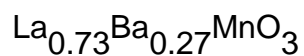


Anomalous temperature dependence of the spontaneous magnetization of single-crystal



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Anomalous temperature dependence of the spontaneous magnetization of single-crystal $\text{La}_{0.73}\text{Ba}_{0.27}\text{MnO}_3$

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Abstract

Detailed measurements of the spontaneous magnetization of single-crystal $\text{La}_{0.73}\text{Ba}_{0.27}\text{MnO}_3$ are presented; these show a marked reduction in this property below 60 K which is consistent with a clearly discernible moment reduction, one of the few definitive conclusions about such effects provided by bulk measurements.

1. Introduction

Over the past decade substantial effort has been directed towards understanding the properties of transition metal oxides—the cuprates and manganites in particular, and, to a lesser extent, the nickelates and cobaltites. In the case of the manganites, the (re)discovery of colossal magnetoresistance (CMR) was the principal focus of much of this renewed interest; this phenomenon results from a metal–insulator transition, the temperature of which displays a marked field dependence as a consequence of concomitant magnetic ordering [1]. While there is currently general agreement that CMR is linked to a strong coupling between the electronic subsystem and the lattice, there are still differing views as regards what is the basic mechanism underlying this effect, despite intensive experimental and theoretical effort—these include percolation in a spontaneous electronic/magnetic phase separation scenario [2] and bipolaron pair breaking [3], amongst others.

In contrast to the many investigations of CMR and the magnetically correlated metal–insulator transition, the low temperature properties of the manganites have received rather little attention.

In keeping with this focus of attention near the metal–insulator/magnetic ordering temperature in doped manganites, detailed magnetization measurements were reported recently on single-crystal $\text{La}_{0.73}\text{Ba}_{0.27}\text{MnO}_3$, the analysis of which yielded [4] a continuous (second-order) paramagnetic to ferromagnetic transition at $T_C = 245$ K characterized by Heisenberg

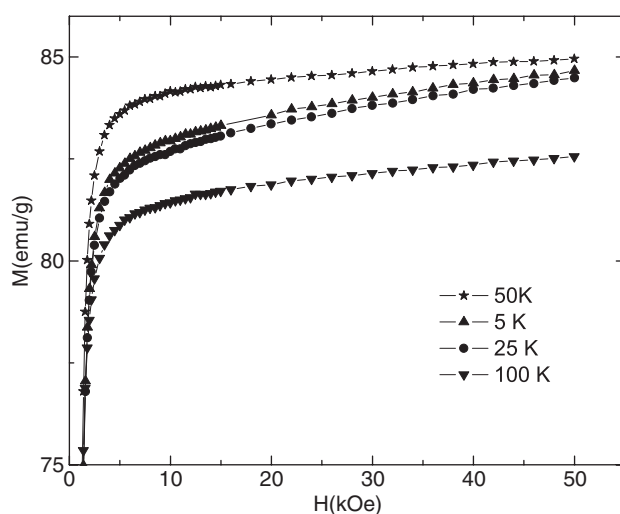


Figure 1. Magnetization isotherms at selected temperatures below 100 K; these are at 50 K (★), 5 K (▲), 25 K (●) and 100 K (▼, bottom).

model exponents [5]. While it appears that the manganites are particularly sensitive to the effects of disorder, specifically the influence of the latter in driving first-order/discontinuous transitions towards second-order/continuous behaviour [2, 6], this does not appear to be the case for this Ba-doped single crystal. Both the critical exponents and amplitudes were consistent with a continuous magnetic phase change, a conclusion supported indirectly by the moderate magnetoresistance reported [7] for this system (as opposed to truly colossal MR in systems exhibiting a lower T_C and a first-order/discontinuous phase change [1, 8]). Below, we report an extension of the study of this single crystal from which the temperature dependence of the spontaneous magnetization is established throughout the ferromagnetic regime. This reveals clear evidence for a marked spontaneous moment reduction below 60 K, a feature previously unreported for this system.

