Ion induced stress relaxation during the growth of cubic boron nitride thin films

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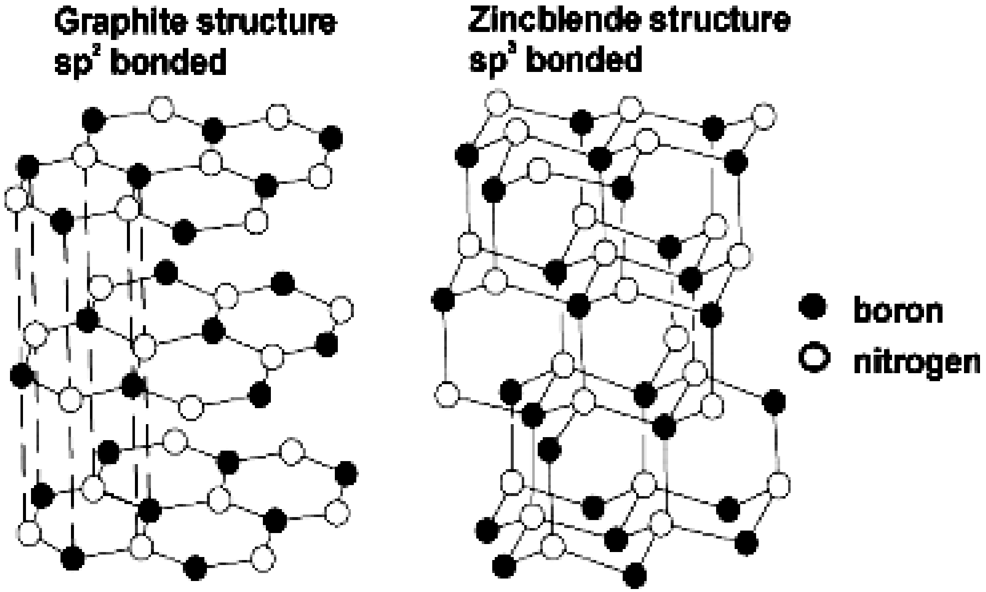
Motivation
Experimental
Stress relaxation
Microstructure
Conclusions & Outlook

Boron nitride thin films



hexagonal
boron nitride (**hBN**)
turbostratic BN (tBN)

cubic
boron nitride (c**BN**)

