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Magnetic and magnetocaloric properties in polycrystalline $La_{0.2}Gd_{0.5}Ba_{0.3}MnO_3$ compound

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ABSTRACT

The detail magnetic measurements of the polycrystalline $La_{0.2}Gd_{0.5}Ba_{0.3}MnO_3$ (LGBMO) compound have been presented in this manuscript. The compound shows high value of magnetocaloric effect (MCE) ($-\Delta S = 9.1$ J/kg-K at 12 K) at low temperature. The experimental results of magnetization and magnetic memory effect confirm the existence of glassy magnetic phase in the compound at the low temperature ($T < 35$ K). The rejuvenation study further substantiates the presence of glassy phase. This high value of MCE in the LGBMO compound has been addressed by the presence of the glassy phase.

1. Introduction

Over the past two decades, the doped perovskite manganites with general formula $R_{1-x}B_xMnO_3$ (where, R = La, Gd, Pr *etc.* and B = Ca, Ba, Sr *etc.*) have been attracting much attention owing to their intriguing properties [1–3]. Moreover, perovskites show a strong correlation among transport, magnetic and structural properties. These compounds show a variety of properties like large magnetocaloric effect (MCE), colossal magnetoresistance (CMR), charge ordering (CO), ferromagnetism (FM) *etc.*, while 'A' site (*i.e.* R & B position) is doped with various elements of different concentrations [1–8]. Therefore, manganite is an important research field from the fundamental as well as application perspectives. MCE is the change in temperature of any magnetic material in response to the external magnetic field in adiabatic condition and therefore, large value of MCE means a substantial change in magnetic entropy or large temperature change of the system in adiabatic condition. Since, our modern civilization is growing, depending upon the refrigeration technology, the MCE is very much important for that cooling applications nowadays [8–11]. MCE is pollution free technique compared to the gas compression technique, as no harmful gases like, hydro-chlorofluorocarbons & chloroflurocarbons are used in it and moreover, the efficiency of MCE is higher than other usual techniques [8–12].

Though the intermetallic compounds such as $La(Fe_{1-x}Si_x)_{13}$, $MnFeP_{1-x}As_x$ *etc.* are well known to show high value of MCE, their production cost is very high [8,13–15]. In comparison to that, manganites are potential alternative due to several advantages, like high resistive nature (*i.e.* eddy current loss is reduced significantly), high chemical stability and furthermore, its production cost to prepare the samples is relatively lower than other materials [8,11,10]. Another advantage for choosing manganites as promising material for refrigeration is its broad working temperature ranges, which can be easily controlled via tuning the doping concentrations with different doping elements [2,10,11]. Generally, the MCE is obtained at the paramagnetic to ferromagnetic phase transition region. From the earlier reports, materials with first-order phase transition experience large change in magnetic entropy ($-\Delta S$) *i.e.* large MCE, but, these kind of materials show thermal & magnetic hysteresis and that indicates the wastage of energy, which could be helpful for cooling purposes [8,15–18]. On the other hand, materials with second-order phase transition exhibit a negligible hysteresis. Therefore, choosing a material that show a large value of MCE with a negligible hysteresis is the prime interest of research these days.

According to Zhang et al. [10] and Phan et al. [17], ferromagnetic manganites ($La_{0.7}Ba_{0.3}MnO_3$(LBMO), $La_{0.7}Ca_{0.3}MnO_3$, $La_{0.7}Sr_{0.3}MnO_3$ *etc.*) consider as potential materials for MCE due to their large magnetic entropy change at the transition temperature,

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but, its hysteresis loss is high. Therefore, the 'La'-site is further doped with other elements in order to modify the magnetic & physical properties of the compounds [2,3,19]. It is established that, when 'La'-site of ferromagnetic LBMO is partially substituted by 'Gd', its long-range ferromagnetic ordering starts breaking [19]. Furthermore, while 'La'-site is completely replaced by 'Gd' (*i.e.* $Gd_{0.7}Ba_{0.3}MnO_3$), its long-range ordering vanishes completely & ground state transforms into a glassy magnetic phase at the lower temperature [20]. Taking all these into consideration, we have chosen $La_{0.2}Gd_{0.5}Ba_{0.3}MnO_3$ (LGBMO) compound, whose phase transition is second-order in nature and ground state is a magnetic disordered state.

