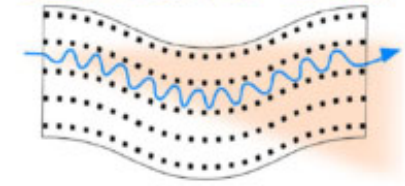


TECHNO-CLS



WP4 - CLS TECHNOLOGY 2ND YEAR REPORT – PART 2

University of
Kent



HELLENIC MEDITERRANEAN UNIVERSITY

JOHANNES GUTENBERG
UNIVERSITÄT MAINZ



UNIVERSITÀ
DEGLI STUDI
DI PADOVA



Outline

- Superlattice: model and manufacturing
- Superlattice: characterization
- Characterization for others partners

SUPERLATTICE: MODEL AND MANUFACTURING

Objectives

- *O4.1 Fabrication of high-quality bent and periodically bent crystals (silicon, germanium) by means of surface modification techniques. Extensive characterization of samples via XRD in parallel with their fabrication.*
- *O4.2 Optimization and characterization of the PLM process to fabricate surface localized stressor alloys on Si and Ge surface; realization of PLM processed PC and PBC optimised for gamma emission.*
- *O4.3 Experimental determination of AW generation and propagation in crystals; monitoring dynamic bending of the crystals.*
- *O4.4 Feasibility studies on laser pulse AW generation and propagation; monitoring the dynamic bending of the crystals.*
- *O4.5 Periodically bent Si-Ge superlattices with parameters suitable for channeling experiments with e- and e+-beams → **extended to Boron-doped diamond superlattices***



Different CU technology for PBCs

→ Strained Superlattice Method

- Crystal planes undulation is not macroscopic and linked to sample shape/stress/strain
- But is directly forced in the crystal lattice by doping of the material, following Vegards' law

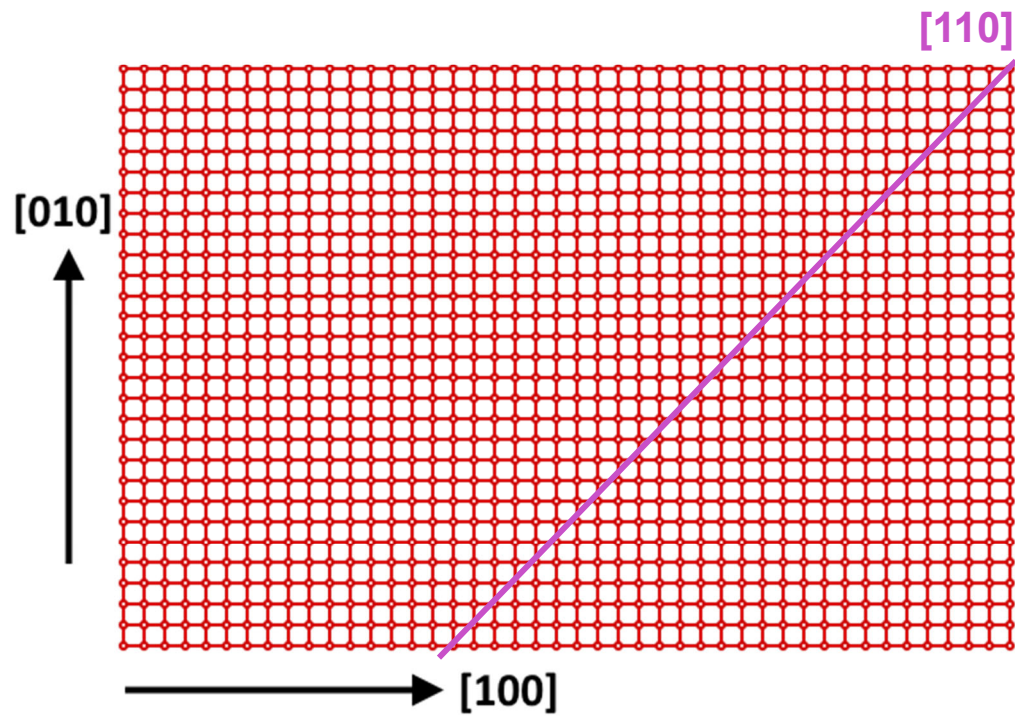
Advantages:

No subsequent mechanical operation → no damage

*Sub-micrometric growth control → **short periods** (λ from few microns or a few tens of microns), suitable for e^- and e^+ for Gamma-ray generation*

