Magnetic and Magnetocaloric Properties of La$_{0.67}$Pb$_{0.33}$-$x$Ag$_x$MnO$_3$ Compounds

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Abstract In this paper, we have studied the magnetic and magnetocaloric properties of the perovskite manganites La$_{0.67}$Pb$_{0.33}$-$x$Ag$_x$MnO$_3$ (0 ≤ $x$ ≤ 0.15), which show a sharp paramagnetic-ferromagnetic phase transition over a wide temperature range ($T = 250–401$ K). The Curie temperature has been analyzed by two methods: using the numerical derivative $dM/dT$ and the thermodynamic model. The experimental results indicate that $T_C$ decreases from 368 to 288 K with increasing Ag substitution independently of the method used to obtain $T_C$. Upon 10 KOe applied magnetic field, the large magnetic-entropy change ($|\Delta_1 S_M|$) reaches values of 2.75, 3, 3.25 and 3.5 J/kg K for $x = 0$, 0.05, 0.10 and 0.15 compositions, respectively, which are comparable to that of Gd. The relative cooling power (RCP) increases with increasing Ag content from 68.75 ($x = 0$) to 156.27 J/kg ($x = 0.15$) for $\Delta H = 10$ KOe. Through these results, La$_{0.67}$Pb$_{0.33}$-$x$Ag$_x$MnO$_3$ materials are strongly suggested for the use of active refrigerants for magnetic refrigeration technology near room temperature.

Keywords Magnetic refrigerator · Magnetic entropy · Manganites · Relative cooling power

1 Introduction

Magnetic refrigeration based on the magnetocaloric effect (MCE) is a viable and competitive cooling technology in the near room-temperature region and it has recently attracted much research interest due to its potential advantage of environmental friendliness over conventional gas refrigeration [1, 2], such as high energy efficiency, less environmental stress, and small volume [3–5]. When a magnetic field is applied to a magnetic material, which is thermally isolated from its surroundings, a change of the temperature (heating or cooling) of the solid may be observed. This phenomenon is known as the magnetocaloric effect or, more precisely, the adiabatic temperature change due to a change of the applied magnetic field. The magnetocaloric effect was first observed by Warburg [6] in 1881: when iron was placed in a magnetic field, it warmed, and removing that field, the iron specimen cooled down and this was explained physically by Debye in 1926 [7] and Giauque in 1927 [8]. The magnetocaloric effect is the result of entropy changes ($|\Delta S_M|$) or temperature ($\Delta T_{ad}$) in a magnetic material with the adiabatic application or removal of a magnetic field. Until recently, the rare earth element gadolinium (Gd) with a large MCE has been considered as the most active magnetic refrigerant in room magnetic refrigeration (MR) [9] since it exhibits a maximum magnetic-entropy change, $|\Delta S_M|$, of 10.2 J/kg K at $T = 294$ K under a magnetic applied field change of 5 T [9]. However, the high price of Gd ($\sim$4000/kg) prevents it from the actual application. Therefore, the search for cheaper working substances and large MCE becomes a main research topic.
Fig. 1 Temperature dependence of the magnetization for La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($0 \leq x \leq 0.15$) sample at $\Delta H = 500$ Oe magnetic field. Inset shows the derivative of magnetisation with respect to the temperature. The magnetic transition temperature, $T_C$, is deduced from the value where $dM/dT$ curve displays a minimum.

In this field, in 1997, Pecharsky and Gschneidner [10] discovered that the giant magnetocaloric effect MCE in the pseudo-binary alloy Gd$_5$Si$_2$Ge$_2$ was twice larger than in Gd. More importantly, this alloy could not only improve the efficiency of large-scale magnetic refrigerators but also open the door to new small-scale applications, such as home and automotive air conditioning. Nonetheless, the Curie temperature of Gd$_5$Si$_2$Ge$_2$ is about 276 K, which is much lower than that of Gd of 294 K, making this alloy difficult to be used in room-temperature magnetic refrigerators [11]. For this reason, there is an extensive search of new materials suited for solid-state cooling machines working in this temperature range, such as Ni–Mn–Ga alloys [12], Mn–As–Sb alloys [13], La–Fe–Co–S alloys [14], Mn–Fe–P–As alloys [15] and various compounds of manganites [5].

In the present study, we investigate the magnetic and magnetocaloric effect related to the effects of Ag doping in La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$, which can be a suitable candidate as a working substance in magnetic refrigeration near room temperature.