In this manuscript, large value of MCE of the polycrystalline LGBMO sample at the vicinity of cryogenic temperature range ($T < 15$ K) has been reported. The experimental results of magnetization, magnetic memory effect, rejuvenation and relaxation study confirm the presence of glassy magnetic phase in the system at the low temperature. Besides, the second-order magnetic phase transition has been confirmed by the study of Arrott plot [21]. The glassy magnetic phase at the lower temperature and the origin of large MCE in this compound have been addressed.

2. Sample preparation and characterization

Polycrystalline LGBMO sample has been prepared by well-known sol-gel method [6]. Preheated rare-earth oxides (La_2O_3, Gd_2O_3), bivalent carbonate ($BaCO_3$) and MnO_2 were taken as starting materials. These oxides and carbonate were greater than 99.99% pure. Appropriate amount of these materials were taken with proper stoichiometry and were dissolved in concentrated HNO_3. Oxalic acid was also added in case of MnO_2 to make it oxalate, as MnO_2 does not simply dissolve in HNO_3. Thereafter, all these solutions were mixed together with millipore water under moderate heating and stirring (for 15 min) condition. After that, appropriate amount of citric acid was mixed to the solution and again stirred for 15 min. Subsequently, the clear solution was put into a heat bath and slowly evaporated at 80–90 °C until the gel was formed. The gel was kept at 200–220 °C to remove organic stuffs and eventually by decomposing the gel, black porous powder was formed. The black powder was again heat treated at 400 °C for 4 h to eliminate remaining organic stuffs. The powder was then used to make pallets and thus, heat treated these pallets at 1300 °C for 36 h to prepare the bulk sample.

The magnetization measurements have been carried out using superconducting quantum interference device magnetometer (SQUID-VSM) within the temperature variation of 3 K to 200 K and up to the magnetic field variation 70 kOe.

3. Experimental results and discussions

The single phase nature of the compound has been confirmed by the room temperature powder X-ray diffraction (XRD) and reported in our previous work [19]. The structure of the compound is orthorhombic with *Pbnm* space group and the lattice parameters are a = 5.503 Å, b = 5.504 Å & c = 7.780 Å.

The temperature dependence of dc magnetization (M (T)) for the LGBMO sample has been shown in Fig. 1(a), where the measurement has been done in both zero-field-cooled (ZFC) and field-cooled (FC) protocol. All the M(T) data were recorded in the warming cycle under the application of external magnetic fields, H = 500 Oe and H = 10 kOe. The M(T) data at 500 Oe reveals that the ZFC and FC curves show a bifurcation at lower temperature ($T < 48$ K). The temperature at which the ZFC-FC curves depart is known as irreversible temperature (T_{irr}) and the temperature, where the ZFC curve shows a cusp is known as spin freezing temperature (T_f). From zoomed portion of M(T) data in Fig. 1(a), it is noted that the bifurcation (T_{irr}) of ZFC-FC curves starts at a higher temperature than the T_f and this type of behavior is usually noticed in the cluster-glass system [22–24]. Conversely, on the application of 10 kOe, the M(T) data shows no ZFC-FC bifurcation as well as no peak in ZFC curve up to low temperature and moreover, both ZFC & FC curves show increasing tendency up to the lower temperature. This could be a possible signature of glassy magnetic phase at the low temperature [19,25]. The temperature dependence of inverse susceptibility ($\chi^{-1}(T)$) has been illustrated in the Fig. 1(b). The transition temperature (T^*) was confirmed from the temperature derivative of magnetization (dM/dT) vs T plot and it has been shown in the inset of Fig. 1(b), where it shows a minimum at 55 K [26,27]. Unlike the sharp minimum in any other paramagnetic to ferromagnetic transition, the present system shows a broad minimum and may be this broadness arises because of all the spins that could not orient along the field direction due to the presence of glassy component in the system. Thus, it gives an indication of having glassy magnetic phase in the system. However, the figure also depicts the linear fitting of the experimental $\chi^{-1}(T)$ curve with the Curie-Weiss (C-W) law of the form $\chi = C/(T - \theta_{CW})$ in the paramagnetic region, where $C = \mu_{eff}^2/3k$ is the Curie constant and θ_{CW} stands for paramagnetic Curie-Weiss temperature. The linear fitting of $\chi^{-1}(T)$ depicts that it does not follow the C-W equation entirely above the T^*. The experimental χ^{-1} curve deviates from the linear curve around the temperature 149 K and this temperature is much higher than the T^*. This type of behavior is indicating that critical fluctuations are present above the T^* [28]. Furthermore, from the linear fitting of C-W equation, the analyzed data gives the value of $\theta_{CW} = 97$ K. This positive θ_{CW} value confirms the presence of ferromagnetic exchange interaction in the sample. The theoretical effective magnetic moment ($(\mu_{eff})_{theo}$) of the compound has been calculated using the following formula,

