Transverse Kerr effect in the (La$_{1-x}$Pr$_x$)$_{0.7}$Ca$_{0.3}$MnO$_3$ ceramics

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Abstract. Magneto-optical properties were measured for (La$_{1-x}$Pr$_x$)$_{0.7}$Ca$_{0.3}$MnO$_3$ ($x = 0, 0.25, 0.5, 0.75, 1$) ceramic solid solutions, including temperature, magnetic field, oxygen isotope content and spectral dependences of the transverse Kerr effect (TKE). The TKE spectra after the transition to the ferromagnetic metallic state depend only weakly on the substitution of La for Pr as long as $x \leqslant 0.5$. A surface magnetism contribution results in a TKE peak near T_C but it can be suppressed by magnetic field or a long-time annealing. A significant oxygen isotope effect is observed for all compositions, being critical for $x = 0.75$. Hysteresis phenomena of two different types were observed in the temperature dependences of TKE. The first type, related to the ferromagnetic fluctuations, was found for $x \leqslant 0.5$ (TKE is larger on cooling). The second type was related to the magnetically inhomogeneous state for $x = 0.75$ and 1 (TKE is larger on heating). The hysteretic variations in the magneto-optical spectrum of (La$_{0.25}$Pr$_{0.75}$)$_{0.7}$Ca$_{0.3}$MnO$_3$ are referred to the possible charge carrier redistribution between the incipient ferromagnetic component and the charge ordered antiferromagnetic component.

1. Introduction

Perovskite manganites demonstrate unique physical properties due to the coupling between electron, phonon and magnetic subsystems [1]. The coupling results in a sequence of unusual effects like colossal magnetoresistance and metal–insulator transitions induced by the oxygen isotope exchange [2, 3], external pressure [4] or irradiation with light or x-rays [5]. The properties of the perovskite manganites R$_{1-x}$A$_x$MnO$_{3+\delta}$ can be tuned by isovalent doping with rare earth elements (R) at a constant level of heterovalent doping with alkaline earth elements (A). The Mn^{4+} content is not affected by the tuning taking into account that $\delta < 0.01$ for R$_{1-x}$A$_x$MnO$_{3+\delta}$ with $0.2 < x < 0.5$ and A = Sr, Ca if the synthesis is carried out in oxygen or air [6, 7]. Respectively, the effect of chemical pressure on the electrical and magnetic properties can be analysed in a straightforward way. Understanding of the colossal magnetoresistance is still lacking nowadays and needs more experimental data on the conductivity mechanism, electronic structure and its variation at the magnetic phase transitions. We report below on the temperature, magnetic field, oxygen isotope content and spectral dependences of the magneto-optical (MO) properties of (La$_{1-x}$Pr$_x$)$_{0.7}$Ca$_{0.3}$MnO$_3$ ($x = 0, 0.25, 0.5, 0.75, 1$) ceramic samples.

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Measurement of the MO spectra in the visible and near ultra-violet spectral range, i.e. in the range of the normal absorption, could provide information concerning electronic structure of the perovskite manganites and its dependence on the variation of the chemical and isotope composition, conditions of the synthesis and so on. In particular, variation of the magnetic ion ratio (Mn^{3+}/Mn^{4+}) is expected to influence MO spectra. The magnitude of the MO response is known to be proportional to the sample magnetization and probability of the electron transitions, therefore the temperature dependence for different magneto-optical effects (MOE) measured at a definite wavelength indicates the variation of the magnetic order in the samples. Temperature and magnetic field dependences of MOE reveal the phase transition temperatures and peculiarities of the magnetically ordered states. Therewith, the penetration depth that contributes to the formation of the MO signal is about 0.1 μm for the material under consideration. Respectively, we observe only the behaviour of the surface layer of the sample, keeping in mind that its magnetic state can be different as compared to the bulk. MO response is sensitive not only to the long-range magnetic order but even to the short-range magnetic order. Thus, formation of the ferromagnetic clusters would manifest itself in the magneto-optical properties.

