ESRF	**Experiment title:** **Investigation of charge stripe annealing in bulk single crystal CMR oxides**	**Experiment number**: HE-903
Beamline: ID15A	**Date of experiment**: from: 06/12/00 to: 12/12/00	**Date of report**: 12/03/02
Shifts: 18	**Local contact(s)**: Thierry d'Almeida	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

S.B. Wilkins[1]*, P.D.Spencer[1]*,, P.D. Hatton[1]*, S-W Cheong[2].

1) Department of Physics, Science Labs, University of Durham, South Road, Durham, DH1 3LE, UK
2) 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 electrical and magnetic properties of the transition-metal oxides.[1] The perovskite manganites have been of great interest recently due to their extraordinary physical properties, especially colossal magnetoresistance (CMR).[2]

We have carried out high energy X-ray scattering studies at ID15A on a single crystal sample of $Bi_{0.24}Ca_{0.76}MnO_3$ grown at Bell laboratories. By probing with high energy X-rays there is a dramatic increase in penetration depth that allows bulk sensitive measurements to be taken with an increase in wavevector resolution, due to the decrease in Darwin width of reflections. Additionally, the greater sample volume probed results in an increase in scattering intensity allowing for the study of weak scattering phenomena. $BiMnO_3$ is a paramagnetic insulator but when Ca is doped into the system to a value of $x = 0.76$ it undergoes a transition into a charge ordered state at $T_{CO} = 240$ K.[3]

The sample of $Bi_{0.24}Ca_{0.76}MnO_3$ was mounted on the triple axis, 4-circle, high-energy diffractometer located on beamline ID15-A. The incident white beam was first partially monochromated by a two bent silicon crystals, in transmission, in the optics hutch. Upon passing into the experimental hutch the beam was then monochromated by a silicon (1,1,3) crystal in transmission. The sample was mounted in a He bath cryostat mounted on a cross tilt stage and the temperature was controlled to an accuracy of 0.1 K. An additional silicon (1,1,3) crystal was used as an analyser, and higher order wavelength harmonics were removed by the use of a Ge solid-state, energy gated detector. The sample was mounted with the

(1,1,0) direction normal to the scattering plane, with the (1,–1,0) direction along the incident beam direction.

The sample was cooled and satellites were located around the (2 2 0) Bragg peak found at a modulation of (0.24, 0, 0) corresponding to structural modulations associated with the Jahn-Teller ordering. Satellites from the true charge order and harmonics from the structural modulation were also located. Measurements were carried out on the structural modulation, charge order and harmonics from 5 K to beyond the transition temperature.

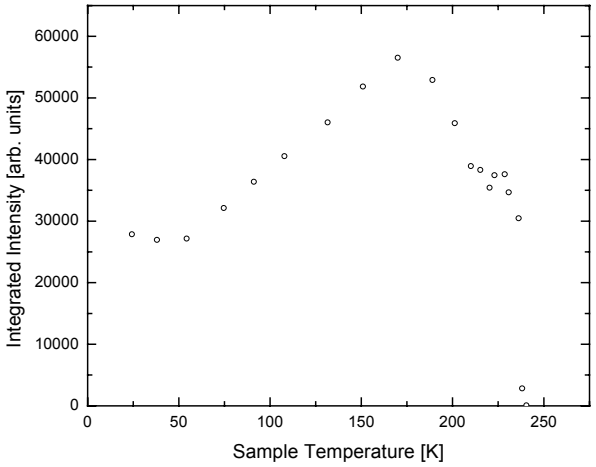

Fig 1 Temp. dependence of (2, 1.75, 0) distortion

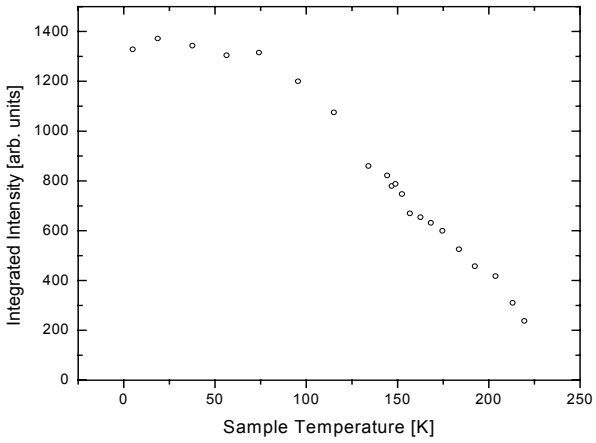

Fig 2 Temp. dependence of (2, 1.5, 0) CO Peak

Figure 1 shows the integrated intensity of the Jahn-Teller distortiont (2.24, 2, 0) as a function of temperature. Upon heating the integrated intensity of the distortion increases until a maximum is reached at the Néel temperature. After this point the intensity drops steadily until the T_{CO} where the sample undergoes a first order structural transition and the Jahn-Teller distortion disappears. The width of the Jahn-Teller distortion was found to be similar to that of the (2 2 0) Bragg peak indicating that structural modulation is as long-range correlated as the lattice. The satellites corresponding to the charge ordering of the Mn^{3+} and Mn^{4+} were located at a modulation of (0.5, 0, 0) around the (2 2 0) Bragg peak. The charge order like the distortion was found to have a width similar to the Bragg peak, and hence it is also as well long-range correlated as the lattice. The temperature dependence of the charge order peak is shown in Figure 2 and the integrated intensity displays a second order like behavior with respect to temperature and like the distortion also disappears at 240 K where the sample undergoes a structural transition.

The results show that both the charge ordering and the structural modulation associated with the Jahn-Teller distortion are bulk effects in the manganites. The high resolution available has shown that the structural modulation has a distinct first-order behaviour with respect to temperature while the charge order has second-order type behaviour. The charge and structural modulation are both as well long-range correlated as the lattice.

This work together with other results obtained on BM28 is being prepared for publication in an internationally recognised referred journal.

[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] Y.Su, C. H. Du, P. D. Hatton, et al., Physical Review B-Condensed Matter **59**, 11687 (1999).