ESRF	**Experiment title:** Structure and superconductivity in $SrRu_2GdCu_2O_8$	**Experiment number**: HE-1412
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Report:

All studies to date suggest that the compound $RuSr_2GdCu_2O_8$ (Ru-1212) exhibits the coexistence of bulk ferromagnetism and superconductivity. This coexistence is unexpected because the magnetic field would be expected to break Cooper pairs. The crystal structure is essentially that of the well-known layered cuprate $YBa_2Cu_3O_7$ with RuO_6 octahedra replacing the "chain" copper atoms and their associated oxygen neighbours. The ferromagnetic moment arises from a small canting of antiferromagnetically ordered Ru spins and the superconductivity arises in the CuO_2 planes, as in other layered cuprate superconductors. However, the superconducting behaviour presents a perplexing question. Samples can be changed from superconducting (SC) to non-superconducting (non-SC) through the choice of annealing conditions. In early work, the effects of annealing were poorly understood, but we now know how to reproducibly make superconducting or non-superconducting samples. No changes in composition, in particular no changes in the oxygen content, can be seen when comparing SC and non-SC samples. The aim of our experiment on ID31 was to search for and determine any differences in the crystal chemistry or microstructure of the two samples.

We carried out measurements on samples of SC and non-SC Ru-1212 using an X-ray energy of 45.2 keV. The samples were packed in 0.6 mm diameter capillaries and data sets were collected over the angular range 1° to 30° at 8 different temperatures between 80 K and ambient temperature using a cryogenic nitrogen blower for cooling. Further data sets were collected between 10 K and 80 K using a liquid-helium-cooled cryostat. We found that good statistics were obtained using data collection times of between 30 and 90 minutes per temperature step.

We had previously carried out a neutron diffraction study on a different sample of SC Ru-1212 that suggested the structure is a $\sqrt{2}a \times \sqrt{2}a \times c$ supercell of the “123” structure, a doubling of the cell-volume resulting from correlated rotations of the RuO_6 octahedra around the *c* axis. However, our investigation on ID31 detected no such superstructure for either sample, and Rietveld refinements were successfully performed at all

temperatures using the space group *P4/mmm* with $a = b \approx 3.83$ Å, $c \approx 11.56$ Å. We carried out repeated scans across short two-theta regions in which superstructure reflections were expected to be present, but no signals were detected for either sample. It appears that formation of the superstructure is extremely sensitive to synthesis conditions.

One significant difference between the SC and non-SC samples was found to be in the lattice parameters; the ratio of *c*/*a* (Figure 1) was greater for the SC sample, which by analogy with $YBa_2Cu_3O_7$ might be consistent with doping, perhaps involving tiny differences in oxygen content. Carrying out Rietveld refinements and allowing the fractional occupancies of the oxygen sites to vary did not reveal any difference between the samples. However, errors are expected to be of the order of at least 1% and it is possible that a difference in oxygen content of less than 1% could still have a noticeable effect on the lattice parameters. Anomalies in the *c*/*a* ratio can be seen for both samples near the magnetic ordering temperature (~140 K).

Another difference between the samples was found in the peak widths. The peaks of the non-SC sample were significantly broader, and Williamson-Hall plots for both samples revealed that the strain, especially in the [001] direction, is greater for the non-SC sample. This is perhaps indicative of a greater concentration of disorder such as stacking faults in the non-SC sample, which could have an adverse effect on superconductivity.

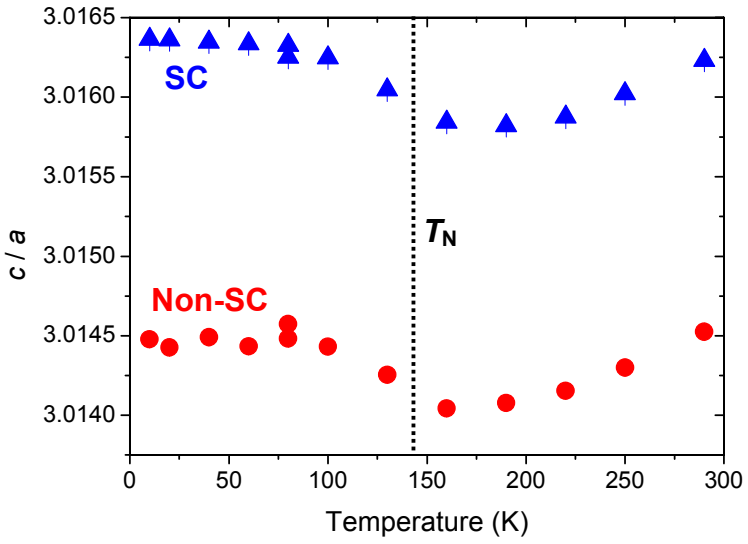

Figure 1: Ratio of lattice parameters *c*/*a* as a function of temperature for SC and non-SC samples. The magnetic ordering temperature T_N is indicated.