Cubic boron nitride

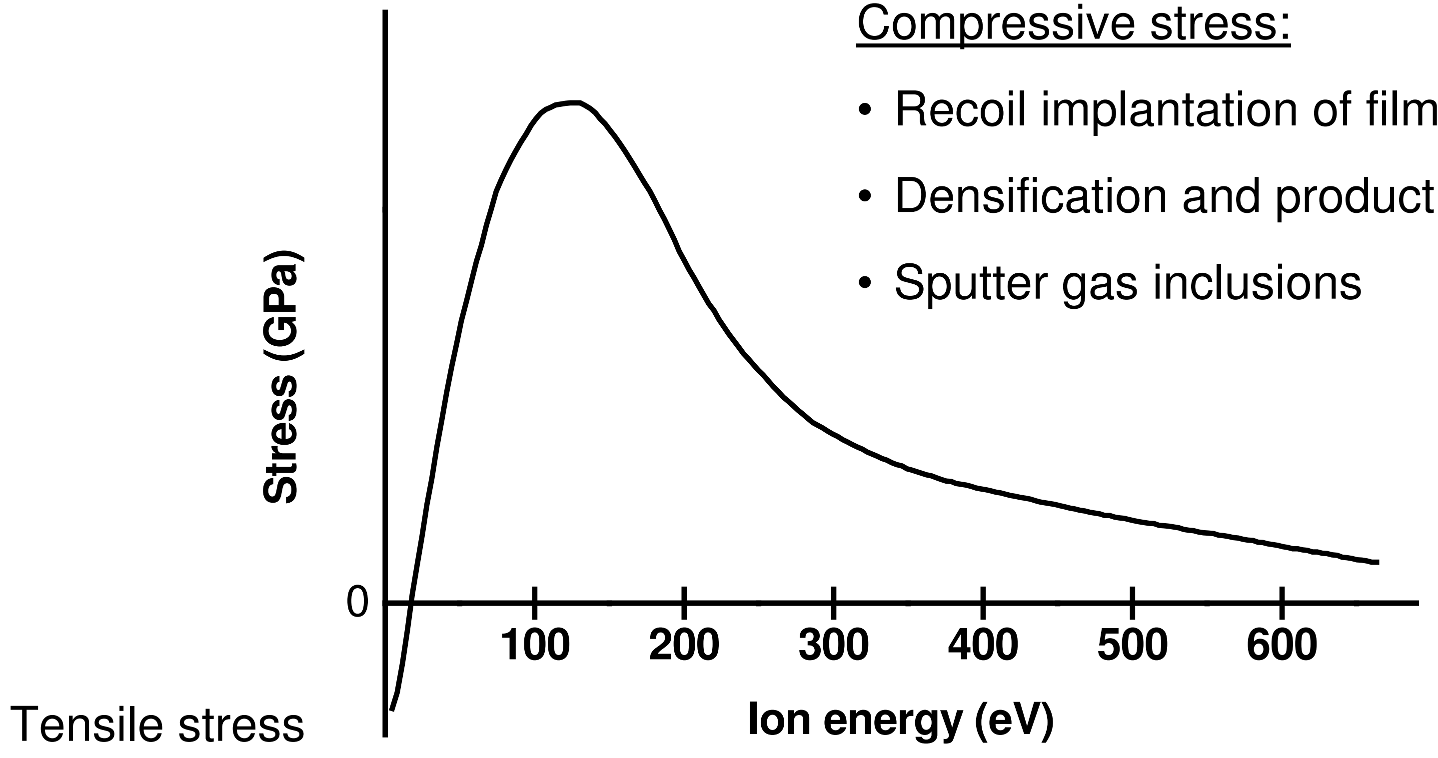
- Super hard material
- Oxidation resistance at high temperatures
  inert against Fe
- Large band gap
- p- and n- type doping

- Ion bombardment
- Layered structure

▼ High intrinsic compressive stress

Motivation

Stress in thin films under ion bombardment



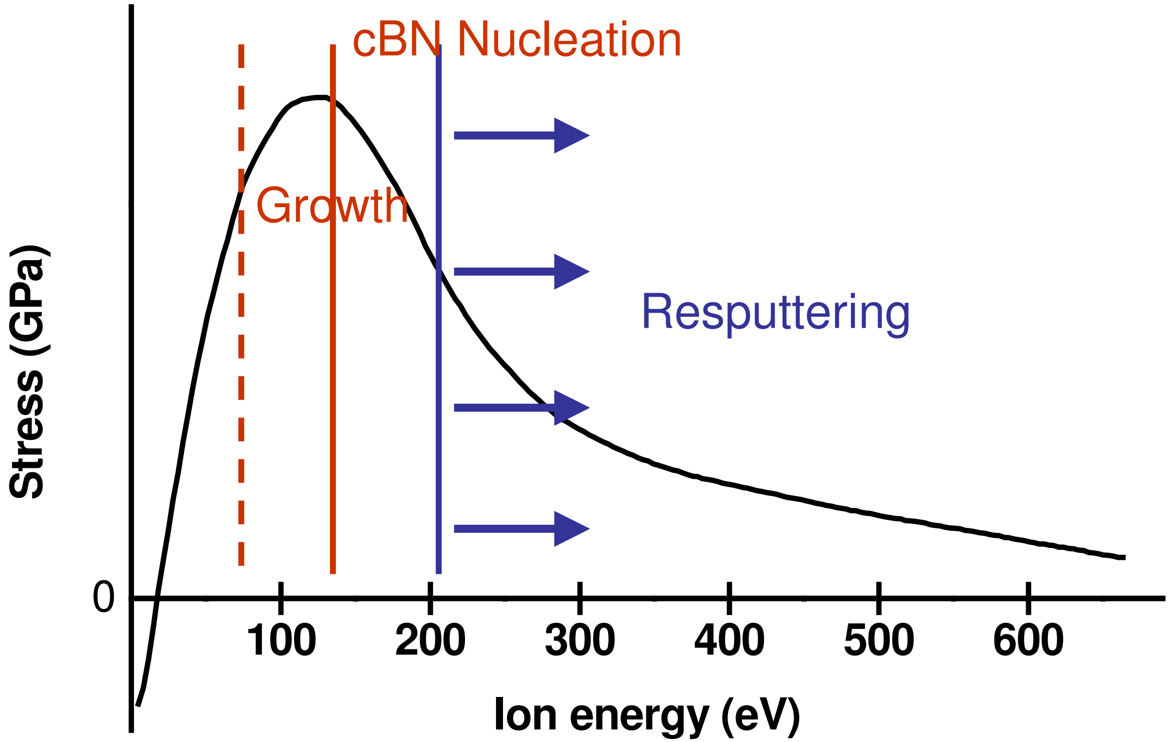
Compressive stress:

