



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

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**Motivation**

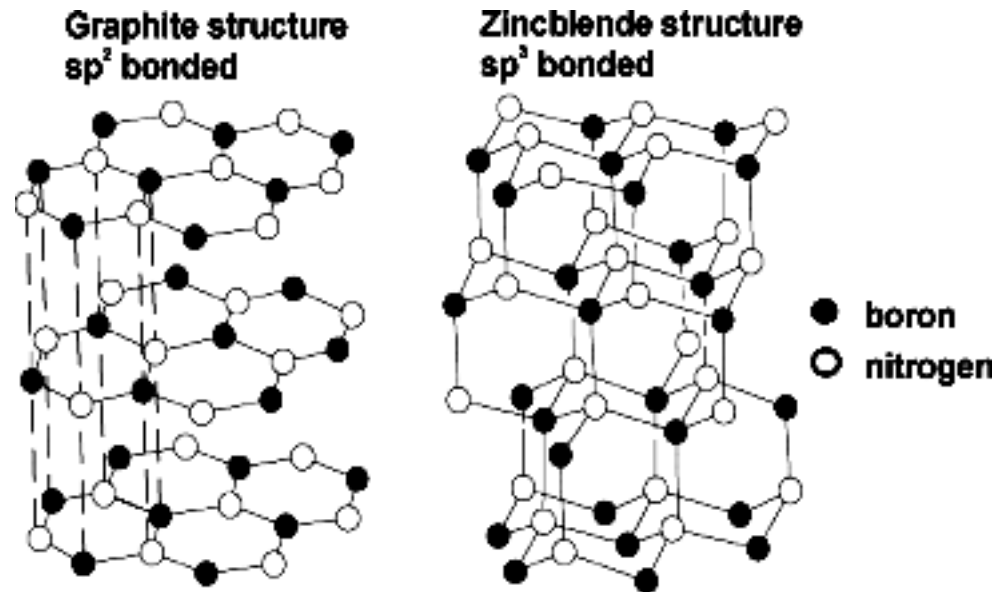
**Experimental**

**Stress relaxation**

**Microstructure**

**Conclusions & Outlook**

# Boron nitride thin films



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

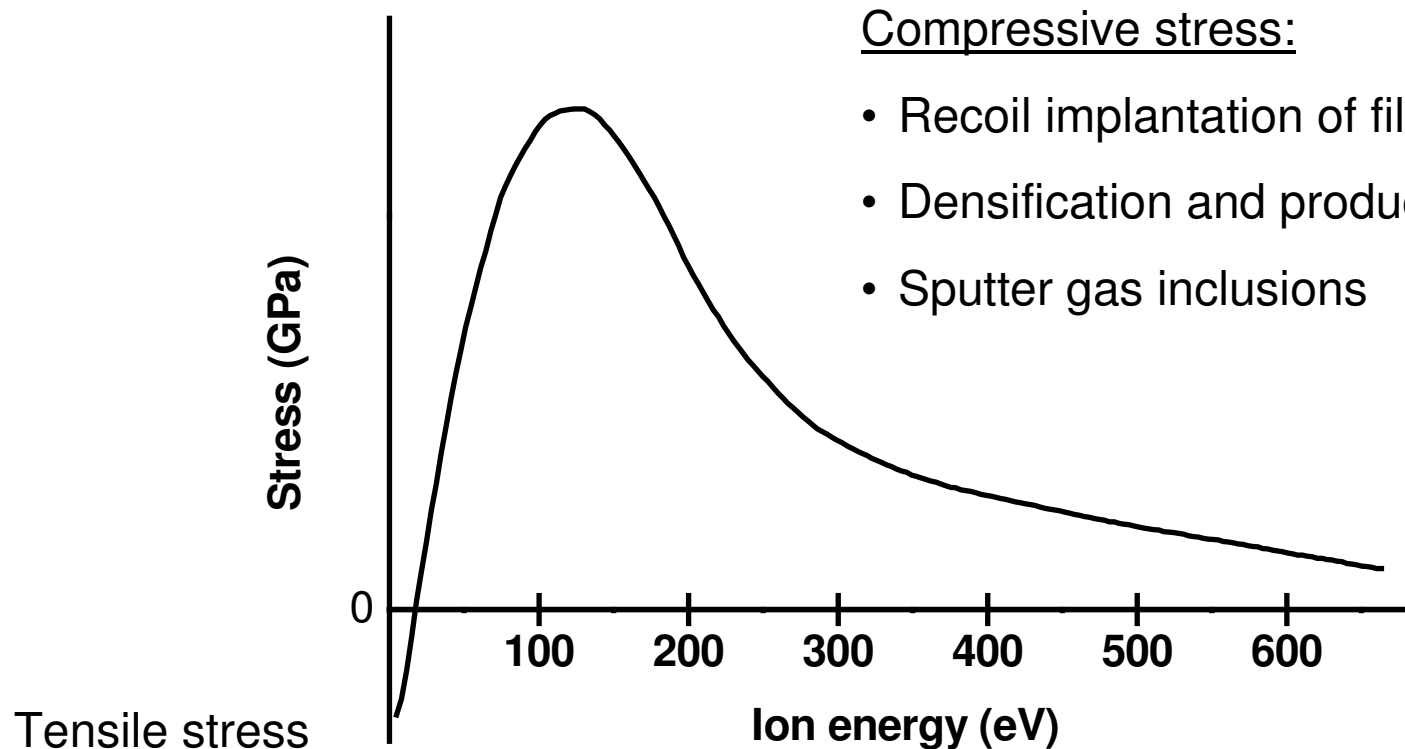
cubic  
boron nitride (**cBN**)