2. Experimental details

The sample in which these effects were observed was that used in the earlier investigation [4] of critical behaviour—a semi-cylindrical rod of length 6 mm and average radius 1.6 mm, weighing 0.163 g. This specimen—grown at the Moscow State Steel and Alloys Institute using a floating zone technique—was of high structural and magnetic quality, with mosaicity of less than 1° and a coercive field of less than 5 Oe (falling to less than 1 Oe above 235 K). A Quantum Design Model 6000 PPMS utilizing five-scan averaging was used to collect magnetization data between 2 and 250 K in applied fields up to 50 kOe (applied along the sample's cylindrical axis), following a previously described protocol for ensuring equilibrium measuring conditions [4]; ac susceptibility data, $\chi_{ac}(H, T)$, measured at 2.4 kHz in a driving field of 0.1 Oe rms applied parallel to the superimposed static/dc biasing field were also collected.

3. Results and discussion

Figure 1 reproduces a selection of magnetization isotherms below 100 K which provide the first indications of anomalous behaviour in this system. From this figure it is clear that

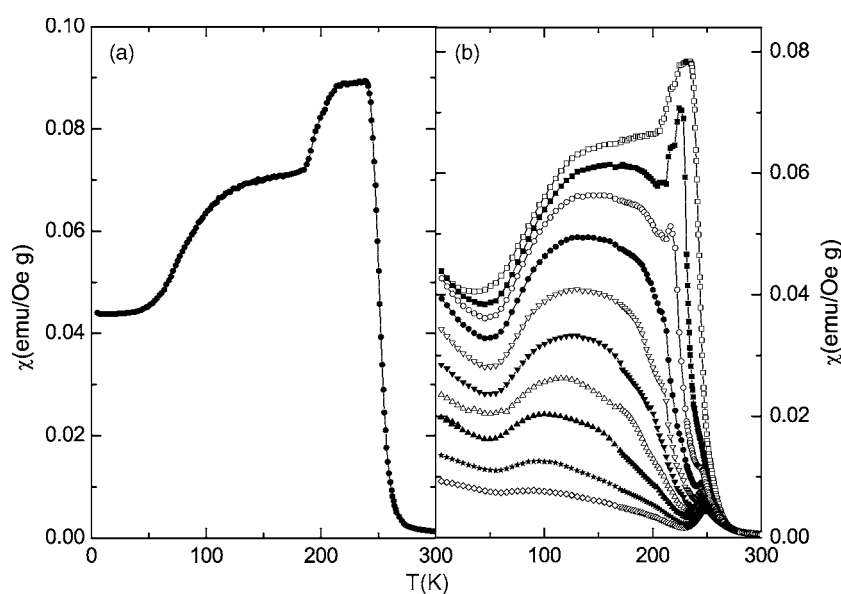


Figure 2. (a) The zero-field ac susceptibility measured on warming following cooling at zero (dc) field. (b) The ac susceptibility measured on warming following cooling at zero (dc) field in progressively increasing static (dc) applied fields of 200 Oe (\square , top), 400 Oe (\blacksquare), 500 Oe (\circ), 600 Oe (\bullet), 700 Oe (∇), 800 Oe (\blacktriangledown), 900 Oe (\triangle), 1000 Oe (\blacktriangle), 1200 Oe (\star) and 1400 Oe (\diamond , bottom).

the magnetization in quite moderate fields (2–5 kOe) increases substantially with decreasing temperature between 100 and 50 K, as expected in a ferromagnetic system. However, *below* 50 K the magnetization in the same field range—a qualitative measure of the spontaneous magnetization—falls. This effect is accompanied by an increasing high field (>5 kOe) slope in isotherms acquired below 50 K, and this enhanced low temperature, high field slope is the reason that the saturation magnetization, extracted from M versus H^{-1} plots (at $H^{-1} \rightarrow 0$), is consistent with the full $\text{Mn}^{3+}/\text{Mn}^{4+}$ moment, as reported in [4].

The behaviour summarized in figure 1, it should be emphasized, is gradual rather than abrupt. Thus there is no evidence of a metamagnetic transition in the data reported here, unlike the situation for $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ at comparable doping levels [9].

Prior to providing quantitative estimates for the spontaneous magnetization which support the assertion made above, it is interesting to review the ac susceptibility data, $\chi_{\text{ac}}(H, T)$, taken for the same specimen; these provide supplementary evidence corroborating the anomalous behaviour evident in figure 1. This is done in figures 2(a) and (b); figure 2(a) shows that in zero static biasing field, $\chi_{\text{ac}}(0, T)$ peaks at a demagnetizing limited value³ just below T_C before falling with decreasing temperature below 200 K. This feature has been comprehensively linked to a structural phase change occurring near this temperature in $\text{La}_{1-x}\text{Ba}_x\text{MnO}_3$ for $0.2 \leq x \leq 0.33$ [10, 11], a structural phase change known to result in a moment reduction, an effect discussed in more detail below. The specific, additional feature in figure 2(a) that needs to be pointed out is the subsequent decline in $\chi_{\text{ac}}(0, T)$ around 100 K before it reaches a plateau below 50 K. The latter correlates with the onset of the anomalous behaviour of the magnetization isotherms, and it, along with the behaviour of $\chi_{\text{ac}}(H, T)$ in finite static biasing