To study MO properties we have chosen measurements of the linear transverse Kerr effect (TKE) that consists in the intensity variation of the light reflected by a sample under magnetization (the magnetic field is aligned parallel to the sample surface and perpendicular to the light incidence plane). Variation of the reflected light intensity for a p wave due to the magnetization of the ferromagnetic sample can be written as [8]

$$\delta_p = 2\sin 2\varphi \frac{A_1}{A_1^2 + B_1^2}\varepsilon'_{xy} + 2\sin 2\varphi \frac{B_1}{A_1^2 + B_1^2}\varepsilon''_{xy} \tag{1}$$

where $A_1 = \varepsilon''_0(2\varepsilon'_0\cos^2\varphi - 1)$, $B_1 = (\varepsilon''^2_0 - \varepsilon'^2_0)\cos^2\varphi + \varepsilon'_0 - \sin^2\varphi$, ε'_0, ε''_0, ε'_{xy}, ε''_{xy}, are real and imaginary part of the diagonal and non-diagonal elements of the dielectric tensor. In the linear magnetic field approximation the dielectric tensor of a gyroelectric medium is

$$\tilde{\varepsilon} = \left\{\begin{matrix} \varepsilon_0 & i\varepsilon_{xy} & 0 \\ -i\varepsilon_{xy} & \varepsilon_0 & 0 \\ 0 & 0 & \varepsilon_0 \end{matrix}\right\}. \tag{2}$$

The diagonal elements describe normal optical properties and off-diagonal elements are related to magneto-optical properties. The linear TKE is uneven in relation to the magnetization.

2. Experiment

The ceramic samples were prepared as follows: ash-free paper filters were soaked with water solution of the metal nitrates, then the ashes formed by burning of the dried paper were pressed into pellets and sintered in air at 1200 °C for 12 h. The ceramic pellets were single phase perovskites with the orthorhombic *Pnma* structure for all x-values according to the powder XRD characterisation. About 1 mm thick pellets with the natural content of oxygen isotopes (98% ^{16}O) were polished mechanically and annealed for 1 h in air at 900 °C for recovery of the sample surface. Afterwards the MO spectra and their temperature dependences were measured (the O-16 series).

The measurements of TKE were made using an automatic MO spectrometer [9]. A dynamic method to record TKE was used. The relative change in the intensity of the reflected light

$$\delta = (I(H) - I(0))/I(0) \tag{3}$$

where $I(H)$ and $I(0)$ are the intensities of the reflected light in the presence and in absence of a magnetic field respectively, was directly measured in the experiment. The magnitude of the alternating magnetic field (39 Hz) in the gap of the electromagnet was up to 1 kOe. The sensitivity of the apparatus was 10^{-5}. MO spectra were recorded in the photon energy range 1.3–3.8 eV at a fixed light incidence angle of $67°$. For low-temperature measurements an optical continuous flow helium cryostat was used, which made it possible to control the temperature in the range 10–300 K.

For the enrichment of the sample surface with ^{18}O isotope, the pellets were put into alumina boats which were placed in a quartz tube mounted in the furnace. The quartz tube formed a part of the closed loops where enforced circulation of gas was applied. The samples were treated in the oxygen gas with the molar fraction of $^{18}O_2$ of 85% under the oxygen pressure of 1 bar for 4 h at a temperature of 950 °C. The penetration of the ^{18}O in the samples was controlled by SNMS depth profiling (Leybold, INA-3). The diffusion profiles obtained were analysed in the frame of the one-dimensional diffusion model. After the subtraction of the intergranular diffusion contribution the intragranular diffusion coefficient for ^{18}O tracer was found $\sim 2 \times 10^{-12}$ cm^2 s^{-1} for all $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ samples. Thus, the effective oxygen exchange takes place mostly in a few μm thick surface layer of the pellet which is much more than the penetration depth of the light.

After the cycle of MO measurements with the samples enriched with ^{18}O (the O-18 series) the surface layer ($\sim 50\,\mu$m thick) was ground off, the pellets were polished again, then subjected to the same recovery annealing and new cycle of the MO measurements (the O-16′ series). The O-16′ series allows us to estimate the contribution from the annealing procedures.

3. Results and discussion

Temperature dependences of TKE $\delta(T)$ were measured at the fixed energy of the incident light ($h\omega = 2.7$ eV) upon sample heating for the series O-16, O-18 and O-16′ (figure 1). The $\delta(T)$ curves reveal characteristic features at the transition to the magnetically ordered state. The position of the abrupt decrease of TKE on the temperature scale corresponds to the Curie temperature T_C, derived from the magnetic susceptibility measurements [10].

