fields of low frequency, or in pulsed magnetic fields [5,13,14]. However, these methods are not completely accurate because of the relaxation processes associated with the formation of the martensitic (austenitic) phase. One of the effective alternative methods is to use a permanent magnetic field and a sample extraction procedure to carry out ΔT_{ad} measurements.

In this paper, we study the dynamics of field-induced martensitic transitions under adiabatic conditions in large static magnetic fields up to 14 T generated by a Bitter-type magnet. The experiments were carried out with a unique magnetic setup that facilitates adiabatic temperature change measurements. Samples of Heusler alloys with different values of latent heat of the martensitic transition were selected for this project.

2. Experimental

2.1. Experimental setup for MCE measurements

An extraction method was used to carry out direct ΔT_{ad} measurements in magnetic fields of up to 14 T in a temperature range of 4.2–350 K [15]. A Bitter-type magnet was employed to generate a steady magnetic field. The sample was encased within a thermal screen to minimize the heat exchange with the environment. The temperature was controlled by a Lake Shore thermo-controller. A Hall sensor was placed in the sample holder to measure the magnetic field. Movement of the sample in and out of the region of maximum magnetic field was carried out with a LinMot actuator. The sample was translated from the center of the Bitter magnet to the outside (or vice versa), a distance of 35 cm, in 1.0 (s). Both the temperature of the sample and the temperature change (due to MCE) were measured using differential copper-constantan and chromel-gold (at low temperatures) thermocouples.

We developed computer software that recorded the adiabatic temperature change (ΔT_{ad}), sample temperature (T), and magnetic field (H) with a time resolution better than 0.1 s. Two plates of thickness of 2 mm and plate area 4 × 4 mm^2 were fabricated from

Table 1
Latent heat (L) of the MST, total entropy changes (ΔS_t) obtained at zero magnetic field, and the phase transition temperatures T_A, T_M, and T_C.

	T_A, K	T_M, K	T_C, K	L, kJ/kg	ΔS_t, J/(kg*K)
$Ni_{45}Mn_{43}CoSn_{11}$	274	266	310	8.67	31
$Ni_{50}Mn_{36.5}In_{13.5}$	358	347	–	11.56	34
$Ni_{50}Mn_{35}In_{14.25}B_{0.75}$	264	245	324	2.7	10