$$(\mu_{eff})_{theo} = \sqrt{0.5 * (\mu_{eff,\ Gd^{3+}})^2 + 0.7 * (\mu_{eff,\ Mn^{3+}})^2 + 0.3 * (\mu_{eff,\ Mn^{4+}})^2} \quad (1)$$

where $(\mu_{eff,\ Gd^{3+}})$, $(\mu_{eff,\ Mn^{3+}})$, $(\mu_{eff,\ Mn^{4+}})$ are calculated with the formula, $\mu_{eff} = g\sqrt{j(j+1)}\mu_B$ and the observed effective magnetic moment ($(\mu_{eff})_{obs}$) has been calculated from the linear fitting of χ^{-1} vs T plot in the paramagnetic region (*i.e.* from curie constant, $C = \left(\mu_{eff}\right)_{obs}^2/3k$), shown in Fig. 1(b). The value of $(\mu_{eff})_{theo}$ is 7.2 μ_B/f. u. and the value of $(\mu_{eff})_{obs}$ is 7.8 μ_B/f. u. Since, $(\mu_{eff})_{obs} > (\mu_{eff})_{theo}$, there may be a short-range FM interaction in the PM region [6].

To get a better notion about the system's ground state characteristic, the field dependence of magnetization (M(H)) has been carried out at low temperatures (T = 5 K & 20 K), which are well below the transition temperature as well as T_f and has been depicted in Fig. 2(a). These M(H) plots, taken with a field variation $0 \rightarrow +70 \rightarrow -70 \rightarrow +70$ kOe, show hysteresis loops and an increase of temperature reduces the value of coercive field as well as the remnant magnetization. Hence, the hysteresis at very low temperature, appears probably due to the alignment of spins below T_f. Moreover, a series of M(H) plots at different temperatures (from T = 5 K to T = 200 K) have been shown in Fig. 2(b) and it exhibits that at low temperatures the magnetization increases with the application of external magnetic field and also they show non-saturating trend with the application of high magnetic field. However, the value of magnetization reduces with increasing temperature and at higher temperatures up to 200 K a linear behavior of magnetization with field was noticed. Another interesting charac-