³ As discussed in [4], two experimental estimates were made for N , the average of which would yield a maximum susceptibility of $0.09(1) \text{ emu g Oe}^{-1}$, very close to that achieved in figure 2(a).

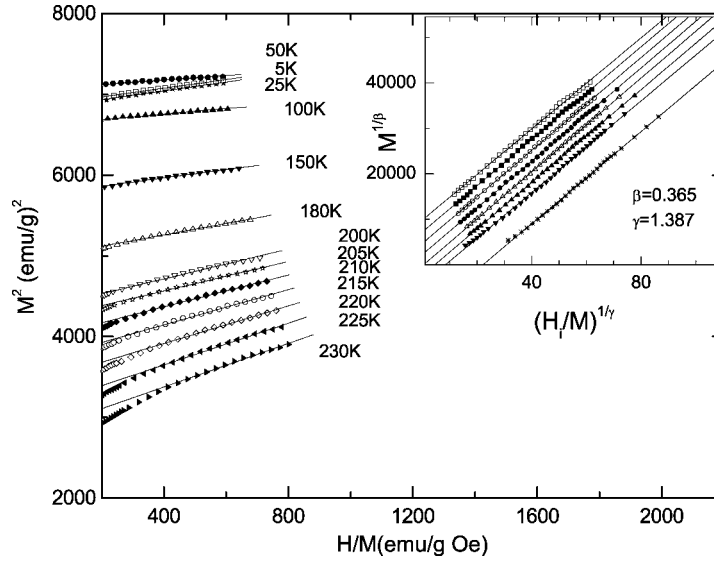


Figure 3. Arrott–Noakes plots (M^2 versus H/M ; equation (1) with mean-field exponents) at temperatures of, sequentially, 50 K (●, top), 5 K (□), 25 K (★), 100 K (▲), 150 K (▼), 180 K (△), 200 K (▽), 205 K (☆), 210 K (◆) and 215 K (○), 220 K (◇), 225 K (◄) and 230 K (►, bottom). Inset: data close to the Curie temperature plotted using the same equation, but with Heisenberg model exponents, at temperatures of, sequentially, 241 K (□, top), 242 K (■), 243 K (○), 244 K (●), 245 K (◇), 246 K (▲), 247 K (▼), 250 K (★, bottom).

fields, figure 2(b), where the appearance of a local minimum near 50 K is evident, provides important ancillary information on the origin of this effect, also discussed below.

Quantitative estimates for the spontaneous magnetization were made using conventional extrapolations from the technically saturated regime on the basis of an Arrott–Noakes equation of state [12]:

$$\left(\frac{H}{M}\right)^{1/\gamma} = \left(\frac{T - T_C}{T_C}\right) + \left(\frac{M}{M_1}\right)^{1/\beta}, \quad (1)$$

M_1 being a material specific constant and H the internal field ($=H_a - NM$ in the usual notation, with N estimated previously [4]). Close to the critical temperature ($241 < T < T_C$) such an equation produces the necessary linearized plots using Heisenberg model values [4, 5] for the critical exponents ($\gamma = 1.386$, $\beta = 0.364$), as the inset in figure 3 confirms, whereas the main body of this figure indicates that mean-field exponents are applicable outside the critical region.

Figures 4(a) and (b) reproduces the temperature dependence of the spontaneous magnetization, $M_S(T)$ —the intercepts in figure 3—so estimated. To emphasize the principal result of this study this plot utilizes the reduced spontaneous magnetization $M_S(T)/M_S(0)$ with $M_S(0) = Ng\mu_B S$ ($\cong 85.36 \text{ emu g}^{-1}$) being the saturation magnetization of a system of N magnetic sites carrying spin S (per unit volume, or mass, as appropriate). The above numerical value for $M_S(0)$ was found from an M versus H^{-1} plot at 2 K and confirmed by calculation using the listed composition [4]. This estimate for $M_S(0)$ is discussed in more detail below. From figure 4(a) the onset of an anomalous decrease below 60 K is clearly visible, providing quantitative evidence of the effects alluded to indirectly from the data in figures 1 and 2. The size of these data points represents the uncertainty in the extrapolated estimate for the spontaneous magnetization in the temperature regime below 150 K. These uncertainties become larger at