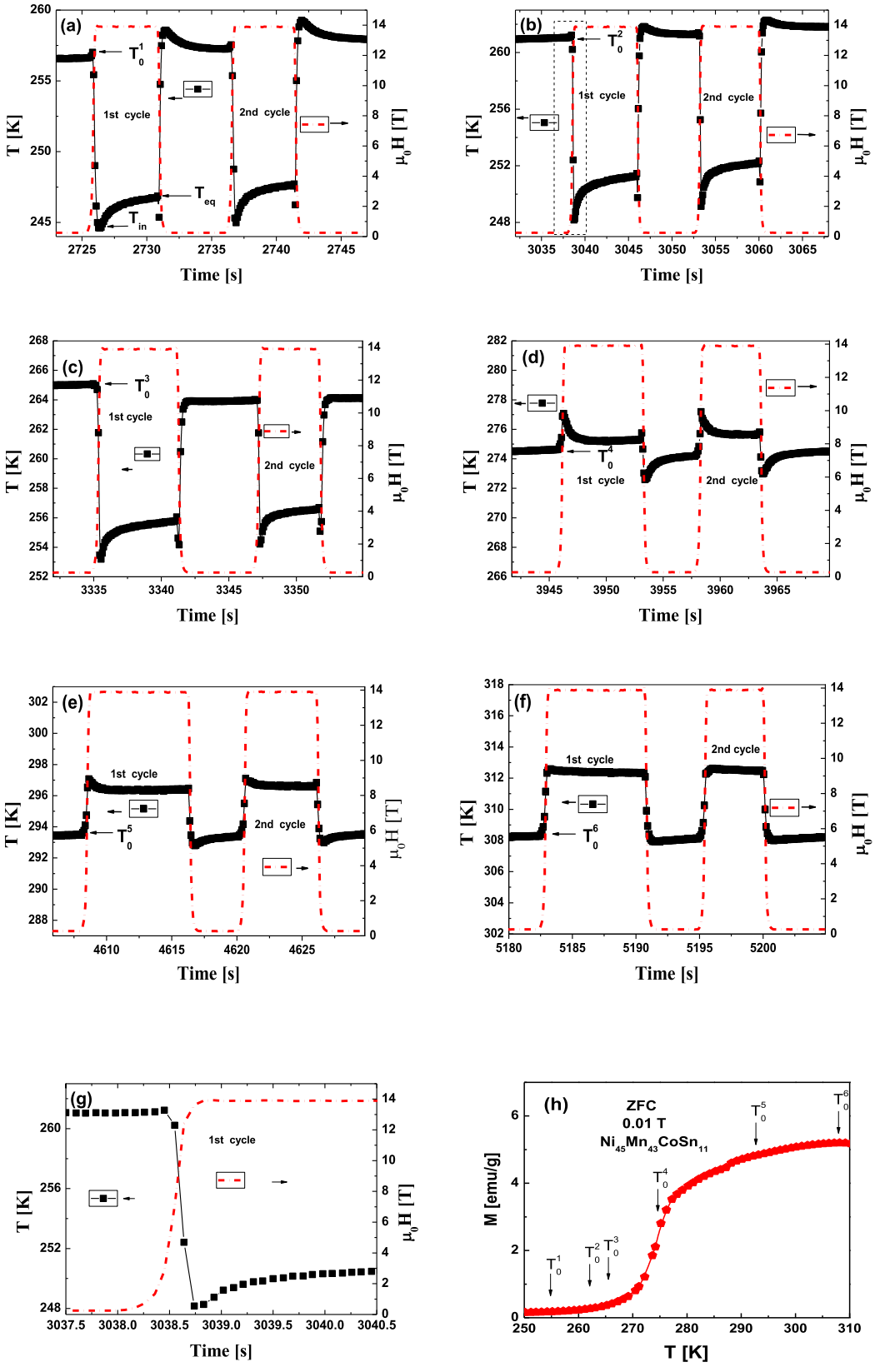

Fig. 2. (a–g):The time dependencies of the temperature changes of $Ni_{45}Mn_{43}CoSn_{11}$ obtained in adiabatic conditions near the MST and T_c; (g): Results obtained during magnetic field increasing; (h): The temperature dependence of the magnetization of $Ni_{45}Mn_{43}CoSn_{11}$ measured while heating at H = 0.01 T. The arrows (Fig. 2h) show the initial temperatures T_0 for the adiabatic temperature changes measurements (shown in Fig. 2a–f).

each compound. The junction of the copper - Constantan thermocouple was placed in thermal contact between two sample plates. Adiabatic conditions were achieved through thermal insulation using a foam polymer, as well as by vacuum (10^{-5} mm Hg).

2.2. Sample preparation

Heusler alloys $Ni_{45}Mn_{43}CoSn_{11}$, $Ni_{50}Mn_{36.5}In_{13.5}$, and $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$ of nominal compositions were fabricated using arc-melting methods (see Ref. [4] for details). The compositions of the compounds were chosen based on preliminary studies and published literature so as to find Heusler alloys with different types of MSTs. The chemical and crystal phase compositions of the samples were studied using energy dispersive X-ray spectroscopy (EDX) and X-ray diffraction (XRD), respectively.

It is know small deviations in the chemical compositions of these types of Heusler alloys can lead to significant changes in their MCE parameters [4,16]. Thus, a non-uniform compositional distribution throughout a sample may result in a longer time required to establish thermodynamic equilibrium. However, the EDX analysis did not reveal such heterogeneity in these samples.

2.3. Magnetic properties

The magnetization ($M(T,H)$) of the samples were measured as a function of temperature in the range 5–400 K and in magnetic fields up to 5 T using a SQUID magnetometer (Quantum Design, USA). The samples were heated to 380 K prior to the measurements at constant magnetic field after the samples were cooled to 5 K in zero magnetic field (ZFC), or in the presence of a constant magnetic field (FC), or during cooling at constant magnetic field (FCC). The transition temperatures were determined from the inflection points of the $M(T,H)$ curves, when possible, or as the center of the transition region.

