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Reversible magnetocaloric effect in materials with first order phase transitions in cyclic magnetic fields: $Fe_{48}Rh_{52}$ and $Sm_{0.6}Sr_{0.4}MnO_3$

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The magnetocaloric effect (MCE) in an $Fe_{48}Rh_{52}$ alloy and $Sm_{0.6}Sr_{0.4}MnO_3$ manganite was studied in cyclic magnetic fields. The adiabatic temperature change in the $Fe_{48}Rh_{52}$ alloy for a magnetic field change (ΔB) of 8 T and a frequency (f) of 0.13 Hz reaches the highest value of (ΔT_{ad}) of -20.2 K at 298 K. The magnitude of the MCE in $Sm_{0.6}Sr_{0.4}MnO_3$ reaches $\Delta T_{ad} = 6.1$ K at the same magnetic field change at 143 K. The temperature regions, where a strong MCE is exhibited in an alternating magnetic field, are bounded in both compounds. In the case of the $Fe_{48}Rh_{52}$ alloy, the temperature range for this phenomenon is bounded above by the ferromagnetic to antiferromagnetic transition temperature in the zero field condition during cooling. In the case of the $Sm_{0.6}Sr_{0.4}MnO_3$ manganite, the temperature range for the MCE is bounded below by the ferromagnetic-paramagnetic transition temperature in zero field during heating. The presence of these phase boundaries is a consequence of the existence of areas of irreversible magnetic-field-induced phase transitions. It is found that the effect of long-term action of thousands of cycles of magnetization/demagnetization degrades the magnetocaloric properties of the $Fe_{48}Rh_{52}$ alloy. This can be explained by the gradual decrease in the size of the ferromagnetic domains and increasing role of the domain walls due to giant magnetostriction at the ferromagnetic to antiferromagnetic transition temperature. The initial magnetocaloric properties can be restored by heating of the material above their Curie temperature. *Published by AIP Publishing*. [http://dx.doi.org/10.1063/1.4968241]

Solid state magnetic cooling technology is considered an alternative to the conventional vapor-liquid technique that is used now in air conditioners and refrigerators in industry as well as in everyday life. There is much of an interest in the search for magnetocaloric materials as the solid state cooling technology is based on them. Presently, one of the major requirements for a material to be used as a prospective magnetocaloric material is its capability to exhibit the giant magnetocaloric effect (MCE).[1] Since a refrigerating machine is a device with periodic sweeps of cycles, there is a substantial need to study the magnetocaloric properties of materials under frequent cyclic exposures to magnetic fields.

Magnetocaloric properties of the materials exposed to alternating and constant magnetic fields may show significantly different behavior for a variety of reasons. Even at low frequencies, the field dependence of the magnetocaloric properties of materials may differ significantly from those measured after a single cycle of field change. First of all, it refers to the first order phase transitions in which the temperature hysteresis can lead to irreversibility of phase transitions induced by an external field in certain temperature ranges. The MCE values on the first and subsequent cycles of the field application in these materials will vary significantly.[2–5] Thus, one of the requirements for the magnetocaloric materials is a large value of the MCE in an alternating magnetic field.

Materials showing magnetostructural phase transitions, known as materials possessing giant MCE, are considered the most promising for the emerging technology of solid state magnetic refrigeration.[6–14] These materials also encompass those in which a sharp quantitative change in lattice parameter occurs even though it is not accompanied by a change of the lattice symmetry.

In this paper, studies on two materials, namely, binary $Fe_{48}Rh_{52}$ alloy and $Sm_{0.6}Sr_{0.4}MnO_3$ manganite, have been carried out. The $Fe_{48}Rh_{52}$ alloy is characterized by a metamagnetic isostructural phase transition, i.e., antiferromagnetic (AFM)-ferromagnetic (FM), with a transition temperature of $\sim$320 K.[15,16] This transition is accompanied by a sharp ($\sim$1%) expansion of the crystal lattice, which has a body-centered cubic structure of the CsCl type. In this alloy, the highest value for the inverse MCE, with $\Delta T_{ad} = -13$ K for $\Delta B = 2$ T, was observed.[17] It is also shown that FeRh exhibits a giant reproducible barocaloric effect.[18]

The $Sm_{0.6}Sr_{0.4}MnO_3$ manganite with Pbnm crystallographic symmetry and exhibiting the first order ferromagnetic (FM)-paramagnetic (PM) phase transition at (T_C)) 130 K is accompanied by a sharp increase in the lattice parameters, without undergoing a change in its symmetry.[19,20] The family of manganites ($Sm_{1-x}Sr_xMnO_3$) also