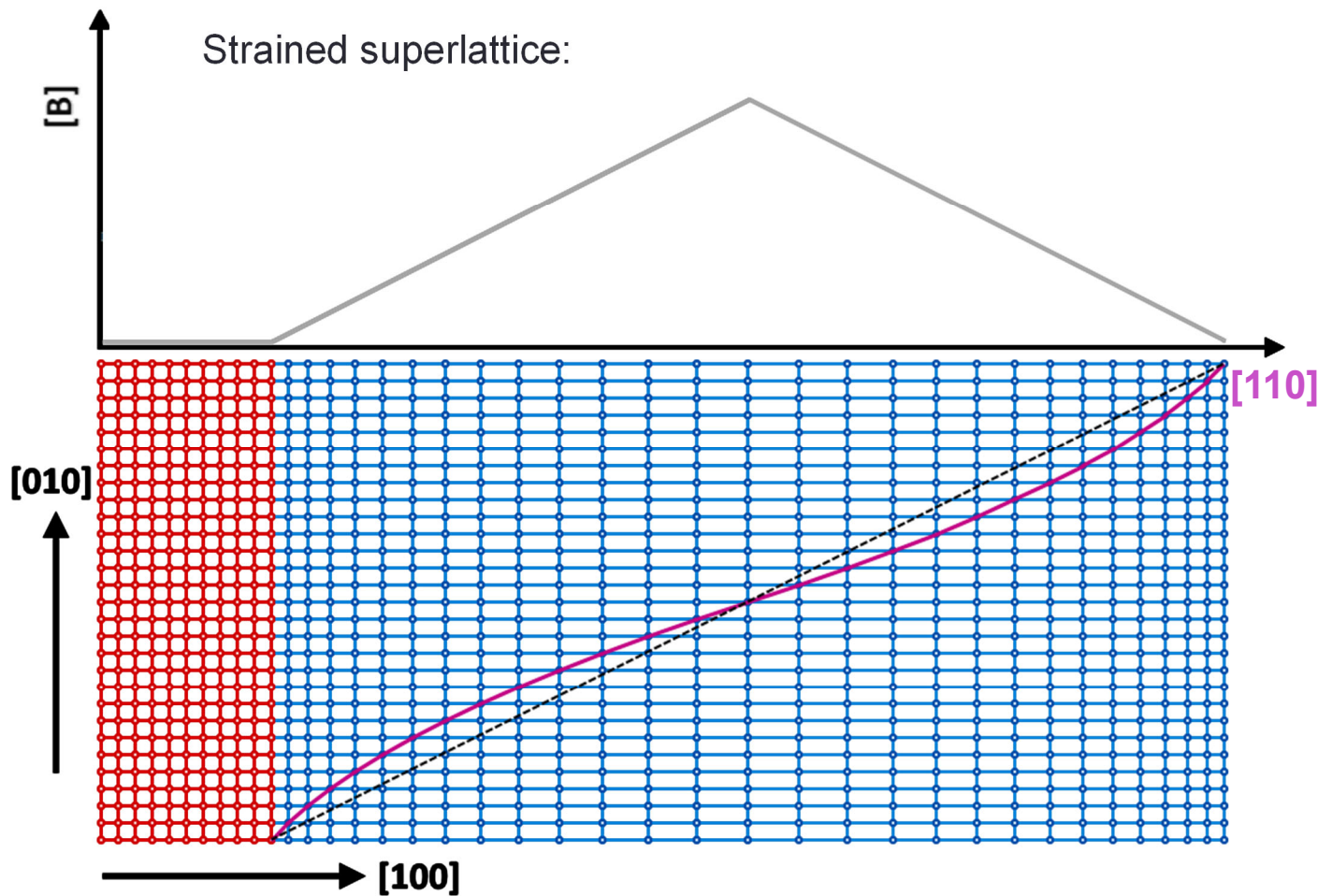
Superlattice principles

Normal cubic crystal: flat planes



Superlattice principles

Strained superlattice:



Undoped substrate

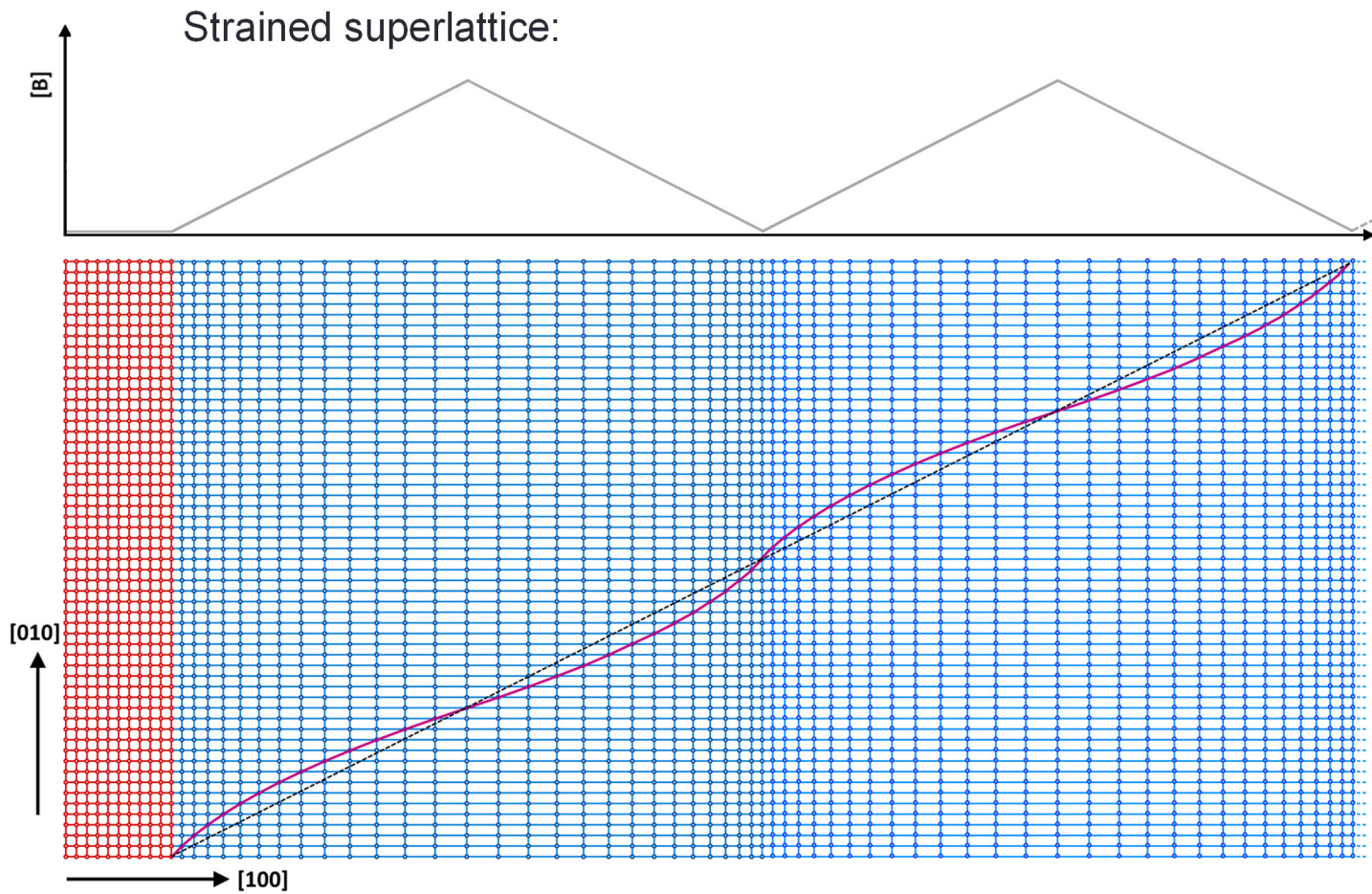
Growth with increasing doping
= increase in crystal parameter
along $[100]$.

Growth with decreasing doping
= decrease in crystal parameter
along $[100]$

**Channeling along $[110]$:
undulation**



Superlattice principles



On several successive doped layers = periods

Key points

➤ Matrix material

➤ Doping elements

**B-doped
diamond**

➤ Number of periods

➤ Period size

➤ Level of doping

= Profiles : two models (from calculation)

Property	[unit]	Si	4H-SiC	GaN	Diamond
Band gap	E_G [eV]	1.1 <i>i</i>	3.23 <i>i</i>	3.45 <i>d</i>	5.45 <i>i</i>
Dielectric constant	ϵ_r	11.8	9.8	9	5.5
Breakdown voltage	F_B [MV/cm]	0.3	3	2	10
Thermal conductivity	λ [W/cm.K]	1.5	5	1.5	22
Sat. drift velocity e ⁻	v_s [10^7 cm/s]	1.0	2.0	2.2	2.7
Sat. drift velocity h ⁺	v_s [10^7 cm/s]	1.0			1.1
Electrons mobility	μ_e [cm ² /V.s]	1500	1000	1250	1000
Holes mobility	μ_h [cm ² /V.s]	480	100	200	2000
Johnson's FOM	JFM [10^{23} $\Omega.W/s^2$]	2	911	490	3064
Keyes' FOM	KFM [10^7 W/K.s]	9	49	16	215
Baliga's FOM	BFM [Si=1]	1	554	188	23017

