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Possible anisotropy gap in $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ detected through specific heat and magnetization measurements

J. López, O.F. de Lima*

Instituto de Física Gleb Wataghin, Universidade Estadual de Campinas, 13083-970, Campinas, SP, Brazil

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Abstract

Magnetization and specific heat measurements, as a function of temperature, were performed on single crystals of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$, under different applied magnetic fields (H). The specific heat in $La_{1.35}Sr_{1.65}Mn_2O_7$ was decreased for $H = 9$ T parallel to the crystal c axis, compared with $H = 0$, possibly due to a suppression of spin-wave excitations (magnons) in that ferromagnetic bilayer structure. On the other hand, the applied magnetic field had no effect in the specific heat of the antiferromagnetic $La_{1.5}Sr_{0.5}NiO_4$. For $H = 9$ T and below the temperature of 4 K the specific heat data, for each crystal, was well fitted by an exponential decay law. This allowed the calculation of energy gaps around 1 meV for both compounds, in close agreement with $\Delta = 2\mu_B H$ for an expected energy gap in the magnon spectrum. Detailed magnetization measurements showed monotonic variations below 4 K and a steep increase close to 2 K. Both magnetization and specific heat measurements suggest the existence of an anisotropy gap in the energy spectrum of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$.
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1. Introduction

The combined ordering of charge (CO) and spin (SO) is proving to be a common phenomenon in transition metal oxides like $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$. The magnetic properties of Mn and Ni perovskites are considered to arise from the strong competition involving ferromagnetic and antiferromagnetic interactions and the spin–phonon coupling [1–7]. The dimensionality of the relevant structure involving the transition metal ions, three-dimensional (3D) or quasi-two-dimensional (2D), also plays an important role. For instance, the bilayer structure compounds $La_{2-2x}Sr_{1+2x}Mn_2O_7$, in which MnO_2 and $(La, Sr)_2O_2$ layers (labeled as ab planes) are stacked alternately along the crystal c axis, have 2D electronic and magnetic properties [8]. Further, magnetic and electrical properties in these families depend on their oxygen contents.

The $La_{1.5}Sr_{0.5}NiO_4$ compound has its magnetic ions (Ni) confined in planes which are insulated by $(La, Sr)_2O_2$ layers and, like its parent compound La_2NiO_4, it is an insulator. In a related family, La_2CuO_4, the antiferromagnetic insulator phase is rapidly destroyed by doping, leading to a metallic superconductor phase at moderate hole concentration [9], while La_2NiO_4 remains nonmetallic up to quite large hole concentrations [10]. A study [11] on the CO in $La_{1.5}Sr_{0.5}NiO_4$, with the neutron diffraction technique, revealed a rearrangement of CO from checkerboard-type to stripe-type as a function of temperature. Also, it was found that the stripe phase persisted up to $x = 0.7$ for highly hole-doped samples of $Nd_{2-x}Sr_xNiO_4$ with $0.45 \leq x \leq 0.7$.

Here, we present the temperature dependence of the specific heat, without external magnetic field (H) and with $H = 9$ T, for $La_{1.35}Sr_{1.65}Mn_2O_7$ ($x = 0.325$) and $La_{1.5}Sr_{0.5}NiO_4$ single crystals. We found that the specific heat data could be fitted by an exponential decay law below 4 K. Detailed magnetization measurements below 10 K showed the existence of a peak close to 2 K. Both results, magnetization and specific heat, suggest the existence of an anisotropy gap in the energy spectrum

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* Corresponding address: Universidade Estadual de Campinas, Instituto de Física Gleb Wataghin, Barão Geraldo, 13083-970 Campinas, SP, Brazil. Tel.: +55 19 37885460; fax: +55 19 37884146.
E-mail address: delima@ifi.unicamp.br (O.F. de Lima).

