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Observation of giant magnetocaloric effect under low magnetic field in $Eu_{1-x}Ba_xTiO_3$



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ABSTRACT

The magnetic properties and magnetocaloric effect in Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1) compounds are investigated. With slight Ba-doping, the antiferromagnetic ordering of pure EuTiO₃ can be significantly changed to ferromagnetic due to the size Ba²⁺ ion bigger than Eu²⁺. A giant reversible magnetocaloric effect and large RC in Eu_{1-x}Ba_xTiO₃ compounds were observed. The maximum value of magnetic entropy change ($-\Delta S_M^{max}$) reaches to 41.4 J/kg K for Eu_{0.92}Ba_{0.08}TiO₃ under the magnetic field change of 5 T. Especially, under the magnetic field changes of 1 and 2 T, the values of $-\Delta S_M^{max}$ are evaluated to be 12.5 and 23.4 J/kg K and the maximum values of RC are 54 and 119 J/kg, without magnetic and thermal hysteresis for Eu_{0.92}Ba_{0.08}TiO₃. Therefore, the giant reversible magnetocaloric effect and large RC make the Eu_{1-x}Ba_xTiO₃ compounds could be considered as a good candidate material for low-temperature and low-field magnetic refrigerant.

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1. Introduction

Recently, magnetocaloric effect (MCE) materials have been attracted the attention of the world due to their high potential use in magnetic refrigerator (MR) technology, and due to its high efficiency, compactness and environmental benignness compared to the conventional gas refrigeration [1–4]. MCE material is one of the critical parts for the development of MR since the effectiveness and leniency scales directly with the magnetic entropy change $(-\Delta S_M)$ for moderate magnetic fields. In the aim, a giant MCE has been reported in several materials such as near room temperature, Gd₅Si₂Ge₂ [5], LaFe_{13-x}Si_x [6], MnAs_{1-x}Sb_x [7] and MnFeP_{0.45}As_{0.55} [8] et al. and low temperature, TmGa [9], TmCuAl [10], ErMn₂Si₂ [11], HoCuSi [12], ErCr₂Si₂ [13] and HoCoSi [14] et al. They mainly to

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be applied to gas liquefaction as helium, hydrogen and natural gas and potential applications in space science [15]. Additionally, if a sufficient MCE of material can be achieved upon application of low magnetic fields (e.g less than 2 T), it is feasible to design a refrigeration cycle using permanent magnets. Therefore, it is desirable to search for materials that have giant MCE with zero or inappreciable thermal and field hysteresis loss under low field ($\Delta H \leq 2 T$).

On the other hand, a strong interplay between magnetic and electric properties has been reported in the perovkites RTiO₃ and (R = rare earth) compounds opening the way for use in spintronics application [16,17]. Cubic perovskite EuTiO₃, which exhibits G-type antiferromagnetic (AFM) order and quantum paraelectric (PE) behavior, has attracted considerable attention since the discovery of its large magnetoelectric (ME) effect [18,19]. An experimental study on the Eu_(1-x)Ba_(x)TiO₃ polycrystalline samples suggests occurrence of ferroelectricity for x > 0.2 and paraelectricity for x < 0.15 [20]. While in a long time ago Eu_{0.5}Ba_{0.5}TiO₃ was predicted to be ferroelectric behavior the sample is AFM with Neel temperature (T_N) T_N = 1.9 K [22]. Since Eu²⁺ ion carries a large spin magnetic moment of $\mu = 7$ μ_B with a zero orbital angular



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momentum (L = 0, J = S), these compounds may show a large isotropic magnetic entropy change [23,24]. Alho et al. [25] theoretically predicted an anisotropic MCE of the same order of magnitude of the usual MCE for AFM systems using Hamiltonian model in the EuTiO₃ compound. Km Rubi et al. reports the $-\Delta S_M$ in magnetoelectric Eu_{1-x}Ba_xTiO₃ compounds for 0.1 < x < 0.9 [26].

In the present paper, we investigated the magnetic properties and MCE in $Eu_{1-x}Ba_xTiO_3$ compounds for 0 < x < 0.1. In EuTiO₃, the competition between the AFM superexchange and an indirect ferromagnetic (FM) exchange leads to a delicate balance between AFM and FM phases, which explain by Akamatsu et al., through first-principles calculations based on a hybrid Hartree-Fock density functional approach [27]. What's more, the EuTiO₃ has strong spinlattice coupling, the magnetic ground state can be switched from AFM to FM, when the lattice constant *a* is increased with the lattice constant *c* relaxed and when *c* is increased with *a* fixed [28]. In $Eu_{1-x}Ba_xTiO_3$ ($0 \le x \le 0.1$) compounds, with increasing the content Ba, the FM dominates in the delicate balance between AFM and FM phases, the magnetic ground state was switched from AFM to FM and giant reversible MCEs were observed. Our results suggest $Eu_{1-x}Ba_xTiO_3$ could be expected to have effective applications in low temperature MR.