2 Experimental

Polycrystalline La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($0 \leq x \leq 0.15$) samples were prepared by standard solid-state reaction method. Details of the sample structure characterization are described in Ref. [16].

Magnetization ($M$) versus temperature ($T$) and magnetization versus magnetic field ($H$) curves were carried out by using a Foner magnetometer equipped with a superconducting coil. The magnetization isotherms were measured with a field step of 10 K Oe and with a temperature interval of 5 and 10 K over a temperature range of 250–401 K.

3 Results and Discussion

3.1 Magnetic Measurements

In order to analyze the effect of Ag substitution on the Curie temperature, two methods have been used to determine $T_C$:

(1) The first one is a determination of inflection point of the transition by using the numerical derivative $dM/dT$ as indicated in the a-inset of Fig. 1.

Figure 1 displays the temperature dependence of the magnetization for the sample La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($0 \leq x \leq 0.15$) under a magnetic field $\Delta H = 500$ Oe. The obtained values of $T_C$ are 361, 332, 311 and 290 K, respectively, for $x = 0, 0.05, 0.1$ and 0.15 [16].

(2) The second one is based on the thermodynamic model: Amaral et al. [17] discussed the magnetic properties of manganites in terms of the Landau theory of phase transitions. Here, the magnetic energy $MH$ has been included in the expression of Gibb’s free energy as given by

$$G(T, M) = G_0 + \frac{1}{2} AM^2 + \frac{1}{4} BM^4 - MH$$

The coefficients $A$ and $B$ are temperature-dependent parameters containing the magnetoelastic coupling and electron condensation energy [18].

By assuming equilibrium condition of the Gibbs free energy: $\frac{dG}{dM} = 0$, the magnetic equation of state is obtained as

$$AM + BM^3 - H = 0$$

(2)
Fig. 2 Isothermal magnetization curves measured at different temperatures between 321 and 401 K for La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($0 \leq x \leq 0.15$)

In this equation, $M$ is the experimentally measured mass magnetization and equal to $M_S + M_i$, where $M_S$ is the spontaneous and $M_i$ is the true magnetization caused by the application of the magnetic field $H$.

By plotting the experimental data in the form $\frac{H}{M} = A + BM^2$, the temperature dependence of parameters $A$ and $B$ can be extracted.

In Fig. 2, we present isothermal magnetization ($M$) versus field ($H$) plots of La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($x = 0$ and 0.1) at various temperatures with an applied field of 10 KOe. It can be seen clearly from Fig. 2 that there is a gradual change in shape when passing from ferro- to paramagnetic phase. In the ferromagnetic phase an abrupt magnetization increases sharply, and then tends to saturation (Fig. 2). The saturated magnetization ($M_S$) can be obtained from an extrapolation of the high field $M$–$H$ curve to $H = 0$. $M_S$ is found to decrease as Ag content increases from $x = 0$ to 0.15.

Since a sharp PM–FM transition occurs around 290 K, which possibly implies a large magnetic-entropy change near room temperature, we performed a measurement of MCE of the present material.

In order to check the nature of the magnetic phase transition of the samples, we use the Banerjee criterion [19]. Ac-
Fig. 3 $\frac{H}{M}$ vs. $M^2$ plot for La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ manganites ($x = 0$ and 0.1)

According to this criterion, the slope of $\frac{H}{M}$ versus $M^2$ curves denotes whether the observed magnetic transition is of the first order (negative slope) or second order (positive slope). Figure 3 is an Arrott plot of $M^2$ vs. $\frac{H}{M}$ curves for La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($x = 0$ and 0.1). Clearly, in the present case the positive slope of $M^2$ vs. $\frac{H}{M}$ curves indicates the phase transition is a second order at the Curie point.

The temperature dependence of parameters $A$ and $B$ can be obtained from the linear fitting of the Arrott plot of $\frac{H}{M}$ vs. $M^2$.

Thermal variation of the parameter $A$ is found to be linear for all the samples (Fig. 4(a)), it varies from positive to negative through zero at $T_C$, whereas the temperature dependence of the parameter $B$ is highly nonlinear (Fig. 4(b)). The Curie temperature $T_C$ is given by

$$ A = A'(T - T_C) $$

where $A$ is negative for $T < T_C$, equal to 0 for $T = T_C$, and positive for $T > T_C$.