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Table 1
Results of fitting the law $C = \beta_1 T + \beta_{3/2} T^{3/2} + \beta_3 T^3$ for 5 K < T < 10 K, $H = 0$ and 9 T

Sample	$H(T)$	β_1	$\beta_{3/2}$	β_3
$La_{1.35}Sr_{1.65}Mn_2O_7$	0	25.0 ± 0.1	1.01 ± 0.04	0.306 ± 0.005
	9	9.42 ± 0.06	2.66 ± 0.08	0.340 ± 0.003
$La_{1.5}Sr_{0.5}NiO_4$	0	15.0 ± 0.1	0.03 ± 0.02	0.250 ± 0.003
	9	16.0 ± 0.1	0.04 ± 0.02	0.242 ± 0.002

The units are mJ/mol K^{j+1}, where j is the coefficient subscript.

of both compounds. Our measurements confirm the complex magnetic excitation and electronic band structure due to charge ordering and the quasi-2D confinement of the magnetic ions. To our knowledge, specific heat measurements for $La_{1.5}Sr_{0.5}NiO_4$ crystals have not been reported yet.

2. Experimental methods

Single crystals of $La_{1.35}Sr_{1.65}Mn_2O_7$ ($3.0 \times 1.0 \times 0.70$ mm^3) and $La_{1.5}Sr_{0.5}NiO_4$ ($4.0 \times 1.2 \times 0.90$ mm^3) were grown by the floating zone method described elsewhere [12]. Specific heat measurements were made with a Quantum Design PPMS calorimeter that uses a *two-relaxation-times* technique, and data was always collected during sample cooling. The specific heat of the sample holder (addenda) was measured both without and with magnetic field. The intensity of the heat pulse applied to the sample was calculated to produce a variation in the temperature bath of 0.5%. Experimental errors during the specific heat and magnetization measurements were smaller than 1% for all temperatures, magnetic fields and samples. Magnetization measurements were done with a Quantum Design MPMS-5S SQUID magnetometer.

3. Results and discussion

Specific heat measurements at low temperatures give valuable information about the ground state excitations. Fig. 1 shows the dependence of the specific heat measurements with temperature, between 2 and 10 K, for $La_{1.35}Sr_{1.65}Mn_2O_7$ (squares) and $La_{1.5}Sr_{0.5}NiO_4$ (triangles) single crystals, studied with $H = 0$ (full symbols) and 9 T (open symbols). The magnetic field was applied perpendicular to the *ab* planes of both samples. The data, plotted as C/T versus T^2, show that the magnetic field strongly affected the low temperature ($T <$ 5 K) behavior of the $La_{1.35}Sr_{1.65}Mn_2O_7$ compound. However, the specific heat curves for the $La_{1.5}Sr_{0.5}NiO_4$ sample did not change with the applied field and were not linear at temperatures below 5 K. Continuous lines in Fig. 1 indicate the fitting of the experimental data, from 5 to 10 K, by the following expression:

$$C = \beta_1 T + \beta_{3/2} T^{3/2} + \beta_3 T^3. \tag{1}$$

Here C is the specific heat, T is the temperature and β parameters represent the contributions of electron interactions (β_1), ferromagnetic spin waves ($\beta_{3/2}$) and the first phonon mode β_3. Antiferromagnetic spin waves could also generate excitations that contribute to the specific heat as T^3. The values of these coefficients for the different samples and magnetic fields are shown in Table 1 with their corresponding errors, evaluated from the fitting procedure. With the exception of the zero-field curve for $La_{1.35}Sr_{1.65}Mn_2O_7$, Eq. (1) does not fit our experimental data below 5 K.

