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Orbital order and a canted phase in the paramagnetic and ferromagnetic states of 50% hole-doped colossal magnetoresistance manganites

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We report neutron scattering results on the spin dynamics in the paramagnetic (PM) and ferromagnetic (FM) states of the 50% hole-doped manganites $Pr_{1/2}Sr_{1/2}MnO_3$ and $Nd_{1/2}Sr_{1/2}MnO_3$. In the PM phase, these systems exhibit two kinds of diffuse scatterings: an isotropic quasielastic diffuse scattering and a dynamical ridge-type, i.e., two-dimensional FM diffuse scattering. With decreasing temperature, both systems enter the pure FM metallic state, but the spin dynamics in the FM state possess a strong *A*-type antiferromagnetic feature. In fact, $Nd_{1/2}Sr_{1/2}MnO_3$ enters a canted antiferromagnetic phase at lower temperature through a second order phase transition. These behaviors in spin dynamics demonstrate the existence of the static $d_{x^2-y^2}$-type orbital ordering in the PM and FM states of 50% hole-doped manganites which have relatively larger one-electron bandwidth.

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I. INTRODUCTION

Recent experimental and theoretical studies on colossal magnetoresistance (CMR) manganites have demonstrated that the one-electron bandwidth (*W*) and an orbital ordering play important roles in determining their physical properties such as spin and lattice structures, transport properties, charge ordering, and spin dynamics.[1–5] In our previous studies, for example, we have studied three perovskite manganites $Pr_{1-x}Sr_xMnO_3$ and $Nd_{1-x}Sr_xMnO_3$ with $x \sim 1/2$, and demonstrated that, instead of the well-known CE-type spin and charge ordered state, some of manganites exhibit a metallic layered *A*-type antiferromagnetic (AFM) state.[5] The change of the magnetic structure from CE type to *A* type can be interpreted as an effect of the widening of *W*. Based on detailed studies of the lattice and spin structures and spin dynamics, we further demonstrated that the *A*-type AFM state have the static $d_{x^2-y^2}$- type orbital order,[6] and this was supported by a large anisotropy of the resistivity.[7]

From a viewpoint of the double exchange (DE) mechanism for the CMR effects, one may expect that the system in the pure ferromagnetic (FM) metallic state has no orbital ordering and shows an isotropic behavior by gaining a maximum kinetic energy. Indeed, isotropic spin wave (SW) excitations were observed in the FM state of $La_{0.7}Pb_{0.3}MnO_3$ and $La_{1-x}Sr_xMnO_3$ with $x \lesssim 0.1$. These results seem to indicate that a transfer integral in these systems is isotropic, and there is no orbital ordering.[8–10] To our knowledge, there is no report on experimental evidence of any type of orbital ordering in the FM metallic phase. In contrast to such an experimental situation, Maezono and Nagaosa showed theoretically that the $d_{x^2-y^2}$-type orbital ordering can be responsible to reproduce the hole concentration (x) dependence of a spin stiffness constant in the FM metallic state.[11] They suggested that the orbital ordering could be even a dynamical resonant (''orbital liquid'') state. By a Monte Carlo simulation, Yunoki *et al.* also claim the existence of an orbital ordered state in the FM phase.[12] Consequently, a detailed experimental examination of a possible orbital ordered state in the FM metallic phase and also in the PM state would be expected to provide indispensable information in order to elucidate the mechanism of the CMR effects.

For this purpose, a study of spin dynamics by neutron scattering technique can give a unique opportunity to detect the orbital ordered state, as was demonstrated in previous works.[6,8,10] In the FM case, it further provides a possibility to distinguish the influence of the orbital ordering from those of the DE mechanism or of the charge ordering. This is because, in contrast to the *A*-type AFM state, the DE mechanism in the FM metallic isospin state allows nonzero charge transfer along all directions, so that the difference of the spin stiffness can be attributed solely to the polarization of the orbital ordering.