2. Experiments

The samples of $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.02, 0.04, 0.06, 0.08, 0.1) were synthesized by a simple method. Details: Europia (Eu_2O_3) was dissolved in nitric acid (HNO₃ 8 mol/L) and stirred for about half an hour. Next, tetra-butyl titanate (Ti(OC₄H₉)₄) and barium chloride (BaCl₂) in stoichiometric proportions was added into the Europium nitrate solution and stirred for about an hour. Then, Glycol ($C_2H_6O_2$) in 1:1 M ratio with respect to the metal nitrates was added as a dispersant. Stirring was continued until a homogeneous solution. The solution was kept at 70 °C for 5 h to form the dried gel. Finally, samples were first pre-treated at 400 °C in air for about 4 h, and then the temperature was slowly increased to 800 °C and kept an hour to remove carbon. The final powders were annealed at 1100 °C in 8% H₂ and 92% Ar atmosphere for 4 h to obtain $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.02, 0.04, 0.06, 0.08, 0.1) materials.

The structures of the Eu_{1-x}Ba_xTiO₃ were investigated by X-ray diffraction (XRD) with Cu K α radiation at room temperature. Magnetizations were measured by employing a commercial superconducting quantum interference device (SQUID) magnetometer, model MPMS-7 from Quantum Design Inc.

3. Results and discussion

Fig. 1 shows the XRD patterns of the products $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.02, 0.04, 0.06, 0.08, 0.1) obtained by thermal treatment at 1100 °C for 4 h in 8% H₂ and 92% Ar. The x-ray powder diffraction pattern confirmed the single-phase nature of the compound (no XRD peaks arising from impurities such as $Eu_2Ti_2O_7$, Eu_2O_3 , and TiO₂ were detected), and crystallizing in the cubic perovskite structure (space group 221). The lattice parameters were determined to be a = 3.908 (2) Å, with Rwp = 14.6% and CHI² = 2.42 for EuTiO₃ by the Rietveld technique using GSAS program. With increasing the Ba concentration, the diffraction peaks just slightly move to the left as shown inset of Fig. 1, because the size Ba²⁺ ion (~1.35 (2) Å) is bigger than Eu^{2+} (~0.94 (7) Å), associated with the doping of Ba at Eu site.

Fig. 2 shows the zero-field-cooling (ZFC) and field-cooling (FC) temperature dependence of magnetization for $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.02, 0.04, 0.06, 0.08, 0.1) compounds under an applied magnetic field of 0.01 T. The ZFC and FC curves for these samples are well reversible, indicating no thermal hysteresis, which is good for



Fig. 1. XRD patterns of the $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.04, 0.08, 0.1). Inset: the corresponding local amplification figure.

application. The peak feature of these *M*-*T* curves for EuTiO₃ results from the AFM to paramagnetic (PM) transition at $T_N = 5.7$ K, induced by Eu²⁺ spins ordering below T_N , which is identical with the G-AFM transition point of previous reported [18,29,30]. With Ba substituting for Eu, the transition temperature was decreased [26]. The EuTiO₃ has strong spin-lattice coupling, when the lattice constant is increased, the magnetic ground state can be switched from AFM to FM [28]. In our previous report, the AFM ordering of pure EuTiO₃ can be significantly changed to be FM with slight Cr-doping [31]. When Ba substitute for Eu, the *M*-*T* curve of Eu_{0.9}Ba_{0.1}TiO₃ compound exhibits a transition from PM to FM and $T_C = 5.6$ K, corresponding to the maximum slope of *M*-*T* curve. Although, with increasing the concentration of Ba, the ground state has been changed from AFM to FM, the transition temperature has not been changed (around 5.5 K) as $x \le 0.1$.

On the other hand, we notice that the reciprocal magnetic susceptibility (χ_m^{-1}) of these compounds follow the Curie-Weiss law $\chi_m^{-1} = (T-\theta_p)/C_m$ above 10 K. Here θ_p is the PM Curie temperature and C_m is the Curie-Weiss constant. The effective magnetic moments (μ_{eff}) are obtained based on the value of C_m . With increasing the content of Ba, the values of μ_{eff} are decreased as shown in Fig. 2(b). It is found that θ_p (3.1 K $\leq \theta_p \leq 4.2$ K) is positive for all the compositions and the effective magnetic moment decreases from 7.6 μ_B for x = 0–7.2 μ_B for x = 0.1, due to Eu²⁺ site dilution by Ba²⁺ ions. But the values of μ_{eff} also belong to a reasonable region ($\mu = 7 \pm 1 \mu_B$), which are found to be consistent to the Eu²⁺ free ion as previously reported [32]. The localized 4f moments on the Eu²⁺ (S = 7/2, L = 0) sites order, and the Eu²⁺ 4f⁷ band is a reason for narrowing of the optical band gap in the title compounds [33].