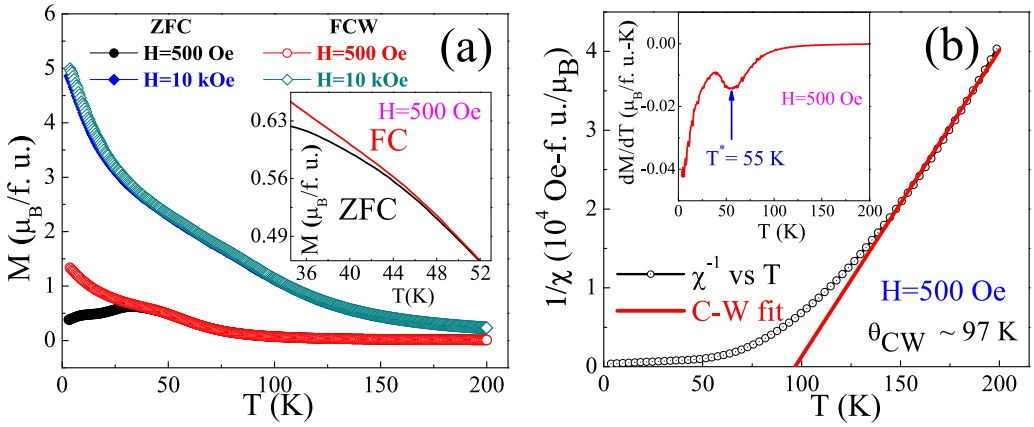

Fig. 1. (a) Magnetization (both in ZFC & FC protocol) as a function of temperature under different external applied magnetic fields of LGBMO sample; Inset: zoomed portion of irreversible region. (b) Curie-Weiss fitting of FCW data of LGBMO compound, measured at 500 Oe; Inset: Derivative of magnetization vs temperature curve, measured at 500 Oe.

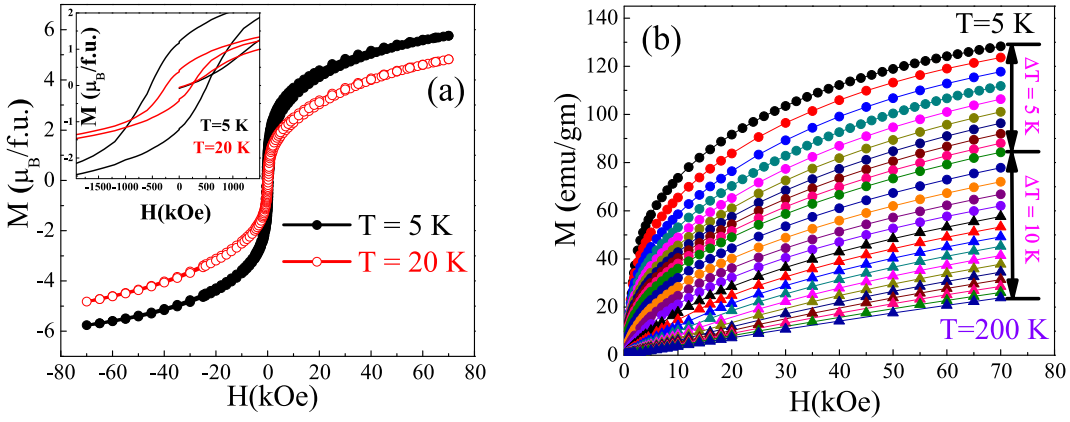

Fig. 2. (a) Field variation of magnetization for the specific temperatures T = 5 K & T = 20 K for LGBMO sample. Inset exhibits the zoomed portion of low field region. (b) Field variation of magnetization of LGBMO compound for various temperatures for 1 Quadrant.

teristic is its non-saturating tendency even after the application of 70 kOe magnetic field at lower temperature. These M (H) results indicate that the system's ground state is a glassy magnetic state at lower temperature (T < 35 K) [20].

In order to know the nature of magnetic phase transition, a very useful and conventional technique, named Arrott method is used. These M-H curves have been plotted as M^2 vs H/M and depicted in Fig. 3. As specified by Banerjee [21], the positive slope of M^2 vs H/M is the indication of second-order magnetic phase transition, while the negative slope indicates the first-order transition. From Fig. 3, it is clear that the magnetic phase transition for this compound is second-order. Unlike first-order transition, the advantage of second-order transition is its broad span of operating temperature. Usually, for ideal system, where the transition is second-order in nature it shows almost negligible thermal or magnetic hysteresis; that means energy loss due to hysteresis is minimum. However, the present system shows a small hysteresis at lower temperature that indicates the existence of short-range ferromagnetic interaction in the system. Furthermore, magnetic relaxation measurement *i.e.* magnetization as a function of time(M(t)) had also been done to corroborate the presence of glassy behavior in the system [29,30]. The M(t) data were taken in ZFC condition and therefore, the sample was first cooled in zero field to the specified temperature *i.e.* at T = 5 K, which is below the freezing temperature. After waiting for a while to stable the temperature, a small magnetic field (H = 50 Oe) was applied and the magnetization data as a function of time (M(t)) were recorded for a time lapse of $t_w = 9000$ s. Moreover, the experimental curve obeys the conventional stretched exponential function of the form shown in Eq. (2) very well.