In this paper, we studied two well-characterized 50%

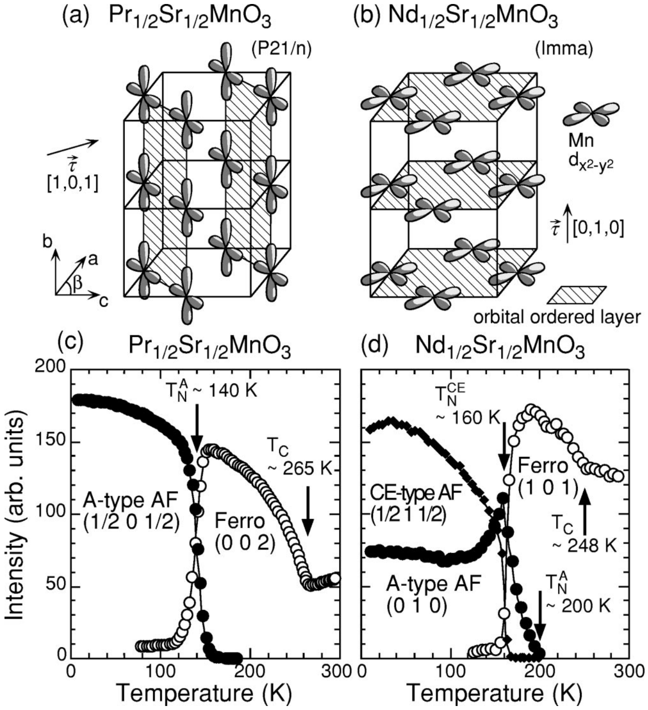

FIG. 1. Upper panels: Expected $d_{x^2-y^2}$-type orbital ordered planes in $Pr_{1/2}Sr_{1/2}MnO_3$ and in $Nd_{1/2}Sr_{1/2}MnO_3$, respectively. Lower panels: Temperature dependences of the FM and AFM Bragg reflections (partially from Ref. 5).

hole-doped manganite systems $Pr_{1/2}Sr_{1/2}MnO_3$ and $Nd_{1/2}Sr_{1/2}MnO_3$. Here, it is useful to point out that the crystal structure of the Pr and Nd compounds may favor a Jahn-Teller (JT) distortion which could induce the $d_{x^2-y^2}$-type orbital order even in their PM and FM states. The crystal structures of the Pr and Nd compounds at room temperature belong to the monoclinic $P21/n$ and to the orthorhombic $Imma$ symmetry, respectively, and the MnO_6 octahedra in both systems consist of two long and one short O-Mn-O bonds.[5,13] Although the JT distortion of the MnO_6 octahedra in their PM and FM states is smaller than those in the A-type AFM state, one can still expect a possibility of the $d_{x^2-y^2}$-type orbital ordering. For a convenience of discussions, we will hereafter employ a terminology of "layer" and "plane" to refer such a $d_{x^2-y^2}$-orbital ordered plane, although the compounds $Pr_{1/2}Sr_{1/2}MnO_3$ and $Nd_{1/2}Sr_{1/2}MnO_3$ do not have a conventional layered structure.

It should be also noted that the JT distortion of MnO_6 octahedra in these two systems occurs along the different crystallographic directions. As illustrated in Figs. 1(a) and 1(b), the directions of the shorter bonds $\vec{\tau}$, which is perpendicular to the $d_{x^2-y^2}$-type orbital planes, are $\vec{\tau}=[101]$ and $\vec{\tau}=[010]$ for the Pr and Nd compounds, respectively. Concomitantly, if the systems show the $d_{x^2-y^2}$-type orbital order, their physical properties should exhibit different directional dependences. For the present study, this feature will provide a further advantage to identify the effects of the orbital ordering experimentally.

In the following, we shall demonstrate that the spin fluctuations of $Pr_{1/2}Sr_{1/2}MnO_3$ and $Nd_{1/2}Sr_{1/2}MnO_3$ show several anomalous behaviors in their PM and FM states. In particular, there exist two types of spin fluctuations: quasielastic isotropic diffuse scattering and dynamical anisotropic (two-dimensional) spin fluctuations in the PM phase. The dynamical spin fluctuations possess A-type AFM spin correlations in their FM states. The clearly distinguishable directional dependences of spin fluctuations help us to unambiguously identify that the anisotropic behavior can be attributed to the $d_{x^2-y^2}$-type orbital ordering in these systems. In addition, we shall demonstrate that the Nd compound further shows a second order phase transition to the canted antiferromagnetic phase.