In addition, a peak on $\delta(T)$ curves was observed (the most profound one for $x = 0.25$ and 0.5) near the transition. The peak was related to the peculiar magnetic state of the topmost surface layer formed as a result of the mechanical treatment. The peak was suppressed by an additional recovery annealing at 1000 °C for 30 h. It also disappeared with the increase of the magnetic field (figure 2). While the TKE below T_C increased with the magnetic field the peak was saturated already in the field of 100 Oe.

For each sample the spectral dependence was measured in the temperature range where $\delta(T)$ was at its maximum that corresponds to the maximum of magnetization. Spectra of TKE for $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ with different x are shown in figure 3. The magnitude of the TKE depends only weakly on the substitution of La for Pr when $x \leqslant 0.5$, and spectra are similar after the transition to the ferromagnetic metallic state. The similarity is evidence that MO activity of the compounds is due to the electronic transitions in the octahedral complexes of Mn^{3+} and Mn^{4+} ions. In the spectral range under study, there is an allowed electric dipole transition in the octahedral complex $(MnO_6)^{9-}$ at $h\omega = 3.5$ eV and spin-resolved d–d transitions in Mn^{3+} ions ($h\omega = 2.5$ eV) and Mn^{4+} ions ($h\omega = 2.6, 3.1$ eV) [11]. Thus, the spectrum of TKE in the range 2.5–3.8 eV is formed by the electronic transitions both in Mn^{3+} and Mn^{4+} ions. The substitution of La for Pr causes tilting of the octahedra rather than their deformation and does not change the Mn^{3+}/Mn^{4+} ratio and position of bands in the MO spectra (in the spectral range of the fundamental absorption) but strongly affects the magnitude of the MOE and the
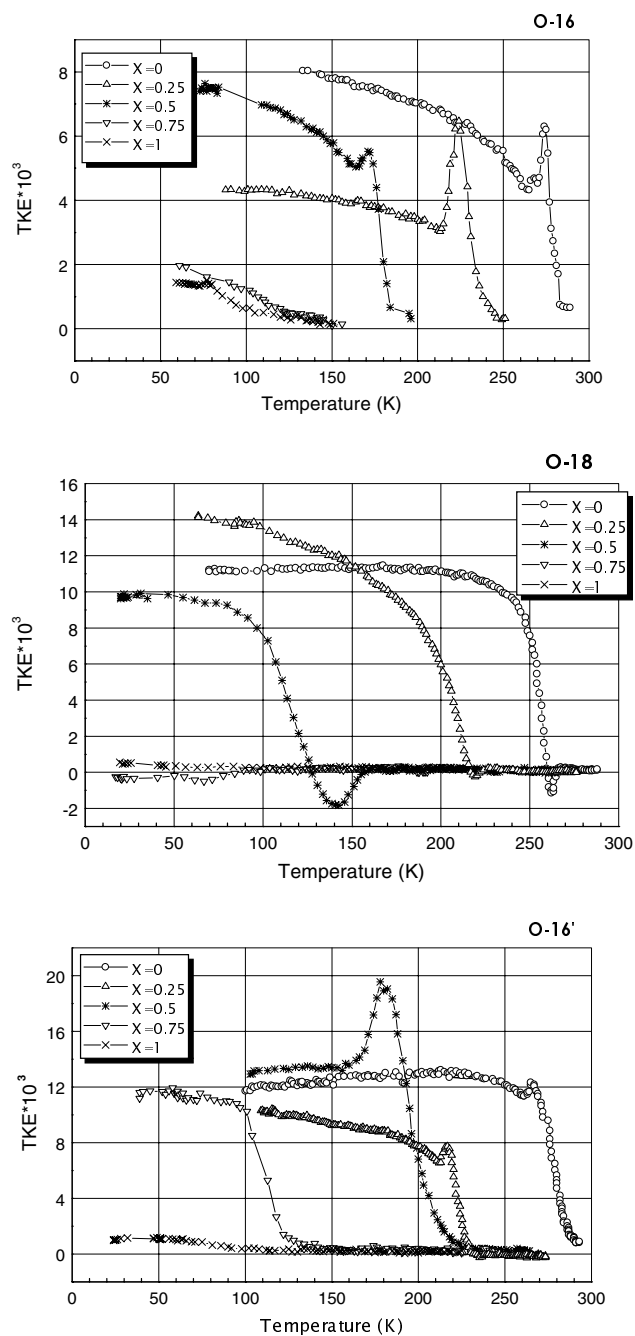
Figure 1. Temperature dependence of the transverse Kerr effect on the chemical composition of the $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ ceramics for the three series: O-16; O-18; O-16′.

