

# WP4 - CLS TECHNOLOGY 2<sup>ND</sup> YEAR REPORT



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**Università  
degli Studi  
di Ferrara**



**HELLENIC MEDITERRANEAN UNIVERSITY**

JOHANNES GUTENBERG  
UNIVERSITÄT MAINZ



**UNIVERSITÀ  
DEGLI STUDI  
DI PADOVA**



# 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 (Pulsed Laser Melting) 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. ->completed in the 1<sup>st</sup> year
- O4.5 Periodically bent Si-Ge superlattices with parameters suitable for channeling experiments with e- and e+-beams -> see Rébecca Dowek's talk

# Objective O4.1-2

## Static BC and PBC



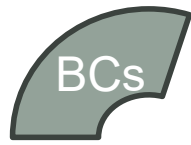
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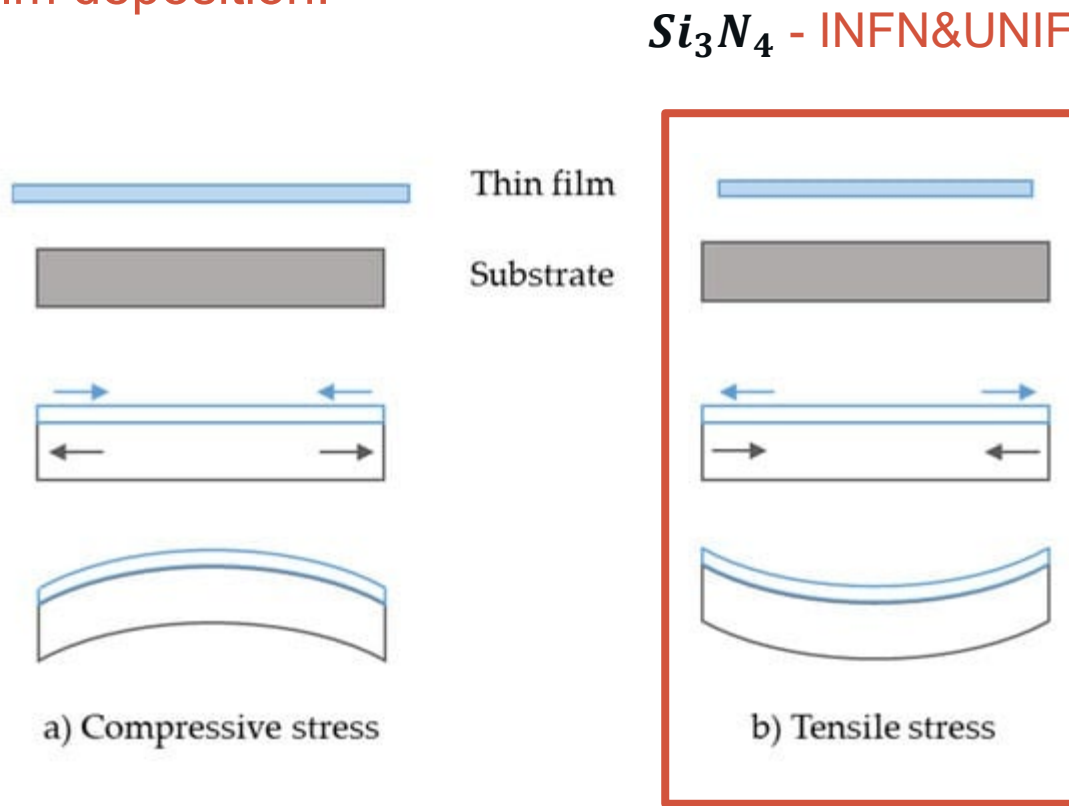
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- O4.1 Fabrication of high-quality bent and periodically bent crystals (silicon, germanium) by means of **surface modification techniques**. Extensive characterization of samples via XRD in parallel with their fabrication.
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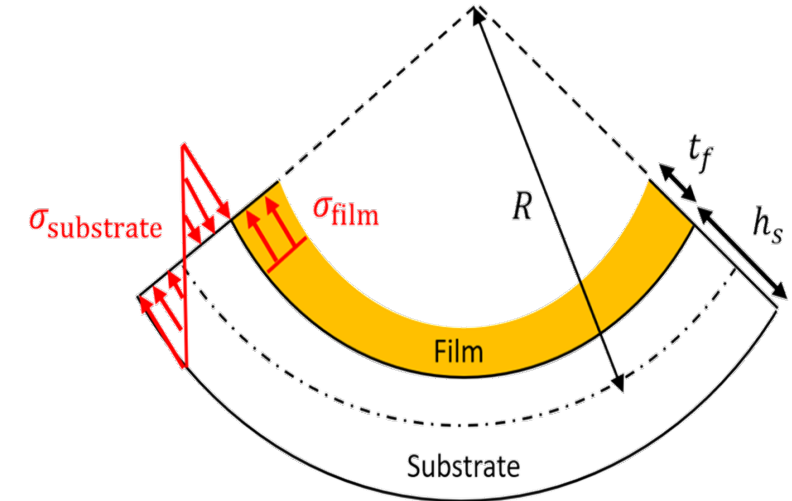
# Bent Crystals by surface modification (BCs)



Thin film deposition:

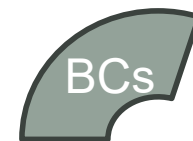


Lanzoni, Mazzolari, Guidi, Tralli, Martinelli, «On the mechanical behaviour of a crystalline undulator», Int. J. Eng. Sci., Volume 46, Issue 9, (2008)



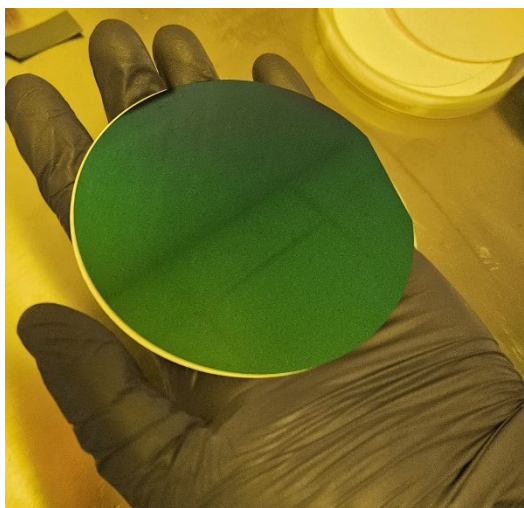
- Deposition of thin layer of  $Si_3N_4$  (Silicon Nitride) occurring at high temperature ( $\sim 1000$  °C)
- Mismatch in Thermal Expansion Coefficient between **Si substrate** and thin film give raise to a thermal stress following the Stoney law:

$$\sigma_f = \frac{\overline{E}_s h_s^2}{6R t_f}$$



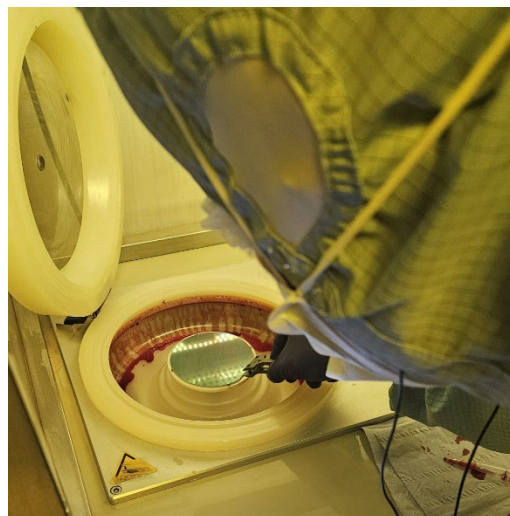
# Bent Crystals with self-standing curvature

Evaluation of film deposition:



2 Sides  $Si_3N_4$

Photoresist  
1 side  
+  
BHF solution



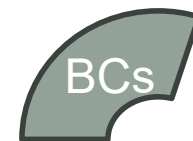
1 Side  $Si_3N_4$

**Optical Interferometry Zygo NX2:**

Profile analysis of the physical surface of the sample with vertical precision  $\sim 1$  nm

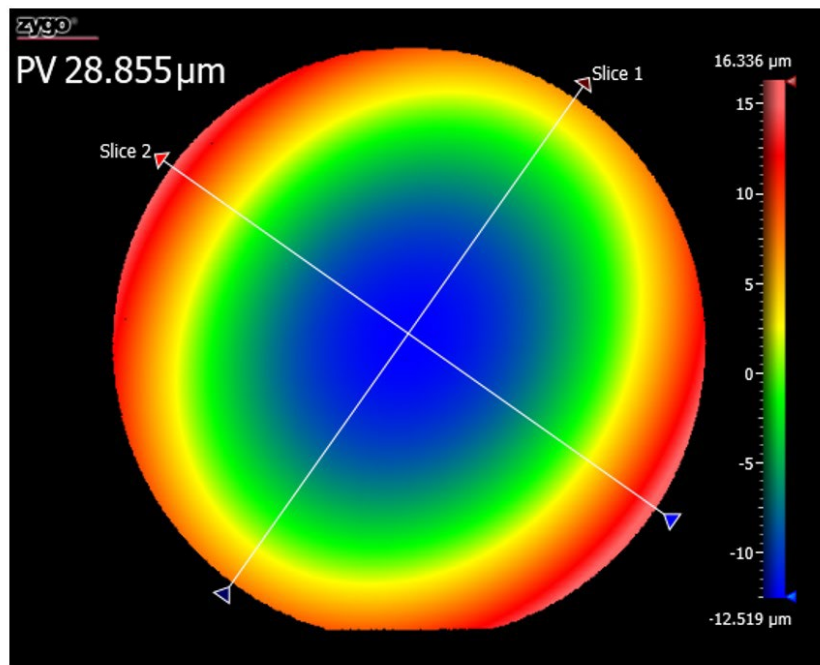


Quality control of film deposition:  
Thickness of Silicon Nitride  $\approx 400$ nm