## 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

## Stress in thin films under ion bombardment

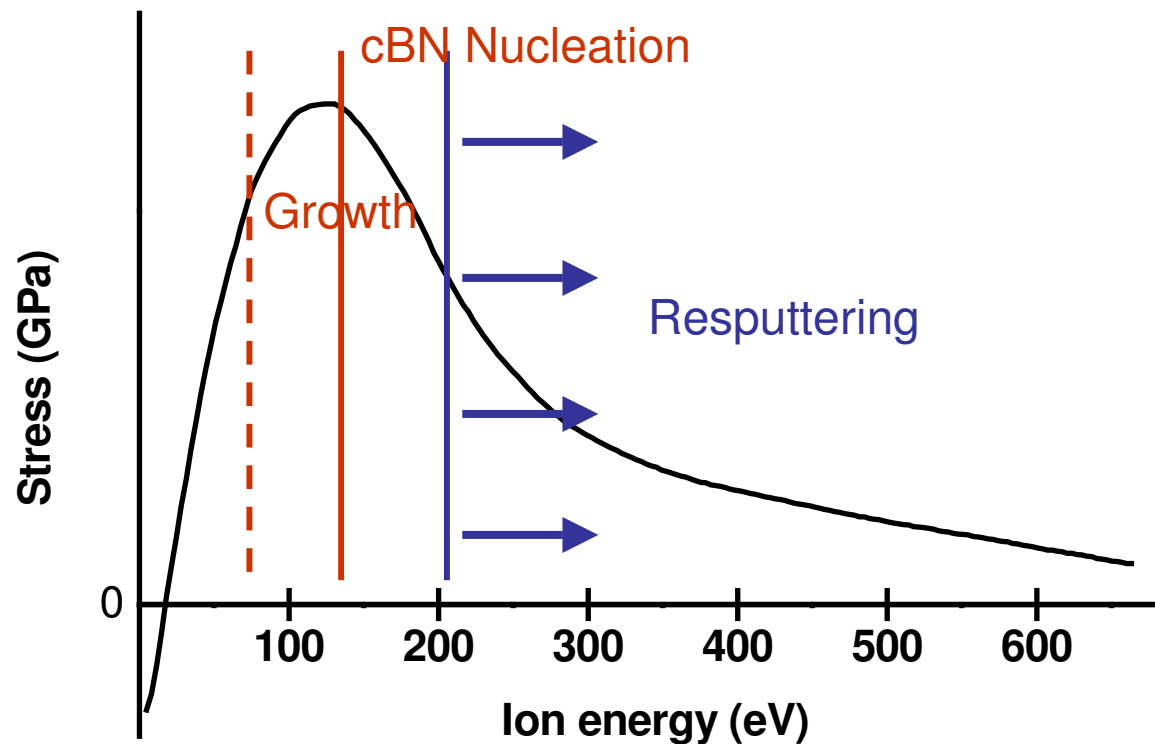


### Compressive stress:

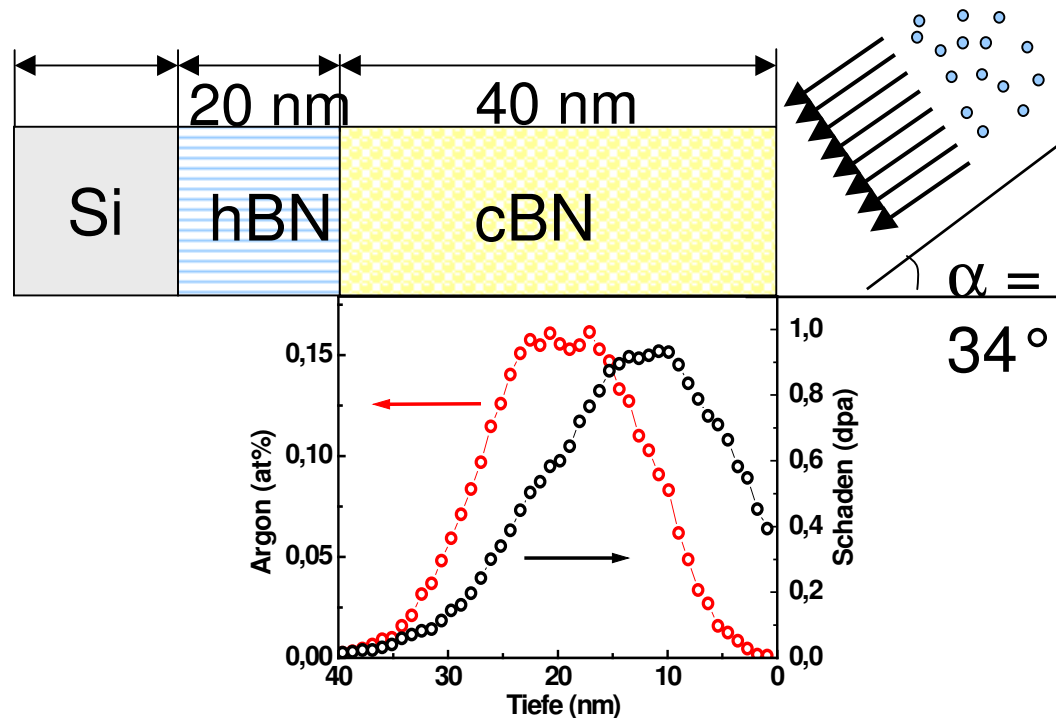
- Recoil implantation of film atoms
- Densification and production of defects
- Sputter gas inclusions