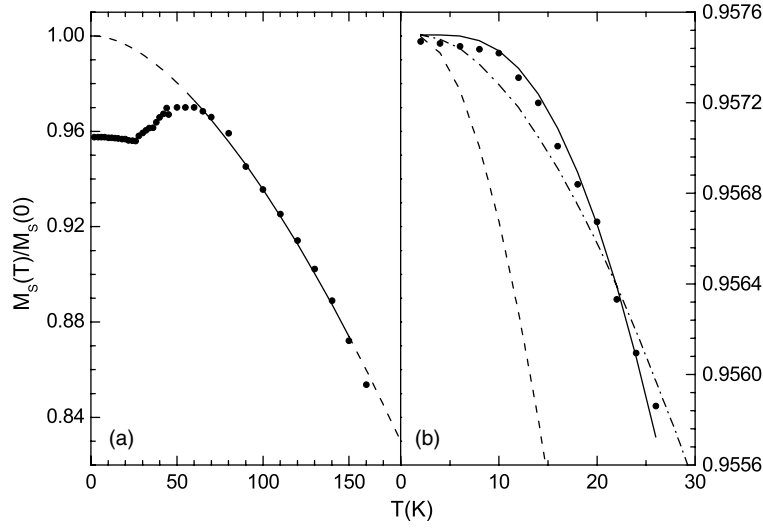


Figure 4. (a) Plots of the reduced spontaneous magnetization ($M_S(T)/M_S(0)$) deduced from the intercepts in figure 3 plotted against temperature. The solid line represents a fit to equation (3) between 60 and 140 K using $D = 65.7 \text{ meV } \text{\AA}^2$ and $\Delta = 0.45 \text{ meV}$; the dashed line extends this fit below 60 K. (b) Spontaneous magnetization data below 30 K; the dashed line uses the D and Δ values utilized in (a) scaled to $M_S(T)/M_S(0) = 0.957(5)$; the solid line utilizes the same value for D (c) as in (a) but with Δ increased to 2.35 meV (as discussed in the text), while the dot-dashed line employs $\Delta = 0.45 \text{ meV}$ as in (a) but with D increased to $159 \text{ meV } \text{\AA}^2$ (also discussed in the text). All fits employ the same scaled ratio of $M_S(T \rightarrow 0)/M_S(0)$.

higher temperatures, specifically in the temperature regime where there is a crossover from mean-field to Heisenberg model exponents, as evidenced by the increasing curvature of the associated isotherms shown in the main body of figure 3. It should be emphasized that these higher temperature data points play no role in the present discussion.

Thus, while the origin of the behaviour displayed in the magnetization isotherms of figure 1 can be identified directly, the underlying physical mechanism cannot. Certainly spin canting in the manganites has been long debated and is a controversial topic [13], although probably not within the framework originally proposed by de Gennes [14]. Nevertheless spin canting and the formation of a spiral spin structure appear as *possible* causes of the results presented above. Indeed data similar to those reported here for systems containing two magnetic species (for example [15, 16]) have been interpreted as indicating spin canting, although probably in the second magnetic species (Nd, for example [15]); the present measurements differ significantly in that they involve Mn ions alone. The difficulty surrounding such bulk measurements however is that they cannot reveal directly microscopic/atomic spin arrangements. This deficiency notwithstanding, we next discuss the model fits reproduced in figures 4(a) and (b).

These lines represent calculations of the spontaneous magnetization based on the premise that the spontaneous magnetization below $T_C/2$ results predominantly from acoustic mode spin-wave excitations described by a gapped dispersion relation, namely

$$\hbar\omega_{ac} = \Delta + Dq^2 \quad (2)$$

leading to a spontaneous magnetization [17]

$$\frac{M_S(T)}{M_S(0)} = 1 - \frac{1}{NS} \left(\frac{k_B T}{4\pi D} \right)^{3/2} \cdot \xi \left(\frac{3}{2}, \frac{\Delta}{k_B T} \right) \quad (3)$$

where ξ is the modified Riemann zeta function.