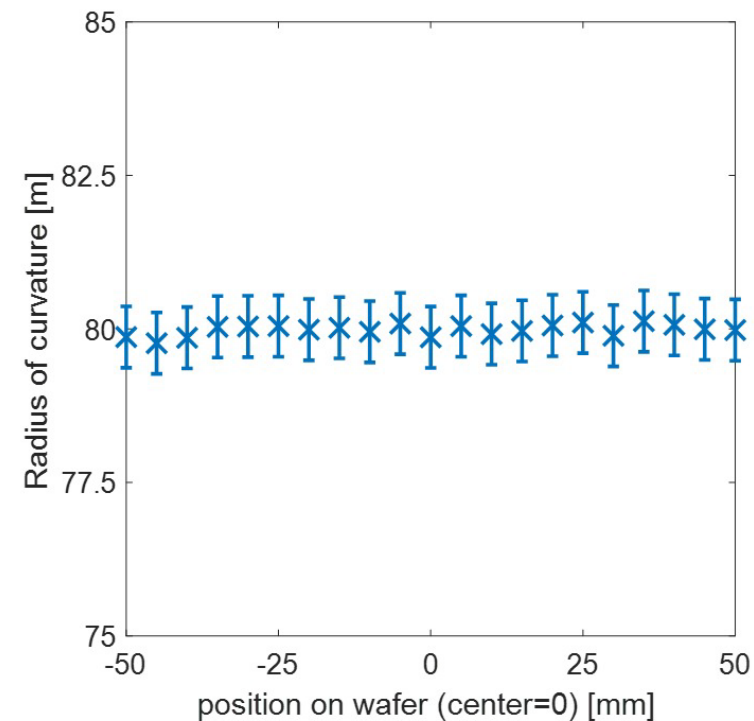


# Bent Crystals with self-standing curvature

- Profile of the wafer surface was measured with Zygo VeriFire HDX interferometer

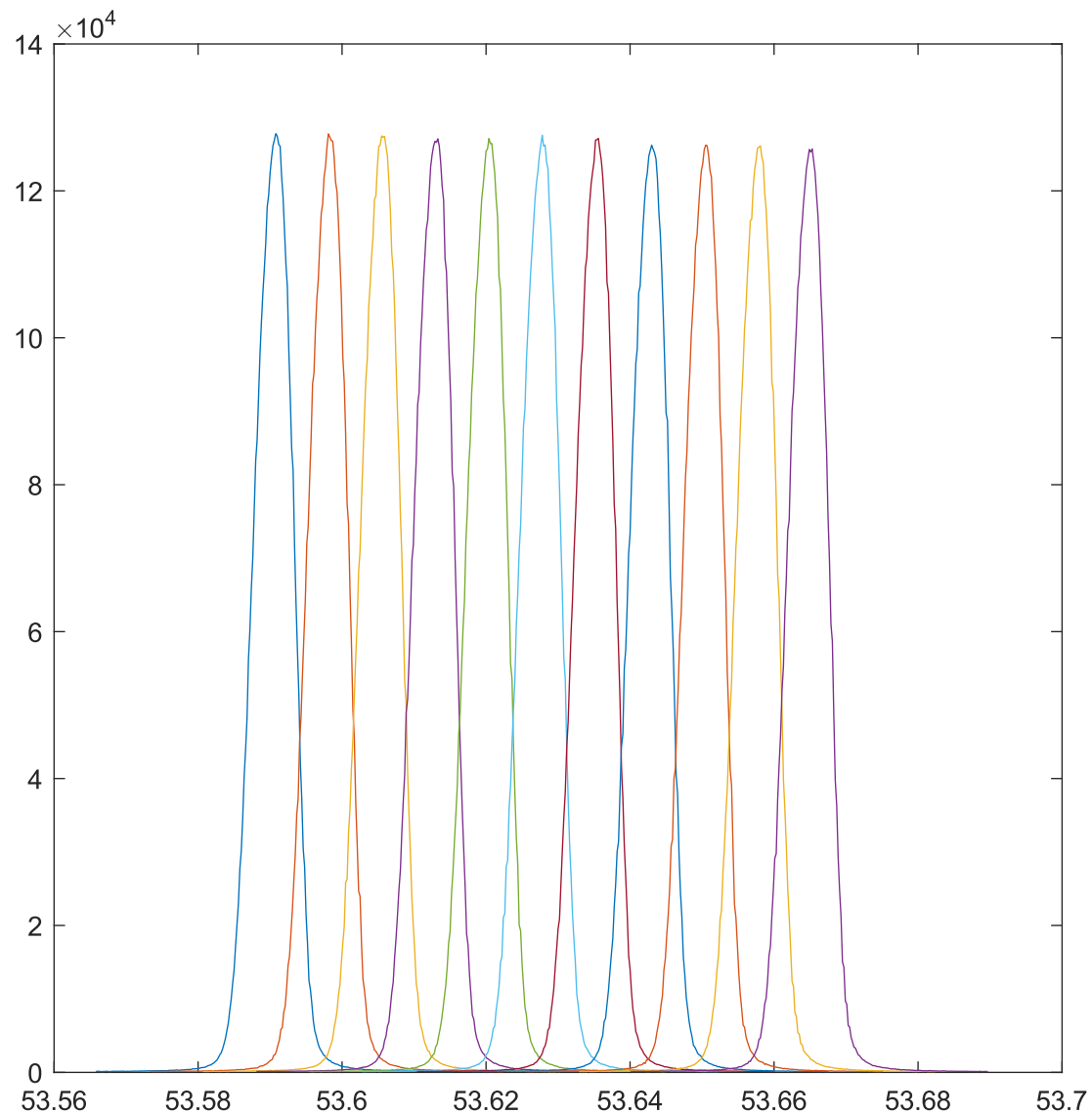
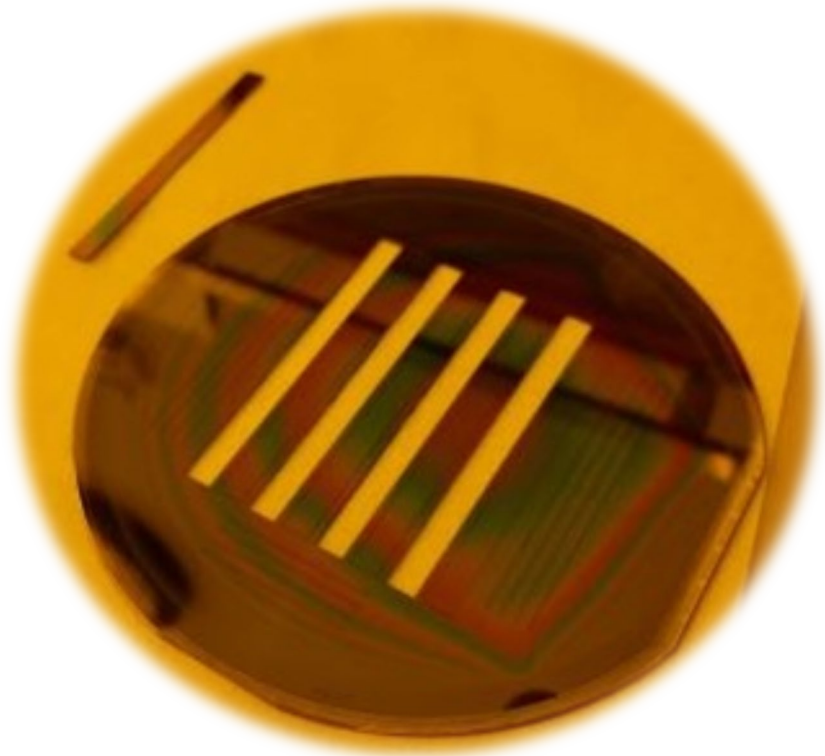


- X-rays diffraction with HR-XRD allowed quantitative analysis of the lattice planes of the wafer

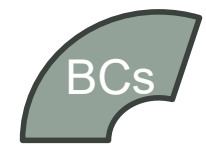


# Bent Crystals with self-standing curvature

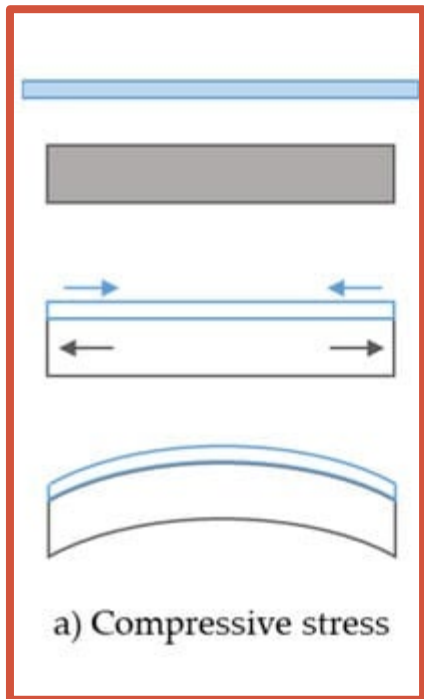
Silicon sample produced  $0.5 \times 4 \times 55 \text{ m}^3$ , bending characterized with HR-XRD: predicted channeling deflection of the beam of  $60 \mu\text{rad}$  (adapted for multi-GeV beams)