II. EXPERIMENTAL DETAILS

Neutron scattering measurements were performed with triple axis spectrometers GPTAS-4G and HER-C11 installed at the thermal and cold-guide tubes at the JRR-3M research reactor JAERI, Tokai, Japan. The GPTAS spectrometer was operated with $k_f=2.57\ \text{Å}^{-1}$ and $40'$-$80'$-$40'$-$80'$ collimators, while the HER with $k_i=1.55\ \text{Å}^{-1}$ and open-$80'$-$80'$ collimators after a monochromator to a detector, respectively. The latter yields an energy resolution of ~ 0.23 meV (FWHM). The measurements were performed with the same crystals used in our previous studies.[6] The details on the sample preparation and the sample quality has been already reported.[5,14,15] The single crystal samples were aligned with the (h,l,h) scattering plane. The FM spin fluctuations were measured at around the nuclear Bragg reflection $Q=(1,0,1)$, while the SW profiles were measured at around the respective A-type AFM Bragg points $Q=(1/2,0,1/2)$ for the Pr compound or $(1,\pm 1,1)$ for the Nd compound, respectively, reflecting a directional difference of MnO_6 distortions, as shown in Figs. 1(a) and 1(b).

III. EXPERIMENTAL RESULTS

A. Magnetic ordering

The magnetic as well as transport phase diagrams for the $Pr_{1-x}Sr_xMnO_3$ and $Nd_{1-x}Sr_xMnO_3$ systems have been reported by Tomioka *et al.*[14] and by Kuwahara *et al.*,[16] respectively. The magnetic transitions and crystal structures of the 50% hole-doped systems, were also reported previously.[5] To illustrate the behavior of magnetic orders, the temperature (T) dependence of the magnetic Bragg intensity in both systems is reproduced in the lower panels of Fig. 1. With decreasing T, these two systems with $x=1/2$ enter a pure FM state at $T_C=265$ and 248 K for the Pr and Nd compounds, respectively, then to the AFM state at $T_N=140$ and 160 K through a first order transition. The previous work on a powder sample showed that the magnetic structures of the low-temperature phase for Pr and Nd compounds are A type and CE type, respectively. The present study on single crystals, however, revealed that the Nd compound developed a weak A-type AFM reflection below $T_N^A \sim 200$ K, and it remains even in the low-temperature AFM phase below $T_N^{\mathrm{CE}} \sim 160$ K and coexist with the CE-type reflections. It was recently reported that a low-temperature phase of $Nd_{1-x}Sr_xMnO_3$ with $x>0.51$ shows such a coexistence.[13]