The solid line in figure 4(a)—a least-squares fit to the data between 60 and 140 K ($\approx 0.55 T_C$)—yields $\Delta = 0.45 \pm 0.02$ meV (5.2 ± 0.2 K) and $D = 65.7 \pm 2.5$ meV \AA^2 ; this estimate for the gap Δ , although small, is approximately twice that found from the analysis of earlier neutron scattering data [18] on a single crystal of slightly higher Ba composition, $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$, a point returned to below; the estimate for D is correspondingly smaller. Such compositional variations are consistent with those reported [19] for the $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ system ($D \approx 46$ meV \AA^2 at $x = 0.2$, rising to 170 meV \AA^2 at $x = 0.33$), while a gap of the size reported above is consistent with the analysis of heat capacity data [20] for $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$. Assuming a simple (cubic) Heisenberg/ferromagnetic nearest-neighbour coupling model yields $2 J_1 S \approx 4.3$ meV, which scales directly with the acoustic spin-wave stiffness estimates compared with the corresponding estimates for $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ [18] and $\text{La}_{0.7}\text{Pb}_{0.3}\text{MnO}_3$ [21]. In turn, the mean-field estimate for T_C for the present sample is 285 K (some 16% higher than the measured value, a similar enhancement to that reported [21] between predicted and measured values for $\text{La}_{0.7}\text{Pb}_{0.3}\text{MnO}_3$).

Above 140 K the curve calculated using the above parameters deviates from the data in a manner consistent with a downward renormalization of D as the temperature increases towards T_C [19].

The principal result of the present study is, however, reiterated by the fits to the data below 30 K shown by the lines drawn in figure 4(b). The spontaneous magnetization displays a clearly identifiable drop from its expected value ($M_S(0) = Ng\mu_B S$) in this low temperature regime to $M_S = 0.9575 (Ng\mu_B S) = 81.7(3)$ emu g^{-1} . The dashed line in figure 4(b) uses the parameters deduced from the higher temperature fit (together with a scaled $M_S(T)/M_S(0) = 0.9575$) and results in a much stronger temperature dependence in $M_S(T)$ below 30 K than is observed. The limited range of these low temperature data combined with their weak temperature dependence precludes precise independent estimates for D and Δ ; two limiting fitting schemes have thus been adopted. In the first (the solid line in figure 4(b)), D has been maintained at its higher temperature value and Δ varied to produce the fit, yielding $\Delta = 2.35$ meV. The second maintains Δ at its higher temperature and varies D ; the dot-dashed line in figure 4(b) utilizes $D = 159$ meV \AA^2 . Irrespective of whether one or all of these fitting schemes is judged to be invalid on a physical basis, the reduced estimate for $M_S(0)$ remains unchanged; it would certainly result from the adoption of alternative extrapolation procedures. However, the current consensus of work on the doped manganites would question gaps on the order of 5 K, let alone the considerably larger value of some 25 K yielded by the better fit to the low temperature data, despite the obvious quality of fit that these parameters yield over the appropriate temperature interval. Thus while the physical origin of the underlying magnetization processes may be questionable, the estimate for $M_S(0) = 0.96(Ng\mu_B S)$ is not. That this signifies an effective moment reduction is unequivocal; the specific mechanism leading to this moment reduction however cannot be identified definitively.

The data presented above are clearly consistent with spin canting, and would yield a (uniform) canting angle θ close to 17° ($\theta = \cos^{-1}(0.958)$). These data are, of course, also consistent with a spiral magnetic structure forming with an axis parallel to the field direction—the long axis of this single crystal—with a similar disinclination angle; measurements of the present type cannot differentiate between these alternatives. Furthermore, given the concerns raised regarding the Δ values estimated from various fits, a discussion of other causes for the observed reduction in $M_S(0)$ is appropriate. As mentioned earlier, structural changes are known to result in a moment reduction in $\text{La}_{1-x}\text{Ba}_x\text{MnO}_3$ between $x = 0.2$ [10] and $x = 0.33$ [11], a range that includes the current composition. This structural change occurs between a higher temperature, higher moment $R3c$ phase and its lower temperature $Pbnm/Imma$ counterpart. Within the composition range $0.2 \leq x \leq 0.33$ this structural change, very clearly revealed by