# Ge Bent Crystals by PLM surface modification (BCs)



$Sb_xGe_{1-x}$  - UNIPD

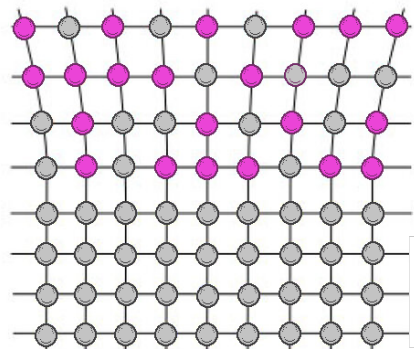


Thin film

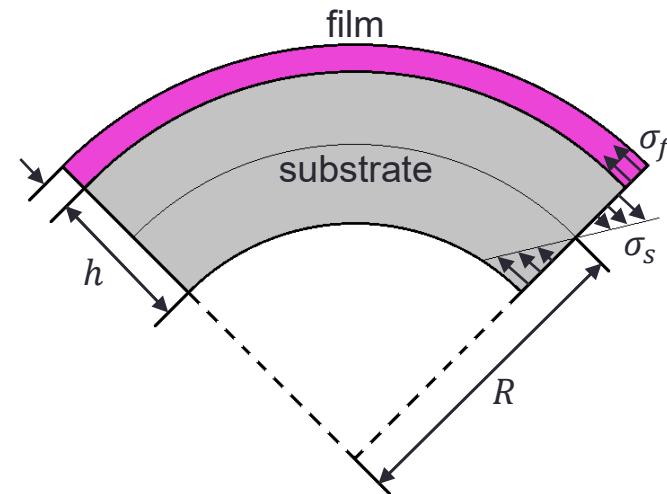
Substrate

Ge-Sb alloy

Ge bulk



$$f = \frac{a_f - a_s}{a_s}$$



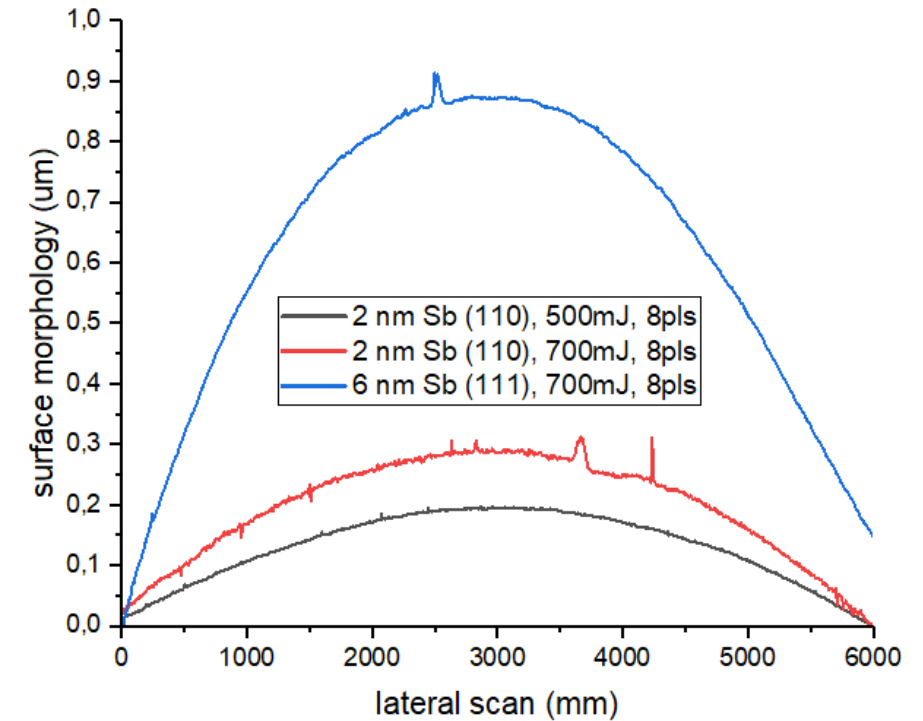
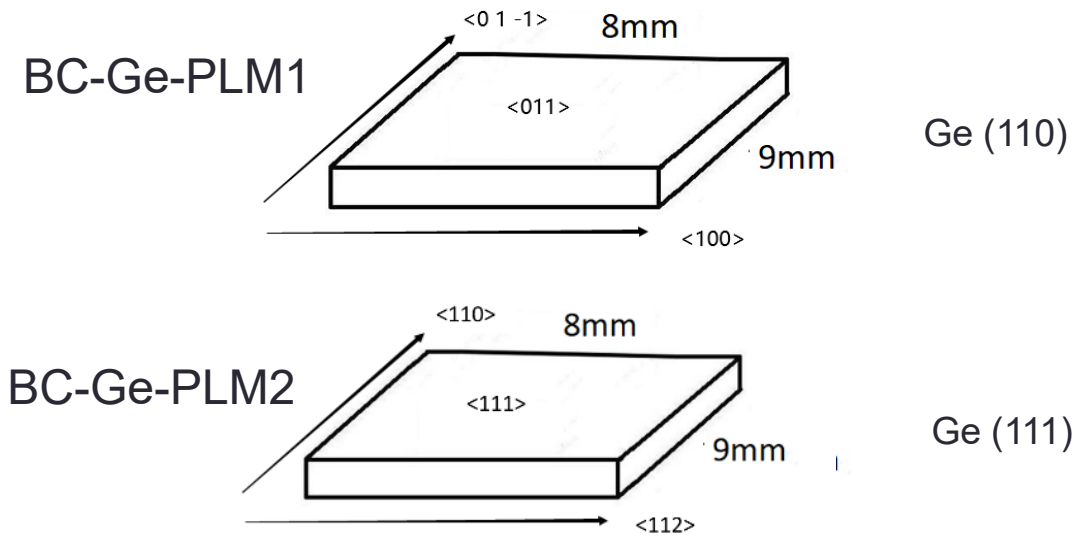
- Creation of a  $Sb_xGe_{1-x}$  thin film by Sb sputtering and Pulsed Laser Melting process that forms a coherent epitaxial constraint.

- Integrated misfit between the film and the Ge substrate induces curvature:

$$\int f dt_f = \frac{h_s^2}{6R}$$



# UNIPD – PLM self-standing curved crystals



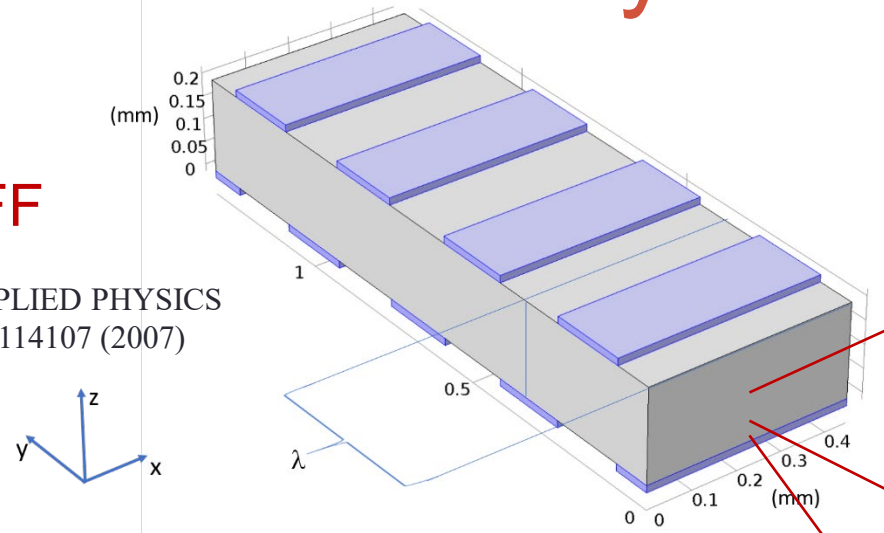
Crystal id.	Plane	Max $\int f dt_f$ (HRXRD) nm	$h_S$ (mm)	R (Stoney + HRXRD) m	R (Stylus prof.) m
BC-Ge-PLM1	(110)	0.46	0.20	14.5	16.6
BC-Ge-PLM2	(111)	0.80	0.15	4.6	4.5

# Periodic stressor layers: 2 patterning designs for STATIC Periodically Bent Crystal

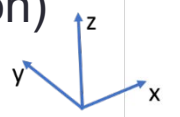
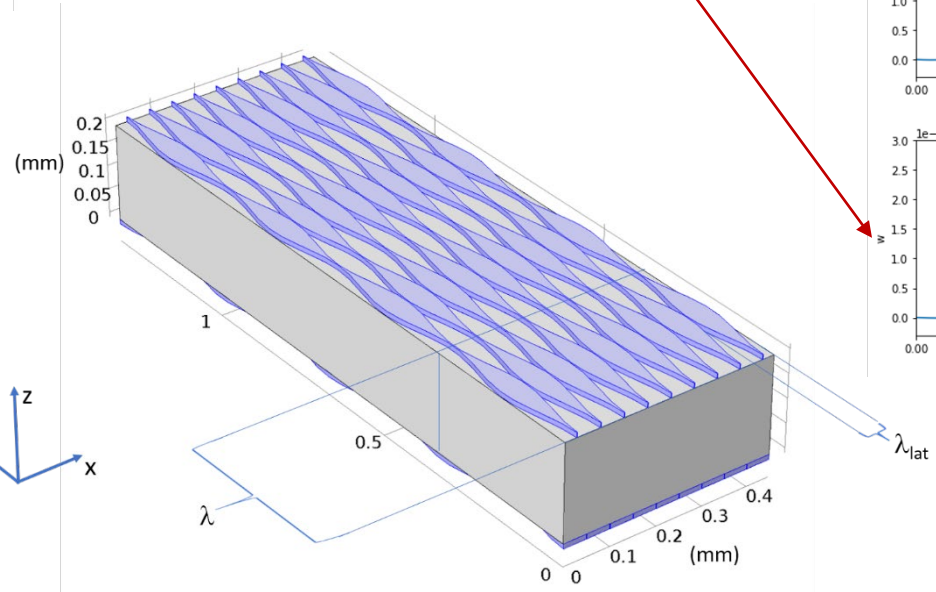


