

Energy disperse detection of new phases in organic compounds

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In situ high-pressure powder-diffraction experiments on organic compounds has been performed at the cubic multi-anvil press MAX80 installed at the F2.1 synchrotron beamline [1]. Energy Disperse X-ray (EDX) analysis has been applied. The white synchrotron radiation combined with a constant diffraction angle $\theta=4.5035^\circ$ has been used. The pressure range in these experiments ranged from atmospheric to approximately 50 kbar. The pressure calibration was based on the compression of NaCl [2]. The hexagonal phase of boron nitride (*h*-BN) has been applied as the pressure medium.

Several different organic compounds have been analysed using an 8 mm in size high-pressure cubic cell. The samples were mounted in a *h*-BN cylinder in the middle of cube made of a mixture of boron and epoxy resin. The cube was placed between six anvils of the MAX80 press.

The samples investigated included perchlorate and tetrafluoroborate salts of 1,4-diazabicyclo[2.2.2]octane, for which phase transitions on cooling were observed before. Although at room temperature both salts are isostructural, their low-temperature behaviour is drastically different. Main structural transformations may be described by conformational changes of the cation, proton transfers between the donor and acceptor sites in the NH^+ -N bonds, and ionic shifts and titling [3-9].

In our experiments several structural changes were observed (*see* Figs. 1 and 2), however the structure could not be elucidated because of the low quality of data. The WinPLOTR software [10] was applied to fitting profiles and determination of the unit-cell parameters and structure.

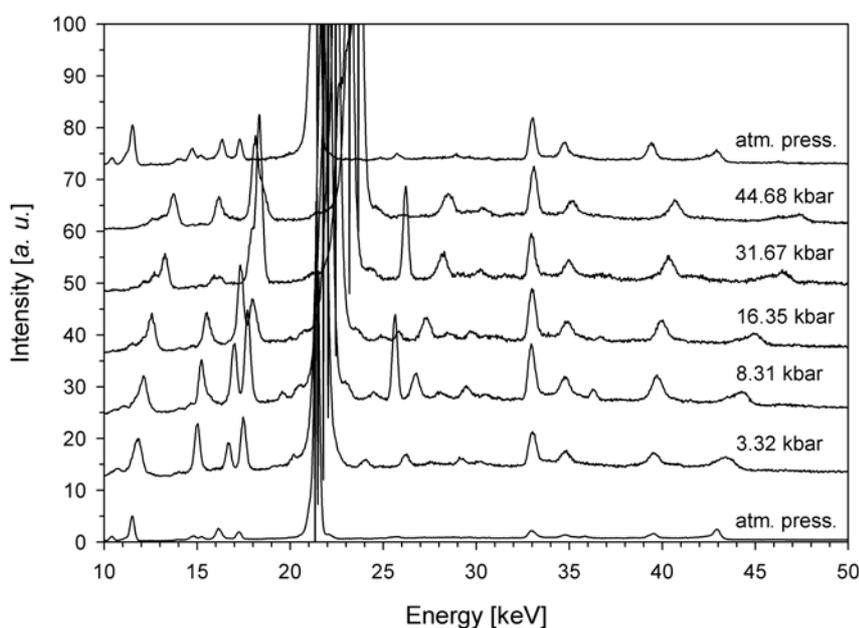


Figure 1: Variation of the EDX diffraction pattern of 1,4-diazabicyclo[2.2.2]octane tetrafluoroborate in the function of pressure.

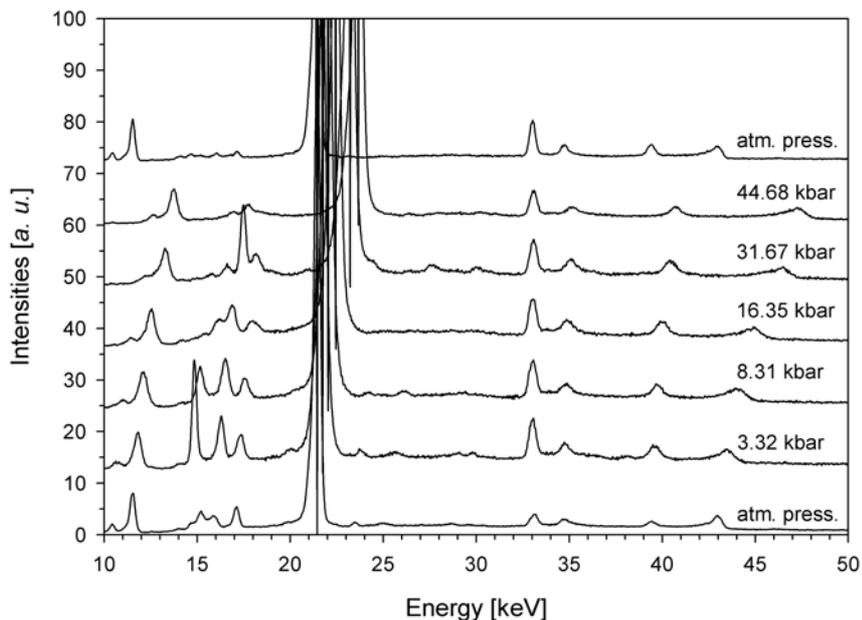


Figure 2: Comparison of EDX diffraction patterns of 1,4-diazabicyclo[2.2.2]octane perchlorate measured at varied pressure.

The preliminary studies have shown that EDX analysis using the cubic multi-anvil press and synchrotron radiation can be successfully applied for studying simple high-symmetry organic compounds at high-pressure, although the signal-to-background ratio and resolution of the measurements should be improved.

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References

- [1] O. Shimomura, S. Yamaoka, T. Yagi, M. Wakatsuki, K. Tsuji, H. Kawamura, N. Hamaya, K. Aoki and S. Akimoto, Multi-anvil type x-ray system for synchrotron radiation. In: *Solid State Physics Under Pressure: Recent Advances with Anvil Devices* (S. Minomura, ed.). KTK Sci. Publ., Tokyo, Japan, 351–356 (1985).
- [2] D. L. Decker, *J. Appl. Phys.* 42, 3239–3244 (1971).
- [3] A. Katrusiak, *Phys. Rev. B* 51, 589–592 (1995).
- [4] A. Katrusiak, M. Ratajczak-Sitarz, E. Grech, *J. Mol. Struct.* 474, 135–141 (1999).
- [5] A. Katrusiak, M. Szafranski, *Phys. Rev. Lett.* 82, 576–579 (1999).
- [6] A. Katrusiak, *J. Mol. Struct.* 552, 159–165 (2000).
- [7] M. Szafranski, A. Katrusiak, *Chem. Phys. Lett.* 318, 427–432 (2000).
- [8] M. Szafranski, A. Katrusiak, G. McIntyre, *Phys. Rev. Lett.* 89, 15507 (2002).
- [9] A. Budzianowski, A. Katrusiak, Lecture notes, International School of Crystallography: Diversity Amidst Similarity Course, Erice (Italy); 9 - 20 June 2004; 370 (2004).
- [10] T. Roisnel, J. Rodriguez-Carvajal, WinPLOTR a graphic tool for powder diffraction. LLB-LCSIM (June 2005).