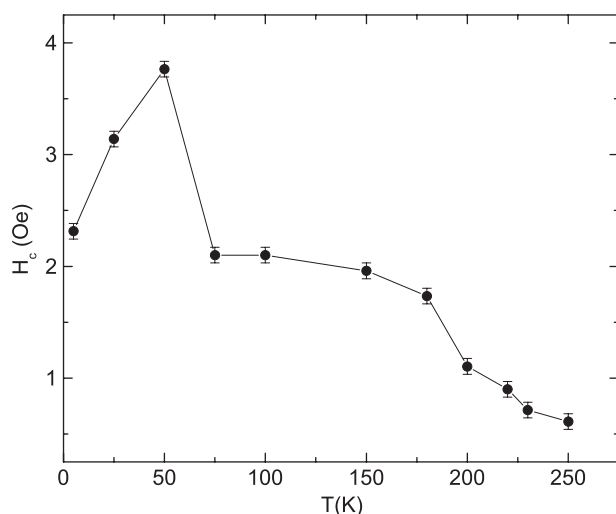


Figure 5. The measured coercive field, $H_C(T)$, plotted against temperature.

neutron data [11], is known to occur near 200 K, and has been linked [10] to anomalies in the resistivity along with a decline in the ac susceptibility, $\chi_{ac}(0, T)$ near the same temperature; this latter feature is clearly evident in the data provided on the present sample in figure 2(a). The present ac susceptibility data contrast markedly with those reported on a variety of other ferromagnets where the so-called Hopkinson maximum appearing just below T_C —and not usually demagnetization factor limited—arises from technical magnetic sources (domain wall motion, coherent domain rotation etc) [22]. The question to be answered is whether the behaviour of χ_{ac} —and of $M_S(T)$ —at low temperatures has a structural or technical origin.

In terms of the latter, the Hopkinson maximum arises when the coercive field, $H_C(T)$, a direct reflection of technical magnetic behaviour, exceeds the ac driving field as $H_C(T)$ increases with temperature below T_C [22]. The ac susceptibility measures the slope of a minor loop averaged over the driving field amplitude; generally this slope exhibits a roughly inverse dependence on $H_C(T)$. The structural phase change notwithstanding, this conclusion that $\chi_{ac}(0, T)$ is not dominated by technical processes near 200 K—and by extension, at and below 100 K—is confirmed by three factors: the temperature dependence of $H_C(T)$, the temperature dependence of the zero-field-cooled (ZFC) magnetization and the (lack of a) frequency dependence in χ_{ac} below 60 K. First, the measured temperature dependence of $H_C(T)$ for the present sample is shown in figure 5. Should the behaviour of $\chi_{ac}(0, T)$ be controlled by technical process, it would correlate approximately inversely with this field. Not only does $H_C(T)$ exceed the ac driving field of 0.1 Oe by close to an order of magnitude essentially *everywhere* below T_C , but, more importantly, it would result in $\chi_{ac}(0, T)$ in the liquid He range climbing to a value close to that measured near 150 K. It does not (figure 2(a)). Second, the ZFC magnetization acquired in fields between 30 and 100 Oe (figure 6), i.e. fields far in excess of $H_C(T)$, still displays the same two-step decrease evident in $\chi_{ac}(0, T)$, as indeed does $\chi_{ac}(H, T)$ (figure 4(b)). The field induced suppression evident in the latter, particularly below 60 K (the regime where $M_S(T)$ is reduced), is often identified with domain wall pinning [22], which leads to the third factor. The ac susceptibility data on a single crystal of slightly lower composition ($x = 0.2$) [10], while also displaying a double-step structure below T_C (only the higher of which, near 200 K, was subject to discussion), exhibited no frequency dependence ($33 \leq f \leq 10$ kHz) in the response below 50 K, contrary to what would result from pinning effects.

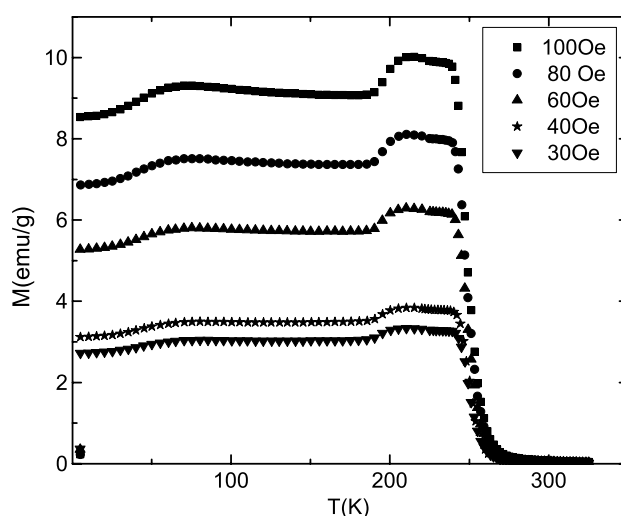


Figure 6. The magnetization, $M(H, T)$, measured on warming following zero-field cooling in static applied fields of, sequentially, 100 Oe (■, top), 80 Oe (●), 60 Oe (▲), 40 Oe (★), 30 Oe (▼, bottom).