**ON/OFF**

V. Guidi et al. APPLIED PHYSICS LETTERS 90, 114107 (2007)

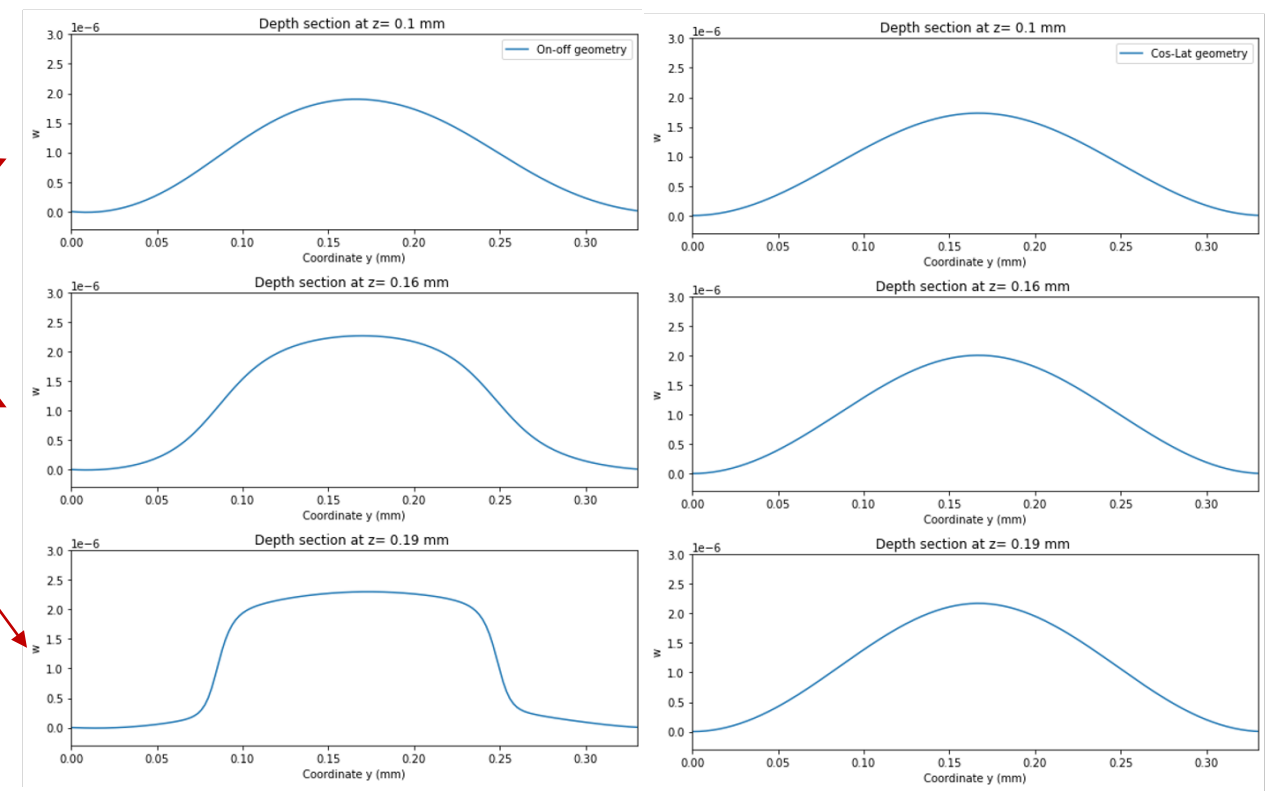


**COS/LAT**  
(feasible with lithography optimization)



On-Off Geometry

Cos-Lat Geometry



$$\lambda_{Lat} = 25 \mu\text{m}$$

Patenting of Cos-Lat design with UNIPD, UNIFE, INFN technology transfer offices has been started

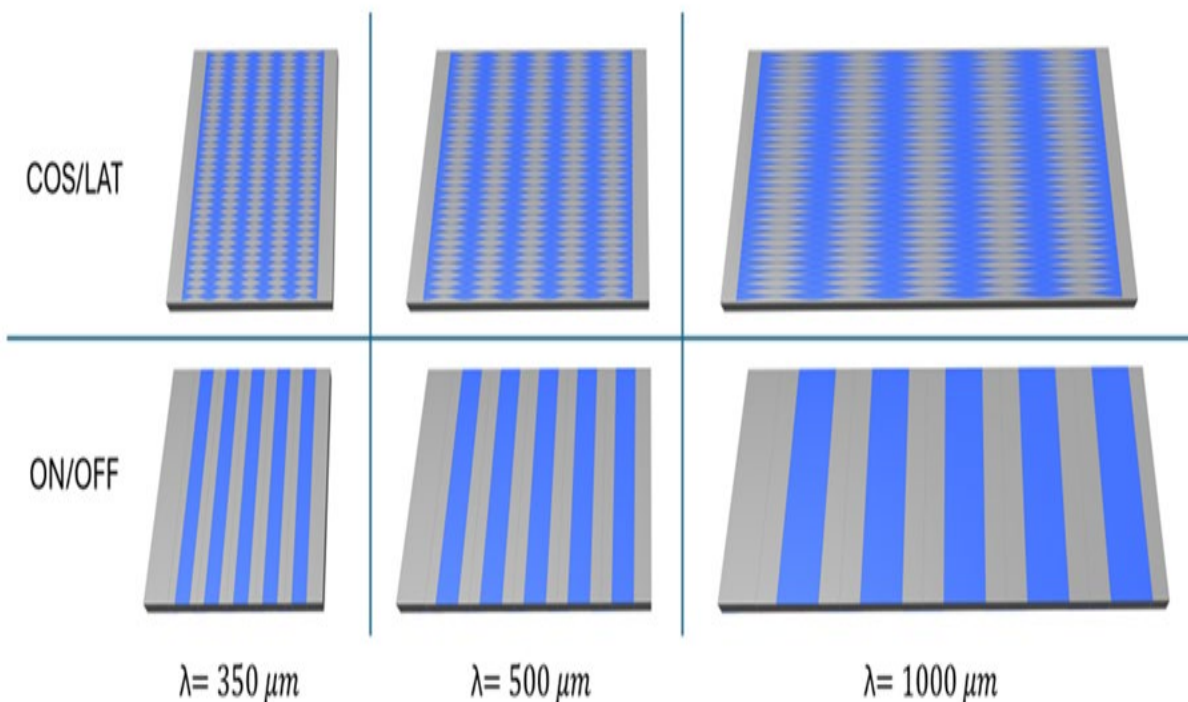
# Periodically bent crystals (PBCs)



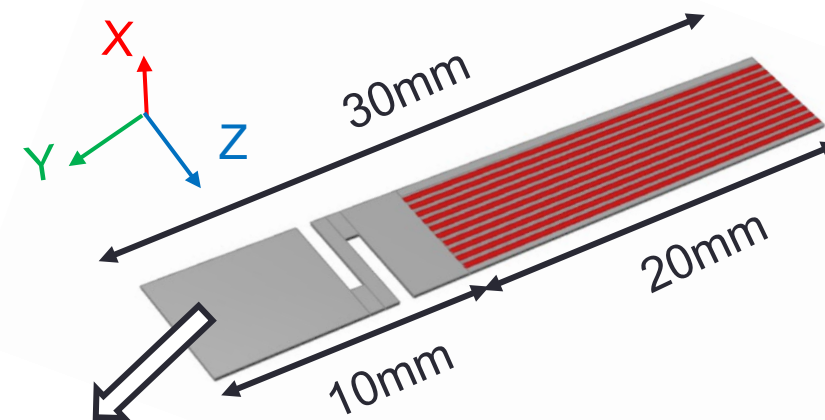
Prototypes manufacturing with both design patterning.

Optimization of parameters is obtained via FEM simulation both for ON/OFF and COS/LAT patterning

- $\lambda_{undulator}$  from 300 to 1000 micron ( $\gg \lambda_{channeling}$ )
- **Amplitude** from  $10*d$  to  $100*d$  (d is the lattice constant)
- **Suitable for positron beam with energy  $> 1$  GeV**



## Example of real undulator design:

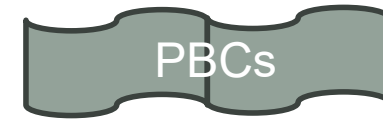


Region clamped in order to handle it during test

Undulator has a length of 30mm divided in:

- Support region of 10mm
- Active region periodically bent of 20mm

# Periodically bent crystals (PBCs)



FEM simulation to obtain spectrum emission:

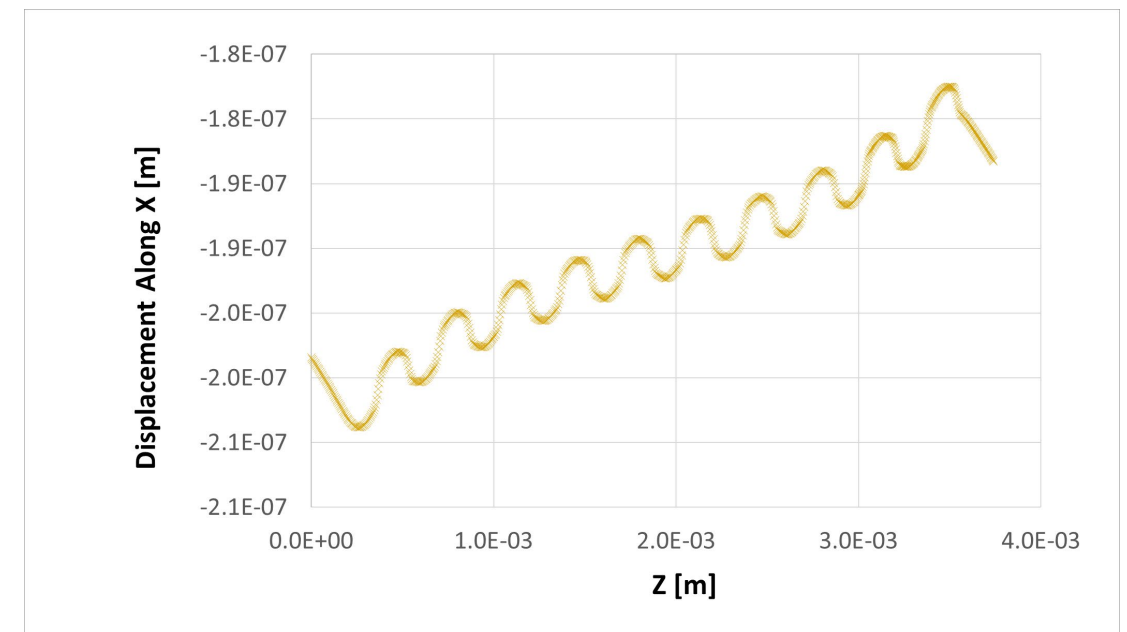
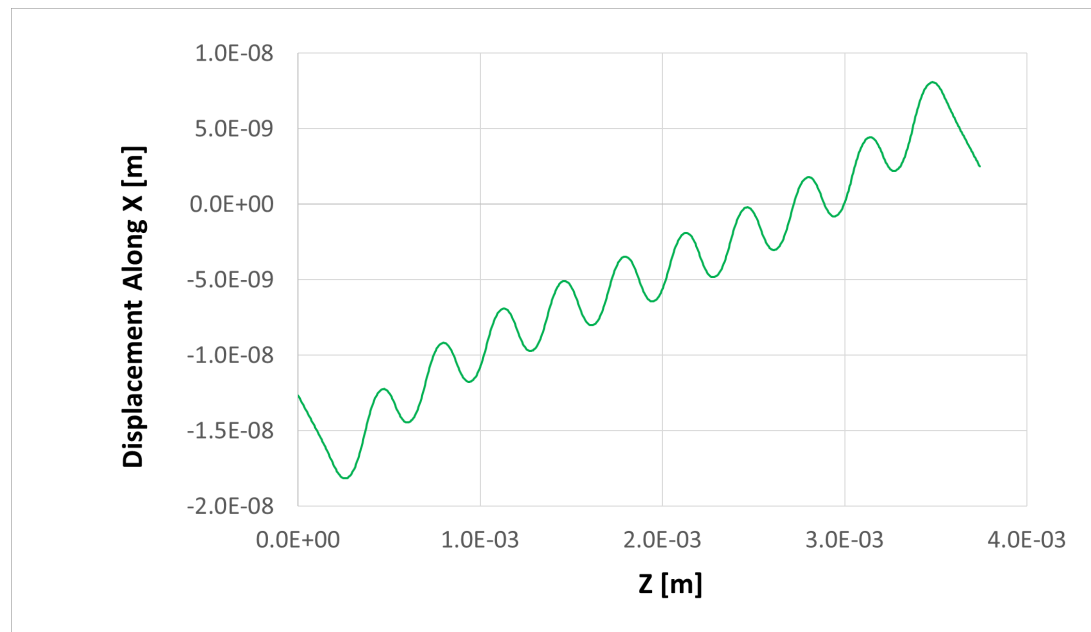
**Activity in synergy with WP2**

**Example of deformation for ON/OFF undulator:**

harmonics ↑

Middle height of undulator

At 200um to the surface of undulator



For each undulator has been performed FEM simulation obtaining the deformation amplitude at different height (0 to 160um) along the beam direction in order to simulate **the emission spectrum using MBN software for 10-20 GeV positrons (LALP CU)**

# Litography of Silicon Nitride tensile PBCs



## Prototypes of mask for photolithography:

For one wafer 2 masks, one for each sides is necessary →  
In order to produce all different prototypes, 4 masks have been manufactured

### Masks 1A+1B:

Number of periods:	Period (um)	334	500	1000
10	ON/OFF	5	5	3
	COS/LAT	5	5	3

### Masks 2A+2B:

Number of periods:	Period (um)	334	500	1000
5	ON/OFF	3	3	3
	COS/LAT	4	3	3

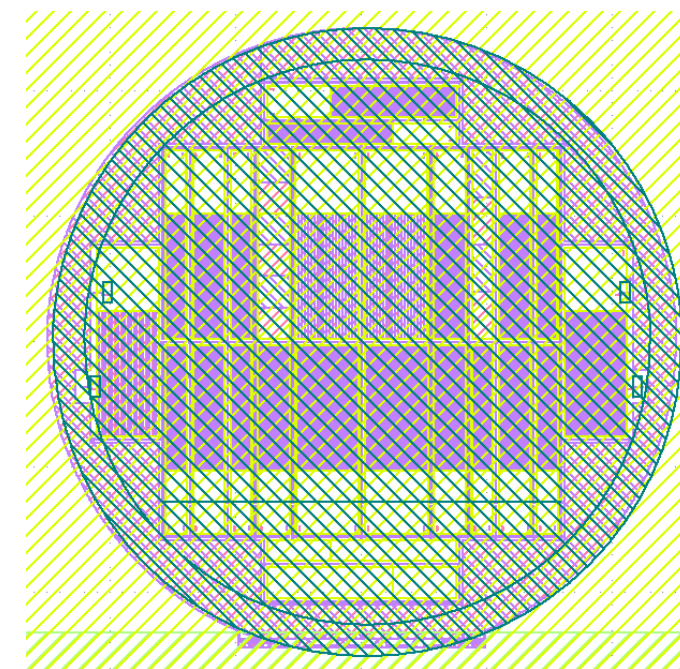
Number of periods:	Period (um)	334	500	1000
3	ON/OFF	3	4	3
	COS/LAT	4	3	3

1 undulator of 4 periods of 7.5mm  
1 undulator of 6 periods of 5mm

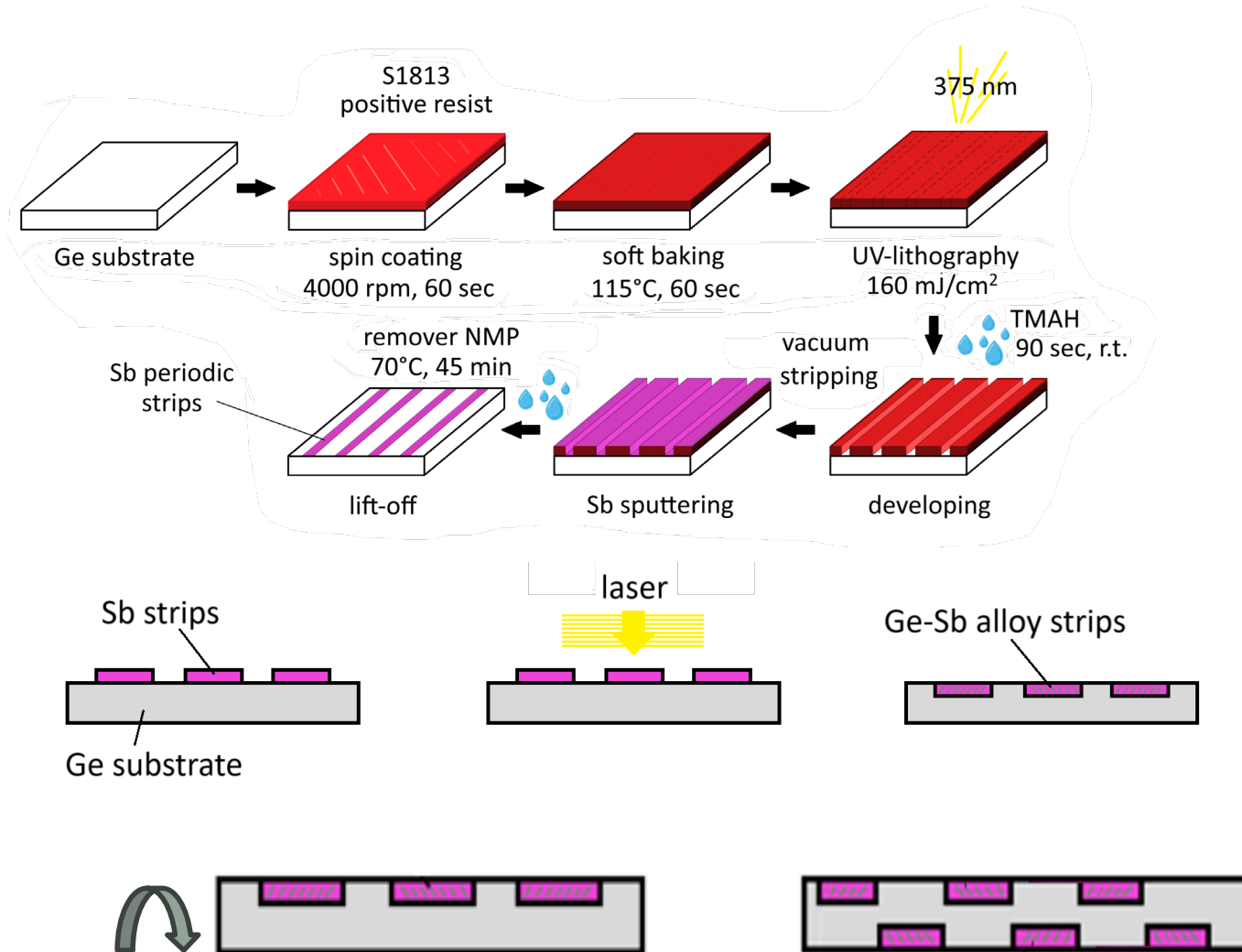


To evaluate the agreement  
with FEM simulation and  
validate it  
by interferometer  
measurements