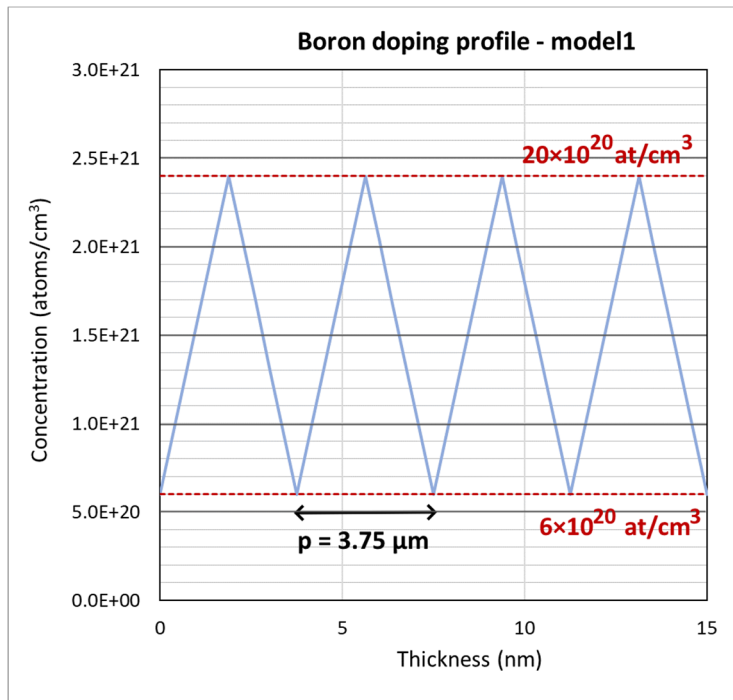
***Excellent thermomechanical properties and
synchrotron adaption for particles channeling purpose***

Two models calculated

JOHANNES GUTENBERG
UNIVERSITÄT MAINZ



@Mainz team



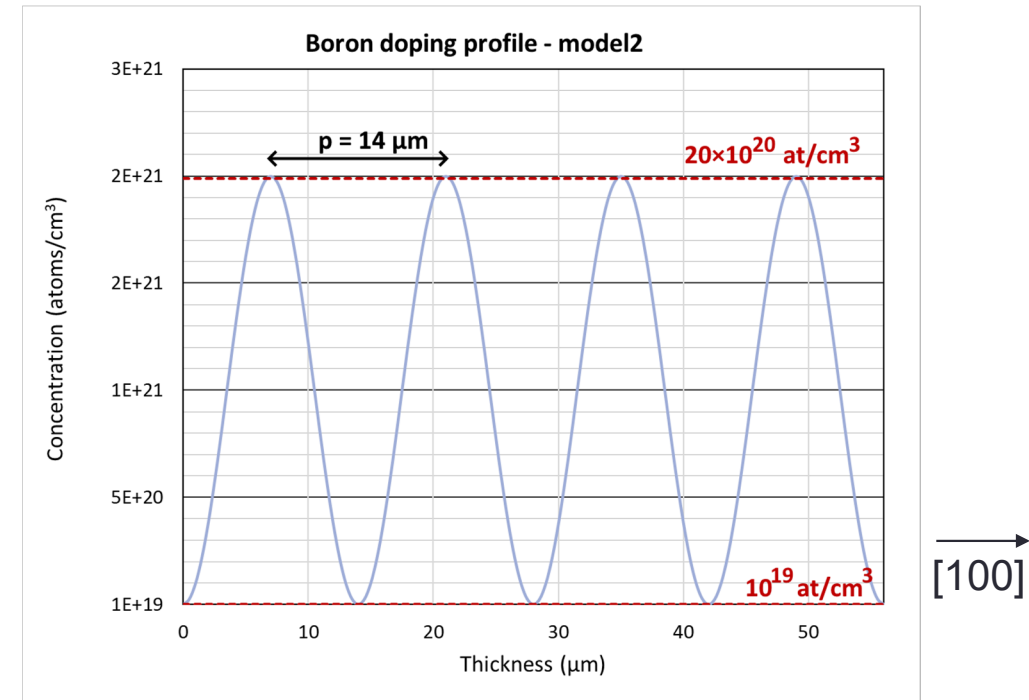
Design for electron channeling

Year I-II: manufacturing two and four periods



MBN
Research Center

@MBN team

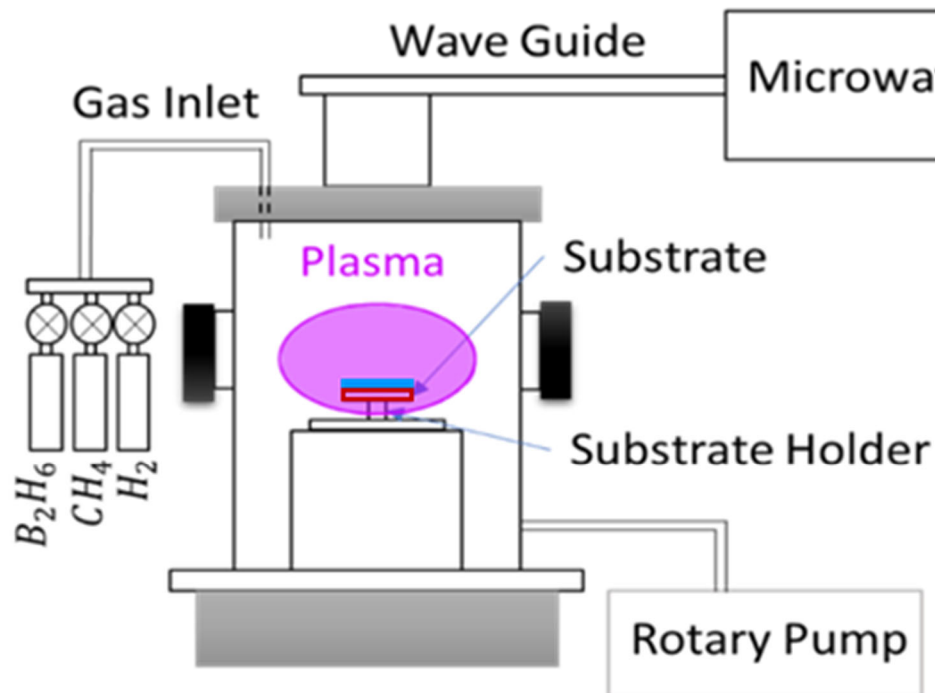


Design for electron/proton channeling

Year II: manufacturing two periods

Boron-doped diamond manufacturing

Microwave Plasma Chemical Vapor Deposition (MPCVD) growth process



Temperature 850°C
H₂ = for plasma environment
CH₄ = Carbon precursor for diamond
*B₂H₆ = Boron precursor for doping, **variable flow***

