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Dependence of the inverse magnetocaloric effect on the field-change rate in Mn_3GaC and its relationship to the kinetics of the phase transition

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We study the dependence of the magnetocaloric effect on the magnetic field-change-rate the first order magnetostructural transition in Mn_3GaC by measuring the adiabatic temperature change ΔT at three different time scales: 11 mT s^{-1}, 700 mT s^{-1}, and ~1000 T s^{-1}. We find that the maximum adiabatic temperature-change of about 5 K is reached in the 11 mT s^{-1} and 700 mT s^{-1} rates, whereas for the ~1000 T s^{-1}-rate the transition lags the change in the magnetic field so that the maximum adiabatic temperature-change is not attained. © *2015 AIP Publishing LLC*.
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I. INTRODUCTION

In recent years, research on materials with a large magnetocaloric effect (MCE) increased due to their potential use in more efficient cooling devices than those which use the gas-compression method.[1,2] Materials that show a large conventional or inverse MCE generally rely on the presence of a first-order magnetostructural transition. These materials can be suitable for magnetocaloric refrigeration provided that the transitional hysteresis is sufficiently narrow, i.e., the magnetic-field-shift of the magnetostructural transition temperature should considerably exceed the temperature-width of the hysteresis.[3,4] Only this assures the reversibility of the adiabatic temperature-change $\Delta T_{ad} = T_{fin} - T_{init}$ on continuously cycling the magnetic field, where T_{fin} and T_{init} are the final and initial temperatures.

The magnetocaloric material Mn_3GaC undergoes a first-order magnetostructural transition at 162 K from a low-temperature antiferromagnetic (AF) to a high-temperature ferromagnetic (FM) state.[5] The entropy density in this system is 17 J kg^{-1} K^{-1},[6,7] and a reversible $\Delta T_{ad} = \pm 2.8$ K is found when cycled in a magnetic field of 3 T.[8] A field dependent shift of the transition temperature of -6 K T^{-1} allows a temperature-shift of the hysteresis that is greater than the 3 K-width of the hysteresis in moderate magnetic fields of about 2 T. These values are obtained from the results of experiments done using a slow field-change rate of 11 mT s^{-1}.[8] In magnetocaloric cooling devices, fast field-change rates are required, and it is necessary to investigate how the material reacts to rapid magnetic field-changes.[13] Therefore, we study ΔT_{ad} for different rates of magnetic-field-change ranging over several orders of magnitude. We measure at rates of 11 mT s^{-1} using a 5 T superconducting magnet, 700 mT s^{-1} using a permanent−magnet setup, and ~1000 Ts^{-1} using a pulsed magnetic field.

II. EXPERIMENTAL

A polycrystalline Mn_3GaC sample was prepared by mixing Mn powder (99.95%), Ga pieces (99.99%), and C powder (99.9+ %) in stoichiometric proportion and additional 10% Carbon. The powder was pressed into a pellet using a hydraulic press and sealed into a quartz tube under argon atmosphere. The mixture was let to react at 800 °C for 5 days. The sample was then ground, and the process was repeated once more. X-ray powder diffraction confirmed the antiperovskite cubic structure (Pm-3 m) with a lattice constant $a = 0.3895 \pm 0.0001$ nm that agrees well with previous studies.[8–10] The sample was single phase, and energy dispersive X-ray spectroscopy (EDX) analysis gave a concentration of 76 at. % Mn and 24 at. % Ga with an error of about 1%. ΔT_{ad} in field-changes of 11 mT s^{-1} was measured in an adiabatic calorimeter located within the bore of a 5 T superconducting magnet. The sample-temperature was determined by a thermocouple placed inside the sample by thermal conductive epoxy. For measurements at intermediate field rates of 700 mT s^{-1}, a device with two nested Halbach magnets was used. By rotating the magnets, fields of 2 T can be ramped up and down with a sinusoidal magnetic field profile. ΔT_{ad} was measured with a differential thermocouple fixed in the center of the sample. A similar differential measurement method was employed to determine ΔT_{ad} in the pulsed-field setup at Dresden high magnetic field laboratory. The field rate here was ~1000 T s^{-1}. We used a 75 ms pulse with a maximum magnetic field of 10 T. The total observation time including pre and post pulse periods was 200 ms. The thermocouple readings were taken every 1 ms. To ensure identical initial conditions for all measurements, the sample was warmed up to the pure FM state and subsequently cooled down to the pure AF state before each measurement. The field and temperature-dependent magnetizations, $M(H)$ and $M(T)$, were measured using a vibrating sample magnetometer and a

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superconducting quantum interference device magnetometer. A field sweep-rate of $20\,\mathrm{mT\,s^{-1}}$ was used in the $M(H)$ measurements.