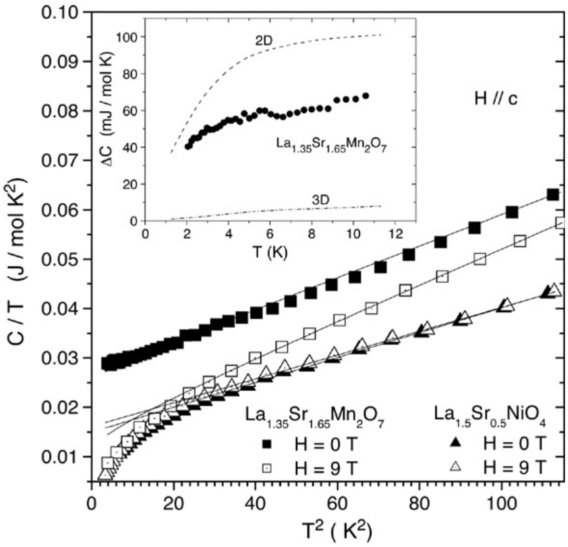

Fig. 1. Specific heat measurements between 2 and 10 K in the single crystals $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$, with $H = 0$ and 9 T parallel to the crystal c axis. Continuous lines represent fittings of the law $C = \beta_1 T + \beta_{3/2} T^{3/2} + \beta_3 T^3$. The data is plotted as C/T versus T^2 to facilitate the interpretation. Inset: $\Delta C = C(H = 0) - C(H = 9$ T) shows a quasi-2D ferromagnetic behavior.

It is worth stressing that, unlike for the $LaMnO_3$ compound, both $La_{1.5}Sr_{0.5}NiO_4$ and $La_{1.35}Sr_{1.65}Mn_2O_7$ have their magnetic ions confined in planes that are insulated by layers of La and Sr oxides. However, $La_{1.5}Sr_{0.5}NiO_4$ shows a different magnetic behavior compared to the $La_{1.35}Sr_{1.65}Mn_2O_7$ sample: the spins of the Ni ions order antiferromagnetically, while the spins of the Mn ions order ferromagnetically. Consistent with this fact, Fig. 1 shows that magnetic fields as high as 9 T do not produce a significant contribution to the specific heat of $La_{1.5}Sr_{0.5}NiO_4$, while a strong change is observed for $La_{1.35}Sr_{1.65}Mn_2O_7$.

From resistivity measurements the $La_{1.5}Sr_{0.5}NiO_4$ sample is an electrical insulator at low temperatures, and applied magnetic fields up to 9 T are not strong enough to destroy this characteristic [10,13]. Therefore, one should not expect a linear contribution from free electrons to the specific heat. However, other kinds of many-body excitations could also produce a linear contribution [14]. On the other hand, the compound $La_{1.35}Sr_{1.65}Mn_2O_7$ is metallic below about 100 K [8].

The authors of Ref. [8] found that the decrease of specific heat at low temperatures, due to an applied magnetic field of 9 T, parallel to the ab planes ($H \parallel ab$), was about ten times larger for a $La_{1.3}Sr_{1.7}Mn_2O_7$ sample than the observed values for $La_{1-x}Sr_xMnO_3$ samples (with $x = 0.3$ and 0.4). They also calculated the theoretical reduction in specific heat, due to an applied magnetic field, for the ideal simple cubic (3D) and simple square (2D) lattices. They concluded that the observed change in specific heat (ΔC) for the bilayered manganite was large, but less than the expected values for an ideal 2D ferromagnetism. In our case the ΔC values for the $La_{1.35}Sr_{1.65}Mn_2O_7$ sample are larger than those of Ref. [8], although they are also reasonably smaller than the expected ones for an ideal 2D ferromagnetism. This result is shown in the inset of Fig. 1, where the ideal 2D and 3D cases are represented by dashed and dot–dashed curves, respectively, as calculated by the authors of Ref. [8]. They also reported $\beta_1 \approx 3$ mJ/mol K^2 for a $La_{1.3}Sr_{1.7}Mn_2O_7$ ($x = 0.35$) sample, obtained from a linear extrapolation to $T = 0$ K, in the low T region ($\leq$3 K) of the C/T data, for $H = 9$ T parallel to the ab planes. Using that same criterion we found a similar value of $\beta_1 \approx 4$ mJ/mol K^2 for the $La_{1.35}Sr_{1.65}Mn_2O_7$ sample. However, since we are interpreting the strongly curved region of C/T (<5 K) as being caused by a gap opening in the spin-wave energy spectrum, it is more reliable to consider $\beta_1 \approx 9.42$ mJ/mol K^2 (see Table 1) evaluated by the fitting of Eq. (1) to the data (5 K $\leq T \leq$ 10 K). This latter β_1 value is at least 2 times larger than the values of 2–5 mJ/mol K^2 observed for the 3D perovskite family of $La_{1-x}Sr_xMnO_3$ ($0.16 \leq x \leq 0.40$) [15]. Therefore, our result suggests that the quasi-2D confinement of the electrons in $La_{1.35}Sr_{1.65}Mn_2O_7$ might be causing an increase of the electron–electron constant β_1, in comparison with a 3D counterpart. It is worth noting that a value of $\beta_1 \approx 7.0$ mJ/mol K^2, closer to our result, was also found [16] for a $La_{1.36}Sr_{1.64}Mn_2O_7$ crystal, measured under an applied magnetic field of 9 T parallel to the c axis.