The $T_C$ estimated from the $M-T$ curves and the Arrott plots are in close agreement with each other, e.g., the $M-T$ curve of $x = 0.1$ sample yields $T_C = 316$ K and the Arrott plots also give almost the same value of $T_C$ (see Table 1).
To evaluate the magnetic-entropy change, the thermodynamic Maxwell relation [20] is used to avoid the difficulty of adiabatic measurements. We used the second method to avoid the difficulty of adiabatic measurements. The variation of magnetic entropy and $M(H)$ isotherms are related by the thermodynamic Maxwell relation [20]:

$$\left( \frac{\partial S_M(T, M)}{\partial H} \right)_T = \left( \frac{\partial M(T, M)}{\partial T} \right)_H$$

(4)

From Eq. (4), the isothermal entropy change can be calculated by means of magnetic measurements [21]:

$$\Delta S_M(T, H) = \int_0^H \left( \frac{\partial M(T, M)}{\partial T} \right) dH$$

(5)

To evaluate the magnetic-entropy change $|\Delta S_M|$ we need a numerical approximation of the integral in Eq. (5). The usual method is to use isothermal magnetization measurement at small discrete field intervals and then $|\Delta S_M|$ can be approximated from Eq. (6) by

$$|\Delta S_M| = \sum_i \frac{M_i - M_{i+1}}{T_{i+1} - T_i} \Delta H_i$$

(6)

where $M_{i+1}$ and $M_i$ are the magnetization values measured in a field $H_i$ at temperatures $T_{i+1}$ and $T_i$, respectively.

Using Eq. (6) and experimental $M-H$ curves at various temperatures, the magnetic-entropy change vs. temperature $|\Delta S_M|$ can be calculated.

Figure 5 shows the variation in $|\Delta S_M|$ magnetic-entropy change as a function of temperature for the samples with $x = 0, 0.05, 0.1$, and $0.15$ at $\Delta H = 10$ KOe. As seen from Fig. 5, the maximum value of $|\Delta S_M|$, namely $|\Delta S_M^{\text{max}}|$ at each field, is obtained close to the respective Curie temperature ($T_C$). The $|\Delta S_M^{\text{max}}|$ value increases from $2.75$ J/kg K for $x = 0$ to $3.15$ J/kg K for $x = 0.15$ at field $\Delta H = 10$ KOe. For all the samples of the present series, $|\Delta S_M^{\text{max}}|$ increases with increasing $x$ (see the inset of Fig. 5).

These values are clearly comparable to the pure Gd [22] ($|\Delta S_M| \sim 3.25$ J/kg K at $\Delta H = 10$ KOe) and other selected manganites [23–31] (Table 1).

This result is of practical importance, because it shows that the present manganite polycrystalline materials could be good working materials for magnetic refrigeration in household refrigerators or air conditioning, because of:

3.2 Magnetocaloric Effect

The magnetic entropy can be measured through either the adiabatic change of temperature by the application of a magnetic field, or through the measurement of classical $M(H)$ isotherms at different temperature. We used the second method to avoid the difficulty of adiabatic measurements. The variation of magnetic entropy and $M(H)$ isotherms are related by the thermodynamic Maxwell relation [20]:

$$\left( \frac{\partial S_M(T, M)}{\partial H} \right)_T = \left( \frac{\partial M(T, M)}{\partial T} \right)_H$$

(4)

From Eq. (4), the isothermal entropy change can be calculated by means of magnetic measurements [21]:

$$\Delta S_M(T, H) = \int_0^H \left( \frac{\partial M(T, M)}{\partial T} \right) dH$$

(5)

To evaluate the magnetic-entropy change $|\Delta S_M|$ we need a numerical approximation of the integral in Eq. (5). The usual method is to use isothermal magnetization measurement at small discrete field intervals and then $|\Delta S_M|$ can be approximated from Eq. (6) by

$$|\Delta S_M| = \sum_i \frac{M_i - M_{i+1}}{T_{i+1} - T_i} \Delta H_i$$

(6)

where $M_{i+1}$ and $M_i$ are the magnetization values measured in a field $H_i$ at temperatures $T_{i+1}$ and $T_i$, respectively.

Using Eq. (6) and experimental $M-H$ curves at various temperatures, the magnetic-entropy change vs. temperature $|\Delta S_M|$ can be calculated.

Figure 5 shows the variation in $|\Delta S_M|$ magnetic-entropy change as a function of temperature for the samples with $x = 0, 0.05, 0.1$, and $0.15$ at $\Delta H = 10$ KOe. As seen from Fig. 5, the maximum value of $|\Delta S_M|$, namely $|\Delta S_M^{\text{max}}|$ at each field, is obtained close to the respective Curie temperature ($T_C$). The $|\Delta S_M^{\text{max}}|$ value increases from $2.75$ J/kg K for $x = 0$ to $3.15$ J/kg K for $x = 0.15$ at field $\Delta H = 10$ KOe. For all the samples of the present series, $|\Delta S_M^{\text{max}}|$ increases with increasing $x$ (see the inset of Fig. 5).

