

**Experiment title:**Phase transformation under pressure in $\text{Sr}_{14}\text{Cu}_{24}\text{O}_{41}$ type compounds**Experiment number:**

HS-348

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ID09

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12

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Report:

Following the discovery of high- T_c superconducting cuprates, the theoretical investigation of the magnetic and transport properties in low-dimensional copper oxide systems has become increasingly active. Recently, the so-called "spin-ladder" systems have drawn much of the attention, due to their predicted striking properties. Spin ladders are quasi-one-dimensional objects resulting from the coupling by corner-sharing of parallel and infinite chains of CuO_4 squares (the ladder legs).

Superconductivity was detected for the first time in a compound containing spin ladders by Uehara et al., who measured the resistivity as function of pressure and temperature in the $(\text{Sr}_{14-x}\text{Ca}_x)\text{Cu}_{24}\text{O}_{41}$ compound. These authors reported the appearance of a superconducting transition in the 3-5GPa range, with a maximum transition temperature of about 12K, in samples with a large calcium concentration ($x=13.6$). The structure of $(\text{Sr}_{14-x}\text{Ca}_x)\text{Cu}_{24}\text{O}_{41}$ can be described as the alternate stacking of layers having the two-leg-ladder arrangement and Cu_2O_3 composition, and layers made of isolated chains of edge-sharing CuO_4 squares, separated by planes containing the alkaline earth cations. This arrangement leads to a composite type of structure, which in a first approximation can be described by using two orthorhombic unit cells with parameters : $a_1 \approx 11.4\text{\AA}$, $b_1 \approx 13.0\text{\AA}$ and $c_1 \approx 2.75\text{\AA}$ for the $\text{Sr}(\text{Cu}_2\text{O}_3)\text{Sr}$ ladder containing slabs, and $a_c = a_1$, $b_c = b_1$ and $c_c \approx 3.92\text{\AA} \approx 10/7 \times c_1$ for the chain layers. The a direction is in the plane of, and perpendicular to the chains and ladders ; the b

direction corresponds to the stacking axis and the c direction is along the chains and ladders.

The present experiment aimed at determining the evolution of the structure as function of pressure and size/charge of the mixed cationic site. For this purpose, five different samples of $(\text{Sr}_{14-x}, \text{M}_x)\text{Cu}_{24}\text{O}_{41}$ general formula were used for the experiment : $x=0$; $\text{M}=\text{Ca}$, $x=6$; $\text{M}=\text{Ca}$, $x=13.6$ (superconducting composition) ; $\text{M}=\text{Ba}$, $x=4$; $\text{M}=\text{Nd}$, $x=6$. The experiment was carried out up to $\approx 10\text{GPa}$ at the ID09 beam line with $\lambda \approx 0.46\text{\AA}$, using the angle dispersive geometry and imaging plate detector. High pressures were obtained by using a DAC, with silicon oil or methanol/ethanol as pressure transmitter medium, and monitored using the ruby fluorescence. The evolution of the cell parameters was determined by following the angular positions of few, well separated Bragg reflections, fitted using pseudo-Voigt line shape. All samples display similar behaviors. The compressibility is strongly anisotropic, with the easiest compression along the stacking direction (b axis), and the hardest one along the a direction.

The most striking feature observed is the presence of a phase transition in the 6-8 GPa range for all samples, consisting in a strong increase of the a parameter value with increasing pressure. The pressure value at which the transition occurs seems to increase with increasing calcium content (and thus decreasing size of the mixed cationic site). This behavior was confirmed for the two pressure transmitting media used, and cannot be attributed to an experimental artefact. Though the observation of the diffraction spectra indicate that the global structural arrangement is conserved through the transition, this lattice anomaly which occurs around pressures where the superconducting state disappears, may be highly relevant for the understanding of the physical properties of this class of materials. Precise low temperature-high pressure diffraction experiments would be of the greatest interest to investigate further the relations between the superconducting state and this structural phase transition.