The magnetization curves $M(T)$ reveal a temperature hysteresis characteristic of first order MST (see in the Fig. 1 for $M(T)$ data obtained using ZFC/FC and FCC protocols). However, the magnetic state of austenitic phase (AP) just above the transition temperature was found to be different. APs with magnetizations of about 60 and 70 emu/g were observed for $Ni_{45}Mn_{43}CoSn_{11}$ and $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$, respectively, (see Fig. 1 (a, c) at $\mu_0 H = 5$ T). A transition with changes in magnetization of about (0.1–0.15) emu/g at $\mu_0 H = 0.01$ T, typical for a paramagnetic-paramagnetic transition [4], was found to accompany the MST in the $Ni_{50}Mn_{36.5}In_{13.5}$ alloy. The transition temperatures (T_c) of the AP, and of direct and inverse MST, T_M and T_A, respectively, are listed in Table 1.

3. Results and discussion

3.1. $Ni_{50}Mn_{43}CoSn_{11}$

The time dependences of the temperature changes with the adiabatic magnetization of $Ni_{45}Mn_{43}CoSn_{11}$ are shown in Fig. 2 near the MST and T_C. As can be seen in Fig. 2(a–c), the rapid changes in the magnetic field at the temperature T_0, which is close to the starting temperature of the transition from martensitic phase (MP) to AP (see Fig. 2), results in the fast decrease in the temperature of the sample to a certain (initial) value of T_{in}. Afterwards, the temperature increases gradually and comes to a stable (equilibrium) value T_{eq}. The maximum temperature change of $\Delta T_r = T_{eq} - T_{in}$, associated with the relaxation time of the system, was observed just below the MST (at a starting temperature of $T_0 = 265$ K $\Delta T_r \approx 2$ K). With the further increase of starting temperature (Fig. 2(d–g)), as expected in the case of direct MCE, the temperature of the sample increases when the field is switched on, and ΔT_r

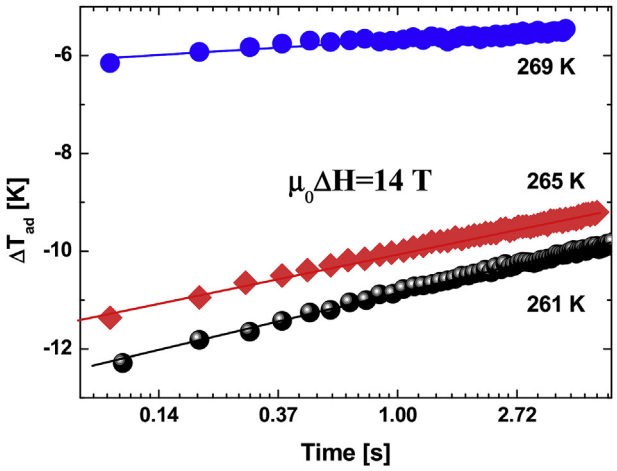

Fig. 3. ΔT_{ad} relaxation time observed for of $Ni_{45}Mn_{43}CoSn_{11}$ after 14 T magnetic field changes.

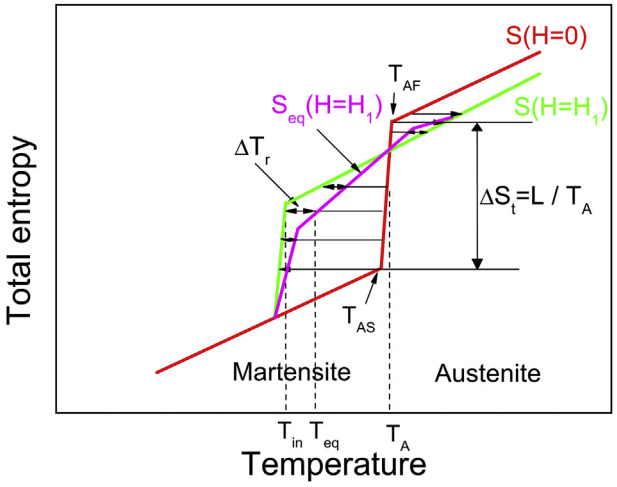

Fig. 4. (S_t-T)- diagram demonstrating the relaxation of ΔT_{ad} in the vicinity of the MST.