Collaboration with
Institut Néel (CNRS)
Grenoble, FRANCE



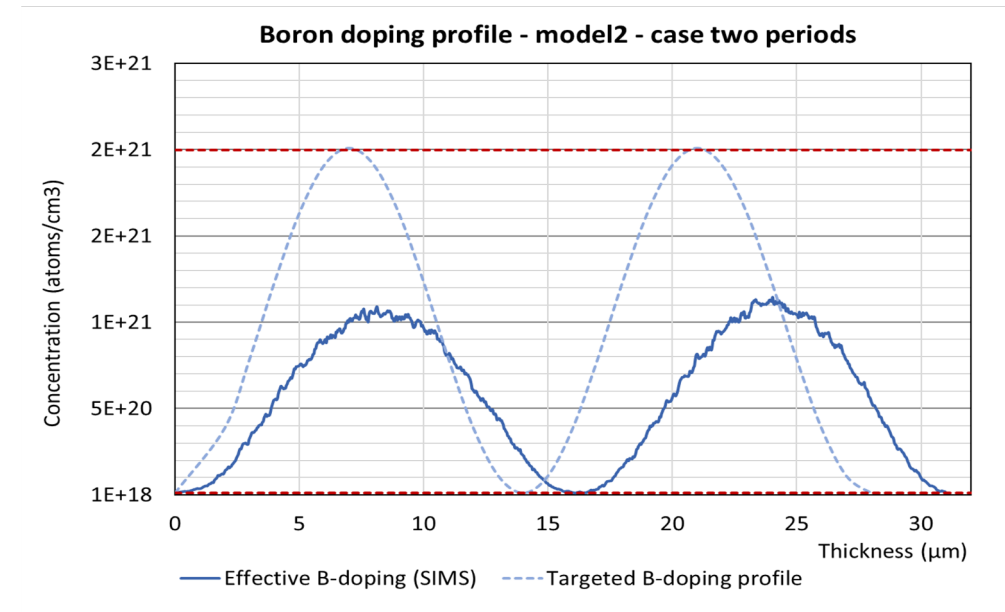
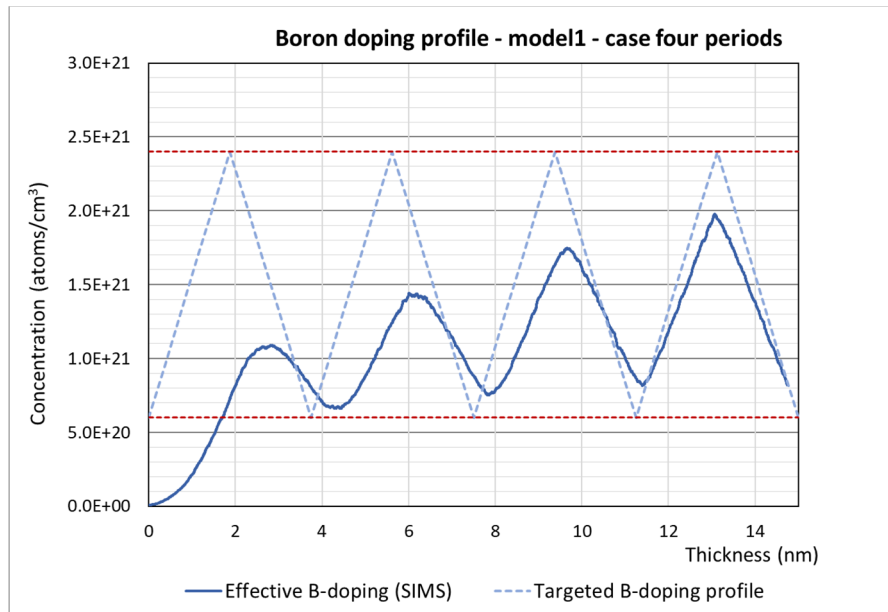
CHARACTERIZATION OF SUPERLATTICES

- Doping profile by SIMS
- Surface characterization by metrology
- Crystalline quality by X-Ray Diffraction Imaging

Pre-characterization of CU samples



Measurement of effective Boron profiles → Secondary ion mass Spectrometry (SIMS)



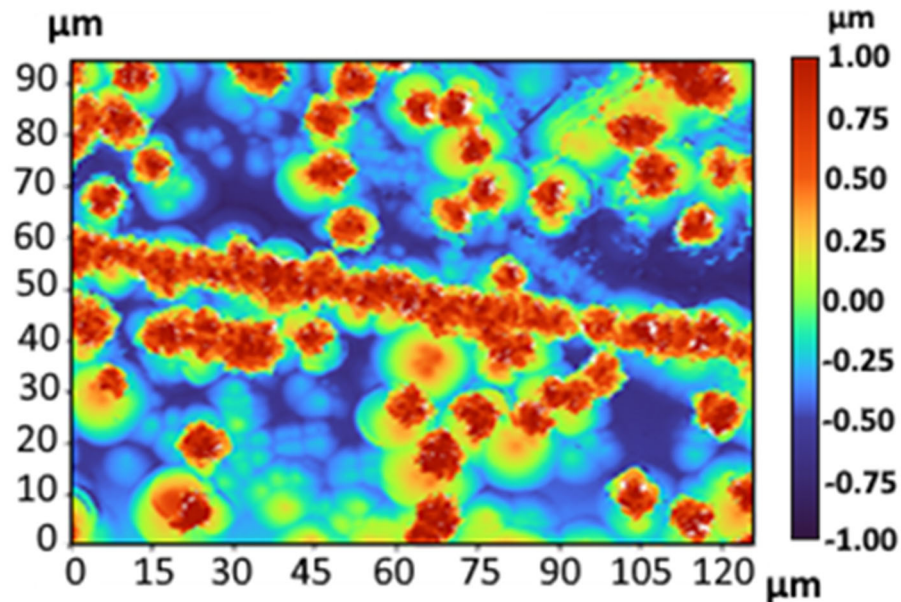
Control of thickness (each period and total). Progress in achieving the targeted, regular profile.
Doping level in expected range.

Pre-characterization of CU samples



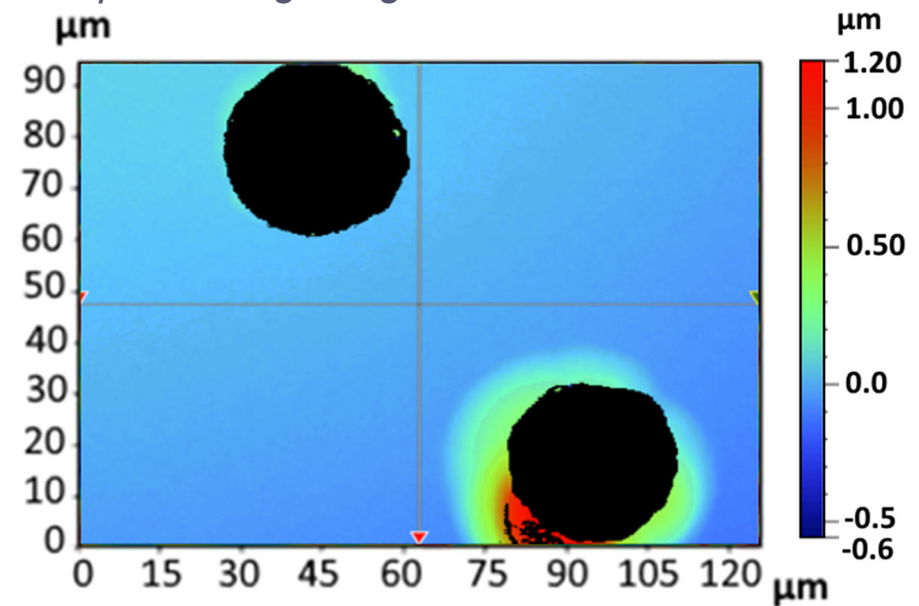
Measurement of roughness and shape of samples → Surface metrology

