ESRF	**Experiment title:** Strain determination of cubic boron nitride films	**Experiment number**: HS-463
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Shifts: 15	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

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Report:

Thin films of cubic boron nitride are superhard (>4000 Vickers) and are considered as coatings for abrasive tools. The most crucial technological problem is the high stress in these films, which leads to limited thicknesses (1-2µm) and often to bad adhesion. Strain and stress determination of cBN films is challenged by the small scattering power and nanocrystalline nature of the material.

We carried out the first determination of the strain tensor of thin (0.2µm) cBN films. The films were grown by DC-biased, plasma-enhanced sputtering methods onto Si(001) substrates. The samples exhibit a layered structure of a hexagonal seed layer (hBN) of a few hundred Å thickness and the cBN film. The experiment was carried out at the Materials Science Beamline (ID11) using an incident energy of 10.5keV and a beam focus at the sample position of $0.2x0.2mm^2$. A Huber 511 four-circle diffractometer equipped with a stepping-motor driven goniometer head and a NaJ scintillation counter was used.

The residual strain tensors of the films had been determined via the $\sin^2\Psi$-method. The surface normal was tilted from being perpendicular to the scattering plane (Ψ=0) to parallel (Ψ=90°). The incident angle was tuned to the critical angle of total external reflection (0.2°) in order to suppress substrate signals. The scattering geometry is depicted in Fig.1. Detector (2θ-)-scans were performed at nine different values of Ψ by varying the angle χ.

Typical count rates for the cBN(111) reflection were 300 cps. Fig.2 shows two detector scans of one of the samples at different angles χ. The shift of the peak at 85° is clearly visible; the shift to smaller angles implies a relaxed lattice spacing towards the surface normal, which is expected for biaxial strain. A complete analysis using the method described by Dölle [1] leads the components of the strain tensor and, if the elastic constants are known, the stress tensor. As a byproduct, also the unstrained lattice parameter can be derived. This was done by way of example for one sample.
Fig. 3 shows the average detector angles as a function of $\sin^2\Psi$. The observation that all points measured are lying on a straight line implies a bi-axial strain. From the slope and the interception of the axis the tensor elements $\sigma_{11}=\sigma_{22}=10.1$ GPa and an unstrained lattice parameter of 2.10812 Å.

The main results of this study are:

- the establishment of the $\sin^2\Psi$-method under grazing incidence conditions.
- the finding that the strain state in cBN films on Si is biaxial and all non-diagonal elements of the strain tensor are zero.
- the determination of the unstrained lattice parameter, which gives a value that is about 1% larger than the bulk value.

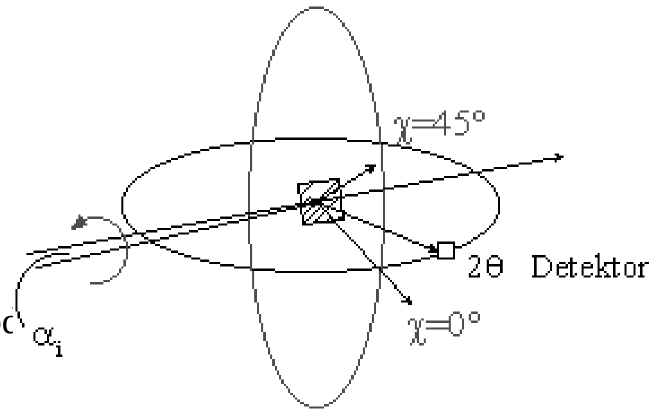

Fig.1: Scattering geometry for the $\sin^2\Psi$ method under grazing incidence conditions.

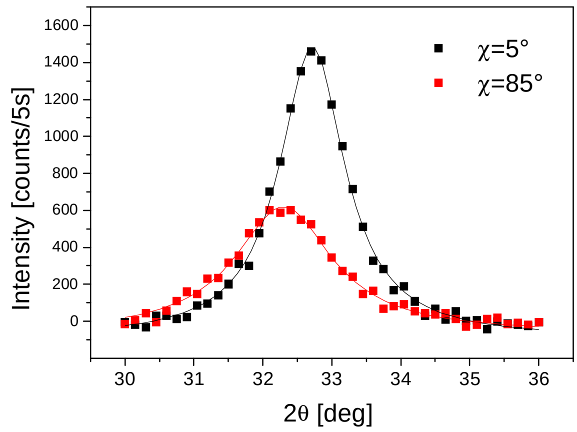

Fig.2: Detector scans through the cBN(111) reflection at fixed incidence angle (0.2°)

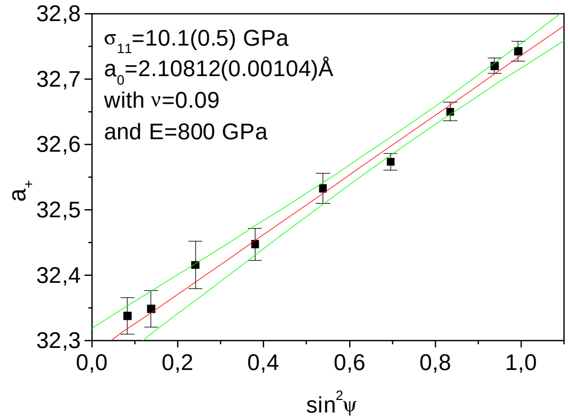

Fig.3: Detector angle as a function of $\sin^2\Psi$. The stress value derived from the linear fit together with the elastic parameters used is indicated.

[1] H. Dölle, J. Appl. Cryst. **12, 489** (1979).