temperatures of the magnetic phase transitions. The analogous situation is known for MO spectra of rare earth orthoferrites where the spectrum is determined by the transitions in the $(FeO_6)^{9-}$ octahedral complexes but the peculiarities of the magnetic state like the temperature
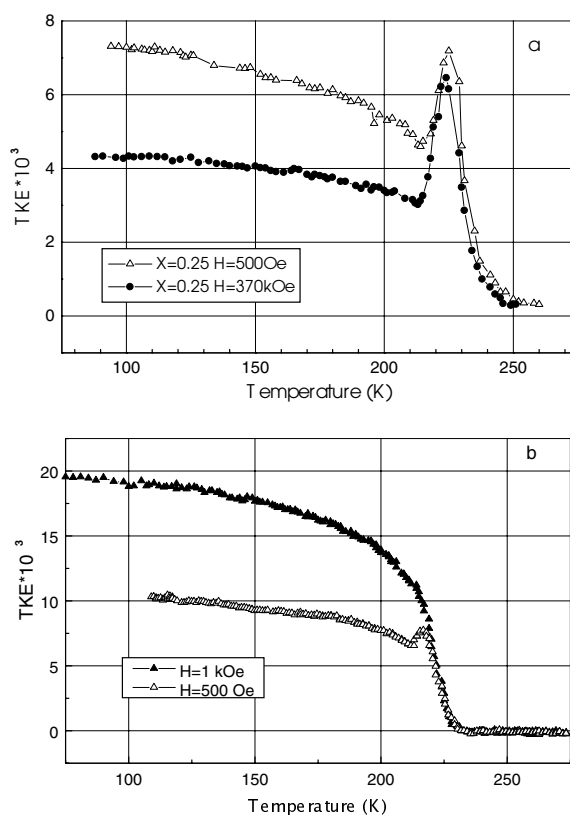
Figure 2. The suppression of the sample surface related peak near T_C by magnetic field and due to the long-time recovery annealing: (a) temperature dependence of TKE for $(La_{0.75}Pr_{0.25})_{0.7}Ca_{0.3}MnO_3$ after 1 h recovery annealing at 900 °C; (b) temperature dependence of TKE for $(La_{0.75}Pr_{0.25})_{0.7}Ca_{0.3}MnO_3$ after 30 h recovery annealing at 1000 °C.

of the spin reorientation transition are related to the interaction of the A and B sublattices of the perovskite structure [9].

The special feature of the $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ system is the tendency to the charge ordering and magnetic inhomogeneity that manifests itself most vividly for $x > 0.5$ [3, 10]. In contrast to the samples with $x \leqslant 0.5$, for the sample $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$ the spectrum was very different depending on whether a cooling or heating process was used to derive the $\delta(h\omega)$ dependence. TKE spectra for $x = 0.75$ at different temperatures are shown in figure 4. The magnitude of TKE at 100 K on heating and cooling at $h\omega = 2.7$ eV was changed by a factor of 15. Another remarkable feature is a strong shift of the main spectral maximum to lower energy (down to 2.4 eV) with appearance of the additional maximum at 3.25 eV on cooling and its return to the value typical for $x \leqslant 0.5$ samples after cooling well below T_C and on subsequent heating.