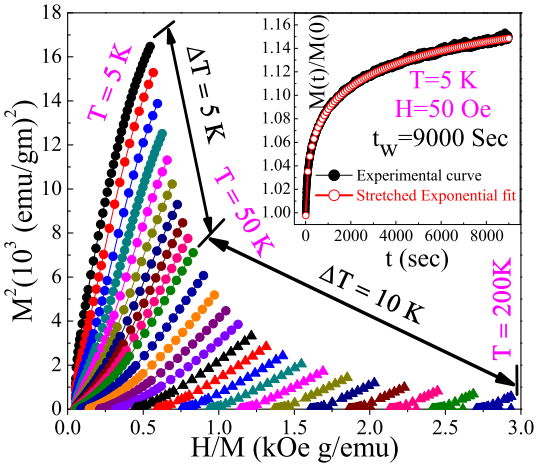

Fig. 3. Arrott plot (M^2 vs H/M) of LGBMO compound at different temperatures; Inset shows the relaxation plot of zero-field cooled (ZFC) magnetization on application of H = 50 Oe at T = 5 K, including the stretch exponential fitting.

$$M(t) = M_0 - M_g \exp[-(t/\tau)^{\beta}] \quad (2)$$

Here M_0, M_g and τ are the intrinsic ferromagnetic component, glassy component and characteristic relaxation time constant respectively and β is the stretching exponent. The experimental as well as fitted data of relaxation measurement have been shown in the inset of Fig. 3. Usually, for glassy system β lies between 0 and 1. In this system $\beta = 0.33$, which is another confirmation of the presence of glassy magnetic phase in the system and it is well reported for other glassy systems [29,30].

It would be worth mentioning that glassy behavior of LGBMO sample can also be confirmed by observing the memory effect experiment [22,29,31]. The FC protocol of memory effect reported by Sun et al. [31] was followed to investigate memory effect and has been shown in Fig. 4. During cooling cycle, temporary intermediate halts were taken at $T_{stop} = 80$ K, 40 K, 20 K, 10 K with a relaxation waiting time $t_w = 1$ h and then, after reaching at 2 K, the sample was subjected to a heating process up to room temperature keeping the temperature ramping rate and applied magnetic field (H = 100 Oe) unchanged. The magnetization M(T) was measured throughout the entire process. During heating cycle, the M(T) exhibits almost same step-like behavior as it was recorded in the cooling cycle for the temperatures T = 10 K, 20 K and 40 K. It concisely shows that the LGBMO compound exhibits "Memory Effect". On the other hand, at T = 80 K no change has been observed during heating cycle as the temperature is much higher than spin freezing temperature (T_f), where the spins get pinned due to the competition between temperature and magnetic field. The cooling and heating cycles are denoted by black and red line symbol respectively in Fig. 4.

In addition to that, the experimentation of magnetic relaxation and the influence of temperature cycling on it were introduced to get a notion about the mechanism of memory effect. The experiment was done under both ZFC and FC protocols followed by Sun et al. [31]. Fig. 5(a) and (b) respectively show the M-t plots of ZFC and FC. Their insets show the magnetization curve during t_1 plus t_3 time consecutively, indicated by black and red colour respectively. Though at T = 10 K magnetization falls down (for ZFC) and raised up (for FC), it comes back to its previous magnetization level again when the temperature was returned to 20 K and insets relaxation curve show an almost continuous line recorded during t_1 and t_3 time span. Thus, it confirms the memory effect as well as the presence of glassy magnetic phase in the sample. The memory effects at lower temperature were previously explained with hierarchical model [32] and droplet model [33].