III. TEMPERATURE-CHANGE AT CONSTANT ENTROPY AT A FIRST-ORDER MAGNETOSTRUCTURAL TRANSITION WITH HYSTERESIS

To support the discussions given below, we provide in Fig. 1 an outline for the adiabatic temperature-change process in the inverse MCE when a first-order magnetostructural transition with hysteresis is present. The outline is general and can be considered for systems with any hysteresis width. Fig. 1 shows schematically a sequence of the temperature-dependence of the total entropy with occurring temperature-changes in the vicinity of a magnetostructural transition when a cyclic magnetic field is applied. The field-changes occur at constant entropy S_0. State A is the high-temperature, high-magnetization state, and state B is the low-temperature, low-magnetization state. The system can be in a mixed state along the forward (solid line) and reverse (dashed line) transformation branches of the hysteresis. These are also shown by arrows in Fig. 1(a). The height of the branch is a measure of the A to B ratio. The initial and final states before and after changing the field are shown with open and filled circles, respectively, in all panels of Fig. 1. The magnetic field is increased and decreased following the sequence shown in Figs. 1(a)–1(e). Each initial state is the final state of the preceding panel as of Fig. 1(b).

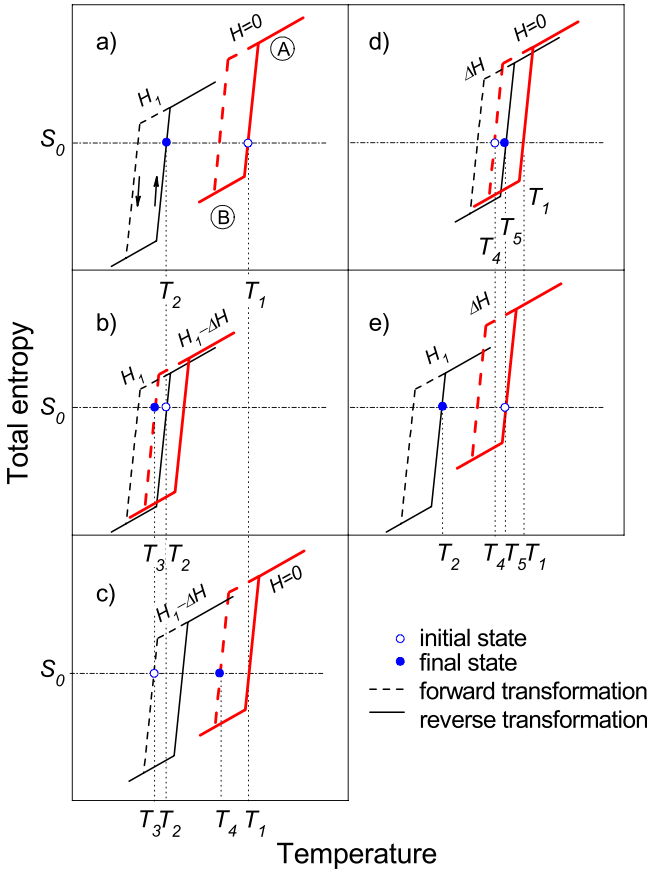

FIG. 1. Schematic sequence of temperature-changes seen in the temperature-dependence of the total entropy under cyclic magnetic-field changes. The field-changes occur at constant entropy S_0. The sequence (a)–(e) is described in the text.

