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Unusual Single Bilayer E-Type Antiferromagnetism in Mn-Substituted Sr₃Ru₂O₇

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Neutron scattering studies reveal an unusual *E*-type antiferromagnetic (AFM) structure in Mnsubstituted single crystals of Sr₃Ru₂O₇ (x = 0.16). The material behaves as quasi-two-dimensional (2D) antiferromagnet with in plane (*ab*) long-range ordering and exhibits only single-bilayer (5 - 6 Å) ferromagnetic correlations along the *c* direction below $T_N = 78$ K. However, the critical behavior of the staggered magnetization, the AFM order parameter, does not reflect the expected behavior of a 2D magnetic phase transition. Such an unusual magnetic structure deviates from what would be expected from the conventional spin-lattice coupling scenario in this class of materials.

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The Ruddlesden-Popper (RP) series $Sr_{n+1}Ru_nO_{3n+1}$, $(n = 1, 2, ...\infty)$ display an array of physical phenomena such as a metal-insulator transition (MIT), spin-orbital ordering, exotic superconductivity, metamagnetic transitions, and quantum criticality [1]. For example, the single layer member of the series, Sr_2RuO_4 (n = 1), exhibits characteristics of both Fermi liquid behavior above 1.5 K and an unconventional spin-triplet superconductivity below 1.5 K [2]. The three dimensional (3D) compound, $SrRuO_3$ (n = ∞), is a FM metal with a Curie temperature (TC) of 160 K [3] and displays characteristics of non-Fermi liquid behavior [4]. The bilayered perovskite $Sr_3Ru_2O_7$ (n = 2) with a slightly distorted tetragonal lattice structure [see the schematic crystal structure in the inset of Fig. 1(a) consists of two layers of RuO₆ octahedra with properties different than that of sister compounds (n = 1 and ∞). Sr₃Ru₂O₇ is metallic and paramagnetic [5] down to the lowest temperature measured. There is no evidence of long/short-range AFM or FM ordering in the range of temperatures from 1.4 to 125 K, with an upper limit of 0.05 $\mu_B/\text{Ru-atom}$ for any possible ordered moment [6]. Yet strong magnetic fluctuations with both FM [7] and AFM [8] character exist in this material. Quantum critical behavior related to a metamagnetic (i.e. magnetic field-tuned) transition has been observed [9]. The AFM fluctuations are enhanced near the critical field [10] in contrast to what should be expected for a metamagnetic transition [see for example Ref. [11].

In systems with strong magnetic fluctuations, chemical substitution is often used to stabilize a magnetic structure, thus allowing for a glimpse at the nature of the magnetic interactions in the parent compound. In this context, it has been demonstrated that a partial substitution of Mn for Ru at the B-site in $Sr_3Ru_2O_7$ induces an AFM order with concentrations of Mn starting as low as 5 % [12 -14] as illustrated in the latest reported phase diagram shown in Fig 1(a). However, the nature of the induced magnetic order and its relevance to the magnetic properties in the parent compound are yet to be elucidated.

This letter describes a comprehensive single-crystal elastic neutron scattering investigation of the magnetism of Mn-substituted $Sr_3(Ru_{1-x}Mn_x)_2O_7$ (x = 0.16) (SRMO16). This concentration of Mn was chosen because this is a unique doping regime. Magnetic susceptibility measurements indicate that at this concentration the AFM ordering temperature reaches a maximum value and a distinctive anomaly in the specific-heat measurements shows that this is a true second-order phase transition. The extracted entropy is also maximum at this concentration. The magnetic order, however, is accompanied by a dramatic increase in the resistivity indicating that the insulating behavior is enhanced by the development of magnetism. The rotational distortion of the RuO_6 octahedra present in the parent compound gradually diminishes with Mn substitution and drops suddenly to zero for x > 0.16. The derived Curie-Weiss temperature shows a cross over from AFM to FM ordering at about x = 0.16 for the in-plane susceptibility, while the c axis susceptibility still favors AFM [13]. Thus the 16 %concentration of Mn is clearly on the borderline between quite different regions of the phase diagram. The data presented here shows that these circumstances lead to an unusual in-plane E-type AFM spin configuration with moments ferromagnetically aligned along the c axis and with only one single bilayer ordering in the c direction.

Single crystals of SRMO16 were grown by the floating zone method and subsequently characterized by powder, single-crystal X-ray diffraction, and magnetic susceptibility measurements [13]. All single crystals used have mosaics spreads of about 1° and masses up to 1.63 grams. The crystal structure is described using an orthorhombic cell consistent with the space group *Pban* with room temperature lattice parameters a = b = 5.50(1) Å and c = 20.72(1) Å [6]. A schematic crystal structure and the phase diagram [13] of SRMO16 are shown in Fig. 1(a) (for simplicity, only the tetragonal unit cell is presented). In this symmetry, the two RuO₆ octahedra are rotated about the *c*-axis (same angle and in opposite directions) [6].