The magnitude of the TKE for $Pr_{0.7}Ca_{0.3}MnO_3$ ($x = 1$) correlates with the magnetic neutron diffraction data revealing a small fraction of the ferromagnetic component in the sample ($\sim$10%) [12]. For comparison, $(La_{0.75}Pr_{0.25})_{0.7}Ca_{0.3}MnO_3$ (i.e. $x = 0.25$) is known to contain only a ferromagnetic component according to the neutron diffraction (magnetic moment 3.5 μ_B per Mn site) [13].
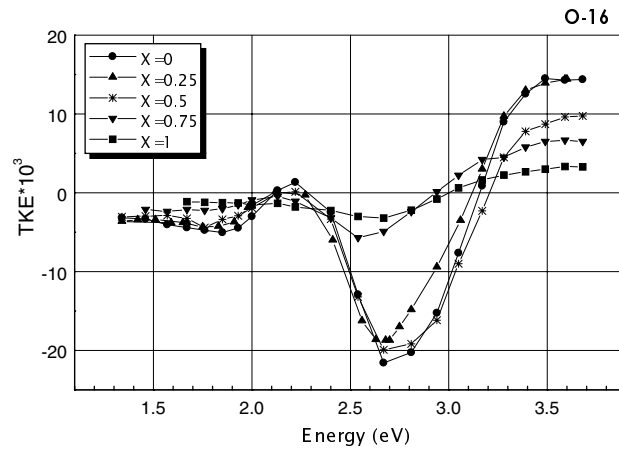
Figure 3. The transverse Kerr effect spectra of the $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ ceramics for the series O-16 at the maximum magnetization, angle of light incidence $\varphi = 67°$.
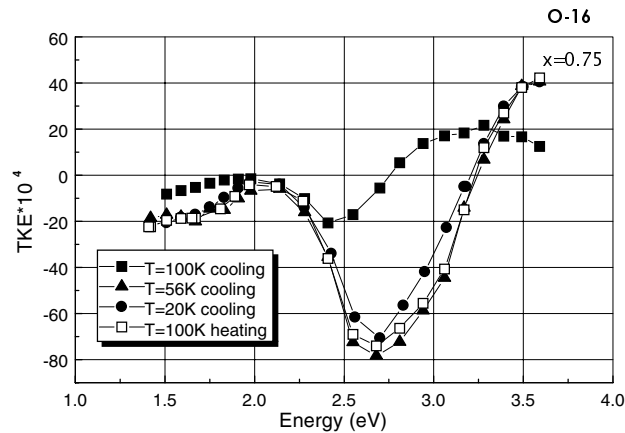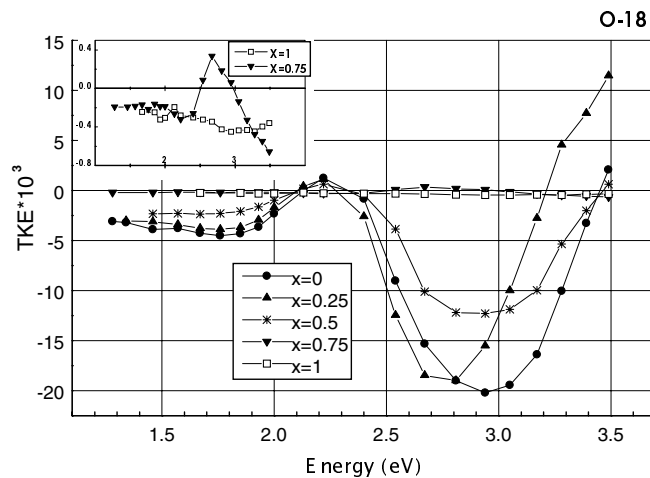

Figure 4. The transverse Kerr effect spectra for $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$ at different temperatures recorded on cooling and heating.

Next we consider effects of the isotope exchange.

Isotope exchange influenced $\delta(T)$ curves for all samples (figure 1(b)). The compositions with $x \leqslant 0.5$ revealed a shift of T_C values to a lower temperature but nearly the same maximum value of the TKE. Simultaneously some broadening of the band near 3 eV and a shift of the spectral maximum to the higher energy was registered (figure 5). The phenomenon is worthy of further study. Now we only can notice the more diversified isotope content of O-18 samples as compared to O-16 samples.

The most significant variation of both spectrum and magnitude of TKE was observed for $x = 0.75$ and 1 (figure 5, inset). For the sample with $x = 0.75$ we have found more than an order of magnitude decrease of TKE with simultaneous switching of the effect sign in the energy range $\hbar\omega > 2.5$ eV. Earlier it was shown [3] that the composition with $x = 0.75$ with natural isotope content of oxygen becomes ferromagnetic and metallic below 100 K. At the same time the composition enriched with the ^{18}O isotope is an antiferromagnet and insulator down to 4 K.

Figure 5. The transverse Kerr effect spectra for the various chemical compositions of the $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ ceramics in the series O-18, at $T = 60$ K for $x = 0$, 0.25 and 0.5 and at $T = 40$ K for $x = 0.75$ and 1; angle of light incidence $\varphi = 67°$.

Neutron diffraction study of the magnetic order had shown that by cooling the ^{16}O sample is subjected to the subsequent antiferromagnetic ($T_N = 150$ K) and ferromagnetic ($T_C = 100$ K) transitions, whereas the O-18 sample showed pure antiferromagnetic order down to 4 K [14].