- Recoil implantation of film atoms
- Densification and production of defects
- Sputter gas inclusions

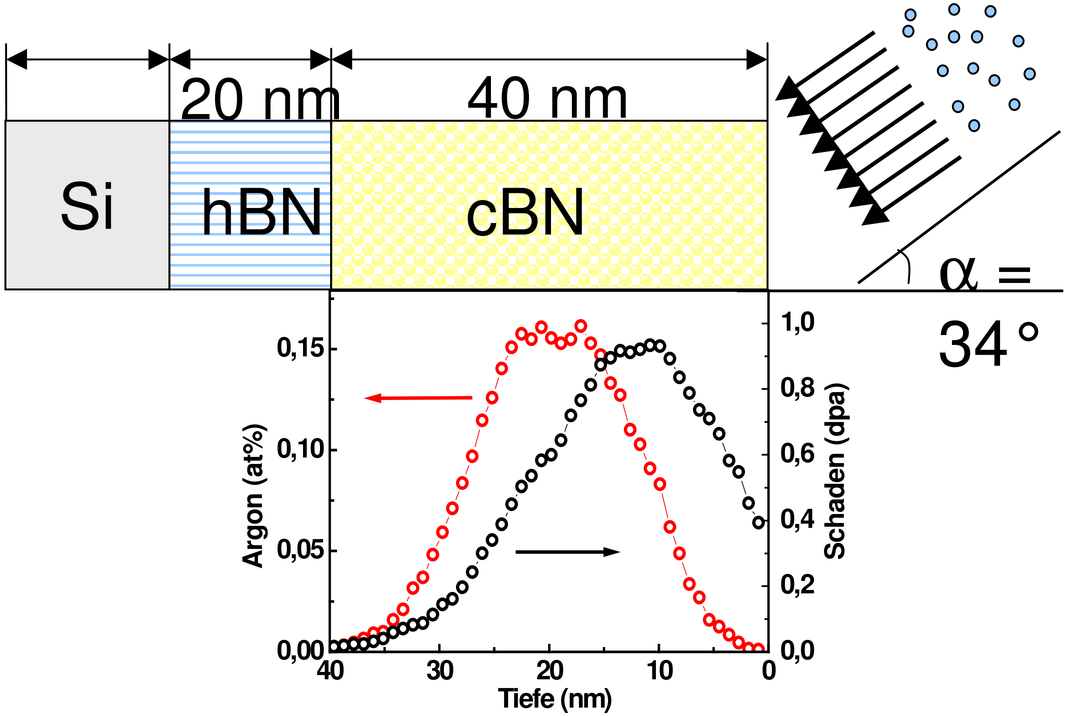
Davis, Thin Solid Films 1993

Motivation

Deposition of cBN



Stress relaxation by ion implantation



IBAD

$Ar^+ + N_2^+$ 500 eV

Incorporation of defects close to the surface

Relaxation:

$N_2^{+,}$ Ar^+, E > 35 keV

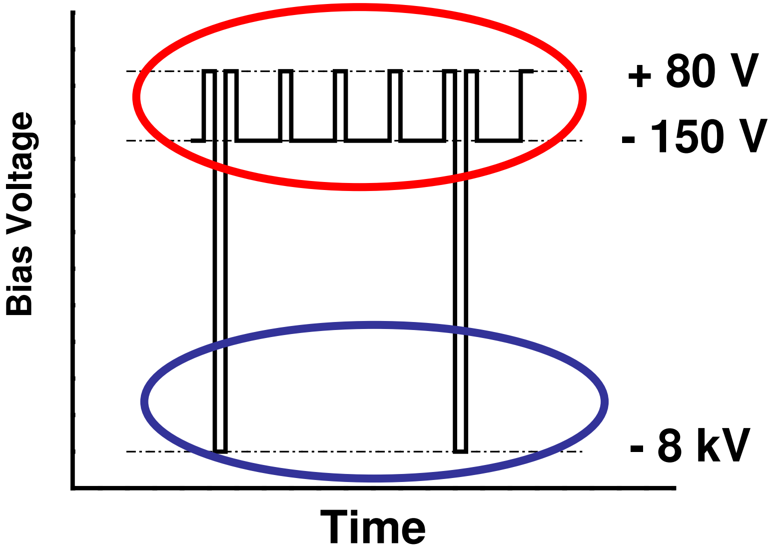
Atomic displacements below the surface

Ullmann et al., JAP. 83 (1998): 1 MeV Ar^+
Boyen et al., APL 76 (2000): 350 keV Ar^+
Fitz et al., APL 80 (2002): 35 keV N_2^+

E > 2.5 keV
cBN/hBN interface
not stable!

Relaxation during magnetron sputter deposition

- unbalanced, RF (13.6 MHz)
- hBN sputter target
- Ion species: Ar^+, N_2^+, N^+
- Ionen density ~ 1 x 10^{10} cm^{-3}



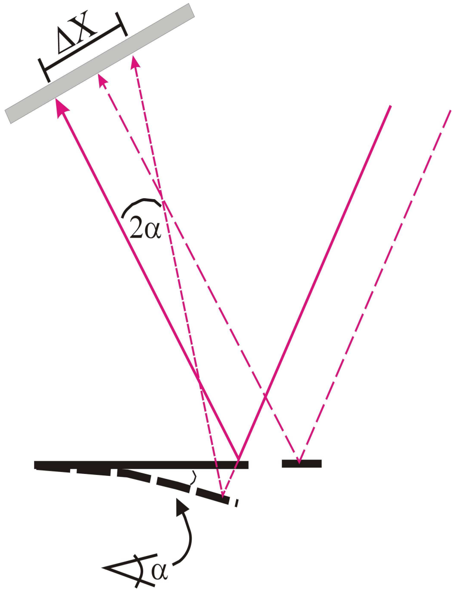
- Growth: -100 - 180 V
- Relaxation: -2.5 to -8 kV
- Surface discharge: +80V

Assumption: Ion flux to the film surface does not vary with bias voltage

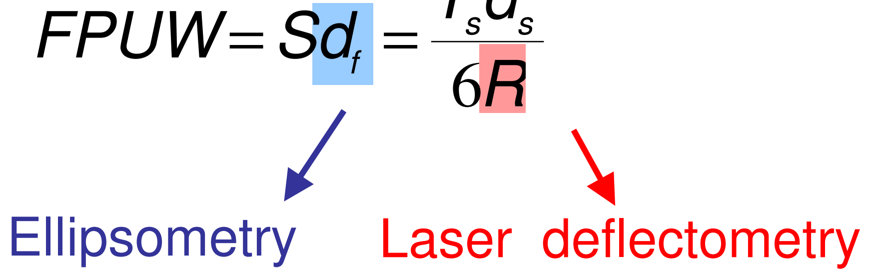
Duty Cycle: $$\frac{t(\text{highVoltage})}{t(\text{lowVoltage})} = 0.3 - 1.2\%$$