Davis, Thin Solid Films 1993

## Deposition of cBN



# Stress relaxation by ion implantation



IBAD

$\text{Ar}^+ + \text{N}_2^+$  500 eV

Incorporation of defects  
close to the surface

Relaxation:

$\text{N}_2^+$ ,  $\text{Ar}^+$ ,  $E > 35$  keV

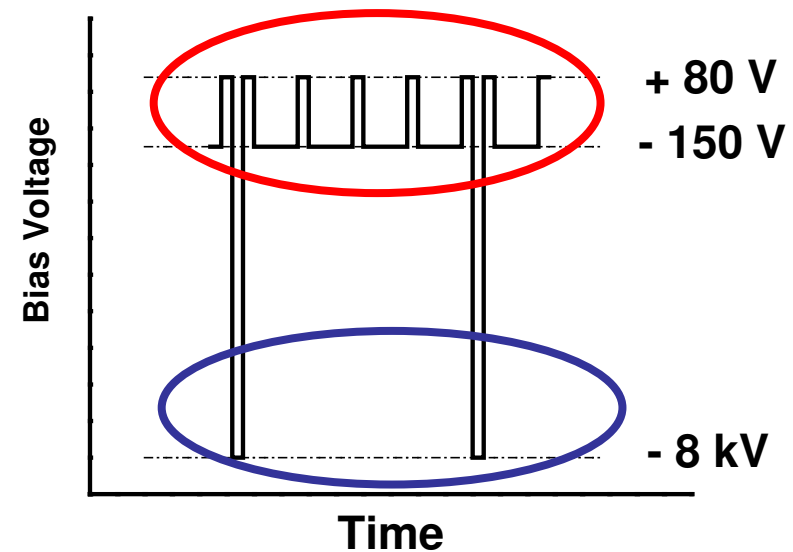
Atomic displacements  
below the surface