In Fig. 1(a), the system is at the initial temperature T_1 and for $H=0$. The state of the system is initially located on the reverse transformation branch at a given ratio of A and B. When a field H_1 is applied, the transition temperature, and thereby the hysteresis, shifts to a lower temperature T_2 so that the initial adiabatic cooling is expressed as $\Delta T_{ad}=T_2-T_1$. Since the process occurs at constant entropy, the state of the system is carried to a higher point (higher amount A) on the reverse transformation branch. When the field is now decreased by ΔH as shown in Fig. 1(b), the proportion cannot change in favor of state B since the state of the system is located on the reverse transformation branch. Therefore, the system exhibits the conventional MCE and cools to T_3 while the field decreases and the hysteresis shifts at the same time such that the forward transformation branch coincides with T_3 when the field is $H_1-\Delta H$. Now, the ratio can change in favor of state B as the field is further decreased, and the sample warms with the inverse MCE. At $H=0$, the state of the sample remains on the forward transformation branch at $T_4<T_1$ shown in Fig. 1(c).

At $T=T_4$ in Fig. 1(d), and similar to the case shown in Fig. 1(b), the state of the system cannot change when the field is reapplied. The state of the system must first fall on the reverse transformation path. Therefore, up to a field ΔH, the system exhibits the conventional MCE and warms further to T_5, which is less than T_1. At T_5, the state coincides with the point on the reverse transformation branch. Further increasing the field from here leads to cooling to T_2 since now the state of the system can change giving rise to the inverse MCE. The cycle is thus completed.

The initial temperature-change T_1-T_3 is always larger than the following temperature-changes T_5-T_3 in subsequent cycles. The narrower the hysteresis, the less significant is the difference T_2-T_3 and T_5-T_4.

IV. RESULTS AND DISCUSSION

Fig. 2 shows the magnetization of Mn_3GaC in the temperature range $10\le T\le 380\,\mathrm{K}$ in a field of 5 mT for

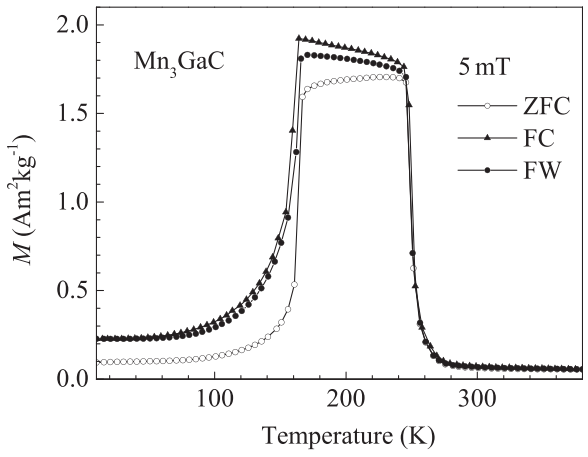

FIG. 2. Temperature dependence of the magnetization under 5 mT for ZFC, FC, and FW measurements. FC and FW measurements show a thermal hysteresis of 3 K.

zero-field-cooled (ZFC), field-cooled (FC), and field-warmed (FW) measurement protocols. The sample undergoes a first-order magnetostructural transition at $T_i = 163$ K with a thermal hysteresis of about 3 K measured between the FC and FW curves. The transition temperature is shifted by an external magnetic field to lower temperatures by about -6 K T^{-1}. This value is slightly larger than other literature values[8,9] and can be related to differences in purity or stoichiometry.[11] The Curie temperature T_C of the sample is 253 K.

The field-dependent magnetization $M(H)$ around T_i is plotted in Fig. 3. $M(H)$ for increasing field is shown with filled symbols, and the open symbols indicate $M(H)$ for decreasing field. The magnetization gradually increases until the critical field H_{S1} is reached (shown for the 145 K-data) where the system starts to transform to the FM phase. A further increase of the field by about 0.3 T leads to a sharp rise in $M(H)$, and the sample transforms to the pure FM state at H_{F1}. The saturation magnetization of 70 A m^2 kg^{-1} agrees well with other reported values.[9] When the field is decreased, a magnetic hysteresis of about 1 T is observed, measured between the critical field H_{F1}, where the transition is completed, and H_{S2}, where the reverse transformation starts. The reverse transformation is completed at H_{F2}. The $M(H)$ curve at 160 K shows for increasing field a transition to the FM state. When the field is ramped down, the reverse transformation to the AF state is not realized, and the system remains in the FM state. The decrease of the magnetization in this case is related to the demagnetization of the FM phase.