From the β_3 values we estimated the Debye temperature ($T_D \propto \beta_3^{-1/3}$), above which the specific heat approaches its classical value [17]. T_D decreased from 434 ± 9 K at zero applied magnetic field to 416 ± 5 K at $H = 9$ T, for $La_{1.35}Sr_{1.65}Mn_2O_7$. The errors involved in the evaluation of T_D were about 2% for $H = 0$, and about 1% for $H = 9$ T. Then, with reasonable confidence we concluded that a weak magnetoelastic (or magnon–phonon) coupling is possibly producing a small softening of the lattice vibrations, when H goes from 0 to 9 T along the c axis direction. This agrees with the anomalous reduction in the ultrasonic velocity along the c axis, observed at $T = 10$ K in $La_{1.2}Sr_{1.8}Mn_2O_7$, when $H \parallel c$ is increased from zero to values above 5 T [18]. Consistently, a small reduction of thermal conductivity along the c axis (for $H \parallel c$) has been observed [19] at a fixed low temperature, for the same compound. On the other hand, for the antiferromagnetic compound $La_{1.5}Sr_{0.5}NiO_4$ a constant value of $T_D = 382 \pm 5$ K was found for both $H = 0$ and $H = 9$ T, which indicates a lack of magnetoelastic coupling.

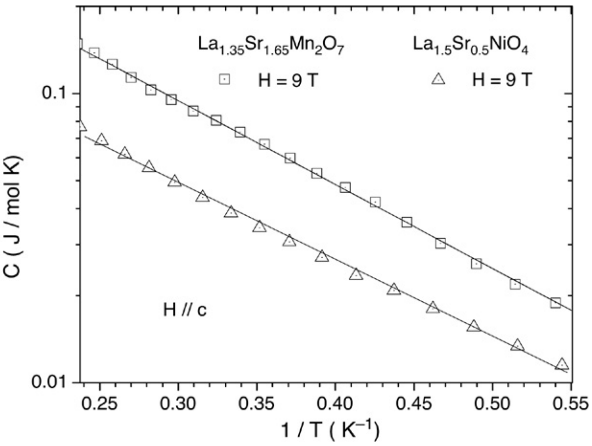

Fig. 2. Re-scaled specific heat data in the low temperature interval (below 4 K) and $H = 9$ T, for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals. The x axis displays $1/T$ on a linear scale and the y axis displays C on a logarithmic scale. The solid lines represent fits to the exponential decay law $C = A\exp(-E_{\mathrm{gap}}/kT)$.

As expected, a term in the specific heat depending on $T^{3/2}$ (Eq. (1)) appears only for the $La_{1.35}Sr_{1.65}Mn_2O_7$ sample, due to its ferromagnetic interactions. For the antiferromagnetic $La_{1.5}Sr_{0.5}NiO_4$ we found $\beta_{3/2} \approx 0$ (Table 1), within the experimental errors, for both $H = 0$ and $H = 9$ T. A study [20] of the specific heat in $La_{1-x}Sr_xMnO_3$, with x between 0.1 and 0.3, has found $\beta_{3/2}$ values in the interval 0.9 to 3.7 mJ/mol K$^{5/2}$. Our $\beta_{3/2}$ values (Table 1) for $La_{1.35}Sr_{1.65}Mn_2O_7$ are approximately equal to these values reported for the 3D counterpart and show an increase when the sample is under a 9 T magnetic field.