Hillock = defect due to highly Boron doped during the growth



$$R_a = 0.38\mu m; R_p = 2.05\mu m$$

Case with dense hillocks population



Recent sample: wide areas without hillock and low roughness

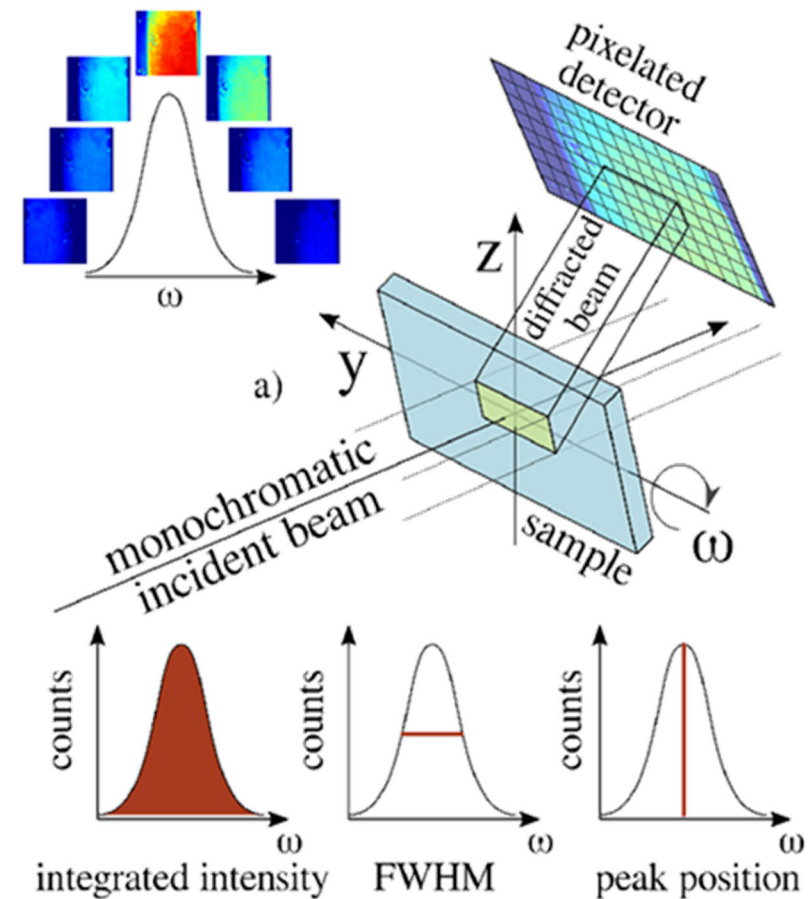
Characterization by X-ray diffraction imaging

@ESRF – BM05 beamline

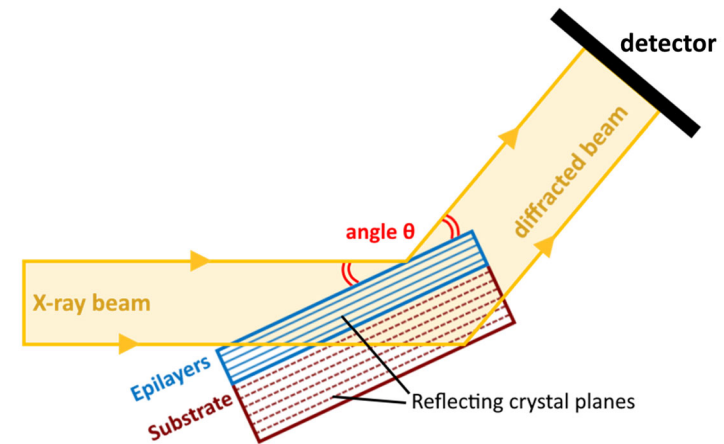
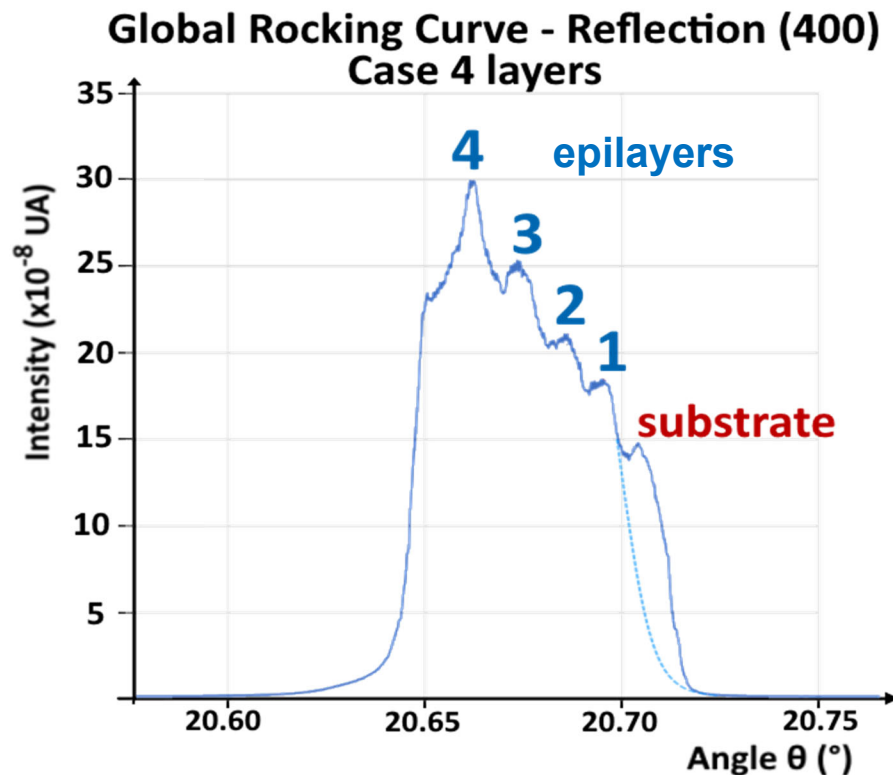
→ Powerful technique for crystal lattice characterization:

- *Crystalline quality
- *Lattice parameter variation
- *Curvature, strain
- *Defects