Neutron diffraction experiments on SRMO16 were car-



FIG. 1: (a) A schematic representation of the latest reported phase diagram for various substitution concentrations (x) of the Sr₃(Ru_{1-x}Mn_x)₂O₇ series (unit cell representation shown in the inset) [13]. Region I is a paramagnetic metallic (PM-M) phase; II a paramagnetic insulating (PM-I) phase; III a metallic phase with AFM correlation (AFMC-M); and IV a long-range AFM insulating phase (LR-AFM-I). Neutron scattering studies were carried out in Region IV (b) T-dependence of integrated magnetic scattering intensity at chosen AFM Bragg peaks for the Sr₃(Ru_{0.84}Mn_{0.16})₂O₇.

ried out at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory with samples mounted inside a closed-cycle refrigerator. Initial measurements were taken on the US/Japan Wide Angle Neutron Diffractometer (WAND) installed at the beam port HB2C at HFIR with a Ge (111) monochromator used to produce an incident neutron with wavelength of 1.48 Å. The HB1A Triple Axis Spectrometer with a fixed incident neutron wavelength of 2.359 Å and horizontal collimation of 48'-48'-sample- 40'-68' as well as the HB3A Four-Circle Diffractometer with a fixed incident neutron wavelength of 1.536 Å were subsequently used in the study. Finally, the Rietveld Refinement package FullProf [15] was used in order to perform a magnetic representation analysis using symmetry adapted propagation wave vectors to obtain the spin configuration in the ground state. We index all diffraction peaks following the orthorhombic unit cell notation.

Our measurements show that magnetic order in SRMO16 has a characteristic wave vector given by $\vec{Q}_M = (0.5, 0, 0)$, as previously reported for 5 % from powder neutron diffraction experiments [12]. The Bragg inten-

sity (I_B) of the observed magnetic superlatice peak (0.5, 0, 0) is a measure of the square of the staggered magnetization of the system (M^2) , which is the order parameter. The *T*-dependence of the order parameter is presented in Fig. 1(b) and inset, this plot indicates that the AFM transition temperature $T_N = 78$ K, which is consistent with magnetic susceptibility measurements [13].

In order to determine the spin configuration neutron diffraction profiles on single crystals of SRMO16 were measured and are presented in Fig. 2. Figures 2(a) and 2(b) show the contour plot of diffraction patterns (intensities in logarithmic scale) for T = 100 K and 10 K in the (H, K, 0) scattering plane, respectively. Fig. 2(a)displays the diffraction pattern at $T > T_N$. The nuclear Bragg peaks appear at the reciprocal lattice positions (h k 0) that satisfy the h + k = 2n (n integer) diffraction condition for this symmetry. The less intense peaks present in the diffraction pattern not satisfying the higher I4/mmm space group conditions indicates the lowering of the symmetry to *Pban* space group which originates from the rotations of the RuO_6 octahedra about the caxis [6, 7, 16]. The diffraction pattern shown in Fig. 2(b), which was taken at the base temperature (T = 10 K), includes both nuclear and magnetic scattering. The magnetic peaks at \vec{Q}_M and equivalent positions are present in the low-Q region at T = 10 K. Since our sample is 90° twinned in the *ab* plane, magnetic reflections at $\vec{Q} = (0,$ (0.5, 0) and equivalent positions are also present in the diffraction pattern.

The exact structure of the spin order in the low-TAFM phase can be unambiguously identified from these single-crystal diffraction results. Figure 2(c) shows the expanded difference scattering pattern (intensity image in linear scale) between low-T AFM phase [Fig. 2(b)] and PM phase at 100 K [Fig. 2(a)], displaying only the magnetic diffraction peaks. Constrained by the orthorhombic symmetry of the lattice structure, there are only two high-symmetry AFM spin structures which can be used to describe the observed low-T magnetic diffraction patterns in the ab plane: the so-called CE- and E-type AFM structures. Our analysis indicates that the absence of the (0.5, 0.5, 0)-type magnetic diffraction peaks excludes the CE-type AFM ordering. Furthermore, the magnetic propagation wave vector \vec{Q}_M indicates the doubling of the chemical unit cell along either a- or b-axis direction, which is suggestive of the zigzag chains of the *E*-type structure. Extensive data collection along high symmetry scattering planes has excluded the possibility of other spin ordering patterns. While neutron powder diffraction [12] and recent resonant elastic soft X-ray scattering [14] studies have proposed the *E*-type AFM order in the ground state, neither of the previous work provided a clear conclusion and both stated the need for a thorough diffraction study, like the one presented here.