Number of periods:	Period (um)	334	500	1000
2	ON/OFF	3	4	3
	COS/LAT	4	3	3



# PLM PBC patterning: processing design and optimization



**Lithographic process** (S1813 commercial resist) adapted and optimized for our system

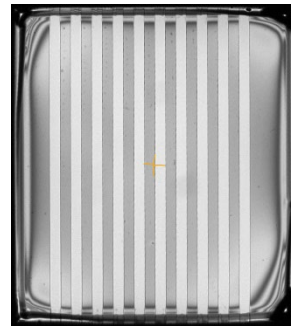
**PLM treatment:** optimized laser parameters from self-standing curvature study

**Rotate the sample** and **repeat** again the same procedure on the **other face** with a controlled offset (half period)

# PLM patterning manufacturing

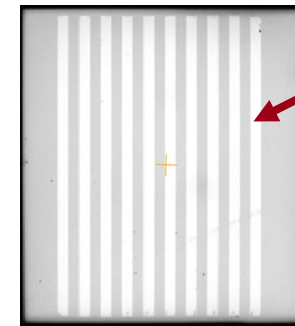
## Optical Images

Maskless lithography  
ON-OFF design

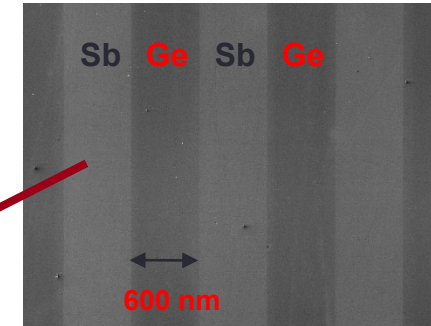


Sputtering  
& lift off

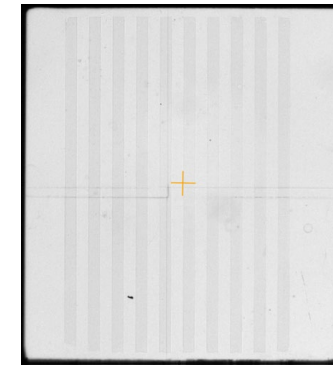
PCB-Ge-PLM2



SEM



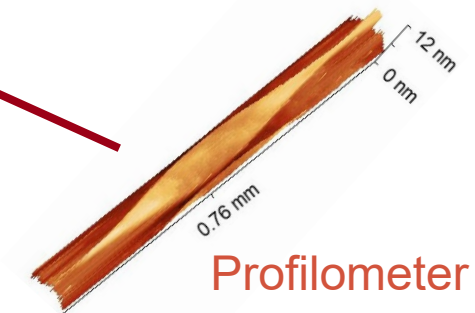
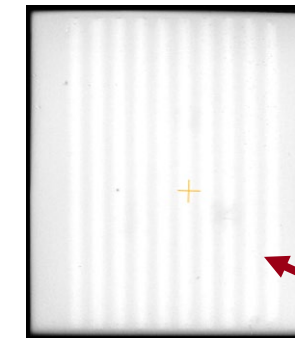
PLM



PLM

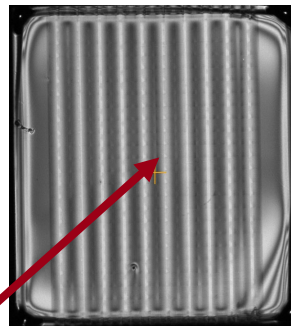


PCB-Ge-PLM3

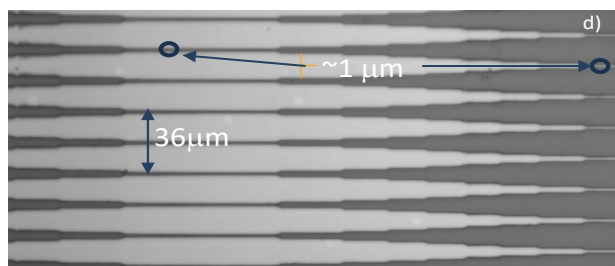


Profilometer

Maskless lithography  
Cos/lat design

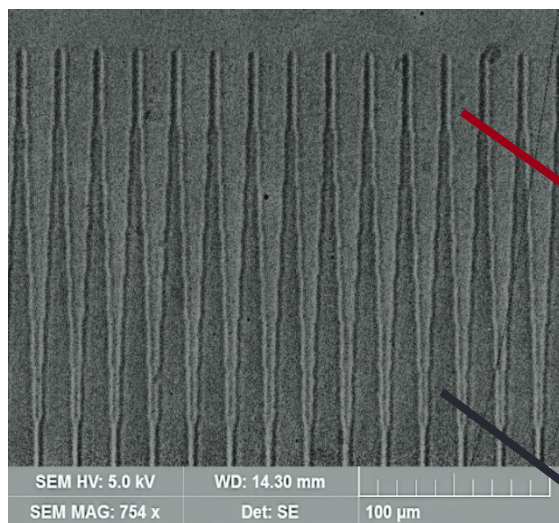


Photoresist

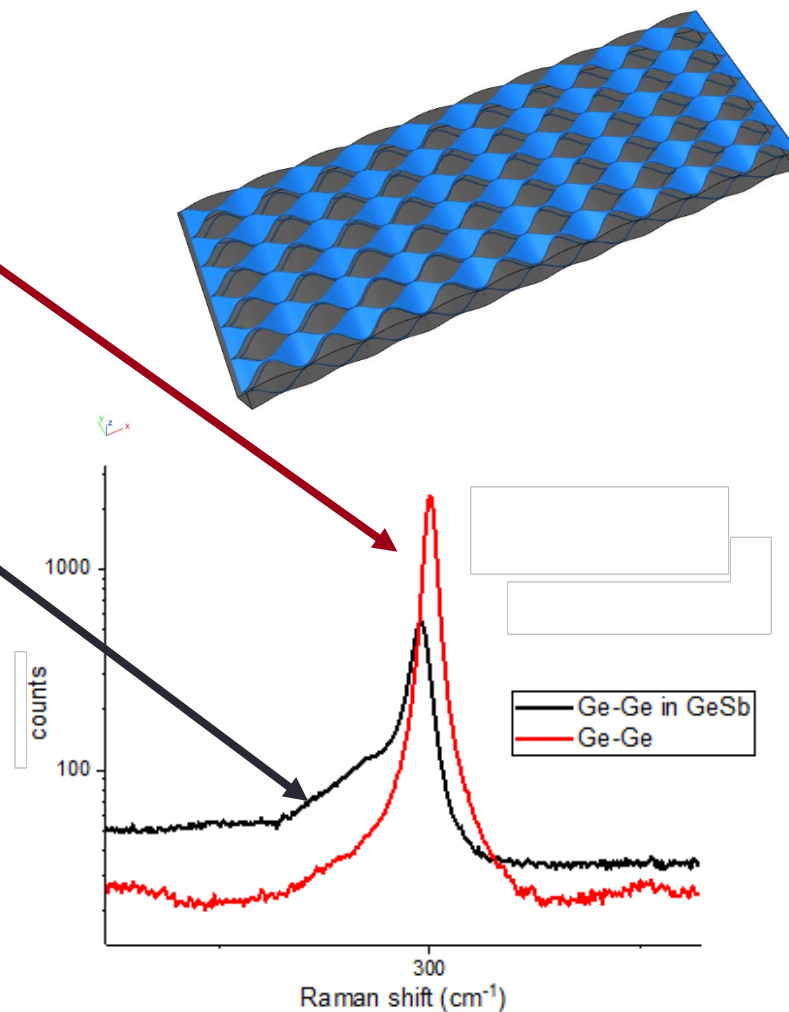


# UNPD undulator characterizations

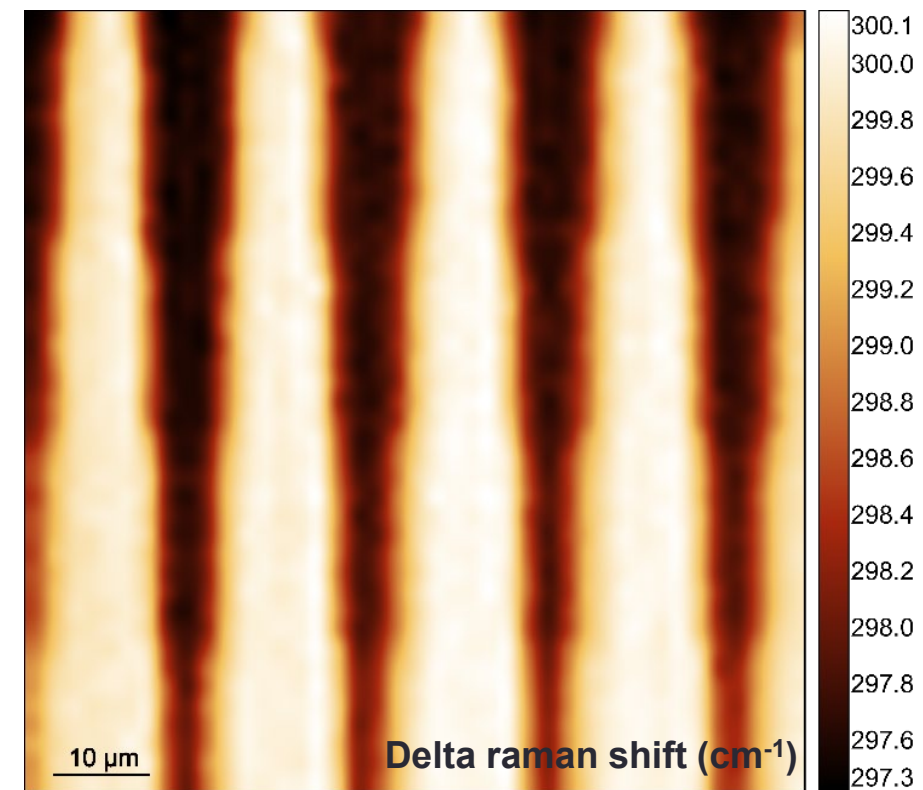
The local strain can be monitored by looking the raman shift of Ge-Ge stretching peak.



