Magnetic field dependent neutron powder diffraction studies of Ru0.9Sr2YCu2.1O7.9

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Nigam, Rashmi; Pan, Alexey V.; Dou, S. X.; Kennedy, S J.; Studer, A J; and Stuesser, N: Magnetic field dependent neutron powder diffraction studies of Ru0.9Sr2YCu2.1O7.9 2010, 1-3.  

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Citation: J. Appl. Phys. 107, 09E134 (2010); doi: 10.1063/1.3365576
View online: http://dx.doi.org/10.1063/1.3365576
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(Received 31 October 2009; accepted 3 December 2009; published online 5 May 2010)

Temperature and magnetic field dependent neutron diffraction has been used to study the magnetic order in Ru0.9Sr2YCu2.1O7.9. The appearance of (1/2, 1/2, 1/2), (1/2, 1/2, 3/2), and (1/2, 1/2, S/2) peaks below TM=140 K manifests the antiferromagnetic order. Neutron diffraction patterns measured in applied magnetic fields from 0 to 6 T show the destruction of the antiferromagnetic order with increasing field. There is no evidence of spontaneous or field-induced long range ferromagnetic order. This latter result contradicts the vast majority of other experimental observations for this system.

Temperature and magnetic field dependent neutron diffraction has been used to study the magnetic order in Ru0.9Sr2YCu2.1O7.9. The appearance of (1/2, 1/2, 1/2), (1/2, 1/2, 3/2), and (1/2, 1/2, S/2) peaks below TM=140 K manifests the antiferromagnetic order. Neutron diffraction patterns measured in applied magnetic fields from 0 to 6 T show the destruction of the antiferromagnetic order with increasing field. There is no evidence of spontaneous or field-induced long range ferromagnetic order. This latter result contradicts the vast majority of other experimental observations for this system.

The present neutron data of ~1.58 g of the powder Ru1212Y sample were collected at two different neutron facilities, viz., the focusing diffractometer (E6) at the Berlin Neutron Scattering Center (BESSY) at Helmholtz-Zentrum-Berlin using wavelength \( \lambda = 2.45 \) Å and the high intensity powder diffractometer (Wombat) installed at the Opal research reactor at Australian National University and Technology Organization (ANSTO) with the incident neutron wavelength of 2.41 Å. Neutron diffraction pattern of Ru1212Y was collected by Wombat from 2θ=16° to 135° in the temperature range of 20–160 K with 1 K step size. Figure 1 shows a portion of the diffraction pattern. In addition to the nuclear Bragg peaks indexed as (0 0 2), (0 0 3), and (1 1 0), additional peaks appear below TM=140 K at 2θ=27.5°, 33°, and 41.5°. These peaks can be indexed as (1/2, 1/2, 1/2), (1/2, 1/2, 3/2), and (1/2, 1/2, S/2), respectively. The appearance of additional peaks along with the Bragg peaks suggests antiferromagnetic (AFM) order with doubling of the unit cell in all directions. The comparison of present neutron data of Ru0.9Sr2YCu2.1O7.9 sample in the temperature range of 20–160 K collected at ANSTO.