No magnetic hysteresis can be observed from the field dependences of magnetization data, which collected during both



Fig. 2. (a) Temperature dependences of ZFC and FC magnetizations of $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.04, 0.08, 0.1) under the magnetic field of 0.01 T; (b) the temperature variation of the ZFC inverse susceptibility fitted to the Curie-Weiss law.

increasing and decreasing field model at 2 K in Fig. 3. With increasing the content of Ba²⁺ ions, the values of M were increased at low field, which suggests a FM coupling between the local Eu 4f spins dominate in the delicate balance between AFM and FM phases. And, the magnetic moment calculated per Eu²⁺ atom in Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1) compounds are showed in the inset of Fig. 3. With doping Ba²⁺ ions, the magnetic moment decreases from 6.8 μ_B (x = 0) to 6.5 μ_B (x = 0.04), and from 6.57 μ_B (x = 0.04) to 6.53 μ_B (x = 0.04) the magnetization data is decreased, due to decreased of Eu²⁺ content. The increase from 6.5 μ_B (x = 0.04) to 6.57 μ_B (x = 0.04) maybe result from the magnetic ground state switched from AFM to FM, because the lattice constant was increased.

In order to further confirm the inference, the field dependence of magnetization in a temperature range from 2 to 4 K with an increment of 1 K, in magnetic fields ranging from 0 to 5 KOe were gave in Fig. 4. It can be seen that there exist intersections among the curves in Fig. 4(a). The low temperature magnetization is smaller than the high temperature value in low fields. On the contrary, this



Fig. 3. The field dependences of magnetization data both increasing and decreasing field model at 2 K in the Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1). Inset: magnetization data both increasing and decreasing field model at 2 K under low field.

condition reverses in higher fields. This agrees with the AFM ordering of the compound below T_N [34]. However, there are not intersections among the curves in Fig. 4(b–d), which imply the magnetic ground state changed from AFM to FM as Eu substituted by Ba.

We have also plotted M^2 versus H/M isotherms (known as the Arrott plots) in Fig. 5. Based on the Banerjee criterion [35], a magnetic transition is expected to be of the first order when the slope of Arrott plot is negative, whereas it will be of second order when the slope is positive. The negative slope of the Arrott plots below T_N , which is shown in Fig. 5(a) and (b), indicating the occurrence of the first order. The positive slope of the Arrott plot suggests that the phase transition in x = 0.08 and x = 0.1 is second order.

As is well known, the ΔS_M value can be calculated either from the magnetization isotherms by using the Maxwell relation $\Delta S(T,H) = \int_0^H (\partial M/\partial T)_H dH$ in which, T is the absolute temperature and *H* is the applied field [36]. Fig. 6 shows the values of $-\Delta S_M$ for $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.04, 0.08, 0.1) as a function of temperature under different magnetic field. The value of $-\Delta S_M$ is found to increase monotonically with applied magnetic field increasing. Under the magnetic field change from 0 to 5 T, the values of $-\Delta S_M^{max}$ reach 40.3 J/kg K for EuTiO₃, 39.9 J/kg K for Eu_{0.96}Ba_{0.04}TiO₃, 41.1 J/kg K for Eu_{0.92}Ba_{0.08}TiO₃ and 32.9 J/kg K for Eu_{0.9}Ba_{0.1}TiO₃ around the liquid helium temperature. Especially, the giant values of $-\Delta S_M$ are observed in the Eu_{1-x}Ba_xTiO₃ compound under field change of 1 and 2 T, which can be realized by a permanent magnet. The negative values of $-\Delta S_M$ for EuTiO₃ compound can be observed at low temperature under low magnetic field change, which also confirm the antiferromagnetic order below T_N . And the negative values of $-\Delta S_M$ for Eu_{0.96}Ba_{0.04}TiO₃ compound only can be observed at 2.5 K under low magnetic field change (0-1 KOe), but it can't be observed in Eu_{0.92}Ba_{0.08}TiO₃. It indicates that the magnetic ground state changed from AFM to FM with increasing the content of Eu substituted by Ba.