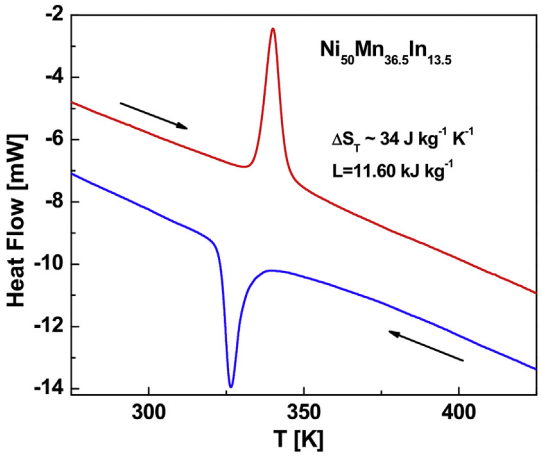

Fig. 5. DSC heat flow curves as a function of temperature for $Ni_{50}Mn_{36.5}In_{13.5}$ obtained for cooling (red), and heating (blue) lines. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

changes sign and decreases in the AP near the Curie temperature of the AP. Thus, far from the MST (Fig. 2 f), temperature relaxation is nearly absent. A slight relaxation in this case is caused by heat transfer between the sample, the sample holder and the thermocouple. For example, in the region of the Curie temperature, the value of ΔT_r does not exceed 0.2 K. This value is an order of magnitude smaller than the value observed in the region of the magnetostructure transition. The contribution of these heat transfer processes to the total relaxation of the sample temperature can be estimated by the formula $\Delta T_r(t) = \Delta T_{ad}(1\text{-}\exp(t/\tau))$, where τ is the characteristic relaxation time due to nonadiabaticity [17], and this contribution is certainly small in the total change of