SEM imaging after PLM



RAMAN MAP





# Objective O4.3

## Dynamic PBC



*Leader*

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- 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 Characterization of crystalline samples under sinusoidal excitation at various frequencies from 1MHz to 15MHz and 25MHz to 60MHz**

# Acoustic Wave Crystalline Undulators



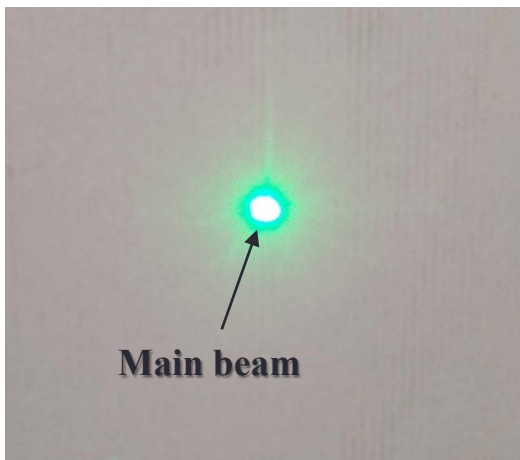
- Experimental detection and characterization of acoustically induced modulation of the lattice in crystal samples
  
- Development and calibration of diagnostic systems
  - **Acoustic wave detection**
    - *CW laser beam deflection*
    - *CW laser optoacoustic Bragg scattering*
  
  - **Acoustic wave characterization**
    - *Nanosecond laser interferometry*
    - *Nanosecond laser refractive imaging*
      - *Computational model*

# Development of Acoustic wave detection techniques

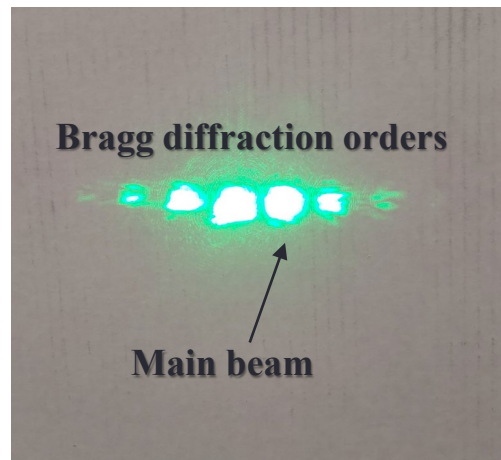
Detection of Traveling Acoustic Waves (AWs) in crystals generated by MHz piezoelectric transducers