ΔT_{ad} for Mn_3GaC under a field-change rate of 11 mT s^{-1} is shown for the temperatures 144, 150, and 160 K in Fig. 4. The fields characterizing the transition are shown for the 150 K-data, which is discussed first. At this temperature, $\Delta T_{ad} = 0$ as long as the field is lower than the critical field H_{S1}. Between H_{S1} and H_{F1} the system shows an inverse MCE and ΔT_{ad} becomes strongly negative. In the case of a complete transition to the FM state, ΔT_{ad} of -4.4 K is reached at H_{F1}. Afterwards, as the field is further increased, a conventional MCE is observed where ΔT_{ad} steadily decreases until the field reaches the maximum value of 5 T. Here, the sample is kept at this field for 10 min to check the

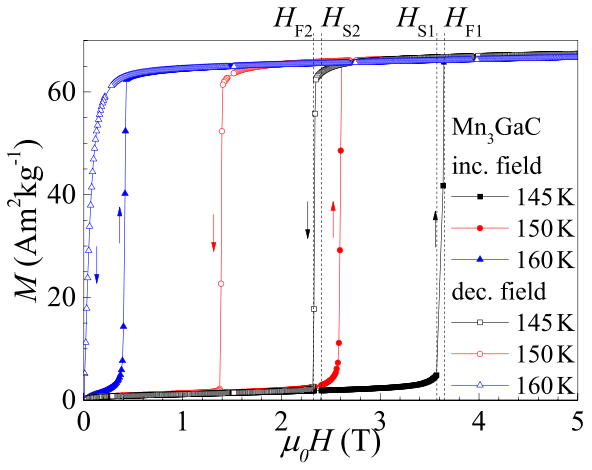

FIG. 3. Field dependence of the magnetization at different temperatures below T_i measured with a field rate of 2 mT s^{-1}. Full symbols: increasing field, open symbols: decreasing field.

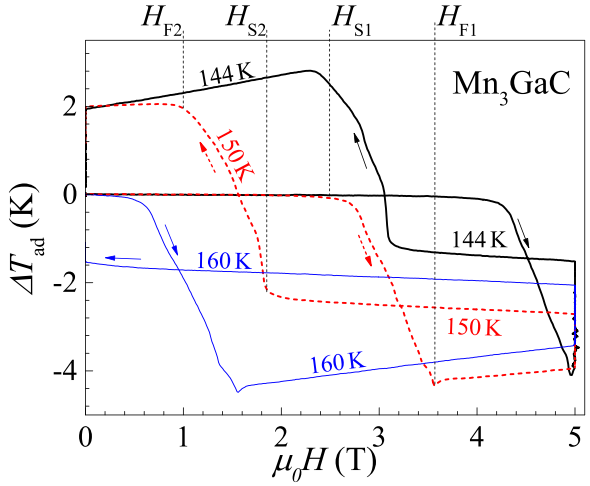

FIG. 4. Temperature-change on applying and removing a magnetic field of 5 T with a field rate of 11 mT s^{-1}. Mn_3GaC shows an inverse MCE below T_i and a conventional MCE in the FM regime.

adiabatic conditions in the calorimeter. During this time, the sample warms by about 1 K. The field is then decreased. Down to H_{S2}, the conventional MCE with a slight decrease in ΔT_{ad} is observed. This decrease is a net effect due to the superposition of the thermal drift in the calorimeter and the small conventional MCE.

Comparing the slope of ΔT_{ad} at 150 K in increasing field from 3.6 T to 5 T and in decreasing field from 5 T to 2 T, a smaller slope for the decreasing field can be observed. This is because for the decreasing field, the conventional MCE causes a decrease in the temperature, while for increasing field it causes an increase. The superposition of thermal drift and the conventional MCE leads to the different slopes in these ranges.

Between H_{S2} and H_{F2}, the sample transforms back to the AF state. The magnitude of ΔT_{ad} observed for the forward and the reverse transition is similar and indicates that ΔT_{ad} shows a high degree of reversibility as discussed in previous studies.[12] The critical start temperatures H_{S1} determined from adiabatic ΔT_{ad} and isothermal $M(T)$ measurements are in agreement. The rapid transition observed in $M(H)$ in Fig. 3 is observed to be broader in $\Delta T_{ad}(H)$ in Fig. 4. This is due to the temperature change of the sample when a field is applied adiabatically leading to a distribution of critical fields. The behavior of ΔT_{ad} at the other temperatures can be discussed similarly.