In situ stress measurement

- Cantilever bending
- Stress can be measured depth resolved



Stoney:



$$FPUW = Sd_f = \frac{Y_s d_s^2}{6R}$$

Ellipsometry

Laser deflectometry

S: Global (average) stress

Y_s: biaxial Modulus substrate

Si (100): 180.5 Gpa

d_s: Cantilever thickness = 180 µm

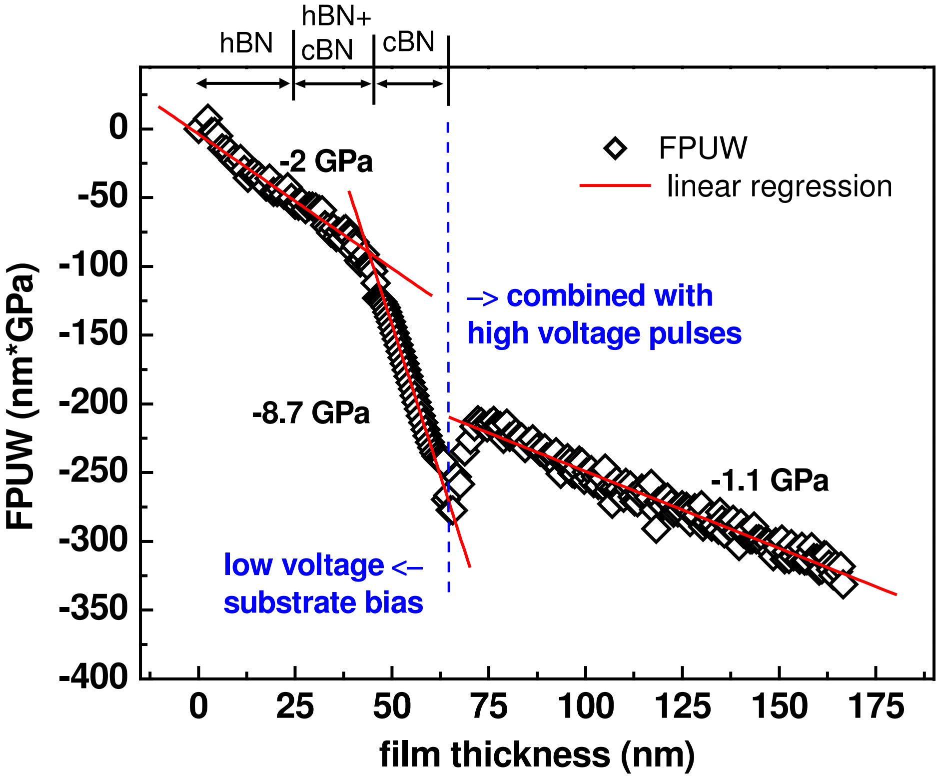
R: Bending radius

d_f: Film thickness

Instantaneous stress:

$$\boxed{\sigma(d_f) = \frac{\delta(FPUW)}{\delta d_f}\bigg|_{d_f}}$$

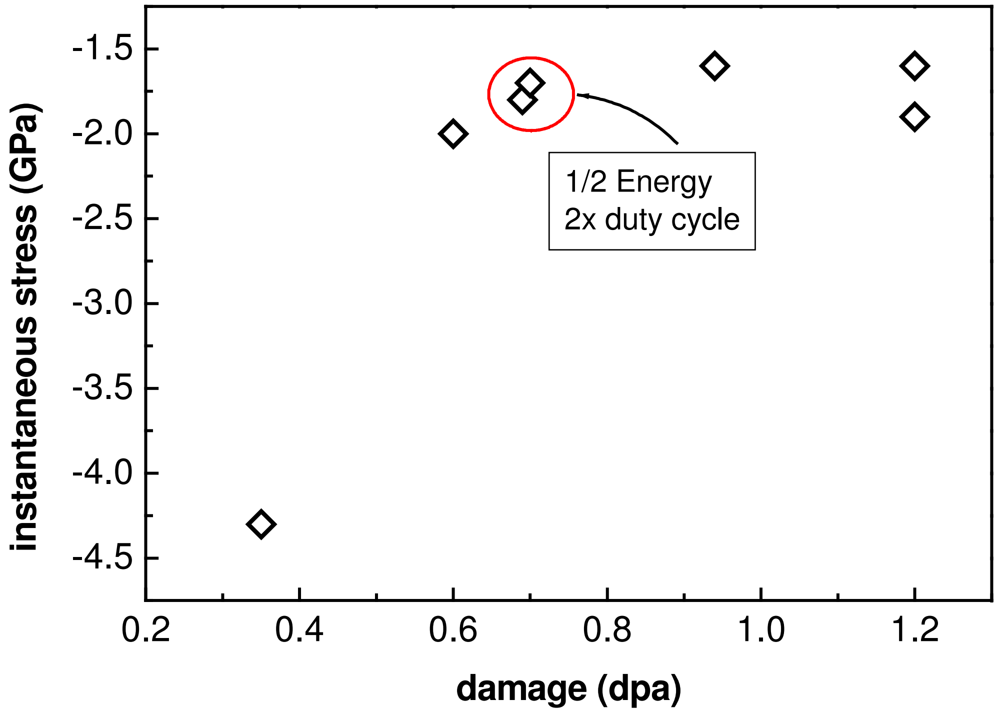
Ion-induced stress relaxation



Growth at -1.1 GPa
E_{Ion} = 8 keV
Equilibrium between defect production and defect annealing

- cBN/hBN interface not stable for $E_{Ion} > 2.5$ keV
- High stress in the depth
- Stress gradients

Ion-induced stress relaxation



Ion-induced damage:
dpa E_{Ion} , duty cycle

dpa < ~0.8:
stress decreases with damage

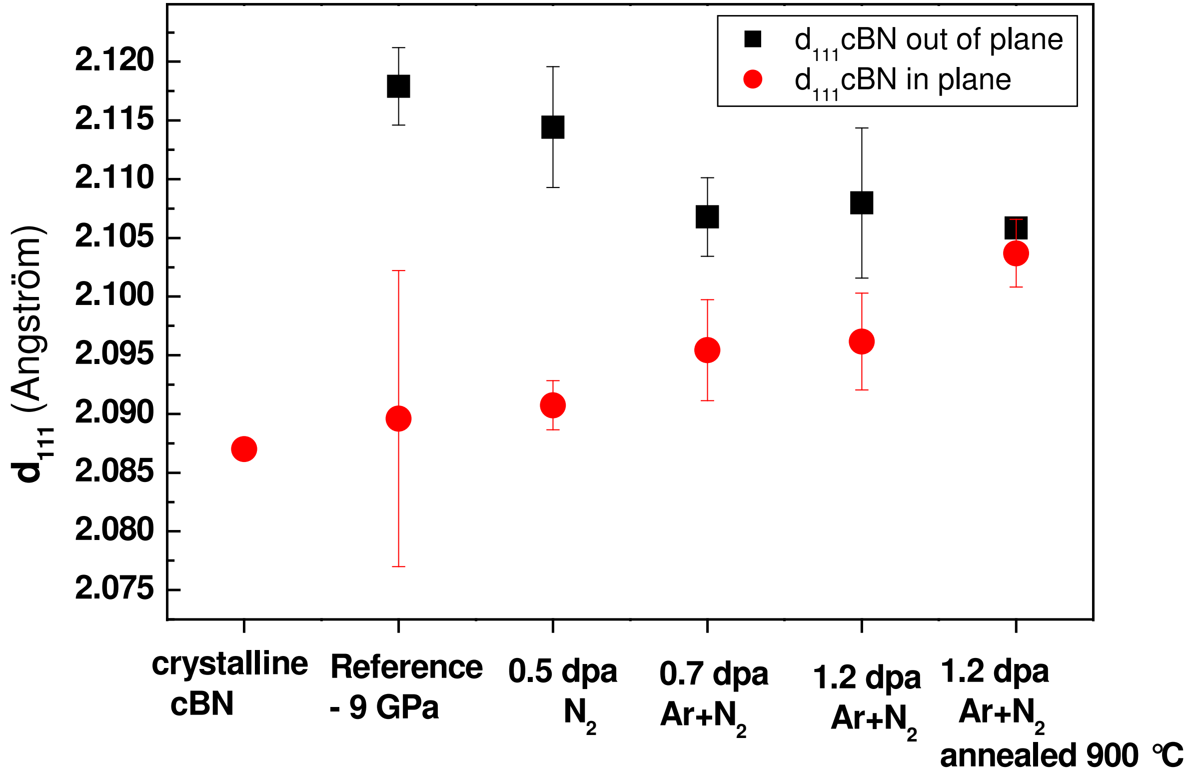
dpa > ~ 0.8:
no further release of stress

