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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Charge Ordered Oxides	**Experiment number**: HE-1628
Beamline: ID31	**Date of experiment**: from: 5/9/03 to: 8/9/03	**Date of report**:
Shifts: 9	**Local contact(s)**: Dr Francois Fauth	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. J.P. Attfield, CSEC, University of Edinburgh

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Dr. J.P. Wright*, ESRF

Report:

Charge ordering (CO) transitions, at which a long range order of two different metal valence states occurs, is of much current interest in solid state physics and chemistry. Much of this interest has been stimulated by work on doped manganite perovskites such as the $Pr_{1-x}Ca_xMnO_3$ system, but there are also many other charge ordered oxides such as the $YBaM_2O_5$ (M= Mn,Co, Fe) materials, in which CO is observed for all three metals. Proof of long range CO comes from a reliable structure determination, but this is often made difficult by the subtle symmetry-lowering distortions that usually accompany the CO transition. This can lead to microtwinning which makes single crystal studies difficult, so high resolution powder diffraction offers some advantages. A joint refinement with X-ray and neutron powder diffraction combines the advantantages of the two techniques, as we have shown in our

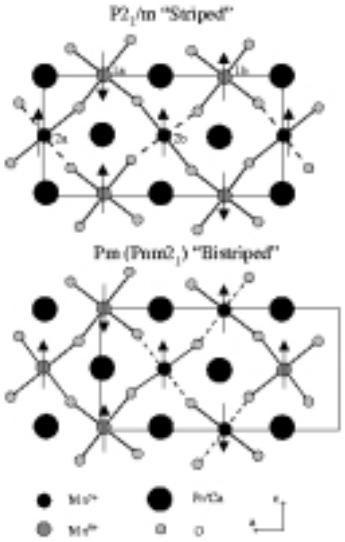
recent determination of CO in Fe_3O_4 below the Verwey transition [1,2], which has been an outstanding problem for more than 60 years.

Experimental Results

1. $Pr_{1-x}Ca_xMnO_3$

Powder diffraction patterns were measured at 180 K and 10 K on both $Pr_{0.5}Ca_{0.5}MnO_3$ and $Pr_{0.5}Ca_{0.5}MnO_3$ using a wavelength of 0.5889 Å with a maximum 2θ of 70°. The four diffraction patterns were collected for about six hours each with longer counting times at higher angles. The samples were held in a 0.4 mm capillary while the measurements were taken.

The low temperature charge ordered structure was determined in $Pr_{0.5}Ca_{0.5}MnO_3$ at 10 K using combined refinements of X-ray and powder neutron diffraction data. The neutron diffraction data was obtained using HRPD, ISIS Facility. We are currently testing the two proposed crystallographic models, $P2_1/m$ and Pm with $Pnm2_1$ pseudosymmetry. These models are shown in the figure, the Mn-O bonds along the elongated dz^2 orbital are dotted and arrows show the direction of the magnetic moments.



Preliminary refinements indicate that the $P2_1/m$ Goodenough model of charge and orbital gives the best fit to the powder diffraction patterns.

2. Fe_3O_4

The powder diffraction pattern was measured using a wavelength of 1.8049 Å with a maximum 2θ of 130° at 90 K. The sample geometry used was half-annular and the diffraction pattern was measured for eight hours with longer counting times at higher angles. The diffraction pattern measured at a wavelength of 1.8 Å is being used in a combined refinement with the previously measured pattern at 0.5 Å and the neutron diffraction pattern measured on HRPD, ISIS Facility. This data gives a better resolution of the monoclinic splitting of the lattice enabling the orthorhombic Pmca symmetry constraints that were used in previous refinements [1,2] to be released and the structure refined in P2/c.

References

1 Wright J P, Attfield J P, Radaelli P G, Phys Rev Lett 87, 266401 (2001)

2 Wright J P, Attfield J P, Radaelli P G, Phys Rev B 66, 214422 (2002)