→ Essential for superlattice characterization but also for other types of CU



Characterization by X-ray diffraction imaging

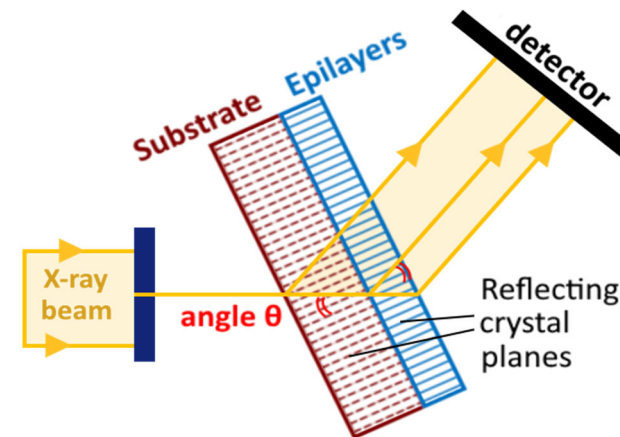
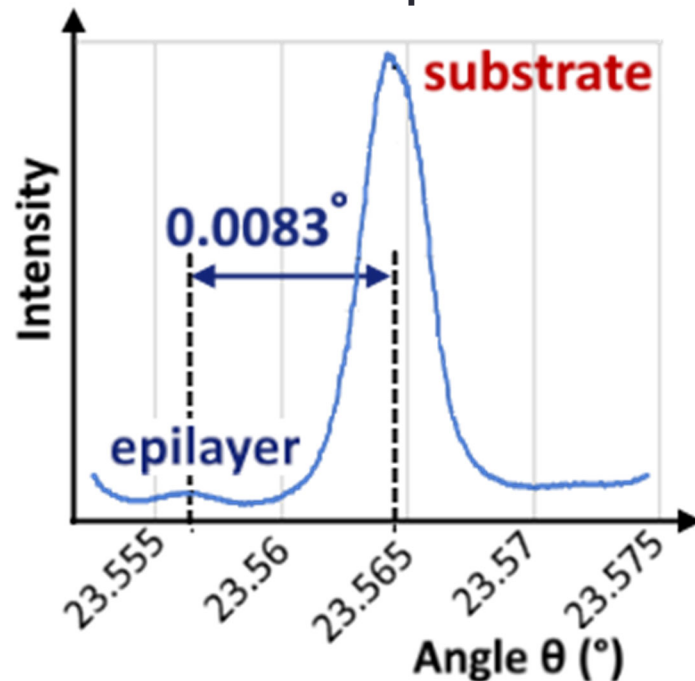


measurement on (400) planes
= parallel to the surface
= direction of growth

→ Estimation of the crystal lattice parameter: increases because of doping as expected

Characterization by X-ray diffraction imaging

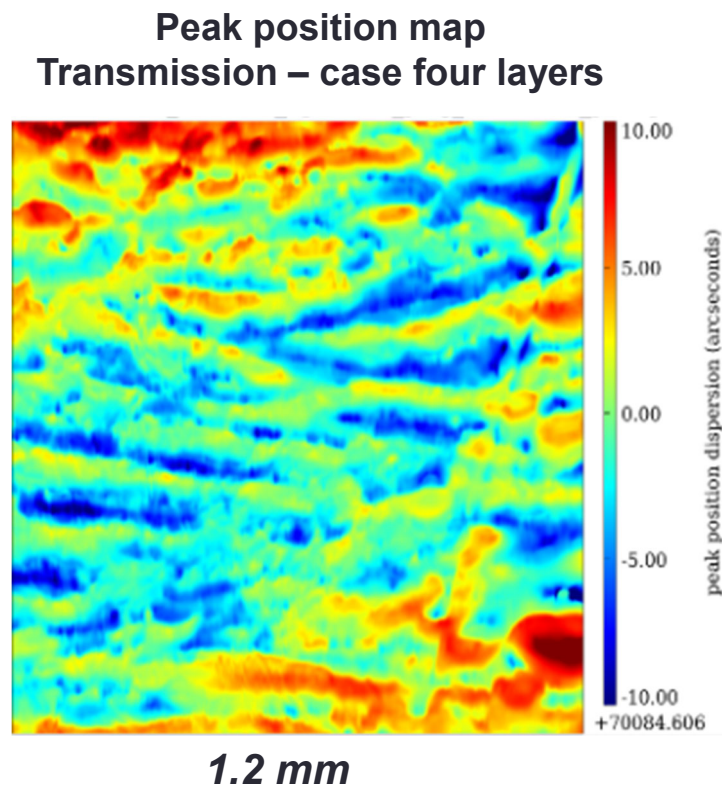
Rocking Curve – Transmission (022)
Case four periods



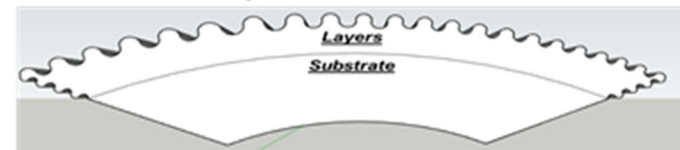
measurement on (022) planes
(by narrow beam)
sensible to the sample depth

→ Estimation of the crystal lattice parameter: increases but less and varies little

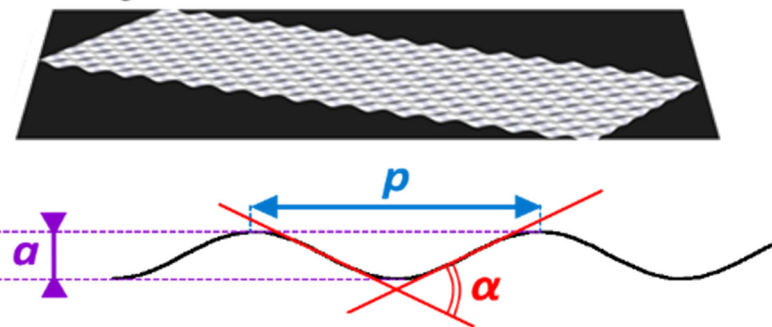
Characterization by X-ray diffraction imaging



cross-section diagram



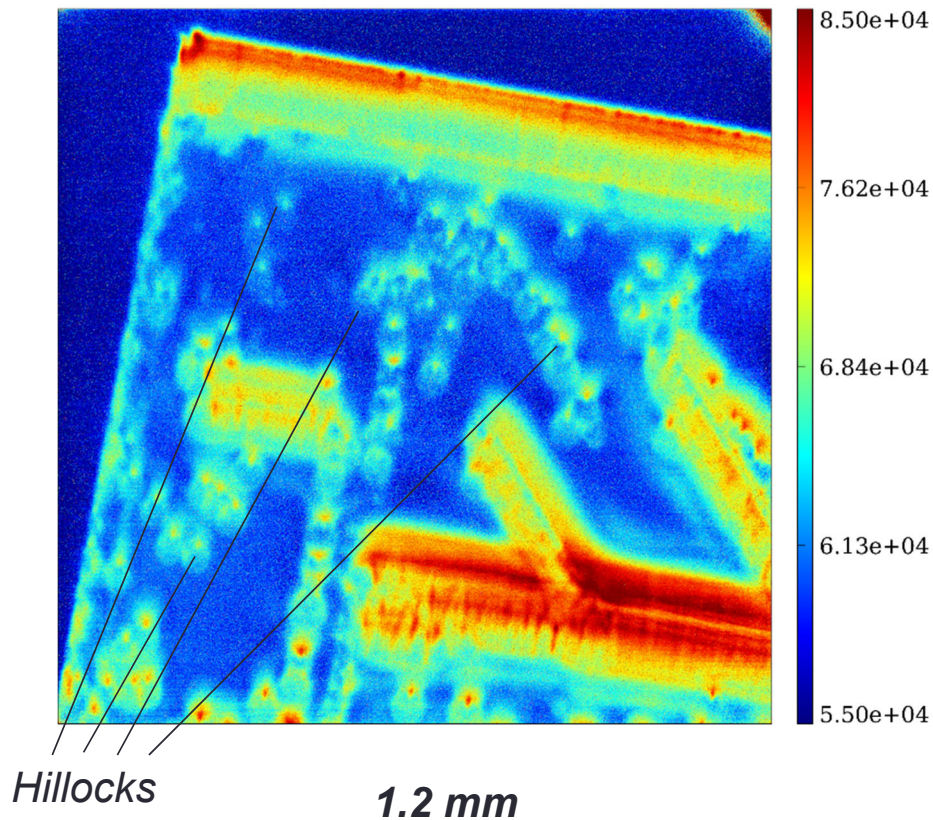
3D diagram



→ Observation of curvature
Average curvature of the sample
“egg crate foam” at smaller scale

Characterization by X-ray diffraction imaging

Integrated intensity map (AU)
Transmission



Recent case.