½ E_{Ion} + 2 x duty cycle equal stress relaxation !

So far lowest ion energy : 2.5 keV

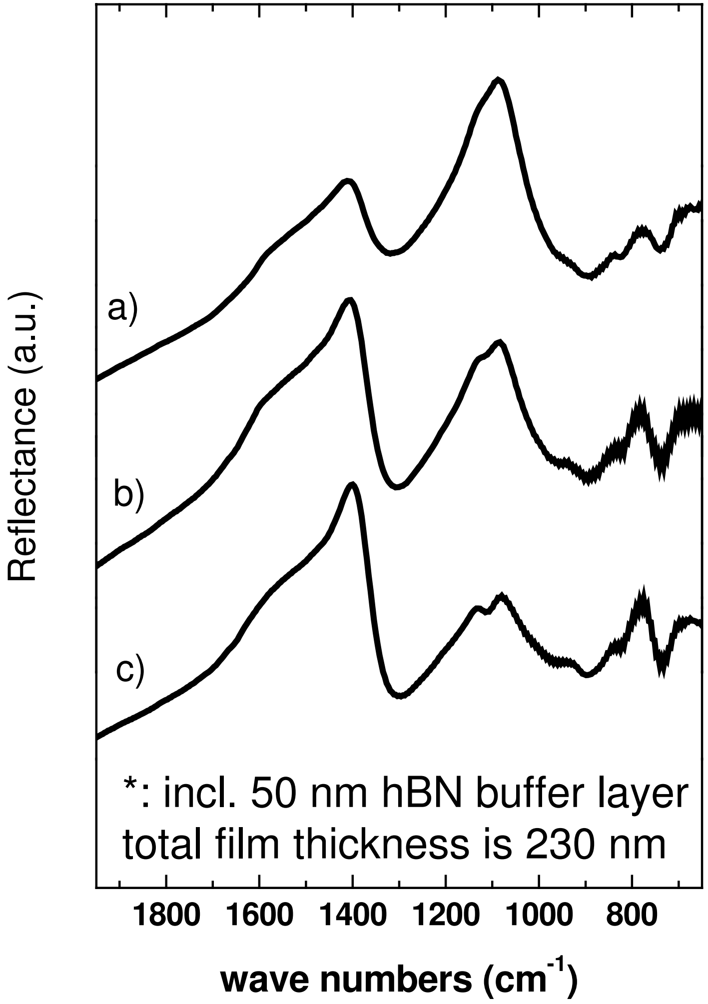
XRD

Grazing Incidence Diffraction measured at ROBL / ESRF



- Large biaxial strain in non irradiated reference sample A -9 GPa
- No biaxial strain in annealed sample: fully released
- In plane lattice constant shifts with increasing dpa towards released value
  relaxation in cBN!
- Biaxial strain remains