$E > 2.5$  keV  
cBN/hBN interface  
not stable!

Ullmann et al., JAP. 83 (1998): 1 MeV  $\text{Ar}^+$   
Boyen et al., APL 76 (2000): 350 keV  $\text{Ar}^+$   
Fitz et al., APL 80 (2002): 35 keV  $\text{N}_2^+$

# Relaxation during magnetron sputter deposition

- unbalanced, RF (13.6 MHz)
- hBN sputter target
- Ion species: Ar<sup>+</sup>, N<sub>2</sub><sup>+</sup>, N<sup>+</sup>
- Ionen density  $\sim 1 \times 10^{10} \text{ cm}^{-3}$
  
- **Growth:** -100 - 180 V
- **Relaxation:** -2.5 to -8 kV
- **Surface discharge:** +80V

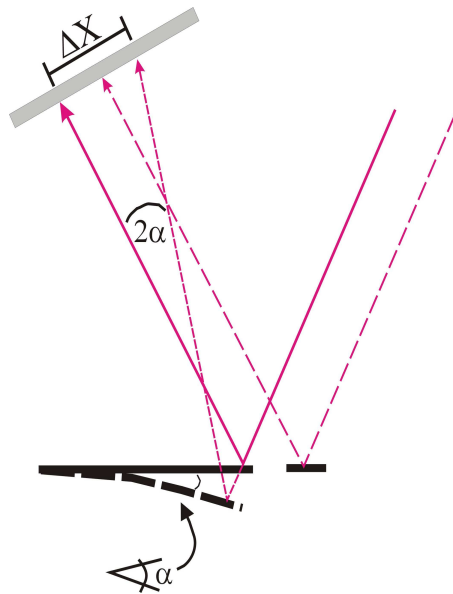


**Assumption:** Ion flux to the film surface does not vary with bias voltage

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:

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

Ellipsometry

Laser deflectometry

S: Global (average) stress

$Y_s$ : biaxial Modulus substrate

Si (100): 180.5 Gpa

$d_s$ : Cantilever thickness = 180  $\mu\text{m}$

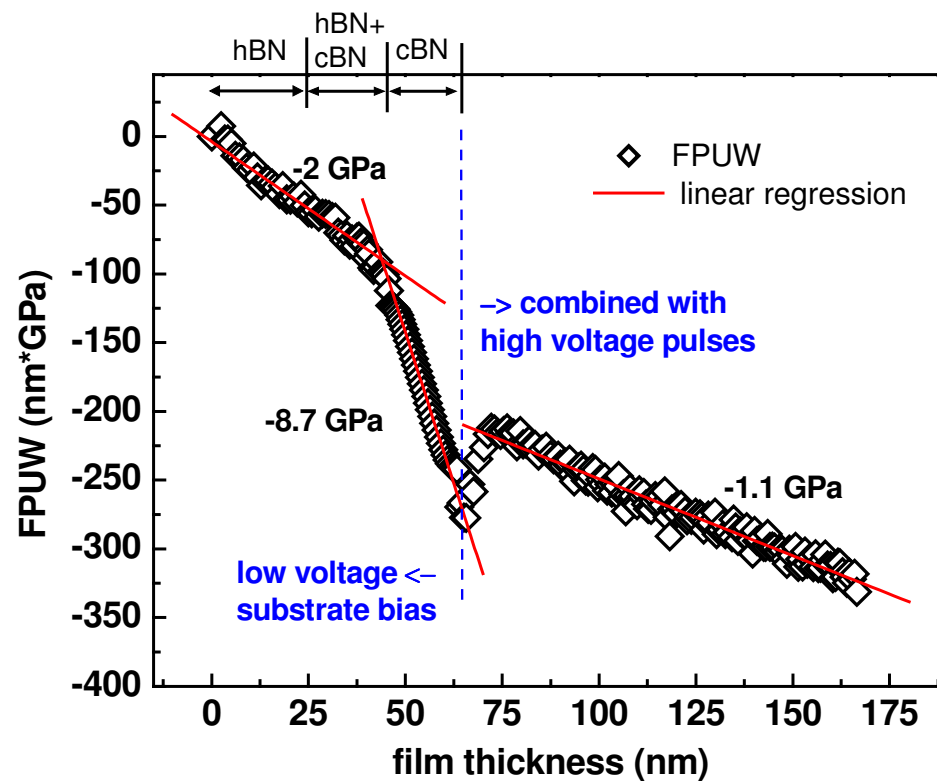
R: Bending radius

$d_f$ : Film thickness

Instantaneous stress:

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

# Ion-induced stress relaxation



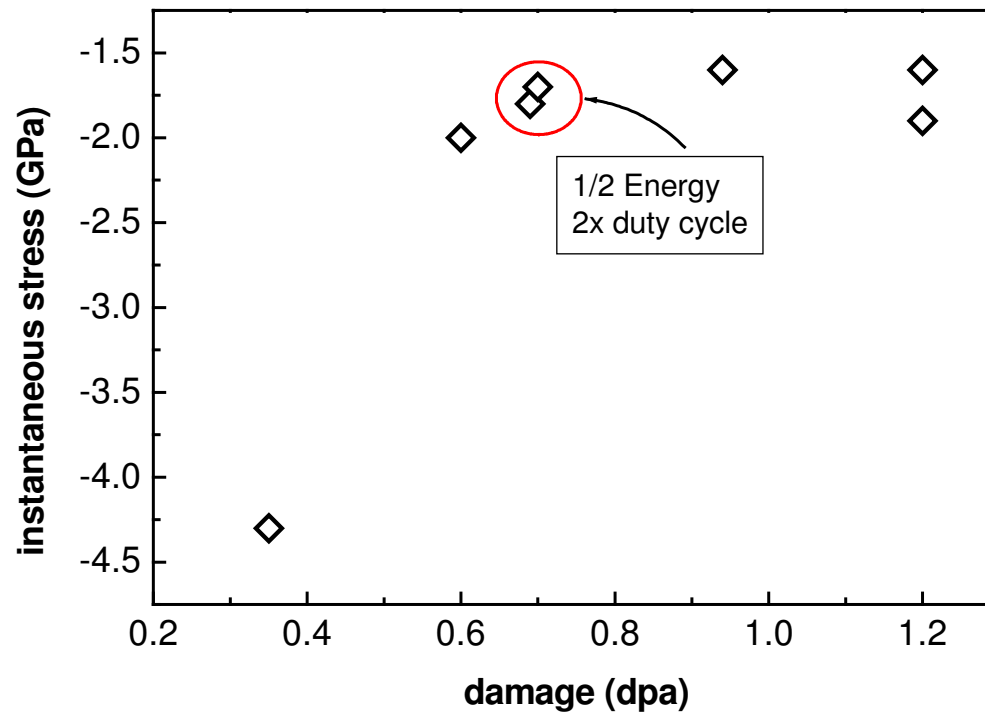
Growth at -1.1 GPa

$E_{\text{ion}} = 8 \text{ keV}$

Equilibrium between defect production and defect annealing

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

# Ion-induced stress relaxation



Ion-induced damage:  
**dpa**  $E_{\text{ion}}$ , duty cycle

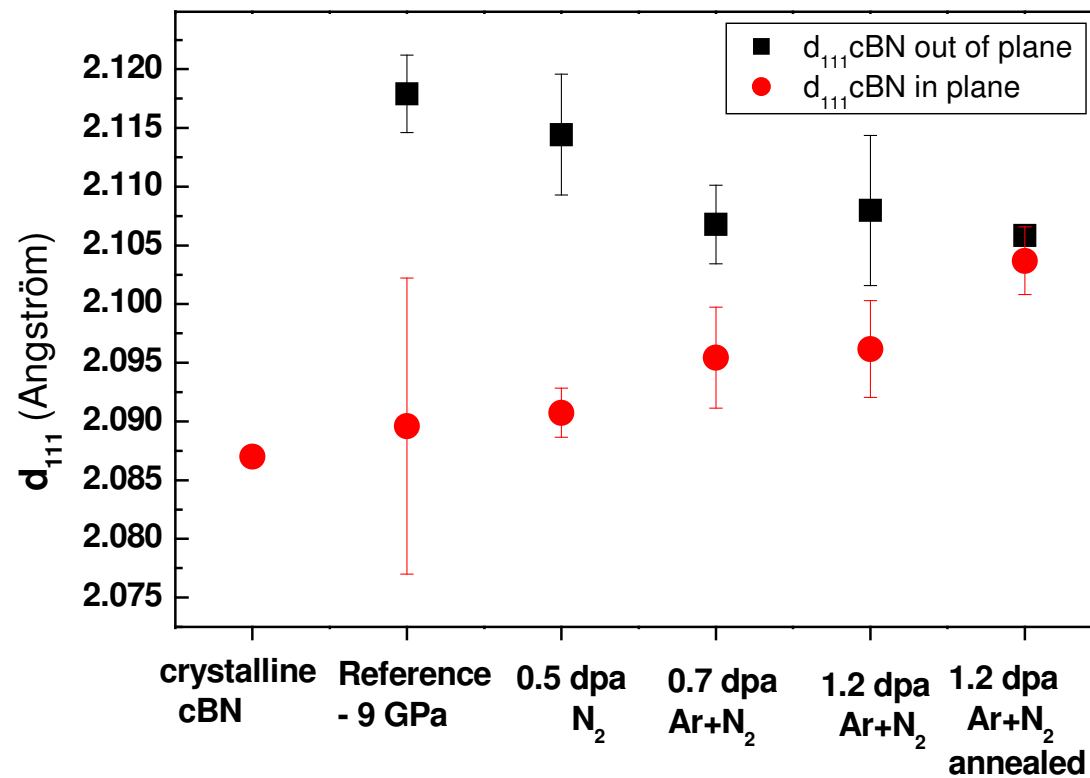
dpa < ~0.8:  
stress decreases with  
damage

dpa > ~ 0.8:  
no further release of stress

$\frac{1}{2} E_{\text{ion}} + 2 \times \text{duty cycle}$  equal stress relaxation !

