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:** High Energy X-Ray Scattering studies of the layered Ruthenates	**Experiment number**: HE-1359
Beamline: ID15A	**Date of experiment**: from: 5/2/2003 to: 10/2/2003	**Date of report**: 28/08/2003
Shifts: 18	**Local contact(s)**: T. d'Almedia	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

In this project we intended to study the layered ruthenate system $Ca_{2-x}Sr_xRuO4$. The system shows a remarkable range of physical properties dependent on the doping level, with the end member Sr_2RuO_4 exhibiting p wave superconductivity below 2 K and is the only known superconductor isostructural to the cuprates[1, 2]. The substitution of Ca for Sr drastically changes the properties of the system and the other end member Ca_2RuO_4 is a Mott Insulator.

In this study we intended to use high energy x-ray scattering studies to firstly investigate the doping regions $0 < x < 0.15$ where the system displays a metal - insulator transition with a huge change in resistivity and a significant change in the structure of the lattice. Secondly, the $x = 0.2$ and $x = 0.5$ samples would be studied, at these doping levels there are significant changes in the properties of the ruthenate system system , $x = 0.2$ the system enters a metal magnetic state and no longer displays the metal-insulator transition, and at $x = 0.5$ there is a change to a paramagnetic state and the magnetic susceptibility has been observed to maximise. Neutron diffraction studied have reported a structurally forbidden reflection that is associated with the $x = 0.5$ and $x =0.2$ samples[3]. We intended to study the lattice distortions at a number of doping levels to see how they are related to the system physical properties.

The crystal were studied at station ID15A using x-rays with an energy of 100 keV. Two silicon (113) crystals were used as the monochromator and analyser to provide high resolution triple axis measurements. The crystals were mounted with the <100> direction normal to the scattering vector and the <001> direction down the beam. The crystals were of a very high quality with a rocking curve width of 0.01°.

The crystals in the doping region $x < 0.15$ undergo a huge change in size of the unit cell and as a result need to be cooled at a rate of 1 K per minute, otherwise the crystal will shatter. Even with this process it is not always possible to get through the transition and during the experiment the sample shattered so we were unable to obtain any results from the doping range $0 < x < 0.15$ even by utilising a cooling rate two times slower that recommended in the literature.

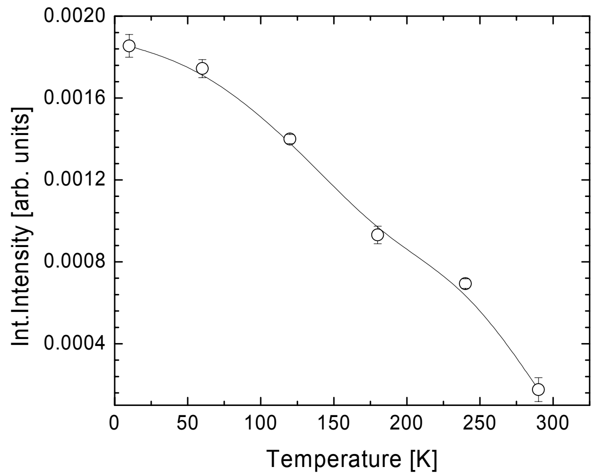

Figure 1 Integrated intensity of the (3, 0, 4) reflection in the $x = 0.2$ doped sample

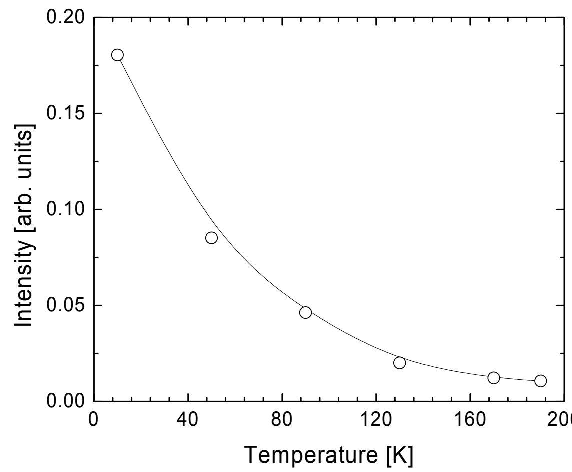

Figure 2 Integrated intensity of the (3, 0, 4) reflection in the $x = 0.5$ doped sample

We studied the $x = 0.2$ and $x = 0.5$ samples and found the structurally forbidden reflection (3, 0, 4) associated with the tilt and rotation of the octahedra around the Ru ions. In the $x = 0.2$ sample this reflection was found to be very strong and as correlated as the lattice, and it was observable up to tempeartures over 300 K and the temperature dependence is shown in Figure 1. The peak broadened as it weakened and it underwent a 2^{nd} order transition above 300 K. In the $x = 0.5$ the (3, 0, 4) reflection was 2 orders of magnitude weaker and it was much broader and hence, less correlated than the lattice and the temperature dependence is shown in Figure 2. It was found to be 2 dimensional in nature with it being relatively poorly correlated between planes. On heating it showed a 2^{nd} order transition and broadened and became less correlated as it approached the transition temperature of 190 K. Due to the superior intensity of ID15A we were able to observe it up to 190 K where previous studies neutron could only observe it up to 65 K due to having less available intensity. What we concluded from this is that the $x = 0.5$ structure is much less distorted than the $x = 0.2$ and the distortion is only present in low temperatures in the $x = 0.5$ sample and there is as yet undetermined relationship between this distortion and the physical and magnetic properties of the sample.

Overall, the results on the $x = 0.2$ and $x = 0.5$ samples shows there is a link between the rotation and tilt of the octahedra and the physical properties shown by the system and it is hoped combing these results with additional transport measurements will explain the link between the forbidden reflections and the physical properties.

1 S. Nakatsuji and Y. Maeno, Physical Review Letters **84**, 2666 (2000).

2 G. Cao, S. McCall, M. Shepard, *et al.*, Physical Review B **56**, R2916 (1997)

3 O. Friedt, M. Braden, G. Andre, *et al.*, Physical Review B **6317**, 4432 (2001)