The small magnitude and switching of the sign of TKE for O-18 sample with $x = 0.75$ (figure 5, inset) does not contradict the antiferromagnetic ordering under cooling. This small TKE is probably due to the magnetic coupling between the surface and the inner part of the sample where ^{16}O is in excess and the T_C value corresponds to the ^{16}O sample (in such a situation, the sign switching can be due to the demagnetization field applied on the sample surface).

The O-18 sample of the composition $Pr_{0.7}Ca_{0.3}MnO_3$ demonstrated only very weak MOE below 60 K which can be related to the appearance of the small magnetic moment due to the magnetic ordering of Pr^{3+} ions not being affected by the isotope exchange. The ordering at 60 K takes places in the O-16 state too as follows from the magnetic neutron diffraction data [12].

Another consequence of the isotope exchange is the switching of the TKE at the peak near T_C for the samples with $x \leqslant 0.5$ (just compare figures 1(a), (c) with figure 1(b)). In O-18 samples we can expect that the demagnetization field created by the inner part of the pellet containing ^{16}O will cause the anti-phase magnetization of the peculiar topmost layer responsible for the peak. In fact, on cooling both the inner part and the topmost surface layer become ferromagnetic earlier than a non-ferromagnetic spacer between them. On subsequent cooling the spacer becomes ferromagnetic too. It reconciles the phase of the magnetization of the topmost layer with the external field, which switches the sign of the linear TKE.

The TKE dependences for O-16′ samples agree with those found for O-16 samples with the exception of $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$. In the latter case we observed a difference between the O-16 and O-16′ samples in the magnitude of TKE (by a factor of 2.5). At least, oxygen stoichiometry variation cannot be responsible for the difference as the annealing conditions were the same for O-16 and O-16′; moreover all $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ samples are stoichiometric in oxygen under the annealing conditions. Taking into account the very high sensitivity of $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$ composition to the effects like isotope substitution, weak magnetic field and lattice strain [3, 10] the mechanism of the FM fraction variation can be rather subtle, like annealing of the structural defects during the heat treatment.

Finally, we discuss the hysteresis phenomena found.

The first type of hysteresis is the most pronounced for O-16 and O-16′ samples of $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$. It takes place below 120 K. The magnitude of the TKE was lower on cooling than on heating. Such a behaviour can be described in the frame of the magnetically inhomogeneous state including antiferromagnetic (AFM) and ferromagnetic (FM) components (two different magnetic order parameters). The experimental results imply a variation in the fraction of the FM component. The TKE magnitude for the sample O-16′ correlates with the magnetic neutron diffraction data reported in [14] (about 80% of the FM component). For $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ the FM state is overwhelming for $x < 0.75$ and the AFM state is overwhelming for $x > 0.75$. The hysteresis flattens the boundary. By cooling of the O-16 sample with $x = 0.75$ the TKE started below 150 K and increased gradually down to the liquid helium temperature. On subsequent heating the TKE was nearly constant up to 100 K and then dropped abruptly in the temperature range 100–120 K.

As mentioned above, the spectra taken at the same temperature 100 K on cooling and on heating of $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$ were different (figure 4). On cooling down to 80 K we measured a spectrum similar to that of $Pr_{0.7}Ca_{0.3}MnO_3$, i.e. the composition with the modest FM component in the insulating antiferromagnetic matrix. On cooling below 80 K and on following heating we measured a spectrum that is similar to the behaviour of the ceramics with smaller x, i.e. the behaviour of the ferromagnetic metal-like state. The variation of the spectrum implies a deviation of the Mn^{3+}/Mn^{4+} ratio from the mean value in the FM component. The incipient FM component is depleted in holes. Respectively, the AFM component would be enriched with holes stabilizing 1:1 charge ordering of Mn^{3+} and Mn^{4+} which means a gain in the total free energy. At the lower temperature the homogeneous distribution of the charge carriers is recovered with an increase in the fraction of FM component. Conventional optical and magnetic measurements as well as neutron diffraction providing the averaged picture were not able to detect the transient state.

The second type of hysteresis is typical for the ceramics with $x \leqslant 0.5$. In contrast to the sample with $x = 0.75$ the TKE magnitude was larger on cooling than on heating (compare figure 6(a) and 6(b)). The appearance of such a broad temperature hysteresis at the transition to the magnetically ordered state for $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ ceramics indicates ferromagnetic fluctuations (formation of the ferromagnetic clusters) far into the paramagnetic phase area. Such strong short-range magnetic fluctuations in the paramagnetic phase of the perovskite manganites were recently reported by Lynn *et al* [15] and de Teresa *et al* [16].

