

ESRF

Experiment title:
**Pressure tuning absorption spectroscopy
at photon energies $E > 2.8$ keV**

**Experiment
number:**
HC-553

Beamline:
BL6/ID12

Date of experiment:
from: 26 Feb 96 to: 28 Feb 96

Date of report:
21 Feb 97

Shifts:
7

Local contact(s):
J. Goulon

Received at ESRF:
28 FEB. 1997

Names and affiliations of applicants (* indicates experimentalists):

J. Röhler Universität zu Köln, D-50937 Köln , II. Physikalisches Institut

A. Rogalev European Synchrotron Radiation Facility, F-38043 Grenoble

Report:

We have carried out a feasibility study of high pressure XAFS experiments at low photon energies selecting the K-edge of potassium (3607 eV) in KCl. Potassium chloride has at atmospheric pressure the NaCl structure. Each ion is surrounded by six nearest neighbors of the opposite charge. At 19.2 kbar a first order phase transition occurs to the CsCl structure ($\Delta V/V=0.1$) [3], which increases the number of nearest neighbors to 8. and is expected to show up as a significant modification of the NaCl-type XAFS.

A beryllium gasketed anvil cell [2] with sintered diamonds (1.8 mm diamond culet, fully remote controlled under high vacuum) was installed on a special table. The pressure was determined from the load on the anvils using a calibrated load-pressure relation. The absorber (~ 1 mg/cm²) was prepared from a saturated solution of KCl (analytical grade) and inserted into the 50x700 μm^2 sized slit of the beryllium gasket (effective absorption path ≈ 2 mm). The pressure gradient at the sample site was minimized by collimating the beam to 20 μm x 500 μm (VxH) before the I₀ monitor. An auxiliary collimator system in the pressure cell served to align the pressurized sample in the beam. To reject the harmonics the cutoff energy of the mirrors was set to 4.5 keV. N₂ filled ionization chambers served as detectors and were found to yield at the highest pressure about 5pA

ionization current for the transmitted intensity. Within the limited beam time available the near-edge spectra as a function of pressure could be recorded up to 25 kbar, *i.e.* across the NaCl-CsCl phase transition. Thus we proved the feasibility of high pressure absorption experiments around photon energies as low as 3000 eV. Due to the sharp undulator maximum useful spectra were only obtained up to about 40 eV above the edge. However, due to the relatively small K core level width the XANES are extremely well resolved, and structural information up the next nearest neighbor is obtained from $k \leq 4 \text{ \AA}^{-1}$ data in a straightforward manner. But surprisingly the analysis of the near edge structure of strongly compressed KCl turned out to be extremely difficult, and as yet we did not succeed to fit any type of our calculated spectra to the experimental data. Further work is in progress.

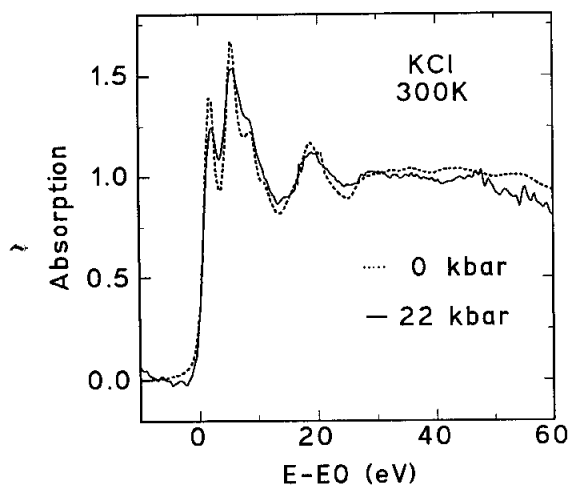


Fig.1 Near-edge spectra of the potassium in K-edge in KCl at ambient pressure, and 22 kbar.

References

- [1] J. Rohler, "High Pressure X-Ray Absorption Spectroscopy", the scientific case, ESRF user's meeting March-89 (unpublished).
- [2] J. Rohler and R. Lubbers, HASYLAB report 1993, pp. 239 and 909.
- [3] E.A. Perez-Albuerna and H.G. Drickamer, J. Chem. Phys. 43, 1381 (1965).

Acknowledgements - We are grateful to the kind and generous support obtained by Drs. D. Haussermann and M. Hanfland during the preparation of the experiment.