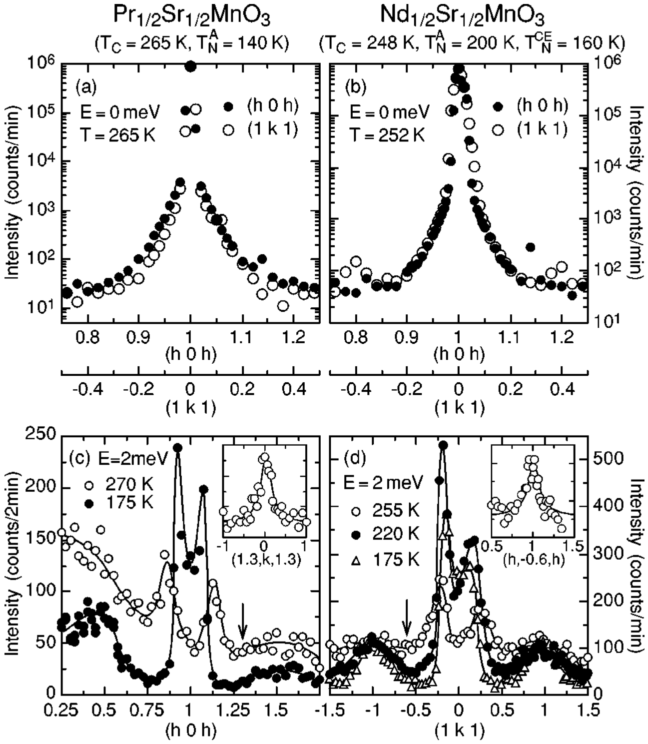

FIG. 2. Constant-E scan profiles observed at $E=0$ (upper panels) and 2 meV (lower panels). (Insets) Profiles observed along the perpendicular to the 2D rodlike scattering at 270 and 255 K for the Pr and Nd compounds, respectively. The arrows in the bottom panels indicate the positions where the profiles in the insets were measured.

We interpret that the slightly different stoichiometry of the sample causes the coexistence of the CE-type and *A*-type reflections in the low-temperature phase of the nominally 50% hole-doped Nd compound. Assuming the same ordered moments for manganese ions in the CE-type and *A*-type states, the volume fraction of the *A*-type region is evaluated to be less than 10% at 10 K. By contrast, the *A*-type magnetic reflection in the FM phase is attributed to a canted AFM order, and should not be interpreted as a phase separation. As is discussed later, this is because *A*-type AFM spin wave excitations are observed in the FM state.

B. Quasielastic and dynamical diffuse scattering

In Fig. 2 are depicted wave vector dependence of spin fluctuations measured at $E=0$ and 2 meV, respectively. These data were taken at the HER spectrometer with an energy resolution of ~ 0.23 meV (FWHM). As shown in the upper panels, the profiles at $E=0$ meV consist of a strong nuclear Bragg component at the center and additional wing scattering. The diffuse component at the wing is practically identical for the h and k directions, indicating that the $E=0$ meV diffuse component is *isotropic*.

In sharp contrast to this, the $E=2$ meV component depicted in the lower panels is clearly *anisotropic*. As shown in Fig. 2(d), the scattering profile for the Nd compound observed in the PM state at 255 K is almost flat along the k direction except for two spurious peaks at $k\sim\pm 0.25$,[17] while the profile along the h direction is narrow (see the inset). This indicates that the dynamical spin correlations are two dimensional. Namely, the spins are strongly correlated ferromagnetically within the planes, but are not correlated between the planes. We interpret that this anisotropic feature is due to the $d_{x^2-y^2}$-type orbital order. These results manifest that the orbital ordering itself has crucial influence on the dynamical spin correlations and causes a distinct anisotropy of the spin dynamics of the manganite systems even in the PM state.

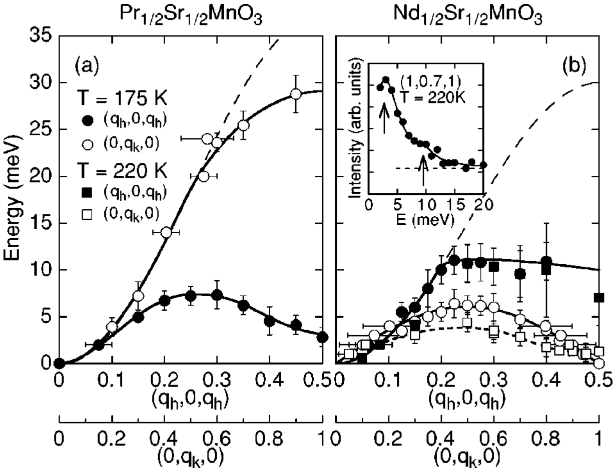

FIG. 3. Dispersion curves within and between plane directions. An inset shows a constant-Q scan profile observed at (1,0.7,1). Solid curves are drawn as guides to the eye.

In the FM state, the system shows a further intriguing feature at around the *A*-type AFM zone center. The profile for the Nd compound observed at 220 K indicates that spins between the planes now exhibit AFM correlations even though the system is still in the FM phase. As discussed below, the AFM diffuse scattering in the Nd compound further developed into real long-range order below $T_N^A \sim 200$ K.