The above arguments support the assertion that the decline in $\chi_{ac}(0, T)$ below 100 K—which correlates with the fall in $M_S(T)$ at low temperatures—is intrinsic, not technical, in origin.

Finally there is the question of whether a second structural phase change in the low temperature (60–100 K) regime causes the reduction in the magnetic response. In addition to the data referred to above indicating a structural phase change near 200 K [10, 11], recent measurements of the thermal expansion ($\Delta\ell/\ell$) above 77 K for single-crystal $\text{La}_{1-x}\text{Ba}_x\text{MnO}_3$ for $x = 0.2$ [23] and $x = 0.3$ [24] provide interesting comparisons. At the lower composition there is a clear anomaly in $\Delta\ell/\ell$ near 175 K, identified with the $R3c$ to $Pbnm/Imma$ structural phase change; by contrast, a barely discernible signature of this phase change can be resolved from this measurement at $x = 0.3$. These are, of course, bulk measurements and as such cannot provide microscopic details directly. More importantly, the composition of the present sample is closer to that of the higher composition specimen mentioned above. Thus, given the fact that neutron data [11] delineate the appearance of a structural phase change far more clearly than thermal expansion results near the composition of interest here, the result that the former measurement yields no indication of an additional structural phase change down to 5 K provides strong evidence that the $Pbnm/Imma$ remains stable throughout the low temperature regime ($5 \leq T \leq 200$ K). Additional structural studies—such as temperature dependent x-ray measurements—would clearly be of considerable help in this regard; such facilities are not currently available to us.

4. Conclusions

In summary, estimates of the spontaneous magnetization—supplemented by ac susceptibility data—indicate a (spontaneous) moment reduction in $\text{La}_{0.73}\text{Ba}_{0.27}\text{MnO}_3$ below 60 K. Evidence is summarized that supports the assertion that this reduction is not associated with a (further) structural phase change nor technical magnetic processes. Furthermore, the inhomogeneous ordering of the type reported in the $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$ system for $x \leq 0.2$ [25], which might initially appear to provide a potential explanation of a moment reduction (possibly

analogous to the mechanism proposed for AuFe [26]), is also an unlikely source on further inspection. The insulating character prevalent at lower doping levels plays an important role in establishing such order; this explanation thus seems improbable in the low temperature *metallic* phase [7] of the present system. These arguments notwithstanding, the bulk nature of the present measurements precludes an identification of the microscopic mechanism underlying this effect. While the low temperature spontaneous magnetization is well fitted by the predictions of a gapped acoustic spin-wave dispersion relation, the associated gap estimates are considerably larger than anticipated from the current consensus of other data. However, survey neutron scattering data [27] on single-crystal $\text{La}_{0.8}\text{Ba}_{0.2}\text{MnO}_3$ —with admittedly few data points in the temperature region of interest here—indicate that spin-wave stiffness estimates based on equation (2) with $\Delta = 0$ do not approach zero temperature monotonically. Coupled with the present data, this might suggest that additional experiments investigating the microscopic/atomic spin arrangement in $\text{La}_{1-x}\text{Ba}_x\text{MnO}_3$ ($0.2 \leq x \leq 0.3$) at low temperature might be appropriate. No comparable detailed studies of canonical double-exchange systems such as $\text{La}_{1-x}\text{A}_x\text{MnO}_3$, $\text{A} = \text{Ca}, \text{Sr}$ containing a single magnetic species have been carried out to date. (As noted earlier, spin canting has been raised in the context of systems incorporating *two* sets of magnetic atoms [15].) However, survey estimates of the *saturation* magnetization in polycrystalline $\text{La}_{0.7}\text{Sr}_{0.3}\text{CoO}_3$ [28] indicate the possibility of a slight decrease in this parameter below about 15 K. The present data thus provide the clearest indication of such effects currently.

Acknowledgment

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