ESRF	**Experiment title:** Depth-resolved glancing angle x-ray diffraction from cubic boron nitride films grown on silicon	**Experiment number:** SI-89
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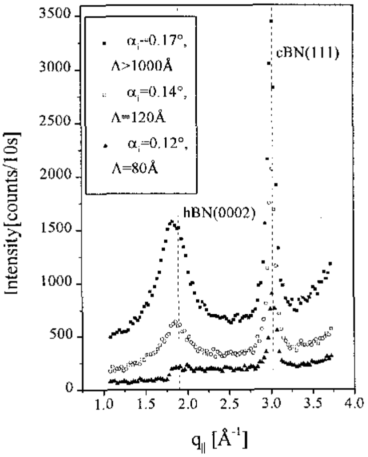
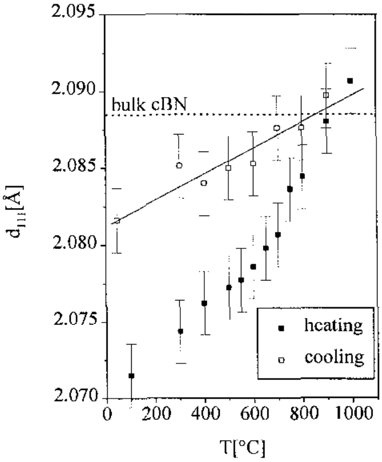
Report:

We performed a grazing-incidence x-ray scattering study of the phase distribution and strain in thin boron nitride films on Si(001) substrates. These films are promising candidates as superhard coatings for cutting tools, as their chemical properties are superior to those of diamond. Similar to diamond there exist at least two crystallographic phases, namely the low density hexagonal phase (hBN) and the high density, covalent bonded and superhard cubic zincblende structure (cBN). The films were sputtered and Ar ion-plated with an energy of 100 eV, resulting in a highly stressed, nanocrystalline material [1]. The samples were 0.2 µm thick with an area of 10x10 mm^2. Measurements at the TROIKA beamline were carried out with an x-ray wavelength of 1 A, using a diamond monochromator at the (111) reflection. We used a horizontal scattering geometry, tuning the incident angle to the critical angle for total external reflection (0.16’). The diffracted photons were collected with a position-sensitive detector in order to resolve the exit angle profiles . The thermal stability of the samples was examined in annealing experiments.

The left graph shows detector scans at three different incident angles and resulting scattering depths: two peaks, associated with the hBN and cBN phase, are visible. The integrated intensities of the two reflections are roughly equal in the upper curve (entire film thickness) and differ by a factor of three in the curve in the middle (120 Å scattering depth). The lattice spacing of the hBN(0002) planes decreases from the substrate to the surface of the film by 1.3% . We propose the following structural model, supported by density profiles extracted from reflectivity data: the samples consist of a layered structure of hBN-rich material near the substrate and a cBN-rich phase at the top of the film. The strain in the film increases from the substrate to the surface and is compressive in the film plane. The right figure shows some of the results of the annealing experiments carried out in situ in a high vacuum chamber. The cBN(111) lattice spacing of the as-grown film differs by 0.9% from the bulk value (dashed vertical line), corresponding to an in-plane stress of 4.2 GPa. Upon heating the cBN-lattice expands linearly until a temperature of about 65O °C, followed by a rapid and irreversible strain relaxation (full symbols). The cooling curve follows the bulk thermal expansion curve with a slope of $3.9x10^{-6} K^{-1}$ (straight line). The relaxed films has an in-plane lattice spacing of 2.081 Å, i.e. a stress of 1.4 GPa.





[1] W. Donner et al., Appl. Phys. A65, 1 (1997)