Fig. 5 shows ΔT_{ad} for a sinusoidally oscillating magnetic field of 2 T with an average field-change rate of 700 mT s^{-1}. The characteristic fields are shown for the 158 K-data. The field is applied in forward positive and reverse directions to the sample. After the field overcomes H_{S1}, ΔT_{ad} becomes strongly negative indicating the beginning of the transition to the FM phase. The transition finishes at H_{F1} with $\Delta T_{ad} = -4.7$ K which is close to the expected maximum value.[9] As the field is reduced, ΔT_{ad} first remains constant after which it begins to increase at H_{S2} as the AF state sets in. In this case, H_{F2} is reached at zero-field without the full stabilization of the AF state. Reversing the field has essentially the same effect of reapplying the field in the same direction.

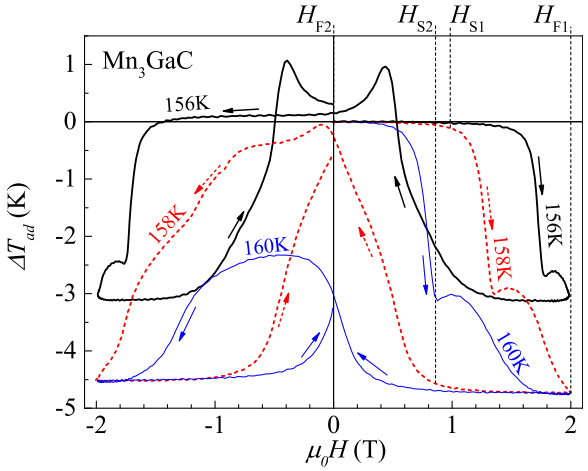

FIG. 5. Temperature-change on applying and removing a magnetic field of 2 T with a field rate of 700 mT s^{-1}. For $T \geq 158$ K the system transforms fully to the FM state.

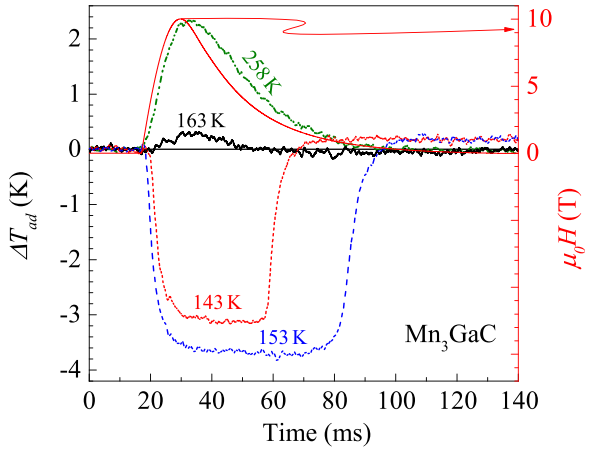

FIG. 6. Time-dependence of the temperature-change (left axis) and magnetic field (right axis) for observation time of 25 ms, 10 T magnetic field pulse (red solid line). Measurements at the second-order transition $T_C = 258$ K and in the FM state at 163 K show conventional MCE. The MCE at 143 and 153 K is inverse.

While increasing the field in the reverse direction, ΔT_{ad} becomes negative and reaches its largest absolute value as the FM phase of the sample increases. On removing the field, the state of the system does not change until the forward transformation to the AF state begins. At this point, the sample begins to warm.

For starting temperatures of 156 K and below the system transforms back to the pure AF state when the field is removed, and the curves for positive and negative field-directions are similar except for a small thermal drift. The critical fields are not affected by the magnetic field direction and agree with those determined from $M(H)$, whereby must be considered that the smooth change of the temperature leads to an enlarged error bar for the critical fields. ΔT_{ad} for the transition from the AF to the FM state in positive field shows an additional superimposed conventional MCE with an increase of about 0.3 K as the field approaches H_{F1}. This superimposed MCE can only occur if the AF/FM ratio remains constant while changing the field. This can happen if the transition pauses. When the transition resumes at higher fields, the ratio changes again in favor of the FM state. A changing ratio indicates the occurrences of the inverse MCE again so that the system cools as the field increases. Comparing this feature with the ΔT_{ad} curve in Fig. 4 at similar temperatures, it is also possible to observe a weak variation in the slope of the curve around similar fields. The lower rate of applying the field in this case can allow the system to relax and, therefore, can smoothen this effect. The cause of this "pause" effect is not clear at the moment, and further investigation are required.