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belongs to the most studied materials and demonstrates a number of interesting physical properties.[21–23]

Attention should be paid to the qualitative difference brought about by a magnetic field on the 1-st order phase transitions in these materials. In the $Fe_{48}Rh_{52}$ alloy, the magnetic field induces the ferromagnetic to antiferromagnetic (AFM-FM) phase transition, causing expansion of the lattice, the inverse MCE ($\Delta T_{ad} < 0$), and a shift in the transition temperature to lower values. In the presence of a magnetic field in $Sm_{0.6}Sr_{0.4}MnO_3$ PM-FM transition, lattice contraction, direct MCE ($\Delta T_{ad} > 0$), and a shift in the transition temperature to higher values occur. In both cases, the magnetic and lattice contributions to ΔT_{ad} have the same sign.

To investigate the magnetocaloric properties of these materials in cyclic magnetic fields, the procedure described elsewhere[24,25] was followed. The samples under investigation were polycrystalline plates with typical dimensions of $2.5 \times 2.5 \times 0.4\,mm^3$. A cryogen-free superconducting magnet system with a maximum field of 8 T served as a source of magnetic field for magnetocaloric experiments. The samples were cooled in a zero magnetic field. After reaching the desired temperature, the cyclic effect of the field was achieved by moving/removing into/out the magnet bore of temperature insert using a linear actuator with a frequency of 0.13 Hz and continued until the upper temperature was reached. The heating rate of the samples was maintained right through the experiments at 1 K/min. To measure the temperature oscillations due to the MCE in cyclic magnetic fields, thermocouples made from constantan and chromel wires flattened to a thickness of about 3 μm were used. The signal from the thermocouple that passed through the SR554 transformer preamplifier was measured by the SR830 Lock-in Amplifier. To test the system in high fields, measurements of the MCE in gadolinium were performed and it was found that the maximum value of the MCE in Gd equals 13.5 K at a field change of 8 T.

The measurement of thermal expansion was performed by the strain gauge technique in constant magnetic fields of up to 8 T. For the measurement, KFL-5-120-C1-11 type strain gauges were used. To reduce the relative error, a change in voltage on the Wheatstone bridge was measured by an SR830 Lock-in at alternating current through the bridge. To obtain absolute values of thermal expansion, voltage changes were measured at a constant current in the same configuration.

The results of MCE measurements in highly and periodically changing magnetic fields are shown in Figs. 1(a) and 1(b) for $Fe_{48}Rh_{52}$ and $Sm_{0.6}Sr_{0.4}MnO_3$, respectively. First of all, the giant values for the inverse MCE (−9, −15, −18, and −20 K) for the $Fe_{48}Rh_{52}$ alloy were observed on exposure to fields of 1.8, 4, 6, and 8 T, respectively. The value of T_{ad} (–20 K) is the highest value cited to date in the literature for MCE measurements at field change of 8 T. In materials with a giant MCE, magnetic and lattice subsystems can contribute to the overall effect.[26,27] Under normal procedures used for MCE measurement, it is impossible to separate these contributions. But in some cases one can still make some estimation. The ratio of contributions in materials can be different, but the magnetic component can reach more than half of the overall effect, especially in high fields. Based on the measurement of the latent heat of transition,[28] it can be concluded that