cBN Stabilität während Ionenimplantation



*: incl. 50 nm hBN buffer layer
total film thickness is 230 nm

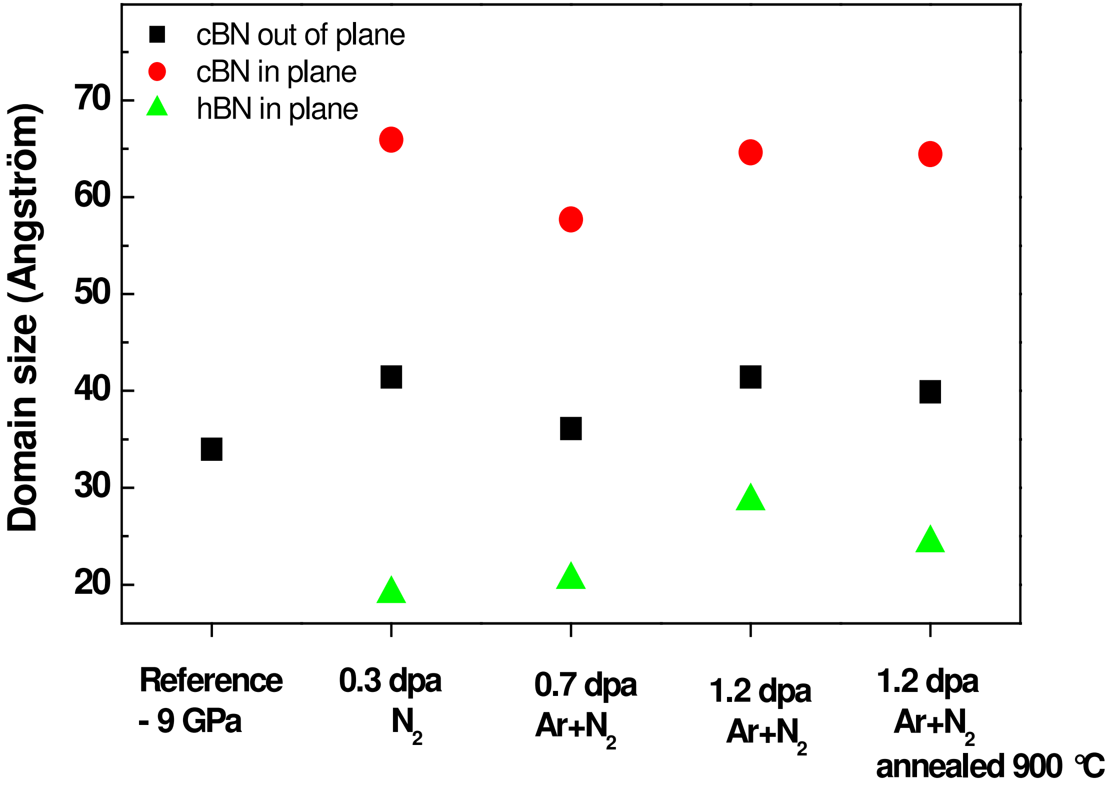
0.6 dpa → Transformation at low rate

0.9 dpa

1.2 dpa

→ cBN hBN Transformation at high rate, rate increasing with dpa

Domain sizes



- Domain sizes are not changed by ion bombardment, annealing or delamination
- Ion bombardment does not induce amorphization
- Increase of hBN is not on the expense of cBN (crystalline)
- Amorphous sp^3 bonded material transformation to sp^2 bonding ?

Conclusions & Outlook

- Stress relaxation by ion implantation (2.5 keV): cBN growth with -1 to -2 GPa stress
- Strain relaxation within the cBN
- Phase transformation: increased hBN content if dpa > 0.8
- BUT: cBN grains appear to be stable

Outlook:

Adhesion seems to be limited by stress and chemistry

Interface engineering, e.g. BCN buffer layer reduces hygroscopy