The authors of Ref. [16] interpreted the specific heat of $La_{2-2x}Sr_{1+2x}Mn_2O_7$ samples ($0.30 < x < 0.50$) as caused by thermal excitations from a two-dimensional gas of ferromagnetic magnons. They fitted their curves assuming the opening of a gap induced by the magnetic field. However, they did not discuss the very low temperature interval (below 4 K). In order to test the hypothesis of a gap opening in our crystals, Fig. 2 presents the re-scaled specific heat data in the temperature interval below 4 K, for the $H = 9$ T data. The x axis is now equal to the inverse of temperature and the y axis is presented with a logarithmic scale to facilitate the comparison with an exponential decay law:

$$C = A\exp(-E_{\mathrm{gap}}/kT) \qquad (2)$$

where A is a constant determined when $1/T$ is extrapolated to zero, and k is the Boltzmann constant. For both samples the data points are reasonably well fitted by straight lines. The estimated energy gaps (E_{gap}) were 0.57 meV and 0.65 meV for $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$, respectively. These E_{gap} values are close to the expected gap value induced by the magnetic field [16], $\Delta = 2\mu_B H \approx 1$ meV, where μ_B is the Bohr magneton and $H = 9$ T.

The occurrence of an energy gap for a crystal of $La_{1.5}Sr_{0.5}NiO_4$ has also been suggested [21] in a study that employed polarized neutrons at 10 K. The neutron energy scans were made with the incident beam fixed in a direction

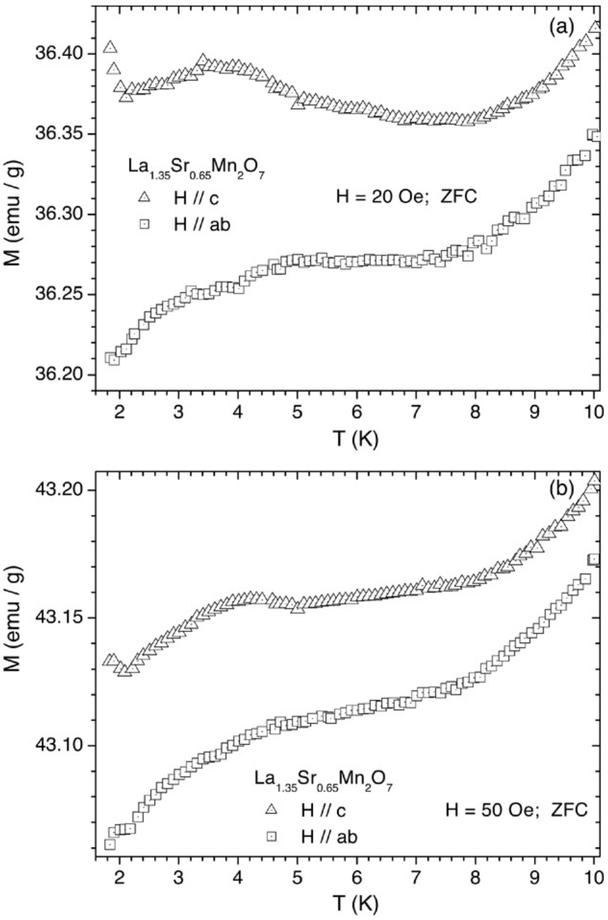

Fig. 3. Magnetization measurements in the $La_{1.35}Sr_{1.65}Mn_2O_7$ crystal, between 1.8 and 10 K, with applied magnetic fields of 20 Oe (a) and 50 Oe (b). Measurements were done with the magnetic field parallel (triangles) and perpendicular (squares) to the c axis. The results reveal an anisotropy attributable to the quasi-bidimensional distribution of the magnetic ions.