FIG. 1. (Color online) Neutron diffraction pattern of Ru0.9Sr2YCu2.1O7.9 sample in the temperature range of 20–160 K collected at ANSTO.
Ru1212Y with the neutron data reported by other groups on the same compound shows that our neutron data exhibit (1/2, 1/2, 1/2), (1/2, 1/2, 3/2), (1/2, 1/2, 5/2), and (1/2, 1/2, 3/2) AFM peaks, whereas only a (1/2, 1/2, 1/2) peak was observed by Takagiwa et al.,\textsuperscript{12} and (1/2, 1/2, 1/2) and (1/2, 1/2, 3/2) magnetic peaks were reported by Yelon et al.\textsuperscript{11} This is further in agreement with the neutron diffraction data reported by Lynn et al.,\textsuperscript{1} on RuSr\textsubscript{2}GdCu\textsubscript{2}O\textsubscript{7-δ}, which showed (1/2, 1/2, 1/2) and (1/2, 1/2, 3/2) reflections at the Ru ordering temperature of about 135 K. Out of these three additional peaks, the (1/2, 1/2, 1/2) peak is the strongest; hence this peak will be used for further discussion. In order to study the behavior of (1/2, 1/2, 1/2) peak with changing temperature, its intensity was plotted as a function of temperature in Fig. 2. Below \(T_\text{M} = 140\) K, the intensity of AFM peak increases with decreasing temperature. Contrary to this evidence of AFM order, \(M(H)\) loops of Ru1212Y exhibit FM-like hysteresis loops;\textsuperscript{11} therefore, it is important to search for the FM component in the system. In neutron diffraction, FM is manifested as additional scattering at nuclear Bragg peaks. No change was observed in the intensity of nuclear Bragg peaks, where a FM component would appear; hence no indication of FM order was given by NPD measurements. Similar observation was made by Lynn et al., and their results gave an upper limit of 0.1\(\mu_B\) to any FM component. Such a small value of FM moment may remain undetected by NPD.

Although the temperature dependent NPD patterns of Ru1212Y have been reported previously\textsuperscript{10,11} no field dependent NPD study has been published. The only study of this type was reported on RuSr\textsubscript{2}GdCu\textsubscript{2}O\textsubscript{7-δ} by Lynn et al.\textsuperscript{1} In order to investigate how the Ru1212Y system behaves in the presence of external magnetic field in the superconducting state, NPD patterns were measured far below the superconducting and magnetic order temperatures of Ru1212Y. Neutron data were collected at \(T = 1.5\) K under the applied field of 0–5 T at BENSC. The measurement at each field took approximately 3 h. Figure 3 shows NPD pattern at \(T = 1.5\) K for \(H = 0, 1, 2,\) and 3 T. It is clear that the magnitude of the AFM peaks (1/2, 1/2, 1/2) and (1/2, 1/2, 3/2) reduces with increasing field. In order to deduce the effect of external magnetic field on the nuclear Bragg peaks, the NPD data collected at 1, 2, and 3 T were subtracted from 0 T NPD data and plotted in Fig. 3. The difference patterns exhibit positive peaks corresponding to the AFM reflections, indicating that the peak intensity decreases when the magnetic field is applied. With increasing field there is a nonlinear decrease in the AFM peaks, which may indicate a diminishing AFM order. Previously, NPD patterns of Ru1212Gd measured by Lynn et al.\textsuperscript{1} in the fields ranging from 0 to 7 T showed similar results. Figure 3 also shows that the external magnetic field increases the intensity of the (0 0 1), (0 0 2), (0 0 3), and (0 0 4) nuclear peaks. For the Ru1212Gd sample, Lynn et al.\textsuperscript{1} could observe additional scattering only at (0 0 2) nuclear peak when external field was applied. The additional scattering of (0 0 1) nuclear peaks in an applied magnetic field may indicate a field-induced FM. Since only the component of magnetic moments perpendicular to the scattering vector can be detected by NPD, the induced ferromagnetism would be perpendicular to the c-axis (consistent with the proposition of Bernhard et al.).

However, we also note that the (1 1 0) nuclear reflection decreases strongly with increasing field. Given that there is no FM order in zero field and that the AFM order is G-type, the only possible interpretation of this is that the field induces grain reorientation with c-axis aligned perpendicular to the magnetic field. The evidence of grain reorientation brings into question whether the application of a magnetic field really induces the disappearance of AFM order or the appearance of FM order.