For the Pr compound, we observed exactly the same behavior with the Nd compound except for the change of the direction of the spin correlations. Namely, the two-dimensional FM spin correlations develop within the (101) planes and *A*-type AFM correlations develop at around $Q=(1/2,0,1/2)$ and (3/2,0,3/2). These are consistent with the idea that the $d_{x^2-y^2}$-type orbital order is formed within (101) planes in the Pr compound. Note that the increase of intensity towards small h in profiles is a contamination due to a direct beam at small 2θ angle.

C. Dispersion relation of spin wave excitations

In order to characterize the spin fluctuations in the FM phase, we determined the SW dispersions. As shown in Fig. 3, there are several remarkable features in the SW dispersion relations within and between the planes. First, it is evident that SW stiffness constant D is different for the two directions. For example, in the Pr compound, the SW energy along the [1 0 1] direction (perpendicular to the orbital-ordered plane) is lower than those along the [0 1 0] direction (an in-plane direction within the orbital-ordered FM planes). The present results indicate a difference in electron transfer along the two directions and an existence of an $d_{x^2-y^2}$-type orbital ordering in the FM state, which should be otherwise isotropic. This gives clear evidence of the static orbital ordering in the FM metallic state of the CMR manganites.

The second feature is the anomalous q dependence of the dispersion curve along the in-plane direction. In the Pr case, the SW dispersion curve deviates in the middle of the Brillouin zone from a single cosine-band-type formula given by conventional linear SW theory, which is indicated by the dashed curve. This curve is calculated for the simple FM Heisenberg model with nearest-neighbor couplings, and is identical to the one predicted from the DE model in the limit of large Hund's coupling by Furukawa.[18] This relation was reported to explain well the observed SW dispersion relations in a relatively wide one-electron bandwidth (W) systems such as $La_{0.7}Pb_{0.3}MnO_3$ and $La_{0.7}Sr_{0.3}MnO_3$.[8,9] However, the deviation from the linear SW theory is serious for the Pr compound, and is even more striking for the Nd compound. In the Nd case, the dispersion along the in-plane direction (along the [101] direction for the Nd compound) becomes practically flat beyond $q_h = 0.2$. The inset of the Fig. 3(b) is the profile observed at (1, 0.7, 1). There are two peaks at 3 and 9.5 meV due to the two magnetic domains. One might suspect that a crystal electronic field excitation from Nd ions gives rise to the peak at 9.5 meV. However, we can easily exclude this possibility because the profile measured at the zone center (1,0,1) exhibits no peak at 9.5 meV. A similar anomaly was previously reported for the lower hole-doping compounds $Pr_{0.63}Sr_{0.37}MnO_3$, $Nd_{0.7}Sr_{0.3}MnO_3$, and $La_{0.7}Ca_{0.3}MnO_3$.[19,20] These SW anomalies are theoretically considered as results from either a magnon-electron interaction,[21–23] a magnon-phonon interaction,[24,25] a coupling to the orbital degrees of freedom,[26,27] and/or randomness effects,[28] and none of the stories are indisputable at present.

Thirdly, the dispersion relations perpendicular to the orbital-ordered layers also exhibit anomalous q dependence. In the Pr compound, the zone boundary (ZB) along the $(q_h,0,q_h)$ direction in the FM phase is located at $q_h = 0.5$. However, it seems that the ZB is located at $q_h = 0.25$ and that the periodicity of the dispersion is doubled along this direction, although the SW energy at $q_h = 0.5$ is still finite. This result revealed that the $d_{x^2-y^2}$-type orbital order realized as is expected from the view points of lattice distortions and that the system possesses the A-type AFM fluctuations in the FM phase.

For the Nd compound, the dispersion along the $(0,q_k,0)$ direction exhibits the same behavior, but the SW energy at the ZB with $q_k = 1.0$ vanishes below $T_N^A \sim 200$ K. To see detailed behavior of the ZB SW excitations, we show in Fig. 4 the q dependence of the SW profiles near the ZB at two T's above and below $T_N^A \sim 200$ K. At 225 K in the FM phase (right panel), the ZB profile shows a clear gap, but at 179 K below T_N^A (left panel) the q dependence indicates that the gap vanishes at the ZB of $q_k = 1.0$. Because these results were obtained in the collective SW excitations, one can conclude that the emergence of the A-type AFM Bragg reflections at T_N^A shown in Fig. 1(d) is a second order phase transition and that the Nd compound enters a spin canted phase but not to a phase separated state. We attributed this second order transition to the increase of polarization of $d_{x^2-y^2}$-type orbital with decreasing T.

