	Experiment title: Search for the proposed B10 crystal structure replacing the CsCl structure at high pressures in the more ionic III-V semiconductor alloys	**Experiment number**: HS1380
Beamline: ID24 ID30	**Date of experiment**: from: 26/10/00 to: 2/11/00 from: 16/12/00 to: 18/12/00	**Date of report**: 14/2/01
Shifts: 18 on ID24 6 on ID30	**Local contact(s)**: S. Pascarelli on ID24 T. LeBihan on ID30	*Received at ESRF:*

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Report:

We have performed X-ray Absorption and Diffraction measurements at high pressure on samples of powder InAs. The experiment was carried out on ID24 and ID30 for the XAS and XRD measurements respectively. The initial goal was to verify recent theories which predict the instability of the CsCl phase for the more ionic III-V semiconductors. We have examined this prediction for the InAs compound, because it is among the more ionic III-V semiconductors to have the lowest predicted transition pressure to CsCl (P_t ~ 70 GPa). The pressure behaviour of InAs had been previously investigated up to ~ 30 GPa with rather contradictory results even in this relatively "low pressure" range. The system undergoes a first order phase transition at around 7 GPa to a 6-fold coordinated NaCl structure. The pressure evolution of InAs after this first transition is far from being understood.

In our experiment at the ESRF we have reached pressures of 55 GPa and 96 GPa for the XAS and XRD measurements respectively, using a Diamond Anvil Cell (DAC) (see figures 1 and 2 where we plot some of the upward stroke scans). We used double slope diamonds with 150 μm flats for XRD and standard diamonds with 320 μm flats for XAS. Silicone Oil was used as pressure transmitting media. The data is currently being analized but we have done some preliminary qualitative analysis.

Up to now, our XRD data (collected at λ=0.3738 A) shows the following qualitative evolution:

1 – The ambient pressure four-fold coordinated Zincblende (ZB) phase ends at 14 GPa

2 – The six-fold coordinated NaCl phase starts around 7 GPa and seems to disappear between 30 and 40 GPa. No NaCl peaks are present at 96 GPa.

3 - In the pressure region 7 - 14 GPa, there is a coexistence of the ZB and NaCl phases (maybe due to kinetic effects. This has to be checked by repeating the measurements using a heated DAC.)

4 - Between 14-16 GPa, the NaCl cell starts to distort a bit internally (appearance of the 021 and 221 peaks), towards a Cmcm distortion, but the cell remains cubic.

5 - Between 16-18 GPa, the cubic cell starts to transform to orthorhombic (appearance of the triplets 200,020,002 and 220,202,022). This is the Cmcm phase (previously mentioned in the literature on this system).

6 - As pressure increases, the cell tends to higher symmetry, but it is not clear what the high pressure phase is (are).

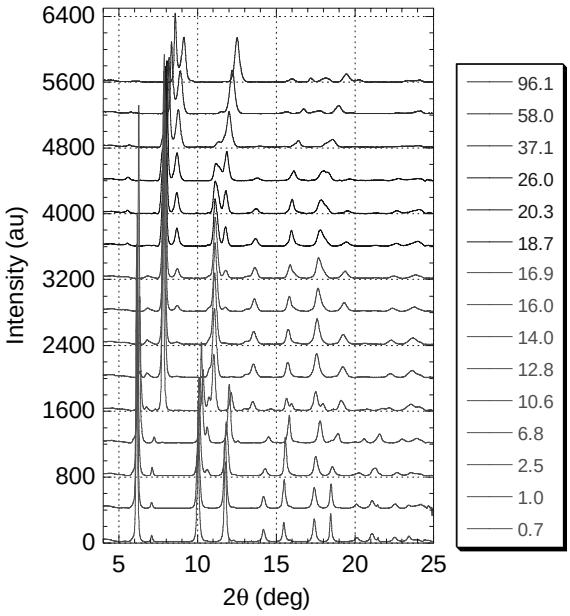

Figure 1: Some XRD profiles collected on ID30 during the upward stroke.

Preliminary 2-shell fits of the As K-edge EXAFS data have been performed, using the ZB and the NaCl model structures only. These fits show:

1 - A clear transition between the semiconducting ZB and the metallic NaCl phase. The transition pressures are in very good agreement with the ID30 XRD experiment.

2 - That the local structure does not seem to change much after that (as expected for second order phase transitions)

The XANES part of the spectra is well known to be more sensitive than the EXAFS signal to structural details of the absorbing site. At the moment, a qualitative analysis shows:

1 - A clear shift of the edge during the semiconductor - metal transition.

2 – That the XANES data is sensitive to both the internal distortion of the NaCl cell and to the orthorhombic distortion of the cell.

3 - The pressure values of the variations of the XANES features agree very well with the XRD data behaviour.

In principle, a complete recovery of the geometrical structure could be obtained from the experimental XANES spectrum. However, the quantitative analysis of this region presents difficulties mainly related to the theoretical approximation in the treatment of the potential and the need for heavy time consuming algorithms to calculate the absorption cross section in the framework of a full multiple scattering approach. We will perform a quantitative analysis of the XANES data using recently developed software (MXAN) that allows to

obtain reliable fitting of the spectra in terms of a well defined set of structural parameters, the initial values of which will be extracted from the XRD data Rietveld refinement.