We also note that there was an overall reduction in the background of the diffraction pattern in applied field of 5 T (see Fig. 3 inset). This indicates that the sample was highly magnetized and levitating inside the sample holder partly lifted out the neutron beam. Returning to zero field, all the peaks recover their zero-field intensities, indicating that the effect of the field is reversible and also that no preferred orientation of the loose powder particles occurred when the field was applied. NPD measurements of Ru1212Gd conducted by Lynn et al. did not report any shift in the back-
ground of the high field data. Their results showed that with an increase in field, the intensity of the AFM reflection begins to decrease, while the induced magnetization increases. When the field was increased from 0 to 7 T, there is no significant AFM intensity observed, but there was induced magnetization with a net moment of $1.4\mu_B$ perpendicular to c-axis. Authors proposed that the Ru moments rotate into another AFM (spin-flop) structure rather than becoming fully aligned with the field. The NPD study of Ru1212Y is consistent with the NPD study of Ru1212Gd sample, except the fact that the Ru1212Y sample gets highly magnetized for fields as high as 5 T. This may be because the paramagnetic moments of Gd may not allow Ru1212Gd sample to magnetize fully at high fields.

In order to extract meaningful and reliable information about magnetism in Ru1212 from neutron diffraction, we repeated the study of magnetic field dependence on the Wombat diffractometer, with a sample clamped tightly inside the vanadium sample holder by a cylindrical aluminum insert. Neutron data were collected at 10 K in the field ranging from 0 to 6 T. No shift in the background was observed for high field neutron diffraction data, which indicated that no grain levitation took place. The response of the (1/2, 1/2, 1/2), (0 0 3) and (1 1 0) diffraction peaks to applied field is plotted in Fig. 4. The intensity of (1 1 0) peak is clearly constant in the entire field range, confirming that no grain reorientation took place. The field dependence of AFM order [indicated by the drop in the intensity of (1/2, 1/2, 1/2) peak (Fig. 4)] shows almost complete destruction of AFM from 0 to 3.5 T. Further, Fig. 4 shows that the intensity of (0 0 3) nuclear Bragg peaks remains unaffected over the entire range of applied magnetic field, neither was any additional intensity detected in other Bragg peaks. This result confirms our conclusion that the neutron diffraction measurements carried out at BENSIC were dominated by grain reorientation and levitation. Zero field, temperature dependent neutron diffraction studies of Ru$_{0.9}$Sr$_2$YCu$_2$O$_{7-\nu}$ sample showed that the system exhibits an AFM order below $T_M=140$ K, but despite the evidence of FM from magnetization measurements, FM order could not be detected by neutron diffraction. In this case diffuse neutron scattering would be a vital tool to detect short range FM in the system. The field dependent neutron studies showed that the AFM order disappears with increasing field. The apparent field induced FM of the loosely packed Ru1212 powder was shown, by the study of the tightly packed powder, to be just grain reorientation, with moments perpendicular to c-axis.

In conclusion, our field dependent NPD measurement results further illustrate a puzzling response to magnetic fields of this system, which exhibits the hysteretic behavior observed in bulk magnetic measurements, but it seems not to manifest in long range FM ordering. Indeed, the vast majority of global and in situ probing techniques consistently exhibit the existence of the FM signal, whereas only neutron diffraction measurements robustly indicate AFM ordering. Clearly, the origin of this controversy is yet quite unclear and requires further investigation.

This work is supported in part by the Australian Research Council and grants from the Australian Institute of Nuclear Science and Engineering. We acknowledge the financial support from the Access to Major Research Facilities Programme (AMRFP), which is a component of the International Science Linkages Programme established under the Australian Government’s innovation statement, Backing Australia’s Ability. This research project has also been supported by the European Commission under the Sixth Framework Programme through the Key Action: Strengthening the European Research Infrastructures [Contract No. RI3-CT-2003-505925 (NMI 3)].

Authors would also like to thank Dr. V. P. S. Awana from National Physical Laboratory, New Delhi, India, and Professor E. Takayama-Muromachi from NIMS, Japan, for providing the Ru$_{0.9}$Sr$_2$YCu$_2$O$_{7-\nu}$ sample.

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