ESRF	**Experiment title:** **Investigation of Charge Stripes in CMR Oxides using Very High Energy X-ray Scattering**	**Experiment number**: HE-664
Beamline: ID15A	**Date of experiment**: from: 28/01/2000 to: 4/02/2000	**Date of report**: 17/02/2000
Shifts: 18	**Local contact(s)**: Thierry d'Almeida	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

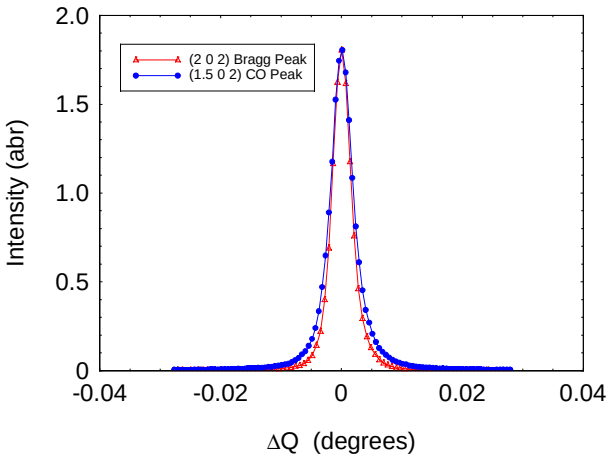
S.B. Wilkins[1]*, M.E. Ghazi[1]*, M. Ohler[2]*, B.D. Fulthorpe[1]*, P.D. Hatton[1], S-W Cheong[3].

1) Department of Physics, Science Labs, University of Durham, South Road, Durham, DH1 3LE, UK
2) High Energy Group, ID15, ESRF‡
3) Department of Physics and Astronomy, Rutgers University, New Jersey 08854, US

Report:

The charge, spin and orbital degrees of freedom play an important role in the electric and magnetic properties of the transition-metal oxides [1]. Recently, the discovery of Colossal Magnetoresistance (CMR) in the manganite compounds [2] has stimulated much interest in this field.

We have performed high-energy x-ray scattering using the high-resolution triple-axis diffractometer at ID-15A on $Nd_{0.5}Sr_{0.5}MnO_3$ [3]. This material shows a transition from a paramagnetic to an ferromagnetic state at 250K followed by a first order phase transition at 160K into a charge ordered Mn^{3+} / Mn^{4+}, anti-ferromagnetic insulating state. The sample was a high quality, single crystal sample of $Nd_{0.5}Sr_{0.5}MnO_3$ grown at Bell Labs. The samples' rocking curve width was ~0.1º measured on a typical Bragg peak.



Neutron scattering has long been the preferred technique for studying such metal-insulator transitions as they can both probe the magnetic and crystallographic structure. However as x-rays are sensitive to the charge distribution they are a very effective technique for studying charge ordering. To date we have used ~10 keV x-rays to study charge ordering utilising the resonant enhancement and polarisation characteristics to also observe spin and orbital ordering. Such measurements probe only the near surface region (the top few microns), limited by the large sample absorption. The purpose of this initial experiment was to see if high-energy x-ray scattering could be used to observe charge ordering. High-energy x-rays have the advantage of much greater penetration allowing the bulk of the crystal to be probed, rather than the near surface region.

‡ Present Address : IMM - Institute of Microtechnology Mainz, Carl Zeiss Strasse 18-20, D-55129 Mainz, Germany

The sample was cooled in a closed cycle He cryostat mounted on the newly installed Eulerian cradle on the triple-axis diffractometer. Using an incident x-ray energy of 100 keV and a matching pair of Si (113) monochromator and analyser crystals, we located satellite reflections corresponding to the charge ordering at modulation wave vectors (0.5 0 0) and (0 0 0.5). Fig. 1 shows the comparison between the charge ordering peak (1.5 0 2) and the corresponding (2 0 2) Bragg peak (normalised to the same peak intensity).