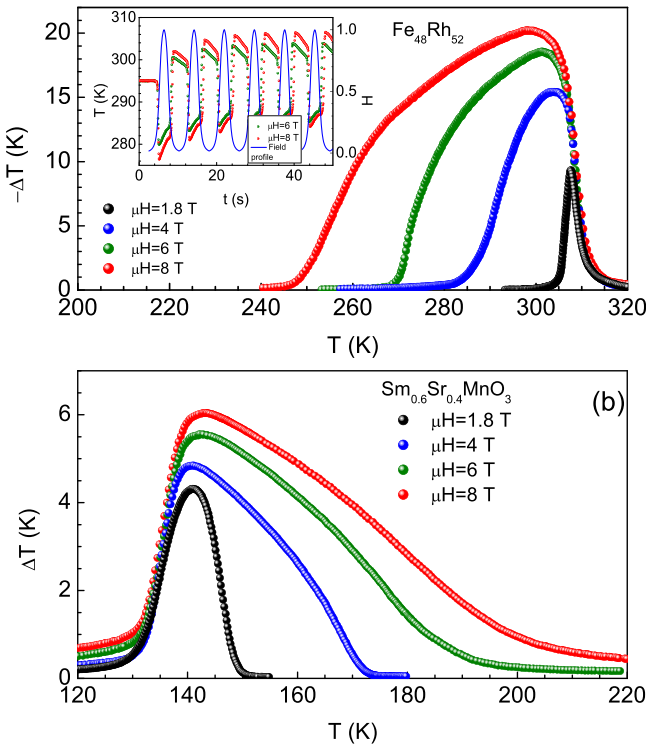

FIG. 1. MCE vs. temperature in different cyclic magnetic fields of 1.8, 4, 6, and 8 T, respectively, for: (a) $Fe_{48}Rh_{52}$; and (b) $Sm_{0.6}Sr_{0.4}MnO_3$. Inset—Temperature of $Fe_{48}Rh_{52}$ and normalized magnetic field as a function of time at $T = 295$ K.

the structural contribution to the MCE in FeRh in high magnetic fields does not exceed 50%, and this percentage decreases with increasing field.

The technique used for the MCE measurement does not allow a study of changes in the temperature profile, as it measures the amplitude of the temperature changes only. The temperature profile of the FeRh alloy was directly measured by connecting the differential thermocouple output to a Keithley 2000 multimeter. The inset in Fig. 1(a) shows temperature profiles at 295 K for cyclic fields of 6 and 8 T. The normalized profile of the magnetic field is also shown. The profiles confirm the obtained values of the MCE in the Fe-Rh alloy.

The MCE in $Sm_{0.6}Sr_{0.4}MnO_3$ is smaller in magnitude but reaches higher values, 4.4, 4.8, 5.5, and 6.1 K in the fields of 1.8, 4, 6, and 8 T, respectively. A noteworthy feature of the temperature dependence of MCE in these compounds is the "mirror" symmetry. One of the lines/boundaries of the temperature vs. MCE plot, where a strong MCE is exhibited, is not dependent on the magnitude of the field. This boundary is in the high temperature region for $Fe_{48}Rh_{52}$, while it is in the low temperature region for $Sm_{0.6}Sr_{0.4}MnO_3$.

In order to understand the temperature dependence of MCE, the thermal expansion of the samples was measured in constant magnetic fields of 1.8, 4, 6, and 8 T. For instance, Figs. 2(a) and 2(b) show the temperature dependence of thermal expansion in a field of magnitude 4 T and in zero field, respectively. These variations clearly reflect on the composition of the samples when there is a temperature change in zero field condition and also under the influence of a magnetic field. In both the systems, the phase transition temperatures

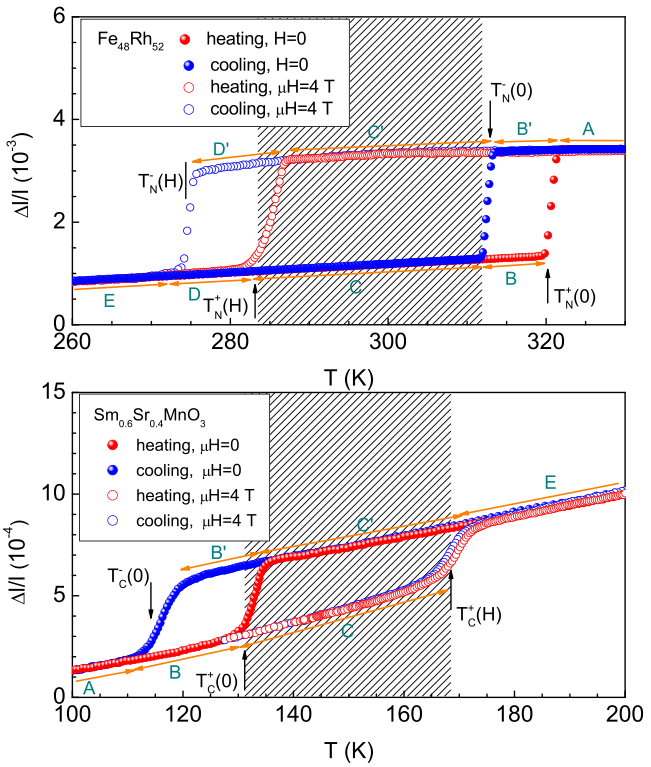

FIG. 2. Thermal expansion vs. temperature in a constant field of 4 T for: (a) $Fe_{48}Rh_{52}$; and (b) $Sm_{0.6}Sr_{0.4}MnO_3$. Hatched areas—regions of the reversible giant MCE.

shift with increasing field intensity and exceed the thermal hysteresis width of the transitions. In $Fe_{48}Rh_{52}$, the hysteresis width is of about 10 K and is weakly dependant on the field. With increasing field strength, the characteristic transition temperatures strongly shift toward lower temperatures.[28] In $Sm_{0.6}Sr_{0.4}MnO_3$, the transition temperatures shift toward higher temperatures with field. The hysteresis is greatly reduced in the field and thus it almost completely disappears at 8 T.