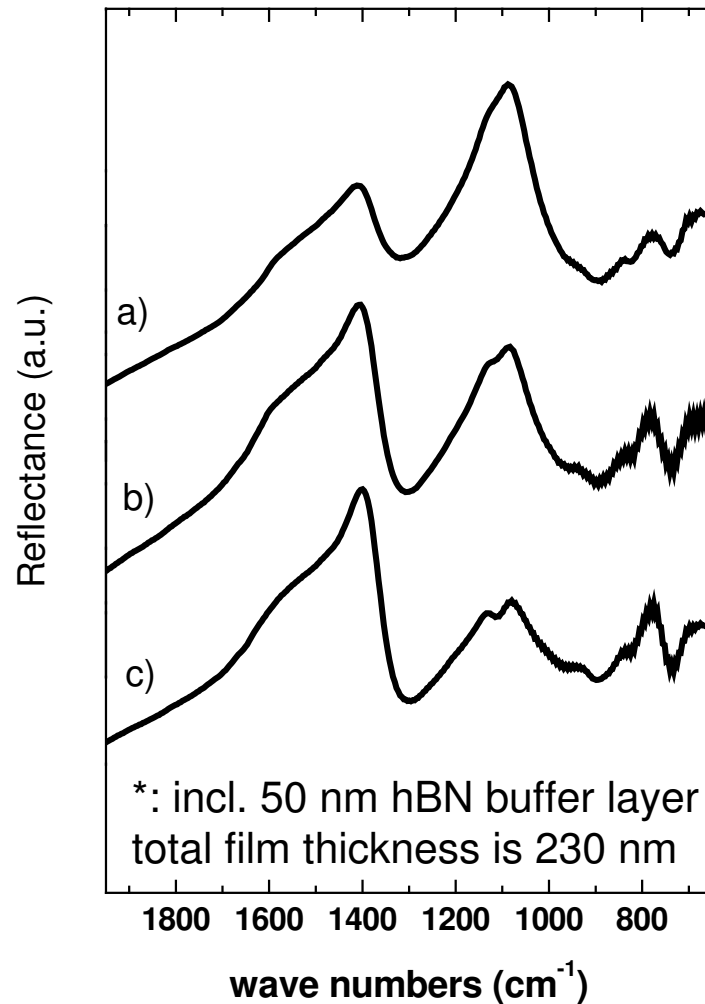
So far lowest ion energy : 2.5 keV

## 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



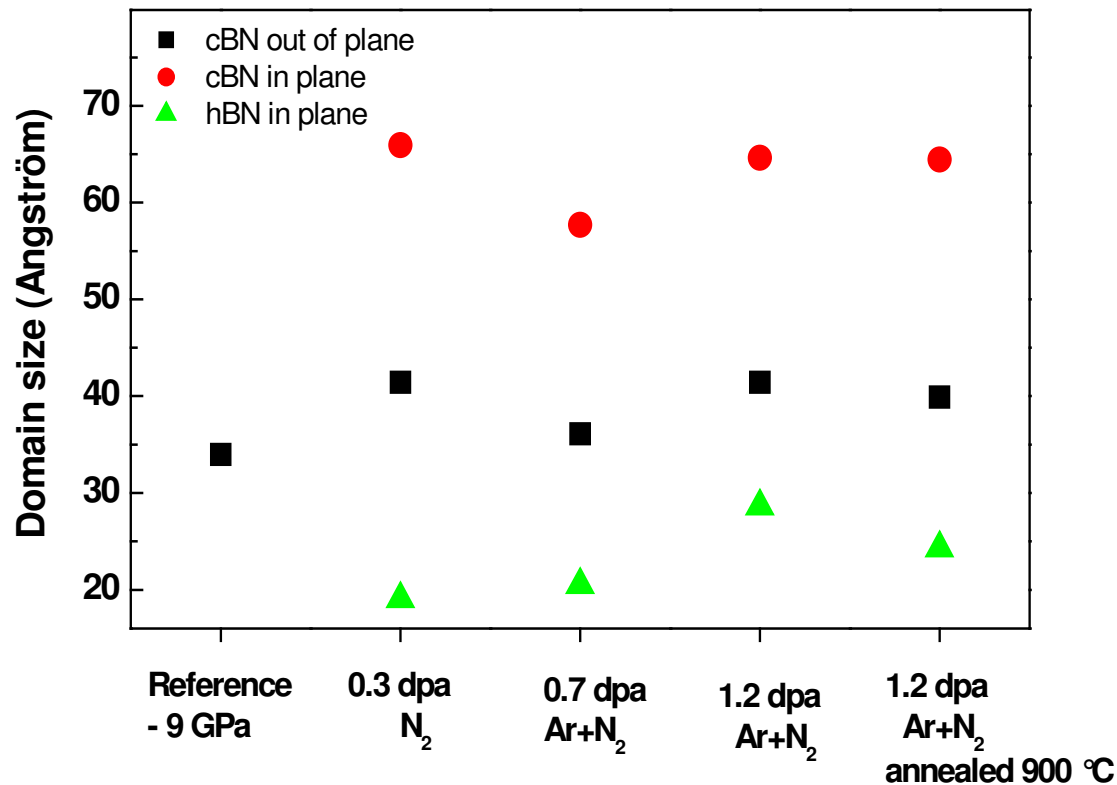
**0.6 dpa** ⇒ Transformation at low rate

**0.9 dpa**

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

**1.2 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<sup>3</sup> bonded material transformation to sp<sup>2</sup> bonding ?

- 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