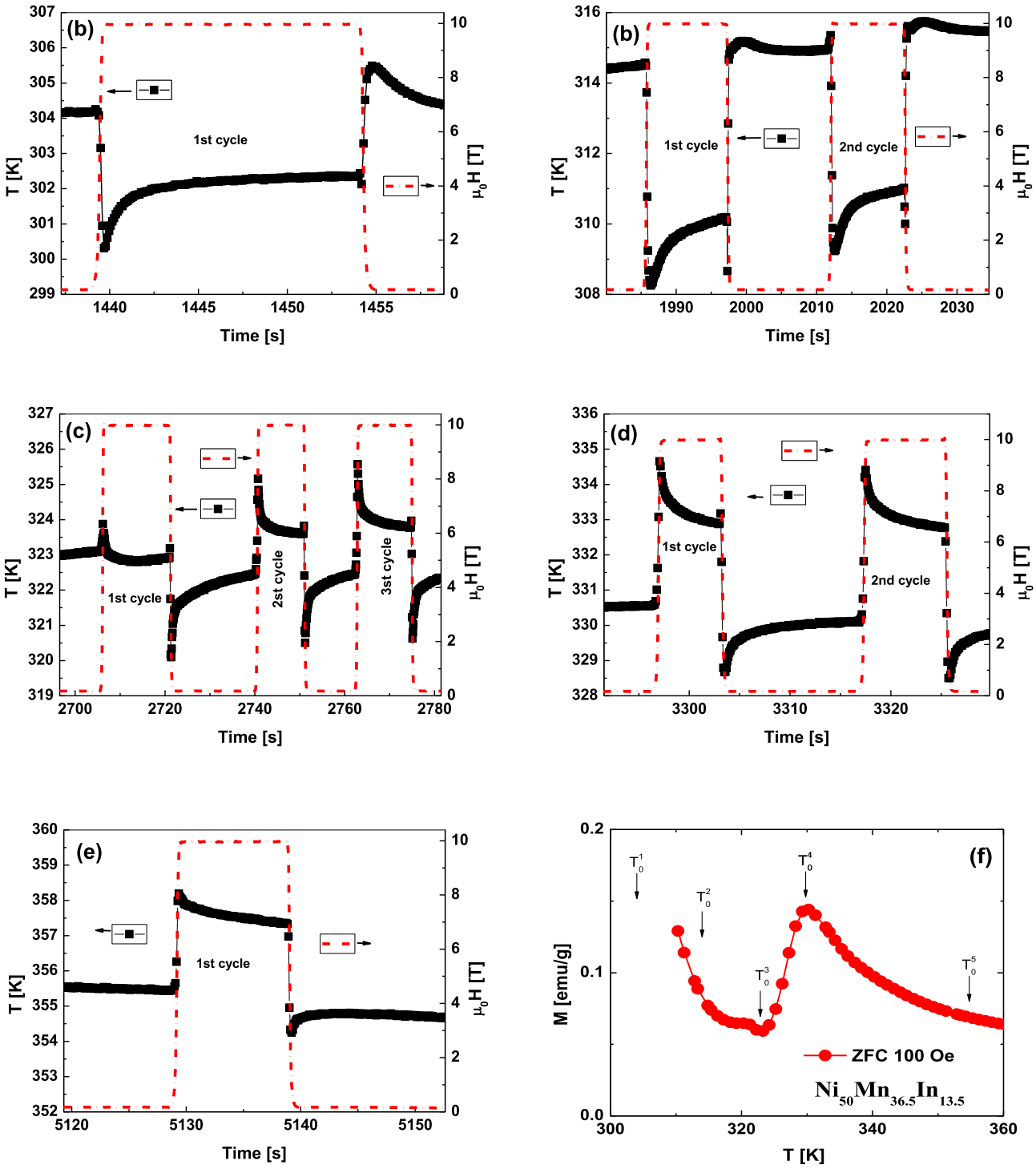

Fig. 6. (a–e): The time dependencies of ΔT_{ad} obtained for $Ni_{50}Mn_{36.5}In_{13.5}$ near of the MST and T_C; (f): The temperature dependence of the magnetization measured while heating the $Ni_{50}Mn_{36.5}In_{13.5}$ sample in a magnetic field of 0.01 T. The arrows show the initial temperatures T_0 for the adiabatic temperature changes measurements (shown in Fig. 6a–e).

temperature in the region of the magnetostructural transition.

The following formula was proposed to describe the time dependence of the.

Magnetization in the close vicinity of the MST [9]:

$$M(t) = M(0)+Zln(1 + t/t_0) \quad (1)$$

where $M(0) = M(t = 0)$, t_0, and Z are parameters determined experimentally. In this case "Z" can be considered as an analog of magnetic viscosity. Thus, it is possible to write adiabatic changes in the temperature as:

$$\Delta T_{ad}(t) = \Delta T_{ad}(0)+Z_1 ln(1 + t/t_0) \quad (2)$$

where $\Delta T_{ad}(0) = \Delta T_{ad}\ (t = 0)$, t_0, and Z_1 are parameters determined experimentally from the time dependence of the adiabatic temperature (see in Fig. 3). As shown in Fig. 3, the logarithmic dependence given by formula (2) is satisfied for all curves (261, 265, and 269 K). It is obvious that the logarithmic law follows from a wide distribution of relaxation times [3], i.e., the presence of an energy landscape with different barrier widths. In Ref. [9] it was found that the relaxation of the magnetization under isothermal conditions lasts for several minutes, whereas in our experiments the characteristic relaxation time ΔT_{ad} is about 2 s. The reason for such a difference is different experimental conditions, different initial thermodynamic conditions and temperatures, and the condition that the relaxation of ΔT_{ad} and magnetization are not proportional to each other. The latter follows from the Maxwell relation:

$$\Delta T_{ad} = -\frac{\mathbf{T}}{\mathbf{C}}\left(\frac{\mathbf{dM(T,H)}}{\mathbf{dT}}\right)\mathbf{dH} \quad (3)$$

Unlike with isothermal conditions, under adiabatic conditions, the magnetically induced transition is hampered by the change in temperature caused by adiabatic magnetization. Therefore, the MST in adiabatic conditions occurs at higher magnetic fields than in isothermal conditions [11]. The behavior of the curves presented in Fig. 2 can be understood from expression (3). A stepwise application of magnetic field leads to a rapid increase in magnetization, since the orientation of magnetic moments in the magnetic field is practically non-inertial (spin-lattice and spin-spin relaxation times are known to be less than one μsec), i.e., the magnetization reaches its maximum value in this field. If the magnetization increase reduces the entropy, the sample temperature should increase and not decrease, as observed in the experiment at temperatures lower than the final temperature of the MST. Thus, nearly simultaneously with the change in magnetization, ions move away from their equilibrium positions, even though the AP has not yet formed. This process occurs quite quickly due to the similar values of austenite and martensite energies and the non-diffusive character of the transition, leading to a decrease in temperature since it is energetically advantageous and $dM/dT > 0$.