These values are clearly comparable to the pure Gd [22] ($|\Delta S_M| \sim 3.25$ J/kg K at $\Delta H = 10$ KOe) and other selected manganites [23–31] (Table 1).

This result is of practical importance, because it shows that the present manganite polycrystalline materials could be good working materials for magnetic refrigeration in household refrigerators or air conditioning, because of:
(2) a modest magnetic-entropy change up on application/removal of a low magnetic field and easily controllable magnetic entropy,
(3) good chemical stability with quite high efficiency, and (4) the possibility of being manufactured at a low price.

From our study it is seen that the perovskites are promising MR materials. A large value of entropy change could be expected in polycrystalline samples. Perovskites are easy to prepare and exhibit higher chemical stability as well as higher resistivity that are favorable for lowering eddy current heating. Beside these, since the Curie temperature of perovskite manganites is doping dependent, a large entropy change could be turned from low temperature to near and above room temperature, which is beneficial for operating magnetic refrigeration at various temperatures.

The large magnetic-entropy change in perovskite manganites is believed to be originated from the role played by spin–lattice coupling in the magnetic ordering process [32]. A significant change accompanying the magnetic transition due to strong coupling between spin and lattice in perovskite manganites has been observed [33] resulting in lattice structure changes e.g. in the ⟨Mn–O⟩ bond distances as well as in the ⟨Mn–O–Mn⟩ bond angle. The significant coupling of spin and lattice degrees of freedom provides a more abrupt variation of magnetization near the magnetic transition occurs and results in a large magnetic-entropy change.

The temperature dependence of the magnetic-entropy change (|ΔS_M|) for x = 0.1 at various magnetic fields is shown in Fig. 6. With the increase of magnetic field, the value of magnetic-entropy change increases and the peak

Fig. 4 (a) Parameters as a function of temperature A and (b) temperature dependence of parameter B.
Fig. 5 Temperature dependence of the magnetic-entropy changes for La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($x = 0, 0.05, 0.1$ and $0.15$) alloys around $T_C$ under magnetic field of 10 KOe. The inset is the $|\Delta S_M|$ as a function of Ag doped La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($x = 0, 0.05, 0.1$ and $0.15$) compounds around $T_C$ under magnetic field of 10 KOe. The inset is the $|\Delta S_M|$ as a function of Ag doped.

Fig. 6 Temperature dependence of the magnetic-entropy change $\Delta S_M$ for different magnetic field intervals for La$_{0.67}$Pb$_{0.33}$Ag$_{0.1}$MnO$_3$ sample. Inset the maximum entropy change $|\Delta S_M|$ and the relative cooling power (RCP) values vs. applied magnetic field.

of the magnetic-entropy changes lightly moves to a higher temperature due to the shift of effective $T_C$ by the applied magnetic field. At Curie temperature, the $|\Delta S_M^{\text{max}}| = 2, 2.44, 2.84$ and $3.25$ J/kg K follows the magnetic field changes of 0.4, 0.6, 0.8 and 1 T, respectively. But this curve does not follow a linear relationship.

To assess the applicability of our samples for magnetic refrigeration, the maximum $|\Delta S_M^{\text{max}}|$ values determined in the present studies are compared in Table 1 with those reported in literature for several materials having close $T_C$ and considered promising for such application. La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($x = 0, 0.05, 0.1$ and $0.15$) compounds could be a potential candidate for room-temperature magnetic refrigeration because of its advantages in terms of easy preparation method, nearly comparable $|\Delta S_M|$, high chemical stability, tenability of $T_C$ over a wide temperature range, high resistivity, cost effectiveness, etc.

As is known, it is not sufficient to identify the potentiality of a magnetic refrigerant material solely by the large magnetic-entropy change $|\Delta S_M|$. Another important parameter is refrigerator capacity (RC), a measure of how much heat can be transferred between the cold and the hot sinks in one ideal refrigeration cycle [34] defined as

$$\text{RCP}(S) = -\Delta S_M^{\text{max}} \partial T_{\text{FWHM}}$$

(7)
where $\partial T_{\text{FWHM}}$ denotes the full width temperature span of $|\Delta S_M|$ versus $T$ curve at its half maximum.