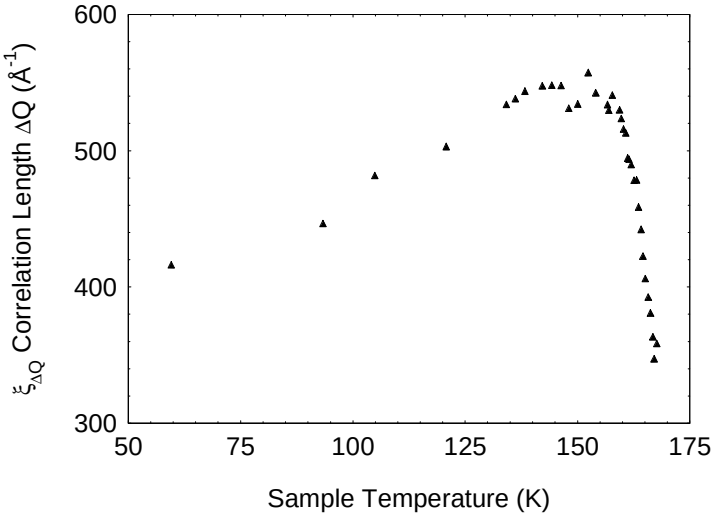


Figure 2

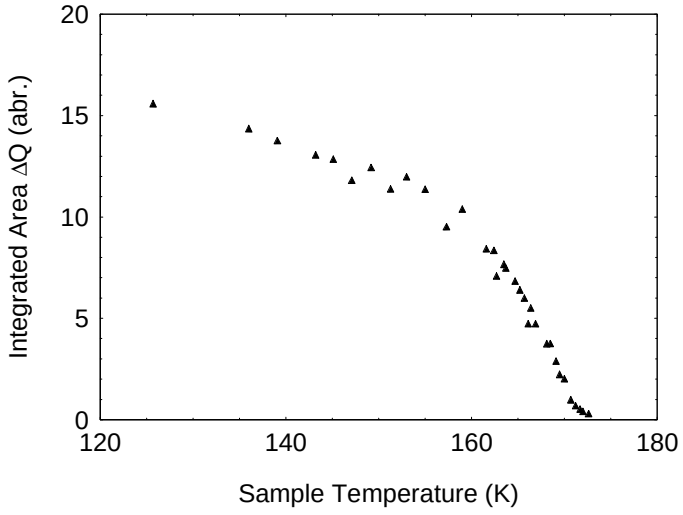


Figure 3

Scans were taken both the longitudinal (analyser rocking) and transverse (sample rocking) directions as a function of temperature on the (1.5 0 2) charge ordering satellite peak. From this data the correlation length of the charge ordering was measured and is shown in Figure 2. The corresponding peak integrated area is shown in Figure 3.

The results obtained during this experiment far exceeded our expectations. The wave vector resolution obtainable with high energy x-rays is far superior to that available with either neutron or ~10 keV x-rays. The intensity of the scattering from the charge ordering was much higher than that observed at more conventional wavelengths. In part, this is due to the increased scattering cross section, however, the major factor is the dramatically increased penetration depth of the whole crystal leading to an increase in the sample scattering volume.

These results are the first observation of charge ordering in the bulk of the sample using high-energy x-rays. These results suggest that the charge ordering is a bulk effect with a high correlation length > 400 Å in the bulk of the sample. In our previous measurements on this sample of $Nd_{0.5}Sr_{0.5}MnO_3$ using x-rays with an incident energy of ~10 keV we found that the correlation length of the charge ordering was only ~120 Å. We believe that the smaller correlation length in the near surface region could be due to surface defects, dislocations etc. induced in the sample.

In summary, our first attempt to observe charge ordering was entirely successful and demonstrates that high-energy x-ray scattering is a technique of major importance in studies of charge ordering.

[1] D.I. Khomskii & G.A. Sawatzky, Solid State Commun., 102, 87(1997).
[2] A.P. Ramirez, J. Phys. Cond. Matt., 9, 8171 (1997).
[3] H. Kuwahara, et. al., Science, 270, 961 (1995); H. Kawano, et. al., Phys. Rev. Lett.,78, 4253.