Intensity hillock / intensity without hillock ~ 3

Crystal quality better for areas without hillocks

And no observation of “egg crate foam”

X-RAY DIFFRACTION IMAGING FOR OTHERS PROJECT'S PARTNERS

Objectives

- *O4.1 Fabrication of high-quality bent and periodically bent crystals (silicon, germanium) by means of surface modification techniques. Extensive characterization of samples via XRD in parallel with their fabrication.*
- *O4.2 Optimization and characterization of the PLM process to fabricate surface localized stressor alloys on Si and Ge surface; realization of PLM processed PC and PBC optimised for gamma emission.*
- *O4.3 Experimental determination of AW generation and propagation in crystals; monitoring dynamic bending of the crystals.*
- *O4.4 Feasibility studies on laser pulse AW generation and propagation; monitoring the dynamic bending of the crystals.*
- *O4.5 Periodically bent Si-Ge superlattices with parameters suitable for channeling experiments with e- and e+-beams*

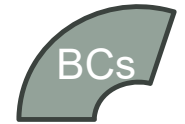
Characterization by X-ray diffraction imaging @ESRF

→ **Support for @UNIFE/INFN/UNIPD team for BCs (PLM) characterization:**

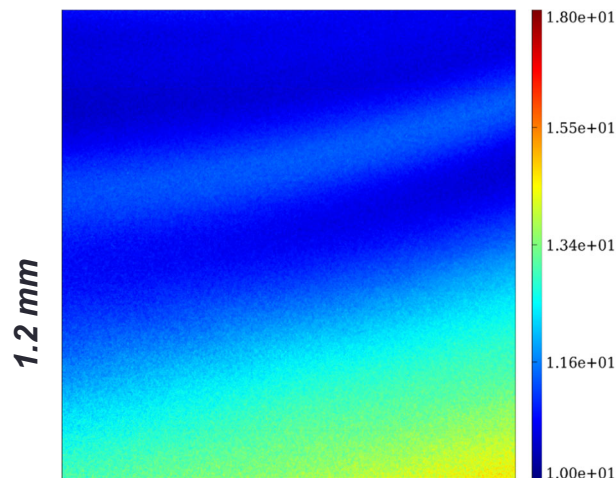
ESRF measurement for **control bulk properties of the material**

→ **Support for @UNIFE/INFN team for PBCs with thin film pattern deposition:**

ESRF measurement for **control of Silicon bulk crystal**



FWHM map (arcsec)



Example for @UNIFE/INFN
Silicon bulk for PBCs fabrication

**Excellent quality: zero
dislocation density**

(FWHM variation due to curvature associated to the way of holding the sample)

X-ray diffraction imaging data treatment and analysis

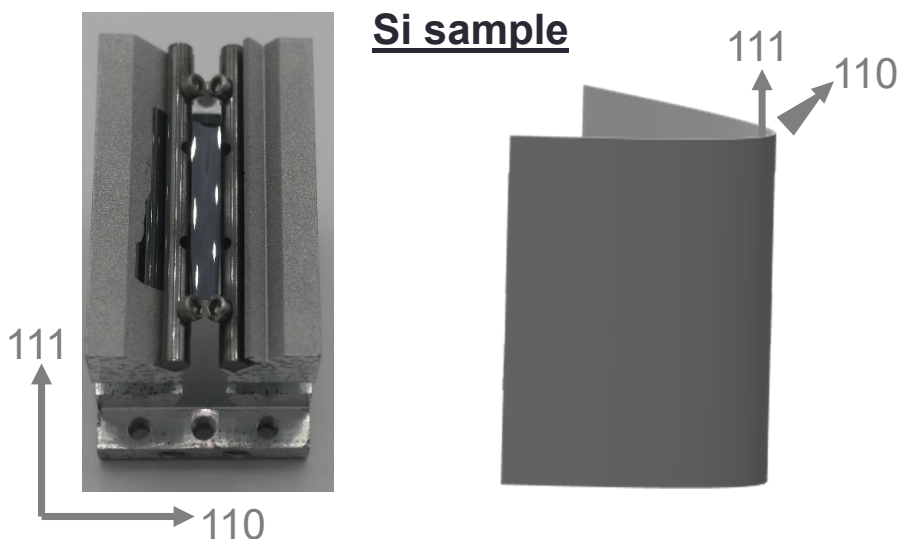
→ @ESRF team support for @UNIFE/INFN/UNIPD teams: Treatment & analysis of data acquired at the **Diamond Light Source** (same type of measurement but different data format)

→ **New python code for analysis**

on analysis of characterization of **Bents Silicon** sample

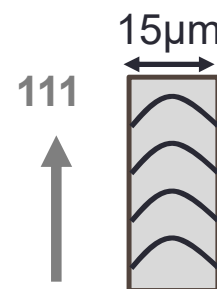


Si sample



Flexion of Si bulk thanks to metal static holder

Cross-section of Si sample



Thin Si sample flexion induces Quasi-Mosaic curvature
→ (111) planes are bent
($R_{\text{curvature}} \approx 3-4\text{cm}$)

(111) planes usable for particles channeling
→ Required characterization of crystal torsion by X-ray diffraction Imaging on (111) planes.

X-ray diffraction imaging data treatment and analysis

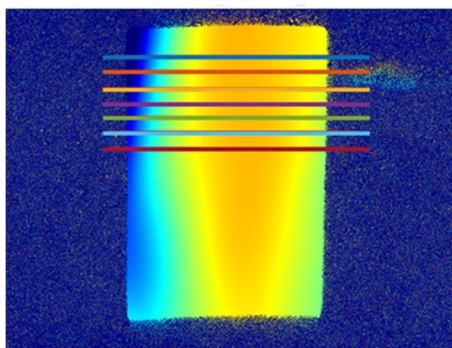
→ @ESRF team support for @UNIFE/INFN/UNIPD teams: Treatment & analysis of data acquired at the **Diamond Light Source** (same type of measurement but different data format)

→ **New python code for analysis**

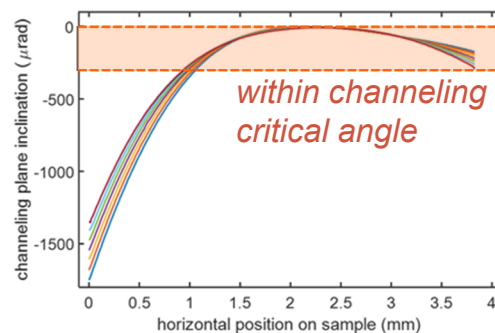
on analysis of characterization of **Bents Silicon** sample