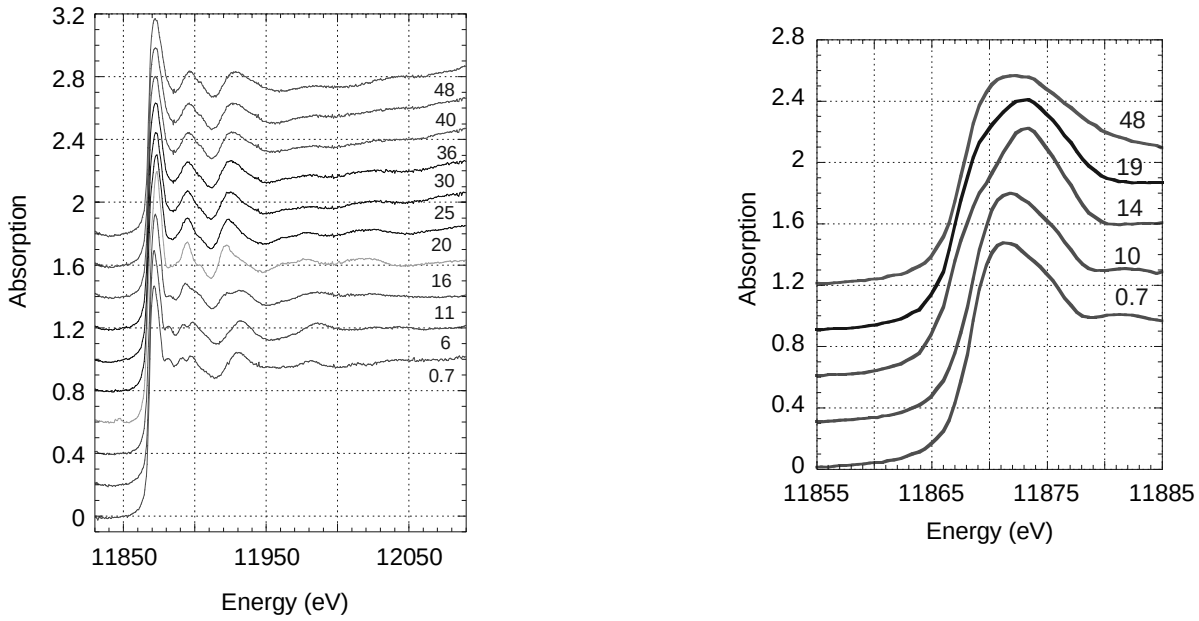

Figure 2: Some of the As-K edge a) EXAFS and b) XANES spectra collected on ID24 during the upward stroke.

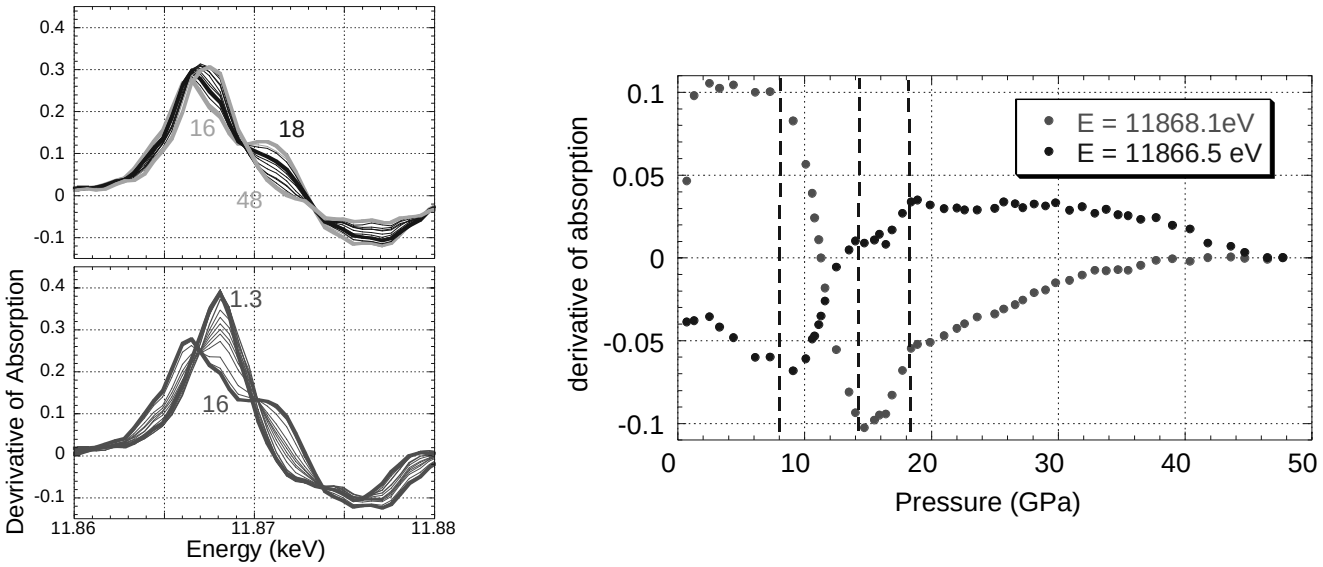

Figure 3: a) Derivative of the XANES spectra during the upward stroke; b)Evolution of XANES features with pressure. Red symbols: maximum of the derivative of the absorption edge at ambient pressure, E= 11868.1 eV; Blue symbols: maximum of the derivative of the absorption edge after the first phase transition, E=11866.5 eV) The semiconductor – metal transition is clearly seen as a shift of the edge towards lower energy (from 11868.1 eV to 11866.5 eV).