ESRF	**Experiment title:** Powder diffraction study of the incommensurate modulated ferroelastic phase of α lead monoxide	**Experiment number:** CH 181
Beamline: D2am	**Date of Experiment:** from: December 6th 96 to: December 9^{th} 96	**Date of Report:** July, 6th 97
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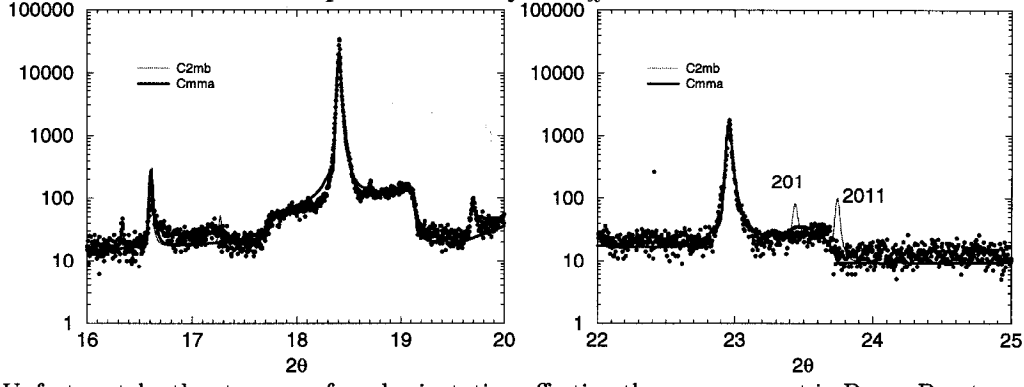
Report:

One entire shift was used to check the alignement of beam, calibrate the monochromator (λ = 0.992 A), set up the closed cycle cryostat and install the specimen.
Two complete diffraction patterns have been recorded in the same experimental conditions: the former at 25 K, the lowest temperature we were able to obtain in the cryostat, the other one at room temperature. The diagrams were collected in the range $10^0 < 2\theta < 70^0$ with step scans of 0.003. It is well known that lead monoxide is very sensitive to carbonates, water adsorption and strain. All these factors can induce the undesired phase transition $\alpha PbO \rightarrow \beta PbO$. Therefore, the preparation of a sample with an even surface for this reflection mode geometry was very painful. The analysis of the main peaks showed that the sample was almost single phased with less than 0.5% of Pb_3O_4. *No* trace of βPbO was detected in the sample. The analysis of the recorded patterns confirms the good crystalline quality of the sample; because of the orthorhombic distorsion and of the lamellar structure characterizing this compound, the profile of the diffraction peaks is anisotropically broadened. In the low temperature diagram, the orthorhombic distorsion is well resolved, even at the beginning of the diffraction diagram. The satellite peaks are slightly larger than the average structure reflections, but an unambigous indexing was possible for most of them.
As the major aim of this work was to clarify the four dimensional space group of the incommensurate modulated structure, the comparison between the room temperature diagram and the incommensurate one was necessary to identify unambiguously the diffraction peaks related to the onset of the incommensurate superstructure.

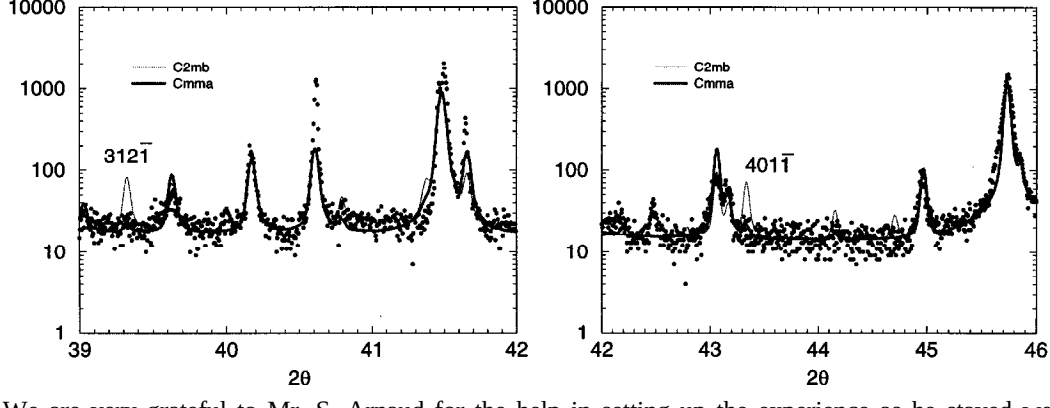
The room temperature and the low temperature diagram were refined using the Rietveld program XND, in order to obtain a very accurate positionning of the satellite peaks. Two model have been tested at present:

- the space group $Cmma$: $s\bar{1}1$ proposed by an electron microscopy study [l]
- the space group $C2mb$: $\bar{1}\bar{1}1$ proposed by previous X-ray and neutron powder diffraction studies [2,3].

As no second order satellite has been observed, the extinctions affecting satellite peaks have been tested only on first order satellites. To push further the analysis, a complete structural determination is needed. The analysis of the two refinements (displayed below) and the comparison with neutron powder diffraction data gives a better agreement for the centrosymmetric space group. It should be underlined that the centrosymmetric group allows the refinement of a very limited number of parameters as most of the modulated displacements are symmetry fixed.



Unfortunately, the strong preferred orientation affecting the measurement in Bragg-Brentano geometry is a major handicap to the improvement of the model. We believe that a new recording using a transmission geometry would avoid this kind of problems.



We are very grateful to Mr. S. Arnaud for the help in setting up the experience as he stayed well after the normal working hours.

References

1 Withers, Shmid, J. Solid State Chem. 113 (1994) 272
2 Moreau, Kiat, Garnier, Calvarin, Phys. Rev. B 39 (1989) 10296
3 Hedoux, Grebille, Garnier, Phys. Rev. B 40 (1989) 10653