The corresponding results for $x = 0.1$ of $\Delta H = 1$ T was presented in the inset (c) of Fig. 6(a). The material with a larger RCP value usually represents a better magnetocaloric substance due to its high cooling efficiency. As shown in the inset (b) of Fig. 6, $|\Delta S_M^{\text{max}}|$ and RCP of La$_{0.67}$Pb$_{0.23}$A$_{0.1}$MnO$_3$ sample exhibit an almost linear rise with increasing $\Delta H$. For $\Delta H = 1$ T, the value of RCP reaches 130 J/kg. The RCP is much larger than that of other manganites and is high enough for magnetic refrigeration. The temperature range $\delta T_{\text{FWHM}}$ is found to be 40 K. This wide temperature range is very beneficial for Ericsson refrigeration cycle as well [35].

Pure Gd, which is considered a good material, exhibits an RCP value of 250 J/kg at 2 T [10], while Gd$_3$(Si$_2$Ge$_2$) which is considered the most conspicuous magnetocaloric material at room temperature, presents an RCP value of 130 J/kg at 2 T [36]. For the samples studied here, the RCP values are found to be 68.75, 84.24, 130 and 156.27 J/kg under the magnetic field variation of 1 T for $x = 0$, 0.05, 0.1 and 0.15, respectively. Compared to other materials considered as good for applications in magnetic refrigerators, our results are interesting enough to pave the way for investigations of materials useful for magnetic refrigeration.

So we can conclude that magnetocaloric materials based on silver-doped manganites can be promising candidates for MR because they show considerable RCP at room temperatures.

Refrigerant capacity ($\Delta Q_M$), which gives a measure of the amount of heat that can be transferred between the hot and cold baths in one ideal refrigeration cycle [37, 38] defined as

$$\Delta Q_M = \int_{T_{\text{cold}}}^{T_{\text{hot}}} \Delta S(T)_{P,\Delta H} dT$$

By performing numerical integration to evaluate the area subtended by the peaks in $\Delta S_M$–$T$ plots, $\Delta Q_M$, can be calculated for temperatures around $\pm$ 10 K of $T_C$ by

$$|\Delta Q_M(T_C \pm 10 \text{ K}, 10 \text{ KOe})|\int_{T_C-10}^{T_C+10} |\Delta S_M(T, 10 \text{ KOe})| dT$$

The value obtained at 10 KOe, around $\pm$ 10 K of $T_C$, increases with increasing Ag content (Table 1) and typically found for $x = 0.1$ to be $\sim$47 % to that of pure Gd (122 J/kg at $T_C \pm 25$ K). However, compared with Gd- or Gd-based compounds, manganites are inexpensive, easier to fabricate, are chemically stable and have a respectable $|\Delta S_M|$ in a relatively low field range (10 KOe) and therefore, could be considered as a potential solid refrigerant in magnetic refrigeration technology for consumer applications.

According to Landau Theory, $|\Delta S_M|$ can be evaluated through the following formula:

$$S_M(T, H) = -\frac{1}{2} A'(T) M^2 - \frac{1}{4} B'(T) M^4$$

where $A'(T)$ and $B'(T)$ are the temperature derivatives of the expansion coefficients.

Using the parameters $A$ and $B$ extracted from the data ($\frac{H}{M} = A + BM^2$), the temperature dependence of the magnetic-entropy change $|\Delta S_M|$ is calculated from Eq. (10) under $\Delta H = 10$ KOe and are shown in Fig. 7. The results
obtained based on the proposed model, fit well with the experimental data on magnetic-entropy change. So the temperature dependence of the magnetic-entropy change is understood through the Landau theory of phase transitions.

4 Conclusion

In conclusion, we have investigated the MCE of perovskite manganite La$_{0.67}$Pb$_{0.33-x}$Ag$_x$MnO$_3$ ($0 \leq x \leq 0.15$). The magnetic-entropy change ($|\Delta S_M|$) of all the samples showed a maximum around their respective $T_C$ and its magnitude increases from 2.75 J/kg K ($x = 0$) to 3.5 J/kg K ($x = 0.15$) with increase of Ag content, under 10 KOE field, and the corresponding RCP are 156.27 J/kg ($x = 0.15$) to 68.75 J/kg ($x = 0$). It indicates that such a series of samples can be suitable candidates as room-temperature working substances in magnetic refrigeration technology. The observed temperature dependence of $|\Delta S_M|$ is in accordance with Landau theory.

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