As MOEs are sensitive not only to the long-range magnetic order but also to the short-range order, an appearance and remagnetization of such clusters implies an increase in the MO activity. The number and size of the clusters is expected to be a function of the sample prehistory and leads to the hysteresis of MOE above T_C. Such hysteretic phenomena were also observed for $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ thin epitaxial films on different substrates [17].

Thus, two completely different hysteresis phenomena were observed, depending on x values, in $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$. With sole ferromagnetic ordering for $x \leqslant 0.5$ the hysteresis reflects short-range ferromagnetic fluctuations in the paramagnetic phase. With antiferromagnetic ordering prior to the ferromagnetic ordering for $x > 0.5$ the hysteresis reflects the first-order nature of the metamagnetic transition.

4. Conclusions

The measurements of the MOE provide insight into the magnetic ordering processes in the perovskite manganites and their interrelation with electronic band structure and
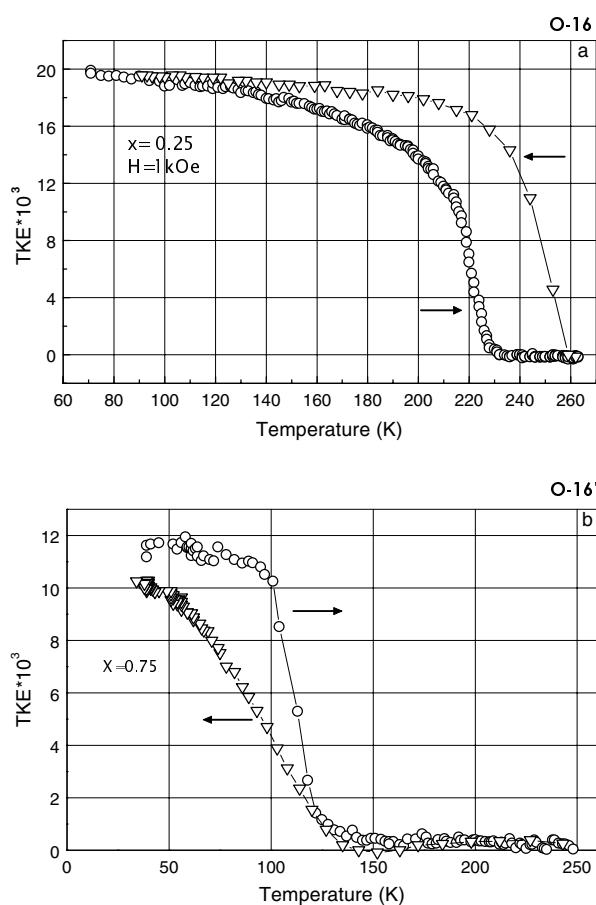
Figure 6. The temperature hysteresis of TKE for $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$: (a) $x = 0.25$; (b) $x = 0.75$. Cooling and heating are indicated by triangles and circles respectively.

electron–phonon coupling. Valuable information can be obtained without sample destruction and with a rather small amount of the material under investigation. The MOE measurements keep track of the FM component in contrast to the conventional optical and magnetic measurements which provide an averaged picture. Curie temperature, magnetization curve, hysteretic phenomena, magnetic and charge inhomogeneous states, surface magnetism (if different from that of the bulk) and even the short-range magnetic order can be deduced from the measurement of the temperature and spectral dependences of TKE.

According to the TKE measurements both the increase in x and oxygen isotope exchange ^{16}O for ^{18}O result in the systematic decrease of the Curie temperature T_C for $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$.

A shift in the TKE spectra occurs in the magnetically inhomogeneous state of $(La_{0.25}Pr_{0.75})_{0.7}Ca_{0.3}MnO_3$, which implies a charge carrier redistribution between the incipient FM component and overwhelming AFM component. The effect has not been detected so far by other techniques.

Two hysteresis phenomena of opposite sign were observed in $(La_{1-x}Pr_x)_{0.7}Ca_{0.3}MnO_3$ depending on x values. They come from short-range ferromagnetic fluctuations and the first-order metamagnetic phase transition, respectively.

The peak of TKE near T_C in low magnetic field is a signature of the surface magnetism and presents interest for practical applications.

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