After the increasing field reaches static value, the relaxation process of newly formed, unstable AP begins (Fig. 2 g). The unstable state of the austenitic phase is due to the fact that it formed under adiabatic conditions. In turn, the relaxation is associated with the destruction of the AP, i.e., the formation of the martensitic phase. Moreover, the formation of the martensitic phase is accompanied by an increase in temperature (Fig. 2a–c). Above 275 K, a direct MCE is observed (Fig. 2d and e). This is due to the fact that the main contribution to the MCE in this temperature range is the MCE of the paraprocess (dM/dT < 0) of the partially or fully formed AP. However, the AP formed upon application of magnetic field is also not stable under adiabatic conditions. In this case, the destruction of austenite leads to a decrease in temperature, due to a decrease in the quantitative content of austenite, which gives a positive (direct) MCE of the paraprocess.

When the sample is in the AP, the application of the field also leads to an increase in magnetization, but also increases of ion oscillations, i.e., increases the sample temperature.

It can be seen from Fig. 2 that the changes in temperature near the T_C almost immediately (within the available time resolution) follow the changes in magnetization. That is, when the application of magnetic field does not lead to a change in the phase ratio, and there is no release of the latent heat, the relaxation processes certainly lasts less than a tenth or a hundredth of a second. At the same time, the most obvious relaxation processes are concentrated in the region with a strong change in the phase ratio, accompanied by a significant release of the latent heat of the transition.

A similar phenomenon should be observed in the samples with the highest values of the latent heat of the magnetically induced transitions. The $Ni_{45}Mn_{43}CoSn_{11}$ sample shows a latent transition heat of 8.7 kJ/kg [18], which is a rather high for this class of materials. Therefore, it seems appropriate to compare and test this hypothesis to investigate relaxation processes in Heusler alloys with different values of the latent heat of the transitions (the samples composition selected for this study see in Table 1).

A qualitative (S_t-T)-diagram that can explain the experimental changes in adiabatic temperature is presented in Fig. 4. It is known that the total entropy increases sharply in the vicinity of the MST. Thus, the change in the total entropy (ΔS_t) can be defined as:

$$\Delta S_t = L/T_A \quad (4)$$

where L is the latent heat of the MST. As mentioned above, the relaxation of the adiabatic temperature was observed in the MST region (with the co-existence of the AP and MP, i.e., a mixed state). As can be seen from the (S_t-T)- diagram (see in Fig. 4), the region of the structural transition at H = 0 is determined by the value ΔS_t. The temperature of the transition region is determined by the starting and finishing temperatures, T_{AS} and T_{AF}, respectively. Therefore, the most visible relaxation of ΔT_{ad} should be observed in materials with large values of latent heat (see in Fig. 4).

3.2. 2 $Ni_{50}Mn_{36.5}In_{13.5}$

In $Ni_{50}Mn_{36.5}In_{13.5}$ alloys, the MT occurs in the paramagnetic state (Fig. 1b) at a temperature of $T_M \approx 358$ K [19]. The DSC data (Fig. 5) show a large value of latent transition heat for this alloy

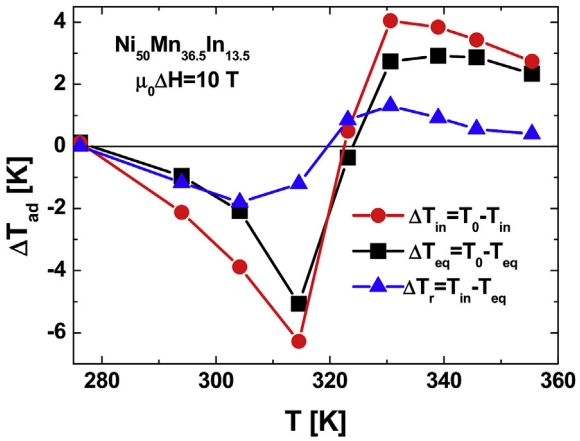

Fig. 7. The temperature dependencies of ΔT_{in}, ΔT_{eq} and ΔT_r observed for $Ni_{50}Mn_{36.5}In_{13.5}$ at magnetic field changes of 10 T.