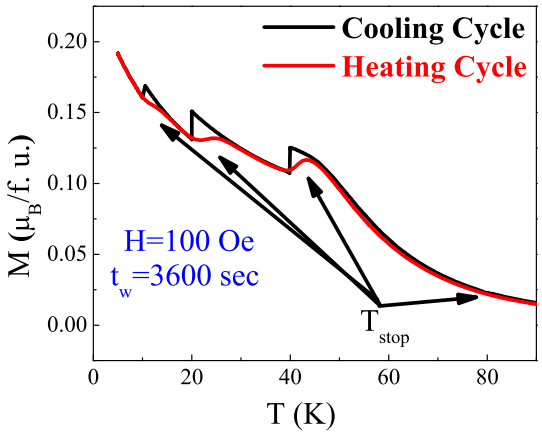

Fig. 4. Memory Effect of LGBMO sample, measured in FC protocol with temperature halts at T_{stop} = 10 K, 20 K, 40 K, 80 K.

Another observation that supports the glassy nature of the system, shown in Fig. 5(a) & (c) and (b) & (d), has been discussed with the hierarchical model. According to this model while the system is at glassy state at lower temperature, there are many metastable states and the states are split further into other sub-level states with decreasing temperature but, merged with increasing temperature. Therefore, while the temperature was quenched to a lower temperature (T = 10 K) from a specific temperature (T = 20 K) and then returned to that specific temperature (T = 20 K), the recorded magnetization curve follows a continuous path for that specific temperature (T = 20 K). This is exhibited by Fig. 5(a) and (b) during ZFC and FC measurements respectively. But, while the temperature is raised to a higher temperature (T = 30 K) from a specific temperature (T = 20 K) and then comes back to that specific temperature (T = 20 K), the recorded magnetization curve did not follow the same path. This has been shown in Fig. 5(c) and (d) during ZFC and FC measurements respectively. This is just because, when the temperature is lowered from the higher temperature, the metastability of the states' increases *i.e.* the probability of getting back to its previous state after taking higher temperature halt, is very thin and thus, in spite of getting back to the same temperature, magnetization curve does not follow the same path. While, in reverse case the metastability diminishes with increasing temperature and hence chances of getting back to its previous state after taking lower temperature halt is very high. Thus, all these observations support that the system's ground state is a glassy magnetic phase. The glassy-ness has been suppressed by applying high magnetic field and thus, high MCE has been achieved in this compound.

The amount of magnetic entropy change (ΔS) of this glassy system can be calculated from the isotherms shown in Fig. 2(b) [29]. Maxwell's thermodynamic relation of the form written in Eq. (3),

$$\Delta S = \int_0^H \left(\frac{\partial M}{\partial T}\right) dH \quad (3)$$

has been used to calculate ΔS. The change of $-\Delta S$ as a function of temperature for various applied magnetic fields has been shown in Fig. 6. Maximum value of $-\Delta S$ has been observed for the application of 70 kOe magnetic field variation and the value is 9.1 J/kg-K at 12 K. Another interesting phenomena to be noticed is that, $-\Delta S$ does not show any tendency to saturate even on the application of 70 kOe magnetic field. However, at low field variation $-\Delta S$ value is low. Generally, for the long-range ordering systems, a symmetric distribution of $-\Delta S$ in the $-\Delta S$ vs T plot is noticed around the transition temperature. Whereas, in this system no symmetric distribution around the transition temperature is noticed. Therefore, the non-saturating tendency and asymmetric distribution of $-\Delta S$ in this compound confirm that, the system has no long-range ordering [29]. Rather, it consists of glassy magnetic phase. This is the cause of getting high magnetocaloric effect with the application of high magnetic field.

4. Conclusions

In conclusion, detail study of all the aforementioned results confirmed the presence of the glassy magnetic phase in the bulk $La_{0.2}Gd_{0.5}Ba_{0.3}MnO_3$ (LGBMO) compound at low temperature. The ground state of the compound developed into magnetically disordered state, as the 'La'-site of the parent ferromagnetic $La_{0.7}Ba_{0.3}$-MnO_3 (LBMO) compound was further doped with another rare-earth element 'Gd'. This induces glassy behavior by breaking long-range ferromagnetic ordering of LBMO and thereby, magnetic entropy was increased as well in LGBMO compound. Thus, applying high magnetic field (70 kOe), large MCE ($-\Delta S = 9.1$ J/kg-K at 12 K) was achieved in the system. Therefore, the presence of glassy

