ESRF	**Experiment title:** High pressure synchrotron powder X-ray diffraction studies of the high temperature superconductor $HgBa_2CuO_{4+\delta}$	**Experiment number**: HS-951
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Shifts: 6	**Local contact(s)**: Dr. M. Hanfland	*Received at ESRF:*

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Report:

Introduction to and aims of this study

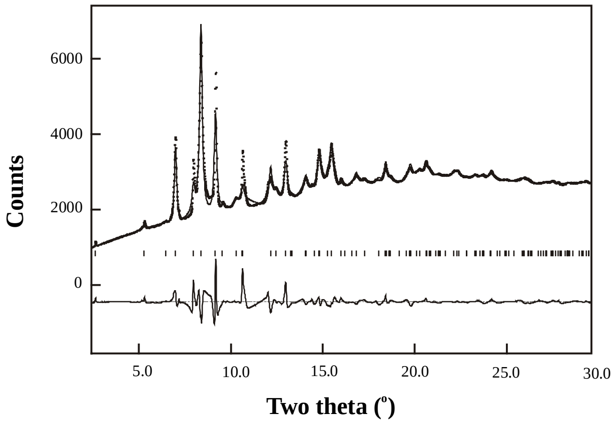
The high temperature superconductor $HgBa_2CuO_{4+\delta}$ (T_c = 96K) is the first member of a homologous series of mercury based superconductors with the general formula $HgBa_2Ca_{n-1}Cu_nO_{2n+2+\delta}$. In this family of superconductors, T_c reaches record high temperatures for the n = 3 member, with T_c = 135K at room pressure increasing to 164K at 31GPa. T_c is observed to increase with applied pressure in all members of this homologous series, plateauing at some maximum value (depending on n), before decreasing. Recently, evidence for reversible phase transitions in $HgBa_2CaCu_2O_{6+\delta}$ and $HgBa_2Ca_2Cu_3O_{8+\delta}$, at pressures of 24GPa and 15GPa respectively, were reported [1]. These transitions appeared to be coincident with clear changes in the pressure derivative of T_c in these materials. The purpose of this study was to examine the structural effect of hydrostatic pressure in the simplest member of the mercury based homologous series, $HgBa_2CuO_{4+\delta}$, with the aim of confirming the presence or absence of a similar phase transition near the observed change in the pressure derivative of T_c (at ~10GPa) in this material. Our previous high pressure diffraction studies of $HgBa_2CuO_{4+\delta}$ (carried out at NSLS and ISIS to 8GPa) had suggested a possible phase transition in this material near pressures of ~8GPa. However, it was not possible to fully clarify these observations due to the pressure limitations (<10GPa) and the development of nonhydrostatic conditions (above 8GPa) in these experiments.

Experimental

High purity polycrystalline $HgBa_2CuO_{4+\delta}$ was loaded into a beryllium backed diamond anvil cell, together with a number of ruby chips and helium as a pressure transmission medium (to ensure a hydrostatic sample environment) at the University of Oxford. Powder diffraction patterns were acquired using an Image Plate system with monochromatic X-radiation of wavelength 0.4362Å. Wavelength and sample-plate distance calibrations were obtained from analysis of the diffraction pattern of a silicon standard. Spatial distortion and tilt corrections and integration of the Debye-Scherrer rings was accomplished using the Fit 2D program. Rietveld profile analysis of selected diffraction patterns was achieved using the GSAS suite of programs.

Results and discussion

Rietveld profile analysis of powder diffraction data acquired below ~8GPa was successfully accomplished. A typical profile fit (for data acquired at 1.95GPa) is shown in Figure 1. As expected, the compressibility of $HgBa_2CuO_{4+\delta}$ was found to be highly anisotropic with the compression parallel to the *c*-axis being much greater than that in the *ab*-plane. The principal effect of high pressure is to compress the apical Cu-O bond, as was anticipated, since at room pressure this bond is rather long (2.769Å). Significant anisotropic broadening of certain diffraction profiles was observed in diffraction patterns acquired at 7.80GPa and above, as shown in Figure 2. In addition, splittings of certain diffraction reflections were observed, for example the (101), (102) and (110) reflections, see Figure 2. It would therefore appear that $HgBa_2CuO_{4+\delta}$ undergoes a rather complex phase transition in the region of ~8.0GPa, near to the observed change in the pressure derivative of T_c in this material. The rather broad nature of the diffraction profiles has made accurate indexing of these high pressure diffraction patterns rather difficult, although this work is still at a preliminary stage. The observed broadening of the diffraction profiles cannot be attributed to the development of nonhydrostatic conditions within the sample chamber, since the ruby fluorescence lines were observed to remain sharp to the highest pressure examined (~30GPa). The observed phase transition, appears reversible, as shown by the data acquired for the decompression cycle, Figure 2.



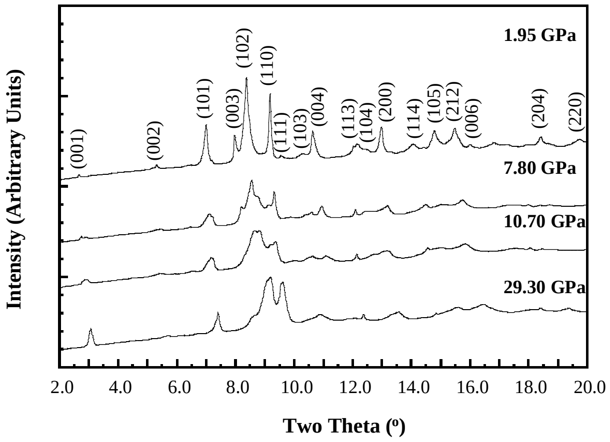


Figure 2

Reference: [1] R. Gatt *et al*, *Phys. Rev. B.*, 57, 13922 (1998).