ΔT_{ad} measured under the fast field-change rate of $\sim$1000 T s^{-1} up to a magnetic field of 10 T are shown in Figs. 6 and 7 as a function of time and magnetic field, respectively, at various temperatures. Fig. 6 includes the time-dependence of the pulsed magnetic field and ΔT_{ad} in a time-window of 140 ms. The field-pulse is given 16.5 ms after the start of recording and reaches its maximum at 30 ms (right scale). To check for response delays between the pulse and the thermocouple, we measure ΔT_{ad} at T_C (258 K) where a sizable conventional MCE is expected due to the FM-PM second-order transition. The data show that ΔT_{ad} follows the magnetic field change with a delay of about 3 ms in rising-field and 6 to 8 ms in decreasing-field which is related to the time constant of the thermocouple setup. At 163 K a smaller conventional MCE is also observed as expected, where the system is purely FM. At 143 and 153 K, which are below T_i, the system shows the inverse MCE above the critical field H_{S1}. A maximum temperature-change of -3.8 K for 153 K and -3.0 K for 143 K is observed. On decreasing field, ΔT_{ad} unexpectedly continues to decrease slowly showing the conventional MCE, which can only be caused by the system being trapped in a state where the AF/FM ratio does not change. At a low-enough field, the system transforms back to the AF state. The maximum in ΔT_{ad} and also the decrease back to $\Delta T_{ad} = 0$ is observed after 12 ms at 143 K and 153 K.

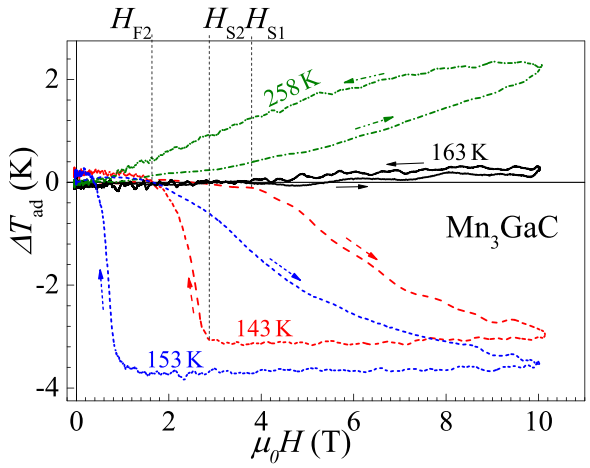

FIG. 7. Temperature change during a magnetic pulse of 10 T with a field rate of $\sim$1000 T s^{-1}. Measurements in the FM state at 163 K and at $T_C = 258$ K exhibit a conventional MCE. The MCE is inverse at 143 K and 153 K. The continued decrease of ΔT_{ad} up to maximum fields along with its reduced absolute value is associated with an incomplete first-order transition at these field-change rates.

To understand the transition process in more detail, we plot ΔT_{ad} against the applied field in Fig. 7. At 258 and 163 K, the conventional MCE is observed, and the temperature-change starts directly when the pulsed field increases. The different paths taken by ΔT_{ad} for increasing and decreasing-fields are related to the time constant of the thermocouple. This is less apparent at 163 K due to the small size of the MCE.