(11.60 kJ/kg). The adiabatic temperature changes as a result of rapid magnetization of $Ni_{50}Mn_{36.5}In_{13.5}$ are shown in Fig. 6. The relaxation is pronounced and the relaxing part of the adiabatic temperature change reaches a relativity high value of about 2 K. If the starting temperature T_0 is in the (294–314) K interval where $dM/dT > 0$, the application of field leads to cooling (Fig. 6a–c), and when $dM/dT < 0$ the sample temperature increases. It is interesting that, in contrast to the previous case (of $Ni_{45}Mn_{43}CoSn_{11}$ see in

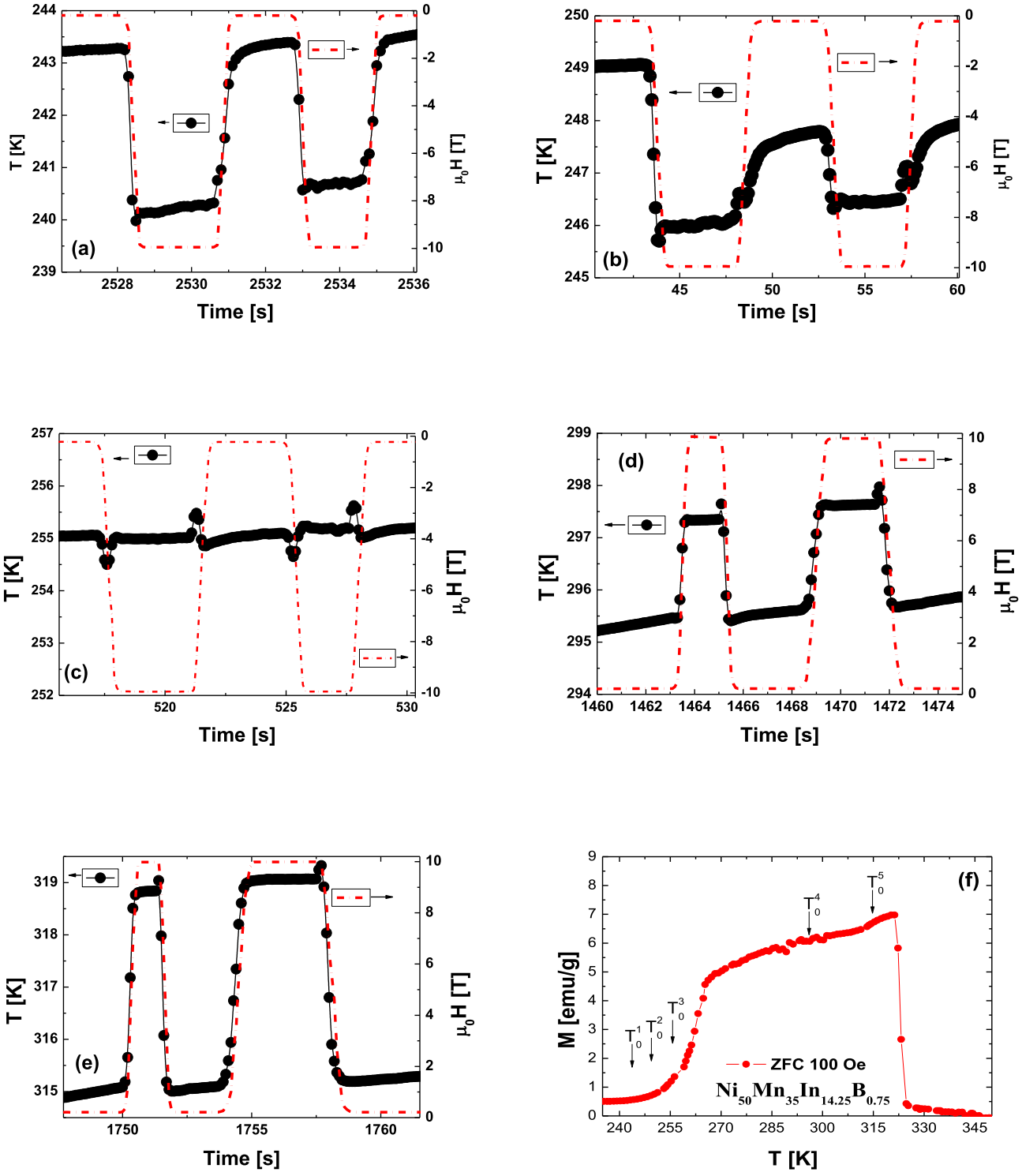

Fig. 8. (a–e): The time dependencies of ΔT_{ad} obtained for $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$ near the MST and T_c. (f): The temperature dependence of the magnetization of $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$ measured while heating a H = 0.01 T. The arrows show the initial temperatures T_0 for adiabatic temperature change measurements (shown in Fig. 8a–e).