Having identified the spin texture in the ground state of the system, the question remains as to what are the orientation and the dimensionality of the spins. In this context, Figure 2(d) yields the first clue regarding the spin



FIG. 2: Neutron diffraction patterns of SRMO16 in the (H,K,0) and (H,0,L) reciprocal planes for temperatures above and below T_N . The color bars represent the intensities in counts per 5 mins and the powder rings observed are those of the Aluminum. The raw data reported on the top panel (on a logarithmic scale) show (a) the structural peaks at $T > T_N$ and (b) the combination of both structural and magnetic peaks at $T < T_N$. Peaks are indexed using the orthorhombic notation [see text]. The differential diffraction patterns at T = 10 K from T = 100 K is shown in (c) and (d) where the studied magnetic Bragg peaks observed at the base T are zoomed in for clarity.

orientation; it displays the differential magnetic diffraction pattern in the (H, 0, L) plane, showing the much stronger intensity for $(1.5, 0, 1 \ 0)$ diffraction peak compared to that of the (0.5, 0, 1) peaks. This drastic difference in intensity indicates that magnetic moments are not aligned along the ab-plane but are parallel to the c-axis, since neutrons can only couple to magnetic moments perpendicular to \vec{Q} . Otherwise, one should expect zero intensity at (1.5, 0, 0) Bragg point in Fig. 2(d) since the magnetic scattering intensity is proportional to $\vec{Q} \times (\vec{S} \times \vec{Q})$, where \vec{S} is the moment and \vec{Q} is the wave vector transfer. Figure 2(d) also yields relevant information regarding the dimensionality of magnetism in this system. To understand this, recall that the integrated intensity for a magnetic Bragg reflection with moments aligned along the c-axis is given by [17]:

$$I \propto |F_M(Q)|^2 (1 - (\frac{L}{Q})^2 |M_c|^2) \cos^2(\pi z L)$$
 (1)

where $|F_M(Q)|^2$ represents the Ru from factor and $z \sim 0.2$ is the reduced distance between the RuO₂ planes in the bilayer. The term enclosed in parenthesis is the polarization factor that unambiguously allows us to detect the direction of the moments, which as explained previously its only term is the out-of-plane magnetic compo-

nent since the moments are aligned along the c-direction. The cosine squared modulation term is not only responsible for the observed Bragg peaks at even values of L for peaks (1.5, 0, l =2n), but also for the variation of the intensity of such peaks, where the next strong peak appears in Figure 2(d) at L= 4. Therefore, the scattering pattern from Figure 2(d) shows that there is some modulation along the c-direction, which is an indication of the layered nature of the magnetic structure in the ground state of this system.

In order to further investigate the dimensionality of the spin order in SRMO16, we systematically characterized both the in-plane [(H, K, 0)] and out-of-plane [(0, 0, L)]magnetic correlations from T-dependent line profiles of superlattice peaks. Figure 3(a) displays the representative H scans across the (0.5, 0, 0) magnetic peak in the reciprocal space at various temperatures. Similarly, Fig. 3(b) presents L scans across the (1.5, 0, 0) peak shown in Figure 2(d). The narrow (resolution limited) linewidth of the H scans across the (0.5, 0, 0) peak at low temperatures is indicative of long-range AFM ordering in the basal plane. The L scans across the (1.5, 0, 0) magnetic peak, on the other hand, exhibit very broad Lorentzianprofiles [see Fig. 3(b)] even at low temperature, indicating short-range AFM correlations along the c-axis. The extracted c-axis magnetic correlation length $\xi(T)$ ploted in Fig. 3(d) inset shows only a maximum value of $\xi \sim 5$ - 6 Å bellow T_N . The average value of the Ru-O apical distance is 2.021 Å [6,13], indicating that the magnetic correlation along the *c*-axis is exclusively restricted to the bilayered-block of the RuO_6 octahedra, which as the insert in Fig. 1 (a) shows, is only half of the unit cell. Thus, the ground state in this system is characterized by a single-bilayer of spins antiferromagnetically ordered in a zigzag chain pattern along the *ab* plane. This is similar to the case of $Sr_3(Ru_{1-x}Ti_x)_2O_7$ (x = 0.4) in which the Ti-induced incommensurate spin-density wave ordering cannot be considered to be three-dimensional due to the finite correlation length along the c direction [18].

We argue that the Mn-induced magnetic structure is an unusual quasi-2D structure. A preliminary fit (i.e. we did not correct for the critical scattering contribution) of I_B -which is proportional to M^2 -to the power-law scaling function resulted in a value of ~ 0.30 for the critical exponent β . This value deviates from the expected value (~ 0.125) for 2D magnetism [19]. Although more detailed measurements would be required in order to obtain the precise value of β , our results are sufficient to indicate that such a Mn-induced 2D magnetic behavior cannot be explained by existing theory such as the 2D Ising model.