In the case of the starting temperature 143 and 153 K which are close to T_i, the decrease in T begins at H_{S1}. The characteristic fields adopted from the features in Fig. 4 are indicated for the data at 143 K. Above H_{S1} about 1 T is sufficient to induce a full transition to the FM state in Mn_3GaC at the slower field-change rates shown in Figs. 4 and 5. This is not observed for the fast field-change rate measurements. ΔT_{ad} decreases continuously up to the maximum field of 10 T without reaching the extremal ΔT_{ad} of −4.5 K. This indicates that the transition is not complete at 10 T (30 ms) meaning that the transition cannot follow the magnetic-field change. As soon as the field begins to decrease, the system locks in a mixed state with a non-changing AF/FM ratio and exhibits the conventional MCE by a very slow continued decrease in ΔT_{ad} (that actually appears almost field-independent) until the field decreases to below H_{S2}. Only below this field, the system can transform back to the AF state. The different slopes in the field dependence of ΔT_{ad} for the transition to the FM and AF states are related to the different field-change rates for increasing and decreasing-fields, which can be seen in the pulse profile in Fig. 6. These results show that the transition cannot follow the field at a pulse where the field reaches its maximum in 12 ms. One explanation for the slow progression of the transition can be related to the high internal stress caused by the volume difference of about 0.5% of the coexisting phases. This is supported by the results of previous neutron-diffraction studies of Mn_3GaC which show a change in the isotropic displacement parameter around the transition which is an indication for the presence of local strain.[10] More investigations on the magnetic and structural changes during the transition are necessary to understand this process in detail.

In Fig. 8, the extremal ΔT_{ad} obtained at all field-change rates are plotted against temperature. Furthermore the magnetic induced entropy change ΔS as a function of temperature are shown. The entropy change ΔS is calculated from the isothermal $M(H)$ measurements. For temperatures above 162 K, the system shows a conventional MCE with positive ΔT_{ad}. At the intermediate rate of 700 mT s^{-1}, ΔT_{ad} of − 4.7 K at 2 T can be reached which is close to that estimated by Tohei and Wada.[9] The ΔS measurements show that the maximum ΔS of 14 J kg^{-1} K^{-1} is achieved for a 2 T, indicating a full transition. The shift of T_i to 153 K in 2 T field would be equivalent to a shift-rate of −5.7 K T^{-1} from isothermal magnetization measurements. Below 153 K, a field of 2 T is not sufficient to induce a transition and the sample remains in the AF state, and no MCE is expected. This is certified by the ΔS measurements of 2 T. The measurements at the field-change rate of 11 mT s^{-1} up to a maximum field of 5 T show similar features except that the conventional MCE in the FM state is larger

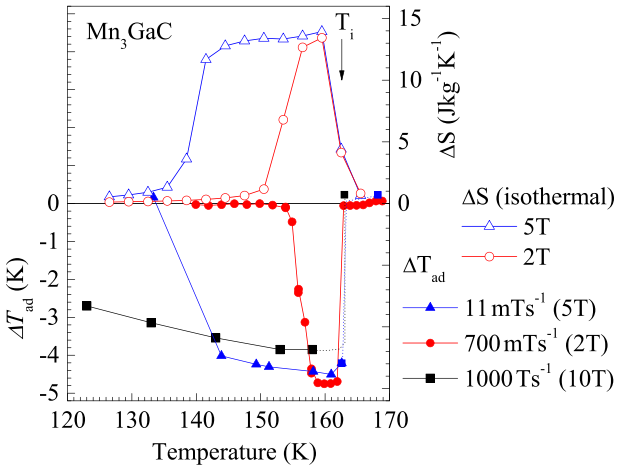

FIG. 8. Target temperature dependence of ΔT_{ad} for different field rates. ΔT_{ad} for the 10 T pulsed field is smaller because of the incomplete transition.

because of the higher applied field which is also confirm with the ΔS-data at 5 T. The inverse MCE extends to lower temperatures as the applied field increases. Since the transition is not complete for the fast field-change rate, the extremal ΔT_{ad} cannot be compared to those attained in the other measurements.

V. CONCLUSION

Mn_3GaC shows a hysteretically narrow first-order magnetostructural transition with reversible extremal ΔT_{ad} of −4.7 K under 2 T and at a field-change rate of 700 mT s^{-1} making this material interesting for cooling devices with a working-temperature around 160 K. For fast field-change rates, the system cannot follow the magnetic field and the material is not able to transform completely from the AF to the FM phase within the time that the field is applied and removed. The reason for the long transition time in Mn_3GaC is thought to be related to the local strain and the crystallographic disorder caused by coexisting different unit-cell volumes at the transition. Further studies on the field-change-rate are required in various systems undergoing first order magnetostructural transitions to understand better the relationship between local strain and the kinetics of the transition.

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