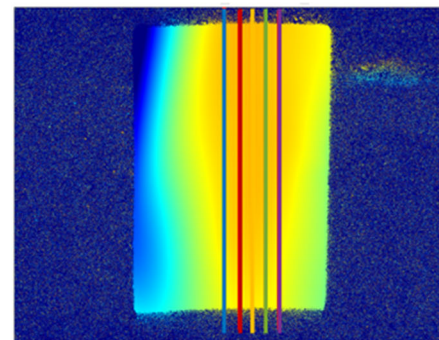
2d map for channeling planes inclination



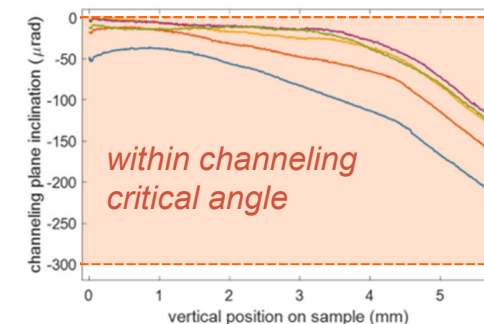
Profile map for channeling planes inclination



2d map for channeling planes inclination



Profile map for channeling planes inclination



Peak position maps and profiles provided.

Except for the 1mm near the edge, **planes remain aligned within the channeling critical angle**

X-ray diffraction imaging data treatment and analysis

→ @ESRF team support for @UNIFE/INFN/UNIPD teams: Treatment & analysis of data acquired at the **Diamond Light Source** (same type of measurement but different data format)

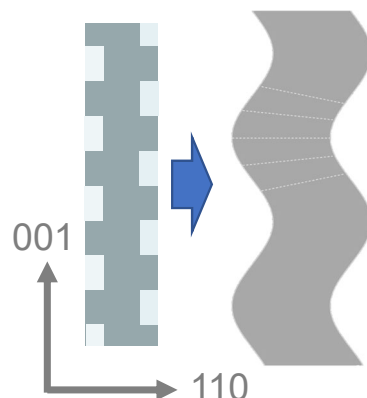
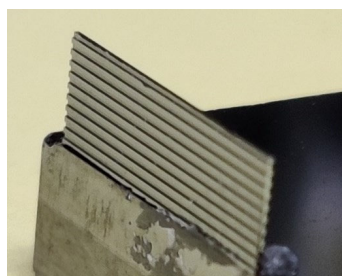
→ **New python code for analysis**

*on analysis of characterization of **Silicon Grooved Crystal** sample*

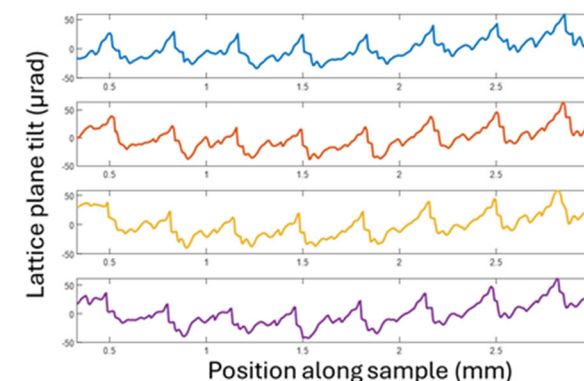
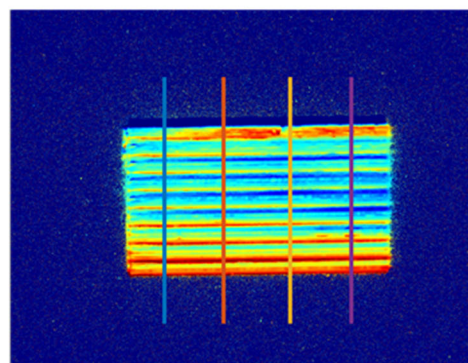


Profile map for map tilt

Si sample



2d map for plane tilt



Double grooving pattern shifted induces crystal sinusoidal deformation

→ Required to measure the crystal sinusoidal bending

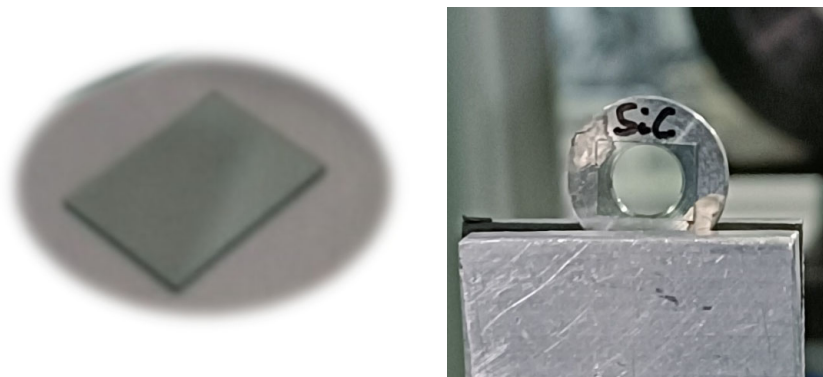
Peak position maps and profiles provided.
Evidence of periodic behaviour. Artifact from not uniform thickness and damage cause by grooving
 (resolved by upgraded techniques of SiN deposition and PLM)

X-ray diffraction imaging data treatment and analysis

→ @ESRF team support for @UNIFE/INFN/UNIPD teams: Treatment & analysis of data acquired at the **Diamond Light Source** (same type of measurement but different data format)

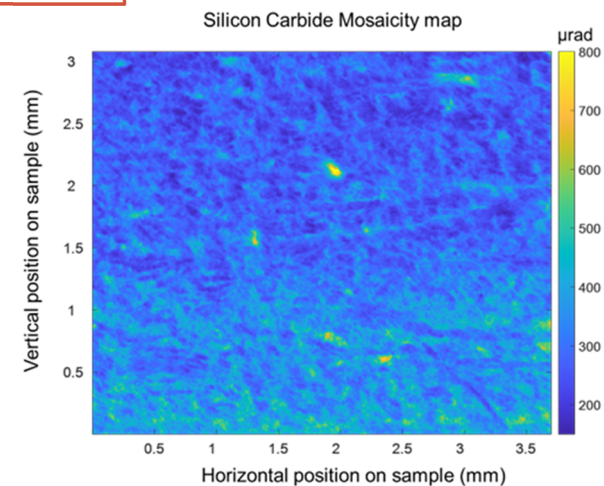
→ **New python code for analysis**

*on analysis of characterization of **Silicon Carbide** sample*



Crystal for radiation enhancement of gamma photon

→ Required to measure the bulk crystalline quality (mosaicity)



Peak position maps provided.
Imperfection detected: average mosaicity of ~400 μ rad.
Phenomena of radiation enhancement persisted (cf WP3)

CONCLUSION

- **Manufacturing and characterization of “superlattice” B-doped diamond**
Good progress on controlling of superlattice parameters
→ Channeling and radiation efficiency tested at @MAMI

- Synchrotron advanced X-ray diffraction technique to characterize other crystals as project support

- Data treatment and analysis as project support
→ Channeling and radiation efficiency tested at @MAMI

Thank you for your attention