Fig. 7 shows the values of $-\Delta S_M$ for Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1) as a function of temperature under magnetic field changed 1 T and 2 T. It can be obviously observed that the value of $-\Delta S_M$ is increased below the transition temperature, which may be caused by the magnetic ground state switched from AFM to FM



Fig. 4. Magnetization isotherms of $Eu_{1-x}Ba_xTiO_3$ collected at 2, 3 and 4 K under low field, (a: x = 0; b: x = 0.04; c: x = 0.08; d: x = 0.1).

as substitution of Ba²⁺ ions for Eu²⁺ions. The value of $-\Delta S_M^{max}$ reaches 12.5 and 23.4 J/kg K for Eu_{0.92}Ba_{0.08}TiO₃ under a magnetic field change 0–1 and 0–2 T, respectively. It is much large and comparable than those of most potential magnetic refrigerant materials in a similar magnetic transition temperature under the

same field change (2 T), such as TmCuAl (17.2 J/kg K) [10], $ErMn_2Si_2$ (20 J/kg K) [11], $ErRu_2Si_2$ (11 J/kg K) [14] and $ErCr_2Si_2$ (24 J/kg K) [37] et al.

Refrigerant capacity (RC) is another important parameter, which is a measure of how much heat can be transferred between the cold



Fig. 5. The Arrott plot of the $Eu_{1-x}Ba_xTiO_3$ compounds at 2, 3 and 4 K under low field, (a: x = 0; b: x = 0.04; c: x = 0.08; d: x = 0.1).



Fig. 6. Temperature dependences of magnetic entropy change for $Eu_{1-x}Ba_xTiO_3$ under different magnetic field (a: x = 0; b: x = 0.04; c: x = 0.08; d: x = 0.1). Inset: magnetic entropy change for $Eu_{1-x}Ba_xTiO_3$ under low magnetic field.



Fig. 7. Temperature dependences of magnetic entropy change for $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.04, 0.08, 0.1) under magnetic field 1 and 2 T.

and the hot sinks in one ideal refrigeration cycle. The RC, defined as a cooling capacity of $RC = \int_{T_1}^{T_2} \Delta S_M dT$, where T_1 and T_2 are the temperatures at half maximum of the peak taken as the integration limits [33]. By using this method, the RC values of the Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1) are evaluated under the magnetic field changes of 1, 2 and 5 T, the results are listed in Table 1. The values of RC in Eu_{0.96}Ba_{0.04}TiO₃ and Eu_{0.92}Ba_{0.08}TiO₃ were improved because the $-\Delta S_{Max}^{max}$ values below the transition temperature were increased. Therefore, Eu_{1-x}Ba_xTiO₃ (x = 0, 0.04, 0.08, 0.1) compounds appear to be a very attractive candidate material for use in a magnetic refrigerator working in low temperature.

4. Conclusion

A giant reversible MCE has been observed in $Eu_{1-x}Ba_xTiO_3$ (x = 0, 0.04, 0.08, 0.1) compounds under low field. Under the magnetic field changes of 2 T, the values of $-\Delta S_M^{max}$ and RC are evaluated to be 23.4 J/kg K and 119.6 J/kg, without magnetic and thermal hysteresis for $Eu_{0.92}Ba_{0.08}TiO_3$ system. Especially, in the $Eu_{1-x}Ba_xTiO_3$ compounds, the giant values of $-\Delta S_M^{max}$ (more than 10 J/kg K) is obtained for a relatively low field change of 0–1 T. The AFM ordering of pure EuTiO₃ can be significantly changed to be FM as slight substitution of Ba^{2+} ions for Eu^{2+} ions, which could

| Table 1 | |
|--|----|
| The values of $-\Delta S_M$ and RC under the field changes of 1, 2 and 5 T for Eu _{1-x} Ba _x TiO ₃ ($x = 0, 0.04, 0.08, 0.1$ |). |

| Samples | $\Delta H = 1 T$ | | $\Delta H = 2 T$ | | $\Delta H = 5 \ T$ | |
|--|------------------------------|-----------|------------------------------|-----------|------------------------------|-----------|
| | $-\Delta S_M^{max}$ (J/kg K) | RC (J/kg) | $-\Delta S_M^{max}$ (J/kg K) | RC (J/kg) | $-\Delta S_M^{max}$ (J/kg K) | RC (J/kg) |
| EuTiO ₃ | 11 | 34 | 23 | 106.3 | 40.3 | 328.1 |
| Eu _{0.96} Ba _{0.04} TiO ₃ | 11.5 | 37.9 | 22.1 | 113.2 | 39.9 | 306.4 |
| Eu _{0.92} Ba _{0.08} TiO ₃ | 12.5 | 54.1 | 23.4 | 119.6 | 41.1 | 322.3 |
| Eu _{0.9} Ba _{0.1} TiO ₃ | 10.1 | 45.1 | 19.1 | 109.1 | 32.9 | 286.3 |

increase the values of $-\Delta S_M$ and RC. In a word, the results suggest the $Eu_{1-x}Ba_xTiO_3$ compounds could be considered as a good candidate material for low-temperature and low-field magnetic refrigerant.

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