parallel, as well as perpendicular, to the NiO layers. Interplane correlations were observed at low energies (≤ 5 meV), in concert with a reduction in the scattering intensity below an energy of 4 meV, for the neutrons polarized along the c axis. Given that neutrons scatter from spin fluctuations perpendicular to the wavevector, these observations indicated that the intensity reduction below 4 meV was due to the freezing out of the c axis component of the spin fluctuations, that could be associated with an energy gap due to a single-ion out-of-plane anisotropy. We found in the $La_{1.5}Sr_{0.5}NiO_4$ crystal a gap value around 4 meV for $H \parallel c$, which is smaller than although of the same order of magnitude as that reported in Ref. [21]. Both results, one probed microscopically and the other macroscopically, seem to confirm the complex magnetic excitation and band structure associated with charge ordering and the quasi-2D confinement of the Ni ions.

Seeking for a better understanding of the magnetic behavior at low temperatures, several magnetization curves ($M \times T$) were measured for all samples. Fig. 3 shows a detailed measurement of the zero-field cooling (ZFC) magnetization in the $La_{1.35}Sr_{1.65}Mn_2O_7$ crystal, between 1.8 and 10 K, with applied magnetic field of 20 Oe (a) and 50 Oe (b). Measurements were done with the magnetic field parallel (triangles) and perpendicular (squares) to the c axis and temperature steps of 0.1 K. The results reveal a clear anisotropy due to the quasi-bidimensional distribution of the magnetic ions. The magnetization for $H \parallel c$ shows a minimum close to 2 K, followed by a steep increase down to the lowest available $T = 1.8$ K. Fig. 4 shows ZFC magnetization measurements in the $La_{1.5}Sr_{0.5}NiO_4$ crystal, between 1.8 and 10 K, with applied magnetic fields of 1 T (a) and 3 T (b). The magnetic field was applied parallel (triangles) and perpendicular (squares) to the c axis and the temperature steps were of 0.1 K. These graphs also display an anisotropy behavior due to the quasi-bidimensional distribution of the magnetic ions. The magnetization here shows a maximum close to 2 K, for the applied magnetic field oriented along the c axis. The absolute value of magnetization is higher in $La_{1.35}Sr_{1.65}Mn_2O_7$, due to its ferromagnetic order, in comparison with the antiferromagnetic order in $La_{1.5}Sr_{0.5}NiO_4$. In both cases, the occurrence of peaks close to 2 K in the magnetization curves for $H \parallel c$ (Figs. 3 and 4) seems to be correlated with the exponential decay in the specific heat curves (Fig. 2). The monotonic decrease (increase) of magnetization for the ferromagnetic (antiferromagnetic) $La_{1.35}Sr_{1.65}Mn_2O_7$($La_{1.5}Sr_{0.5}NiO_4$), in the temperature interval going from 4 K to 2 K, could be