Figure 4 presents a schematic diagram of the Mninduced SRMO16 spin structure in the bilayer block resulting from our FullProf magnetic refinement of the low-T neutron diffraction data. Our analysis revealed that: 1) the symmetry allowed magnetic structure with moments aligned along the c direction provide the best description of the data; 2) the spins are coupled ferro-



FIG. 3: Neutron diffraction profiles at magnetic Bragg peaks (a) (0.5, 0, 0) in the (H,K,0) and (b) (1.5, 0, 0) in the (H,0,L)scattering planes, respectively. The narrow (resolution limited) linewidth of the H scans is indicative of long-range AFM ordering in the basal plane while the very broad Lorentzianprofiles of L scans indicate short-range AFM odering (see inset on Fig. 3b) along the *c*-axis. The solid lines are a guide to the eye.

magnetically within the bilayer and antiferromagnetically along the basal plane; and 3) an upper limit value of ~ $0.70 \ \mu_B/(\text{Ru/Mn})$ for the average ordered moment is obtained from the integrated magnetic scattering intensities.



FIG. 4: Schematic AFM spin configuration in the ground state of SRMO16 resulting from a magnetic refinement [see text]. The single-bilayer magnetic structure of the (Ru/Mn)O₆ octahedra is displayed where the zigzag chains of the E-type AFM order are marked with solid lines. Two un-equivalent spins in the in-plane magnetic unit cell and associated octahedra are illustrated while the top and bottom (Ru/Mn)O₆ layer have identical spin ordering structure.

It is known that there is a close correlation between the magentic ground state and structural distortions in layered ruthenates attributed to a strong spin-lattice coupling. The rotational distortion of the RuO_6 octahedra tends to enhance the FM instability while the combination of tilting and flattening of the RuO_6 octahedra favors the antiferromagnetic fluctuation as manifested in the $\text{Ca}_{2-x}\text{Sr}_x\text{RuO}_4$ [20]. For chemically substituted $\text{Sr}_3\text{Ru}_2\text{O}_7$, the exact magnetic ground state depends on the nature of the substituent. It is intriguing to note that a small amount of Ti or Mn substitution can stabilize AFM ordering, either incommensurate (ICM) as in the case of Ti substitution or commensurate (CM) as in the Mn substitution case, but both different from the dynamic AFM correlations observed in the parent compound. In case of nonmagnetic Ti substitution, a flattening of the unit cell is realized with the shrinking of the c-axis lattice constant and and expansion along the basal plane [18]. While it is not clear if additional rotational distortion of RuO₆ exists as in the Mn-doped sample, Ti substitution enhances the ICM dynamics and ultimately leads to a static spin-density-wave-type magnetic order at $x_{cr} \sim 0.04$ and above [18] The situation is different in Mn-substituted $Sr_3Ru_2O_7$ as the substituent is magnetic with a smaller ionic radius. The introduction of Mn flattens the RuO_6 octahedra by reducing out-of-the-plane Mn/Ru-O bond length without inducing a tilt distortion [13], similar to the case of Ti substitution [18]. This results in an increase of the d_{xz}/d_{yz} bandwidth. However, in contrast to Ti substitution, Mn substitution has little effect on the in-plane Mn/Ru-O bond length, but suppresses the rotational distortion present in the parent compound. This implies that the d_{xy} bandwidth will not decrease with the substitution. Neither of these structural modifications will lead to an insulating ground state expected from first-principles calculations [20] or from a simple Mott-type scenario [21]. The observed AFM insulating state in Mn-doped Sr₃Ru₂O₇ is then puzzling as it is inconsistent with the *conventional wisdom* [20], and its origin is likely related to additional cooperative effects from doped magnetic ions.

In summary, neutron scattering has revealed a truly unexpected Mn-stabilized AFM ordering in the bilayered ruthenate $Sr_3(Ru_{1-x}Mn_x)_2O_7$ (x = 0.16). The AFM order is found to exhibit only single bilayer-thickness correlation along the *c* direction. This is remarkable, as the magnetic susceptibility data is isotropic above T_N . What is evident from the magnetization data, including the extracted Curie-Weiss temperature, mentioned above, is that there is a competition between AFM and FM and that $x \sim 0.16$ is the cross over point [13]. Also x = 0.16 is the point where the rotation angle of the octahedron diminishes to zero rapidly. An obvious follow up experiment is to look at magnetic ordering for lower values of Mn-substitution. In the latter, three-dimensional magnetic ordering may be favored.

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