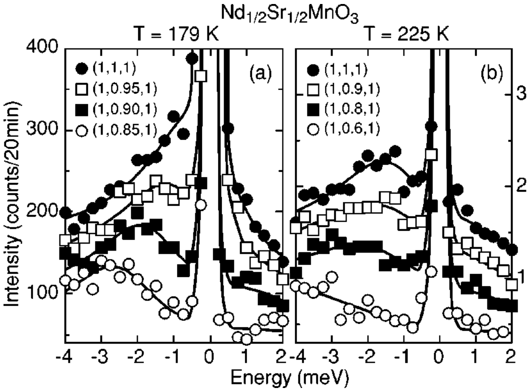

FIG. 4. q- dependence of constant Q scan profiles observed above and below the A-type AFM transition temperature T_N^A ~ 200 K.

IV. DISCUSSION AND CONCLUSIONS

All the results described in the previous section shed light on the nature of the ordering process of two compounds. Even in the PM phase, the dynamical spin fluctuations are anisotropic due to the polarization of the $d_{x^2-y^2}$-type orbital. As T is decreased, these systems enter the FM state, but the lattice distortion is also enhanced at the same time, and results in the change of the polarization of the orbital state. The AFM spin correlations are developed between orbital-ordered planes in the FM state and are progressively increased, and they yield a weak parasitic A-type AFM order (a spin canted phase) in the Nd compound through a second order transition. With further decreasing T, these systems show first order transitions to the pure AFM state, where the Pr compound has the A-type AFM state while the Nd compound has a phase separated AFM state between CE type and A type. The change of the magnetic structures from the (mainly) CE type order in the Nd compound to the A-type AFM order in the Pr compound is attributed to the widening of the W in the latter compound.

Finally, we would like to discuss an issue of a FM central component. Recently, it was pointed out that the CMR manganites which show large CMR phenomena have a strong central component at around their T_C.[29,30] The central component can be also observed in the PM state of the AFM manganites.[6,31,32] Such a central component yields a spin diffusion constant with one order smaller energy scale to its spin stiffness constant.[6,31] The present results shown in Figs. 2(a), 2(b) demonstrate that the correlation length of the central component is an order of ~100 Å. In addition to the central component, however, the system has an additional anisotropic (two-dimensional) component which has a larger energy scale and shorter correlation length than those of the central component. This component must correspond to conventional thermally activated spin fluctuations, indicating the coexistence of slowly fluctuating large clusters and conventional spin fluctuations in the system. The former slow fluctuation is very likely related to the CMR phenomena, while the latter dynamical component clearly manifests an influence of the underlying orbital ordering.

In conclusion, we have found a number of interesting features in the spin dynamics of the 50% hole-doped manganites $Pr_{1/2}Sr_{1/2}MnO_3$ and $Nd_{1/2}Sr_{1/2}MnO_3$ in the PM and the FM phases. (1) The dynamical spin correlations in the PM state are two dimensional. (2) The SW dispersion in the FM state is anisotropic. (3) The SW dispersion along the in-plane direction shows a deviation from a conventional linear SW theory. (4) AFM spin correlations are developed in the FM state. We attribute the results (1), (2), and (4) to the underlying $d_{x^2-y^2}$-type orbital ordering. We demonstrated that the coexistence between FM and *A*-type AFM ordering in the Nd compound should be treated as a canted AFM order but not as a phase separation. The directional dependence of the spin correlations was clearly observed in two systems, which further supports our interpretation that these spin fluctuations are driven by the specific $d_{x^2-y^2}$-type orbital ordering. These results establish that the orbital ordering has crucial influence on the spin correlations in the 50% hole-doped manganites, even in the PM and FM states. The reason of the result (3) remains an open question, and further experimental and theoretical studies are desirable.

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