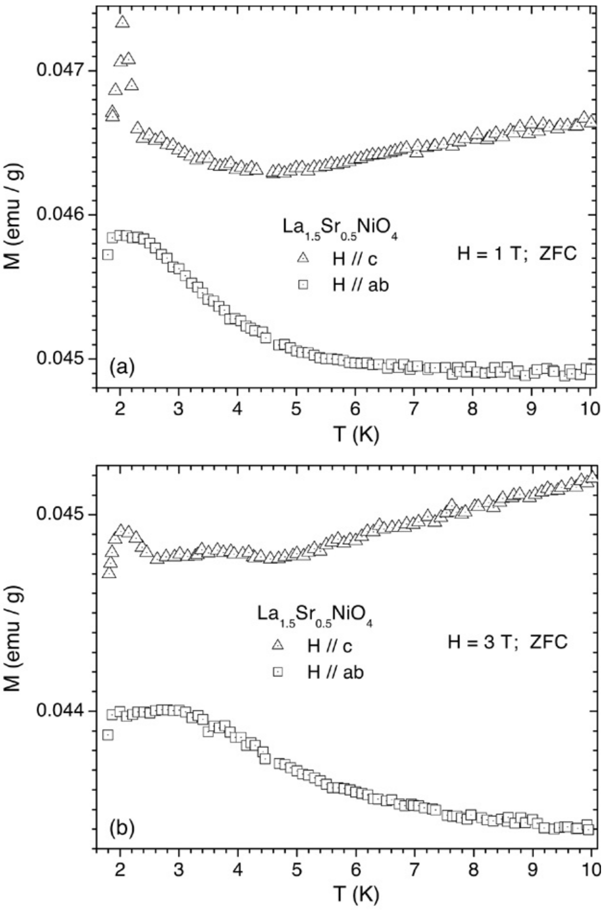

Fig. 4. Magnetization measurements on the $La_{1.5}Sr_{0.5}NiO_4$ crystal, between 1.8 and 10 K, with applied magnetic fields of 1 T (a) and 3 T (b). The magnetic field was applied parallel (triangles) and perpendicular (squares) to the c axis.

an indication for a continuous second-order phase transition associated with a gap opening in the magnon spectrum. However, our present data cannot prove conclusively these hypotheses, so additional studies are clearly needed to better elucidate the problem.

4. Conclusions

Single crystals of $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ were characterized by means of magnetization and specific heat measurements. At low temperatures the bilayer compound $La_{1.35}Sr_{1.65}Mn_2O_7$ presented a ferromagnetic order, with magnetization values around 43 emu/g (Fig. 3), while $La_{1.5}Sr_{0.5}NiO_4$ showed an antiferromagnetic order, with magnetization values around 46×10^{-3}emu/g (Fig. 4).

The specific heat in $La_{1.35}Sr_{1.65}Mn_2O_7$ clearly decreased when a magnetic field $H = 9$ T was applied parallel to the crystal c axis (Fig. 1). We interpreted this behavior as being caused by a suppression of spin-wave excitations (magnons) due to the reduced magnetic dimensionality of the bilayer structure. This effect was larger than in the case of a 3D counterpart (e.g. $La_{1-x}Sr_xMnO_3$ [15]), although not as large as expected for an ideal 2D system [8] (see the inset of Fig. 1). The fitting of our specific heat data also indicated that the quasi-2D confinement of the electrons in $La_{1.35}Sr_{1.65}Mn_2O_7$ might be causing an increase of the electron–electron constant ($\beta_1 \approx 9.42$ mJ/mol K^2), in comparison with its 3D counterpart ($\beta_1 \approx 3$ mJ/mol K^2). On the other hand, the specific heat did not change with application of a 9 T magnetic field in the $La_{1.5}Sr_{0.5}NiO_4$ crystal. However, the electronic excitations were drastically suppressed at very low temperatures, as revealed by a downward turn in the specific heat curve (Fig. 1).

Below 4 K we found that the specific heat data, for the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ crystals, could be fitted by an exponential decay law (Fig. 2). This allowed us to estimate characteristic energy gaps around 1 meV for both compounds, in good agreement with an expected gap $\Delta = 2\mu_B H \approx 1$ meV in the magnon spectrum [16]. In particular, our gap value of 0.65 meV for $La_{1.5}Sr_{0.5}NiO_4$ is of the same order of magnitude as the out-of-plane anisotropy gap found through polarized neutron experiments [21].

Detailed magnetization measurements, in the low temperature region below 4 K, showed a monotonic decrease (increase) for the ferromagnetic (antiferromagnetic) $La_{1.35}Sr_{1.65}Mn_2O_7$ ($La_{1.5}Sr_{0.5}NiO_4$), with a steep increase around 2 K. These features, occurring in the same temperature interval as the exponential specific heat behavior, might also be correlated with a possible gap opening in the magnon spectrum. In conclusion, our magnetization and specific heat data suggest the existence of an anisotropy gap in the energy spectra of the $La_{1.35}Sr_{1.65}Mn_2O_7$ and $La_{1.5}Sr_{0.5}NiO_4$ compounds. However, further studies are desirable to confirm and extend our present results.

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