Fig. 2) the relaxation is strongly visible in the paramagnetic region of the AP. Apparently, this is due to the fact that in this case the application of high magnetic field leads to the formation of the austenitic phase with high magnetization and to shift of the MST to lower temperature (as it is shown in Fig. 1b).

From the obtained data (Fig. 6) it is possible to reveal the temperature dependence of ΔT_{ad} for the initial value of the adiabatic temperature change ($\Delta T_{in} = T_{in}$-T_0), the equilibrium value of the adiabatic temperature changes ($\Delta T_{eq} = T_{eq}$-T_0), and the relaxing part of the adiabatic temperature change ($\Delta T_r = T_{in}$-T_{eq}).

The temperature dependencies of the adiabatic temperature changes of ΔT_{in}, ΔT_{eq} and ΔT_{eq} can be determined from the results presented in Fig. 7. As can be seen from the graph, all three values demonstrate a similar behavior, and change sign in the MST. With an increase in the magnitude of the magnetic field, the temperature range in which the phenomenon of ΔT_{ad} relaxation is observed increases, just as the peak ΔT_{ad} is broadened in the region of the MST (not shown).

3.3. $Ni_{50}Mn_{35}In_{14.25}B_{0.75}$

The temperature of the MST for this compound is $T_A = 269$ K, the Curie temperature $T_C = 324$ K (Fig. 1c), and the latent heat $L = 2.7$ kJ/kg [20]. As can be seen from the graph (Fig. 8), the relaxation effect of the adiabatic temperature change was not observed outside the MT region (Fig. 8 (a), starting temperature $T_0 = 232.5$ K), at the beginning of the transition (Fig. 8b, $T_0 = 243$ K), and near the end of the transition (Fig.8e, $T_0 = 315$ K). An interesting behavior was observed in the middle of the temperature range of the MP-AP transition (Fig.8c, $T_0 = 255$ K). As the field increases, the temperature of the sample first decreases rapidly, and then increases rapidly. Although these changes are insignificant (less than 0.5 K), they show that the process can be described as occurring in two stages. First, ion oscillations occur (as in the direct MCE), most likely due to the presence of ferromagnetic regions of the AP. However, when the field reaches the threshold values for the sufficient displacement of ions from their equilibrium positions, already at an increased temperature, structural changes result in a inverse MCE.

4. Conclusions

In the case of the direct of MCE in the vicinity of the second order magnetic phase transition in $Ni_{45}Mn_{43}CoSn_{11}$, the changes in temperature under adiabatic conditions nearly follow the application of field. Given that the time resolution in the extraction method is about 0.1 (s) or better, it is reasonable to assume that the characteristic times of the energy redistribution between the magnetic and ionic systems are shorter than this time. The situation is different at the inverse MST since the relaxation times of the adiabatic temperature changes can reach 10 s or more, depending on the latent heat of the transition and the initial temperature. It is important to note that, with a significantly large value of the latent heat of transition, the relaxation of the adiabatic temperature change is comparable to the value of the temperature change and, therefore, the measured temperature may be overestimate, when applying pulse techniques. The process of relaxation of the adiabatic temperature changes can be described by the same logarithmic law as that which describes the relaxation of magnetization, but with different parameters. This logarithmic law indicates the presence of a wide range of characteristic relaxation times, but the specific value of the parameters of this law should be justified from microscopic theory.

Author contribution

All co-authors contributed equally to the article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

This work was supported by the Office of Basic Energy Sciences, Material Science Division of the U.S. Department of Energy, DOE Grant No. DE-FG02-06ER46291 (SIU) and DE-FG02-13ER46946 (LSU). YK acknowledges support from a grant from the National Science Center Poland (NCN) under the SONATA program (2016/21/D/ST3/03435). E. L. and A. G. are thankful for the Researcher Mobility Grant of the Academy of Finland (No. 318405).

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