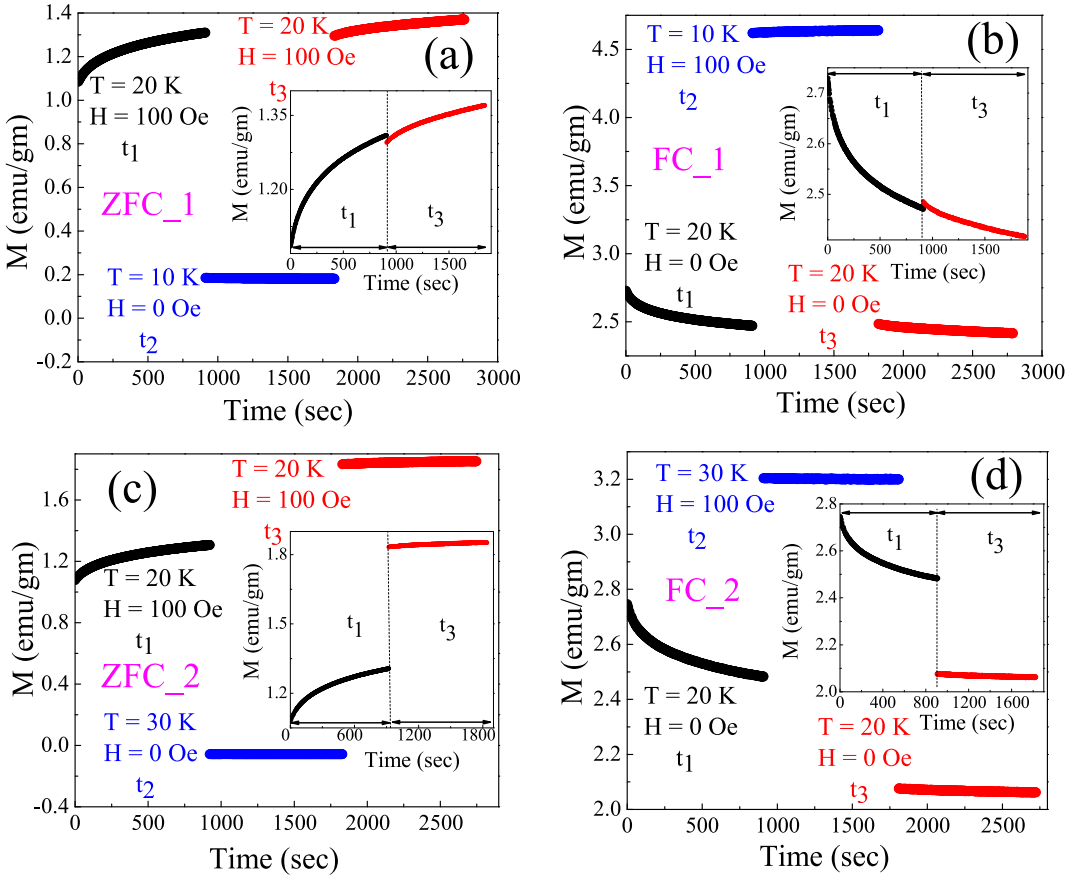

Fig. 5. Rejuvenation measurement.

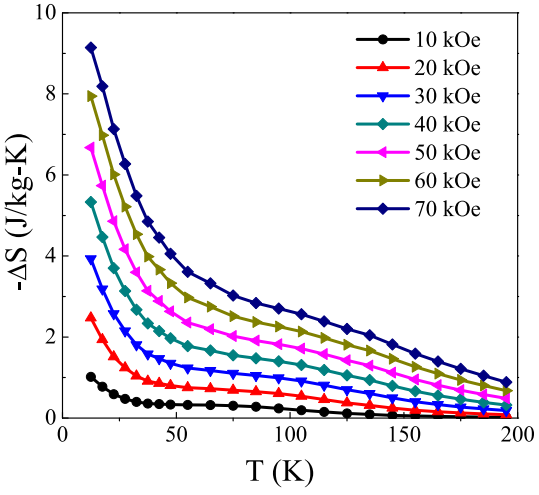

Fig. 6. Magnetic entropy change −ΔS as a function of temperature for the application of different magnetic fields.

magnetic phase is playing vital role in achieving high MCE in the system.

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