The plots in Figs. 2(a) and 2(b) are marked by distinct characteristic temperature regions. Let us consider each of these regions from the point of view of the influence of the cyclic magnetic field on the first order phase transitions. Analogies similar to the ones outlined below based on the $H-T$ or $T-\Delta S$ diagram are given in Refs. 29 and 30. Our arguments are based on the $T-\Delta l/l$ diagram, which is clearer for understanding of the cyclic effect of magnetic field. For both compositions, the samples are ferromagnetic in the temperature region A, and accordingly, application of the cyclic field leads to cyclic modification of the ordered state of the spins. But the structural state is not changed and it leads to a weak direct MCE. In the region B, the $Fe_{48}Rh_{52}$ alloy is antiferromagnetic, and the giant MCE due to phase AFM-FM transition accompanied by a change in the lattice parameters only occurs at the first application of a magnetic field. In subsequent cycles of turning on/off in of the field in the region BB' there will be only a faint MCE caused by changing the degree of magnetic ordering.

Because of ambiguity of phase composition in region BB′ in $Fe_{48}Rh_{52}$ in order to transform it to the low temperature AFM phase it is necessary to decrease the temperature to below the FM-AFM transition point in the cooling mode under the zero field ($T_N^-(0)$) condition and then to raise the temperature. Similarly, the composition of the phase depends on the history of $Sm_{0.6}Sr_{0.4}MnO_3$ in region BB′. If in the absence of a field, i.e., zero field, the sample is heated to a temperature above the ferromagnetic-paramagnetic (FM-PM) transition temperature in the heating mode ($T_N^+(0)$), and then it is decreased, it will bring the sample into the region B′. Subsequently, at the first turning on the field, there will be a significant direct MCE due to a magnetostructural transition. Subsequent cycles of on/off of the field provide only a weak direct contribution by the magnetic subsystem to the MCE.

In the region C, the $Fe_{48}Rh_{52}$ alloy is in the AFM state and the $Sm_{0.6}Sr_{0.4}MnO_3$ manganite is in the PM state. By turning on a strong magnetic field, a phase transition with change in the lattice parameters in both materials can be induced. Turning off the field in the region C′ leads to a return to the state C, since in the absence of a field the equilibrium states are AFM and PM states in $Fe_{48}Rh_{52}$ and $Sm_{0.6}Sr_{0.4}MnO_3$, respectively. Thus, in the temperature region CC' in both materials under cyclic application of the magnetic field a reversible magnetostructural transition should be observed. Accordingly, the giant reversible MCE is one in which contributions from the magnetic and lattice subsystems are summarized. These regions are limited by the temperatures $T_N^+(H)$ and $T_N^-(0)$ in the $Fe_{48}Rh_{52}$ alloy and $T_C^+(0)$ and $T_C^-(H)$ in the $Sm_{0.6}Sr_{0.4}MnO_3$ manganite. These regions are of main interest for the magnetic refrigeration technology. The minimum fields required for the formation of the CC' area are determined by the width of the temperature hysteresis and the shift of the transition temperature in a magnetic field. The field strengths are about 1 T and 0.5 T for $Fe_{48}Rh_{52}$ and $Sm_{0.6}Sr_{0.4}MnO_3$, respectively. The critical fields were estimated from the temperature dependence of thermal expansion measured in different fields in the heating and cooling runs. For the FeRh alloy, the field was estimated directly from Fig. 2(a), as the transition temperature linearly depends on a magnetic field.[15] In $Sm_{0.6}Sr_{0.4}MnO_3$, the transition temperature has nonlinear field dependence. Similarly, it can be shown that in areas DD′ and E, reversible phase transitions under cyclic application of a magnetic field are not observed in both materials.

Thus, in the materials with first order magnetostructural phase transitions, the reversible phase transitions at cyclic application of the magnetic field will be observed in certain temperature ranges only. It will be accompanied by the reversible giant MCE, with the lattice and spin subsystem contributions (hatched area in Figs. 2(a) and 2(b). From these figures, it is clear that one of the temperature boundaries is independent of magnetic field. For $Fe_{48}Rh_{52}$, this boundary coincides with the transition temperature FM-AFM in the cooling mode. In the case of $Sm_{0.6}Sr_{0.4}MnO_3$, this boundary is the transition temperature FM-PM in the heating mode.

