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Experiment title: Structure of the collapsed $Bi_{6+x}Sr_{9-x}Fe_5O_{26}$, related to modulated compounds $Bi_{2.4}Sr_{2.6}Fe_2O_{9+\delta}$ and $Bi_2Sr_2CaCu_2O_{8+\delta}$.

Experiment number: HC255

Beamline:	**Date of Experiment:**	**Date of Report:**
ID11-BL2	from: 27/09/95 to: 30/09/95	17/09/96
Shifts:	**Local contact (s):**	*Received* at ***ESRF:***
9	Heinz Graafsma	20 SEP 1996

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Report:

The Bi-Sr-Cu-O, Bi-(Ba,Sr)-Cu-O and Bi-Sr-Fe-O systems are very well known for the existence of the incommensurately (or commensurately) modulated phases. But, lately, in these systems, new periodic phases, so called collapsed phases, have been also evidenced and characterized only by X-ray powder diffraction or HREM studies, so that their actual and original structure was not already established. Structural similarities with the modulated phases are expected and then require to be specified.

Some structural models, based on microscopy studies, have been proposed. The different authors refer to the related modulated compounds and the correspondences between the two types of structure are interpreted calling up to shearing mechanisms. But the structure of the shearing zone was not still refined. For the first time, a single crystal of the double collapsed phase $Bi_{6+x}Sr_{9-x}Fe_5O_{26}$ was synthesized and allows us to refine the structure. The study of the double collapsed phase $Bi_{6+x}Sr_{9-x}Fe_5O_{26}$, related to the 2212 modulated phase was carried out from a single crystal having dimensions of 18x18x $360\mu m^3$. The tryst al has the monoclinic space group $P2_1/n$ and the unit cell parameters a=16.491(9) Å, b=5.481(3) Å, c=30.086(16) Å, α = 90°, β = 91.39 (2)°, γ = 90°. This crystal is obviously characterized by a strong lattice translational pseudo-symmetry ; as a matter of fact two sets of reflections hkl are observed : with strong intensities for h+l=2n, with weak to medium intensities for h+l=2n+l, suggesting a pseudo B lattice. An electronic diffraction pattern (Fig. 1) shows both weak spots and intense spots.

In order to allow a satisfactory refinement of the structure in the primitive lattice we used synchrotrons radiation.

Data were recorded with a Thomson X-ray image intensifier coupled to a Pricenton slow scan CCD camera. Data were collected in oscillations of 1 degrees with 0.2 degree overlap. Images were corrected using FIT2D and integrated with DENZO resulting in a data set of 2674 reflections (against 1840 for standard data collection).

The structure was solved by direct methods and refined to a final reliability factor of 0.068. The structure of the previously so called shearing zone could be established with accuracy. A projection along the b axis showing the arrangement of the cations in the collapsed phase was given in Fig.2. Thanks to these new results we could determine that a description of this compound from the incommensurate one by a shearing mechanism is not a satisfactory way. Indeed, it appear necessary to consider a more complex relation between the modulated and the collapsed phases.

The existence of this new model should allow to revise the different collapsed phases previously investigated by electron microscopy.

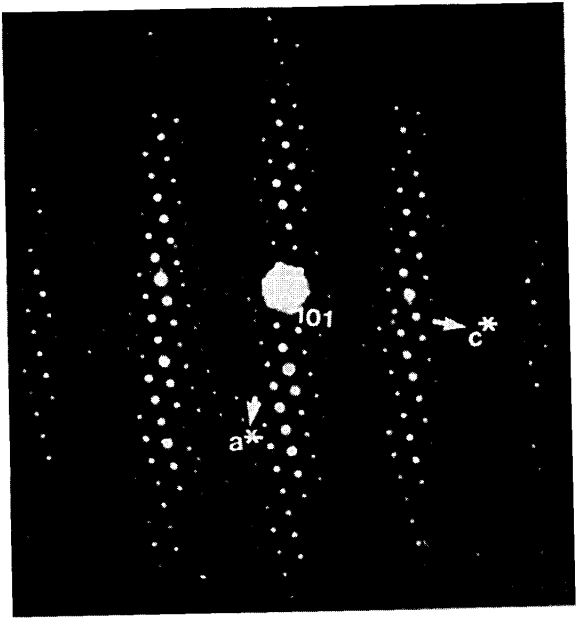

Fig.1

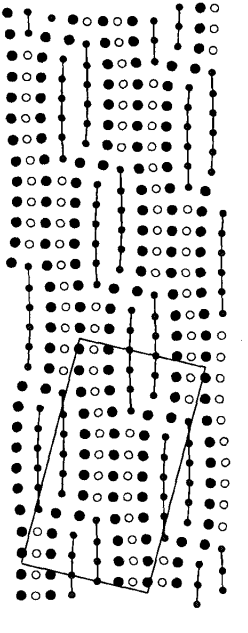

Fig.2