



**Experiment title:** EQUATION OF STATE AND PHASE TRANSFORMATION OF BN POLYMORPHS : r-BN and w-BN

**Experiment number:**  
HS 328

**Beamline:**  
*ID30*

**Date of experiment:**  
from: 10/12/97 to: 18/12/97

**Date of report:**  
*01/03/98*

**shifts:**  
18

**Local contact(s):**  
Mohamed Mezouar

*Received at ESRF:*  
- 3 MAR. 1998

Names and affiliations of applicants (\* indicates experimentalists):

Yann LE GODEC\* - Universite P. et M. Curie (PMC).

J.M. BESSON\* - PMC.

G rard SYFOSSE\* - PMC.

Domingo MARTINEZ\* - Universite de Valence (Spain).

Vladimir SOLOZHENKO - Institute for superhard Materials (Ukraine).

Daniel HAUSERMANN - ESRF.

Mohamed MEZOUAR - ESRF.

---

### Experimental report.

Our research program on boron nitride was initiated last year with a systematic investigation of rhombohedral BN, r-BN which is the less known form of r-BN, although it is the most interesting variety in that it will transform into any of the other three allotrops, depending on the thermodynamic conditions.

During the last run, between December 10<sup>th</sup> and 18<sup>th</sup>, precise equations of state of pyrolytic r-BN have been measured, at different temperatures, up to the transition to wurtzite BN (w-BN). This work has been performed in a large volume Paris-Edimbourg cell, by angular dispersive methods, using the new FASTSCAN imaging plate setup implemented on ID30 (figure 1). With such a setup, the exposure time is reduced by a factor of 15 by comparison with conventional imaging plates.

In addition, integration over the diffraction rings allows to take into account preferred orientation effects and to obtain information on the intensities, with yield the relative quantities of the coexisting w-BN and r-BN phases at the transition (figure 2). After subtracting the background (figure 1b) from the total pattern (figure 1a), the diffraction patterns from the sample (figure 1 c) are clean enough to allow Rietveld profile refinement. This is still under way, at the present time, since the experiments were done at the end of last year only, and the full results will be presented in a complementary report.

The transition pressures at various temperature for the **r-BN**→**w-BN** transformation have been found to be **published**n previously <sup>(1-3)</sup> (figure 3). The reason is that in previous experiments the phase transition had been performed under fixed pressures, after heating for a few tens of seconds and only quenched samples had been studied. In our case by contrast, **in situ** observation of the **r-BN**→**w-BN** is done and it occurs distinctly lower, especially at low temperature (figures 2 and 3), **which** shows that in studying on quenched samples, one suffers from the reversibility of the transition during the quenching process.

The thermoelastic equations of state of r-BN have been established for the first time at 25, 100, 200, 300, 400, 500, 600, 700 and 800°C. Figure 4 shows, as an example, the equation of state at 200°C. The expected anisotropy of r-BN is illustrated in figure 5 showing the compression along the [ 100] and [001] axes. This high degree of anisotropy is of course related to that of the chemical bonds of r-BN which is a layered solid with weak van der Waals interlayer bonds.

## References :

- 1) A. Onodera, K. Inoue, H. Yoshihara, J. *Mater. Sci.* 25,4279 (1990).
- 2) I.A. Petmsha, A.A. Svirid, *High Pressure Research* 9, 136 (1992).
- 3) V.L. Solozhenko, I.A. Petrusha, B. Barbier, *Sverk. Mater.* **18**, no 6,62 (1996).