Magnetocaloric properties of materials with magnetostructural phase transition in cyclic fields can degrade over time. Fig. 3 shows the temperature dependence of MCE in the FeRh alloy after 1000 and 3000 on/off cycles of the field. In order to explain the degradation of the magnetocaloric properties, we suggest the following. In the magnetostructural transition accompanying each cycle of turning on/off of

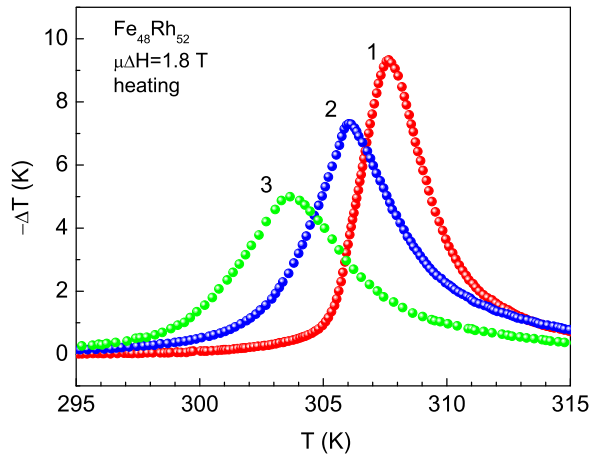

FIG. 3. Variation of MCE with temperature for Fe-Rh alloy: virgin curve after overheating above T_C (1), after 1000 (2) and 3000 (3) cycles of field turning on/off.

the magnetic field, ferromagnetic and antiferromagnetic domain wall motion is initiated due to magnetostriction. This process can eventually lead to a reduction in domain size as well as increased role of the domain boundary areas from cycle to cycle. This leads to a reduction in the number of spins in the domains, decreased magnetostriction, and increase in coercive force. All of these effects result in magnetization decrease in MSE. The process of interaction between domain surfaces can lead to a change in transition temperature of each of the domains, resulting in a general smoothening of the transition. Therefore, in order to restore the initial properties of the alloy, a thermal procedure of heating the sample above its Curie temperature, followed by cooling it to form the new pattern of ferromagnetic domains, is required. The procedure of heating the sample above its Curie temperature for a few seconds and cooling it in air was repeated several times, and every time the original properties were completely restored. In Ref. 28, it was shown that a magnetic field of 14 T is required for complete magnetostructural transition. Therefore, the incomplete phase transitions also can result in reduction of MCE. But this kind of influence should disappear with time, if the sample is kept away from the phase transition temperature to complete phase transition fully. But we observe that for full recovery of initial properties, the sample should be heated above the Curie temperature. As in the FeRh alloy, there are many systems that exhibit a sequence of low-temperature magnetostructural transitions, FM-AFM, and high temperature transitions, FM-PM, especially in some of the Heusler alloys.[2,11,31,32] Degradation of magnetocaloric properties due to magnetostructural transition is probably characteristic for these systems. But these are not observed during experiments due to the fact that the Curie point in these systems is close to room temperature. In the process of measurement of the properties of the materials, they are often heated to above their Curie point, and the virgin properties are restored every time.

In summary, the following conclusions can be drawn from these studies. The giant values of the MCE in alternating magnetic fields of up to 8 T are realized in the $Fe_{48}Rh_{52}$ alloy (–20 K) and $Sm_{0.6}Sr_{0.4}MnO_3$ manganite (6.1 K). In both systems, the giant MCE is associated with a first order reversible magnetostructural transition, and the temperature boundaries are independent of magnetic field, thereby limiting the temperature range of observation of a giant reversible MCE. For a system exhibiting AFM-FM transition, the upper range of temperature is restricted, while the lower range of temperature is limited in a system exhibiting FM-PM transition. The reversible MCE is observed only in fields above a certain critical range. For the $Fe_{48}Rh_{52}$ and $Sm_{0.6}Sr_{0.4}MnO_3$, it is about 1 and 0.5 T, respectively.

Despite the high importance of the observation of giant MCE in alternating magnetic fields, some effects are observed in experiments significantly complicating the use of these materials for advanced magnetic refrigeration systems. Degradation of the magnetocaloric properties of the material under the influence of thousands of cycles of magnetic field is a significant drawback of the $Fe_{48}Rh_{52}$ alloy exhibiting metamagnetic isostructural transition (AFM-FM). In the $Sm_{0.6}Sr_{0.4}MnO_3$ manganite, any degradation effect is not observed. Therefore, materials with the magnetostructural FM-PM phase transition can be considered to be more promising for use in magnetic refrigeration technology.

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