

**Experiment title:**

High resolution powder diffraction study of highly correlated electron systems

Experiment number:

HS-3 13

Beamline:

BM16

Date of experiment:

from: 11/6/97 to: 15/6/97

Date of report:

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Shifts:

9

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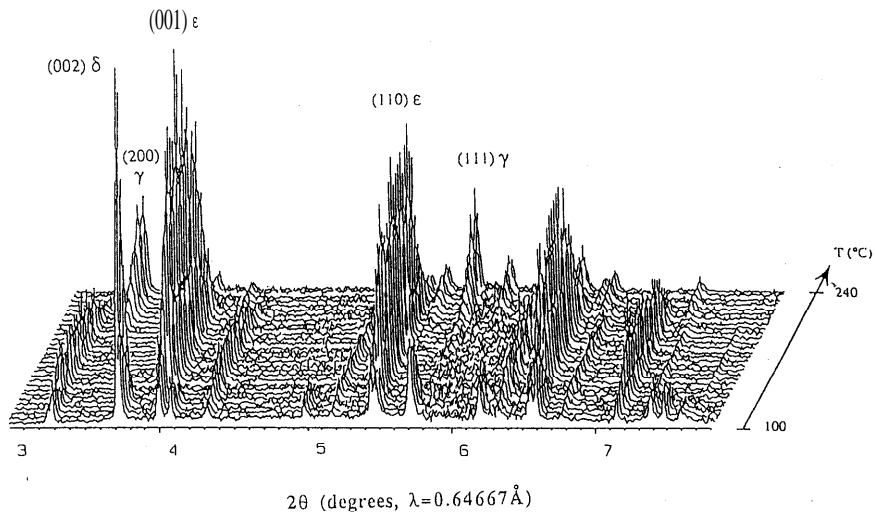
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Report:

The preliminary work plan was modified during our stay at ESRF. It appeared in the literature the single x-ray crystal structure of CaV_2O_5 which gave less interest to look at this compound. However we took the opportunity to use a heating system to study in dynamic the structural transformation of $\delta\text{LiV}_2\text{O}_5$ into $\varepsilon\text{LiV}_2\text{O}_5$ and then into $\gamma\text{LiV}_2\text{O}_5$ with the increase of temperature. This work was very successful and a publication was submitted in january in the Journal of Solid State Chemistry. The main results are summarized in the following abstract:

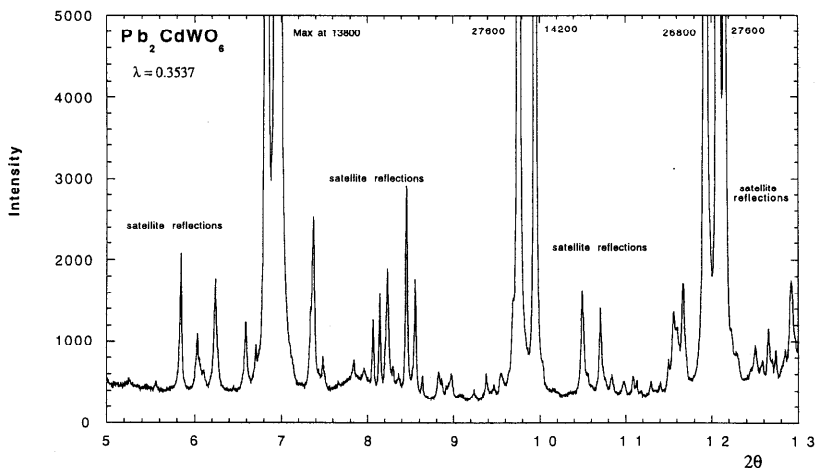
The structural evolution and the domains stability of the $\delta, \varepsilon, \gamma$ phases of the lithium vanadium oxide bronze LiV_2O_5 has been evidenced by synchrotron x-ray powder diffraction in the temperature range 100 - 250°C. It is shown that $\delta\text{LiV}_2\text{O}_5$ is stable below 120°C and that complete transformation in the high temperature form $\gamma\text{LiV}_2\text{O}_5$ is achieved above 220°C. The crystal structure of the intermediate phase $\varepsilon\text{LiV}_2\text{O}_5$ has been determined by Rietveld analysis. It crystallises in the orthorhombic system space group $P2_1mn$ and the cell parameters at 140°C are $a=11.3552(6)\text{\AA}$, $b=3.5732(2)\text{\AA}$ and $c=4.6548(3)\text{\AA}$. The electronic localisation of the vanadium atoms over two different crystallographic sites is found for this phase as for another bronze $\alpha'\text{NaV}_2\text{O}_5$.

The temperature evolution of the x-ray powder patterns of $\delta\text{LiV}_2\text{O}_5$ on heating are presented on this figure.



Concerning the compounds $\text{Bi}_4\text{V}_4\text{O}_{14}$, $\text{Bi}_4\text{V}_8\text{O}_{22}$ and MgV_2O_5 , x-ray powder patterns were collected but they are still to be analysed in view of the recent results obtained in September 97 at the ILL. For MgV_2O_5 additional peaks (small satellites) are present which could indicate the presence of a superstructure.

In the case of the incommensurate phase Pb_2CdWO_6 satellite peaks were clearly identified during this experiment as shown on the following figure.



These data should allow to determine the origin of this modulation.