ESRF

Experiment title:
The interplay between the order-disorder and displacive character of the ortho-I to ortho-II phase transition in $YBa_2Cu_3O_{6+x}$

Experiment number:
HC 256

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Report:

The cell-doubled ortho-II phase is a predominant structural feature of the high temperature superconductor $YBa_2Cu_3O_{6+x}$. It is characterized by short range ordered alternating full and empty Cu-O chains in the basal plane, and associated atomic displacements of surrounding atoms, giving rise to strongly modulated diffuse superstructure peaks. The issue addressed in this experiment was of the connection between the order-disorder and displacive character of the transition from the ortho-I to the ortho-II phase.

The appearance of atomic displacements associated with the oxygen ordering suggests the possibility of phonon soft-mode character which one can attempt to investigate indirectly by separating out the different order parameters (oxygen ordering and atomic displacements). Previous neutron and x-ray investigations had only determined the occupational and displacive parameters at room temperature. By systematically studying this vs temperature, a separation of these effects is possible and one could check for any continuous evolution of the displacements.

A 2 mg single crystal of $YBa_2Cu_3O_{6.5}$ was mounted in a specially designed small oven. The diffractometer was used in 2-axis mode with the small Huber 4-circle mounted at the sample position. 100 keV x-rays were selected using an imperfect Si monochromator. The beam was collimated to illuminate a 1 mm vertical and 0.5 mm horizontal section of the crystal. At various temperatures between 25 and 180 "C, l-scans (5 sec/pt, 40 pts per superstructure) were made along the (2.5,0,1), (3.5,0,1), (4.5,0,1), and (5.5,0,1) with the a and c axes lying in the scattering plane. Unfortunately, the Eulerian cradle was severly restricted in movement to about $\pm$ 10°, so that only a small no. of peaks were accessible (23 in total). The background was determined by a scan out of the k=0 plane (about k = 0.2), accomplished by a small χ rotation which preserves the scattering geometry and minimizes changes in absorption. (0.5,0,1) and (1.5,0,1) series peaks were not measured due to the high remnant background coming from the (shielded) straight through beam. It should be noted that the slow w movement took up more than 50% of the beam time.

The scans at each temperature were fitted to an ortho-II structural model, where the average structural parameters of the ortho-I cell are fixed to those refined by H. Casalta et al. (Physics C, 258, 321, (1996)). The allowed atomic displacements of the cell doubled phase, the line width, and the overall scale factor were determined from the x-ray data by assuming a Lorentzian line shape and a modified structure factor arising from the atomic displacements.

Within the experimental error, the fits show no systematic variation of the atomic displacements vs 2'; they remain essentially equal to the room temperature displacements already refined in a previous study of our crystals (R. Hadfield et al., Physics C 235-240, 1267 (1994)). Qualitatively, one can see this in Fig. 1, where there is essentially no renormalization of one superstructure peak with respect to another vs T. If there are any trends, then these could only appear above 1600 C, where our data is of insufficient quality to draw any conclusions.

Figure 2 shows the T dependence of the scale factor and the line width. Above 150 "C, the data are pretty lousy, so no significance should be given to the value of the scale factor at 180 ^{0}C. The interpretation of the T dependence of the scale factor is probably impossible, since this quantity is very sensitive to the crystal alignment; reproducibility scans showed that the χ and w motors lost steps during course of the experiment.

The determination of the atomic displacements is much less sensitive to this. Thus, it seems reasonable to conclude that the atomic displacements are soley determined by the local oxygen arrangement and there is probably no significant displacive character to the transition. To come to a definitive conclusion, a future experiment would at least require a much larger span for the ω motor to access many more peaks and search for Debye-Waller effects.

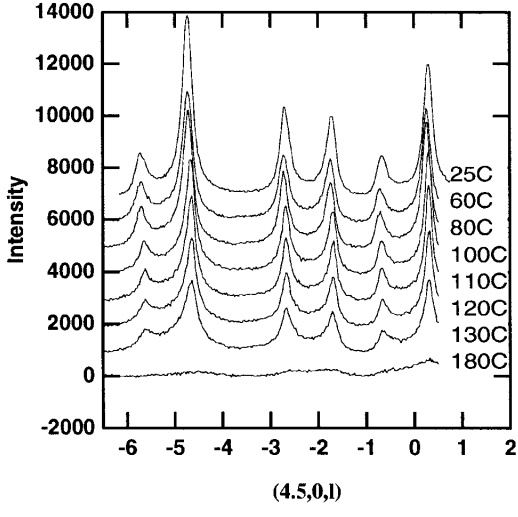

Fig. 1 l-scan along (4.5,0,l) at various temperatures.

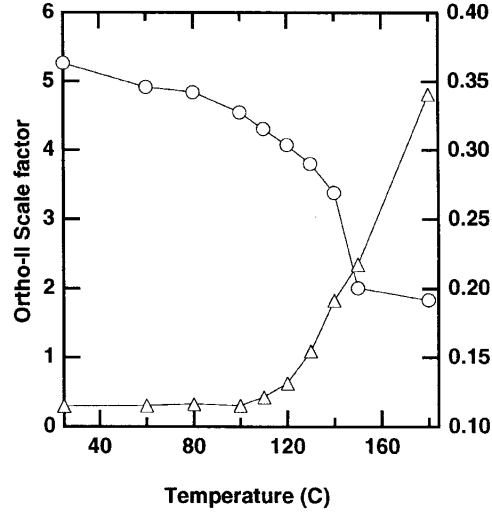

Fig. 2 Ortho-II scale factor (circles) and line width (triangles) vs Temperature.