a) CW Laser Bragg diffraction by MHz acoustic grating

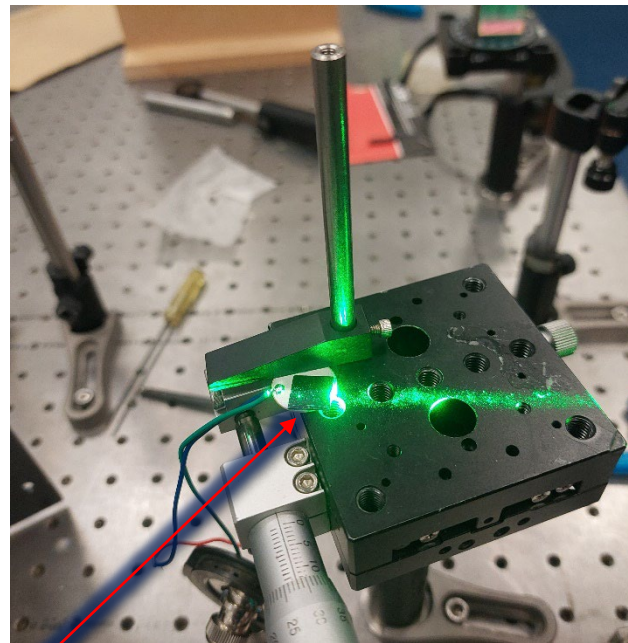
No excitation



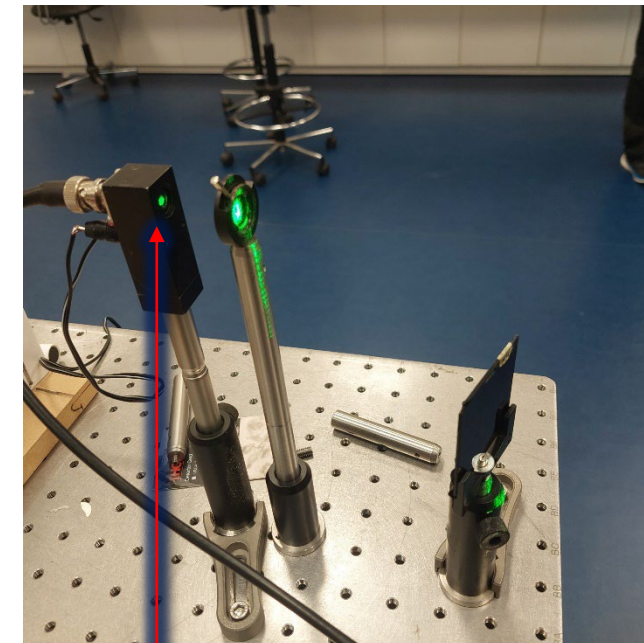
With excitation



b) CW Laser beam deflection by MHz oscillating piezos



*532 nm laser beam deflection from Si crystal*



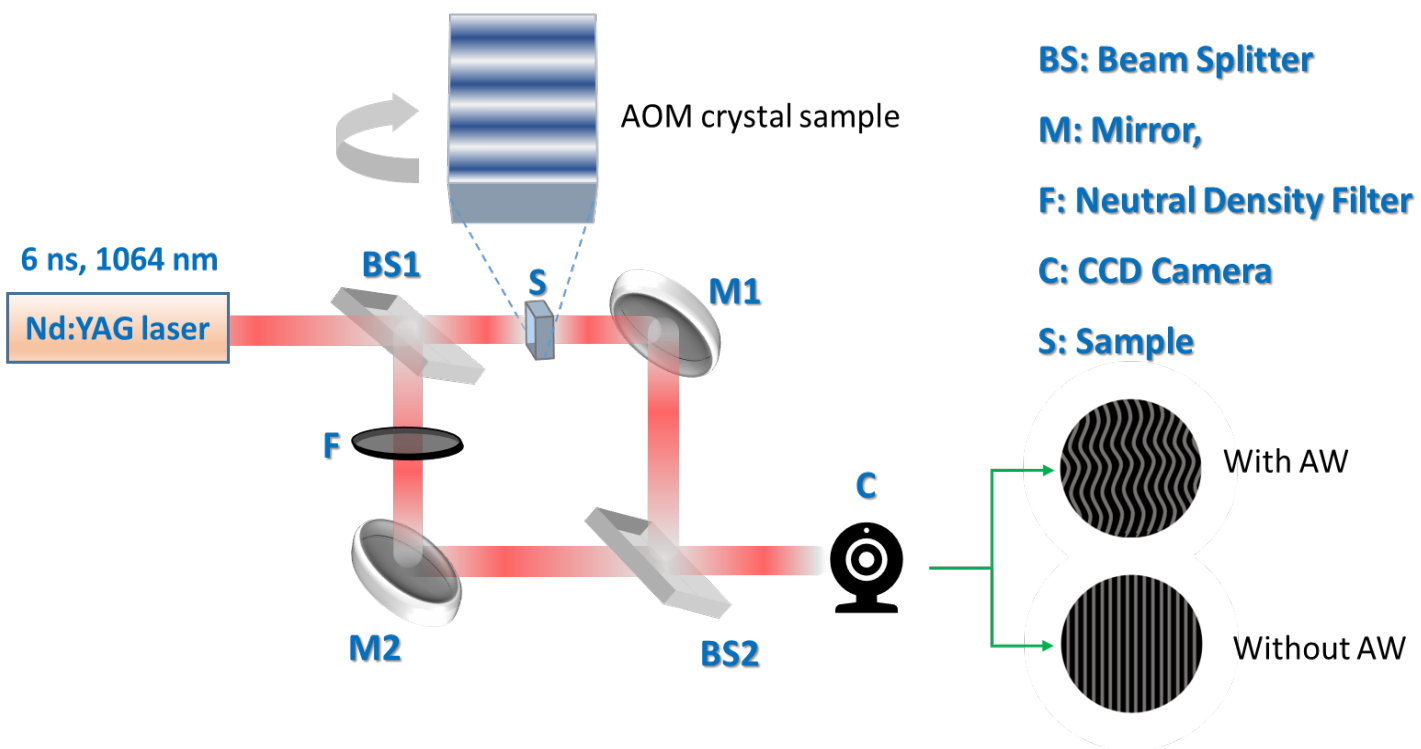
*Ultrafast picosecond photodiode*

# Imaging of Travelling MHz AWs by Nanosecond Laser Interferometry:

## Principle of operation and setup

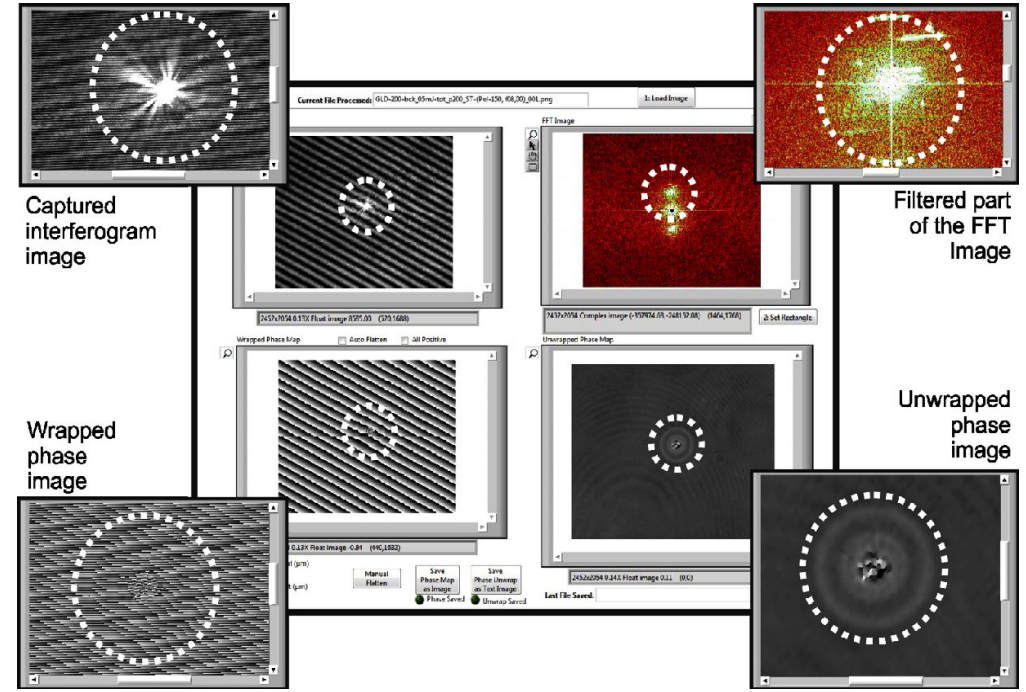
- Imaging Optical interferometry system based on the Mach–Zehnder setup
- High-resolution 2D imaging of the dynamic lattice modulation

### Schematic diagram of a nanosecond Mach Zehnder interferometer



**Fast probing with 6ns laser pulses**

**Capturing of effectively FROZEN travelling waves!!!**



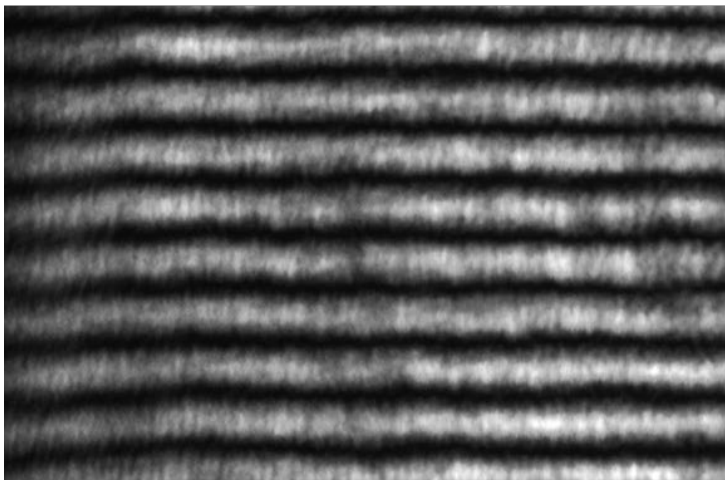
**Interface and process of fringe analysis software**

# Imaging of Travelling AWs by Nanosecond Laser Interferometry: Typical Proof of Principle Experimental Results on 5 cm Quartz Crystal

## Acoustically excited Quartz crystal at 40.65 MHz

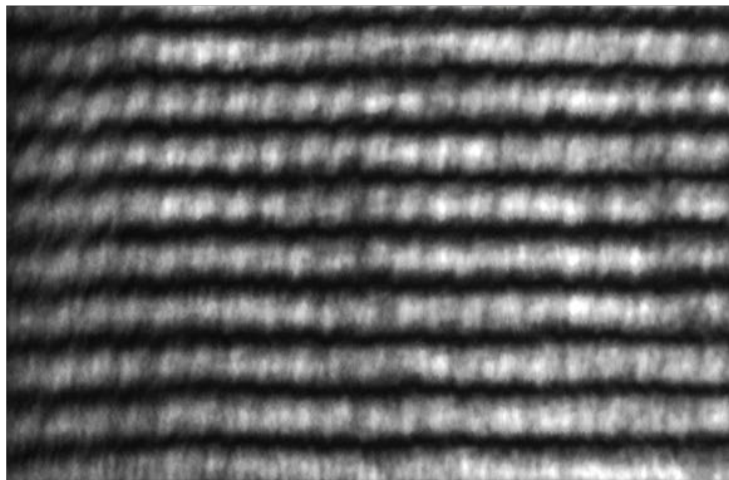
Analysis via in-house Image processing Software

No excitation (Reference)



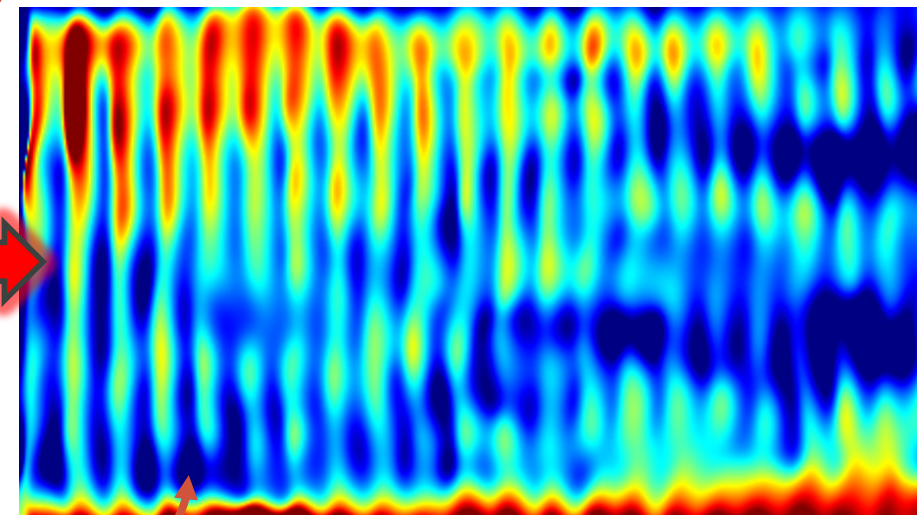
Unmodulated fringes

With excitation (Signal)



Modulated fringes

$f = 40.65 \text{ MHz}$ ,  $\lambda = 140 \mu\text{m}$



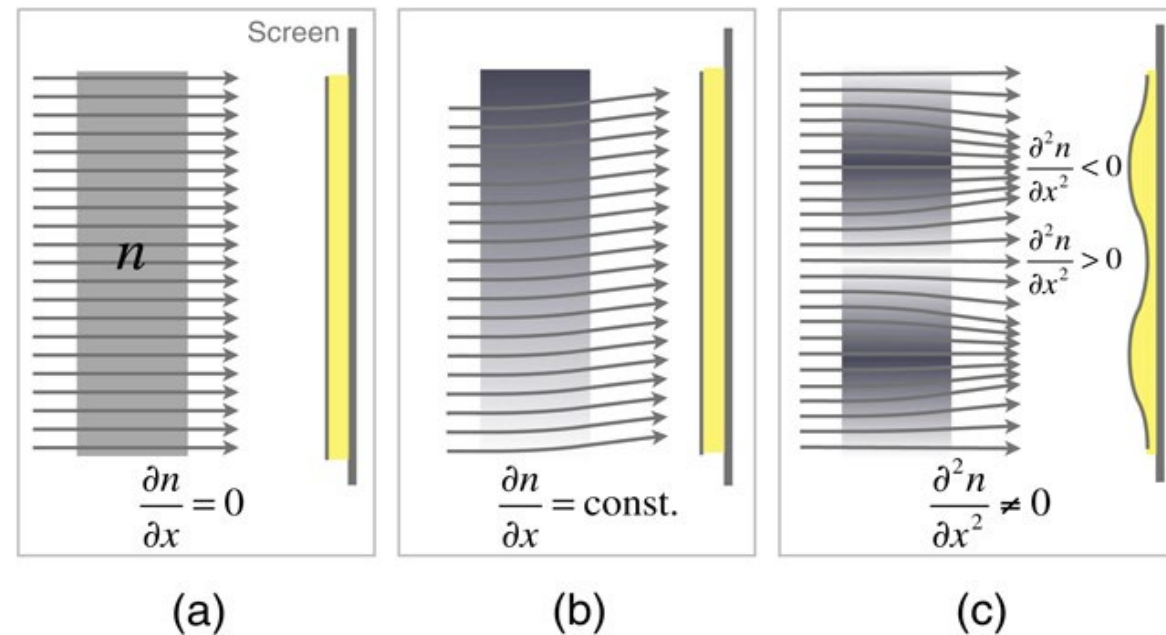
Detected acoustic waves

FROZEN  
travelling AWs!!

## Nanosecond Refractive imaging of travelling AWs:

### Principle of operation

- Homogenous refractive index*
  - *No laser deflection* → *Uniform illumination*
- Refractive index with homogenous gradient*
  - *homogenous laser deflection* → *Uniform illumination*
- Inhomogeneous refractive index gradient*
  - *Inhomogeneous laser deflection* → *Non-Uniform illumination*



\*Nobuki Kudo 2015 Jpn. J. Appl. Phys. 54 07HA01

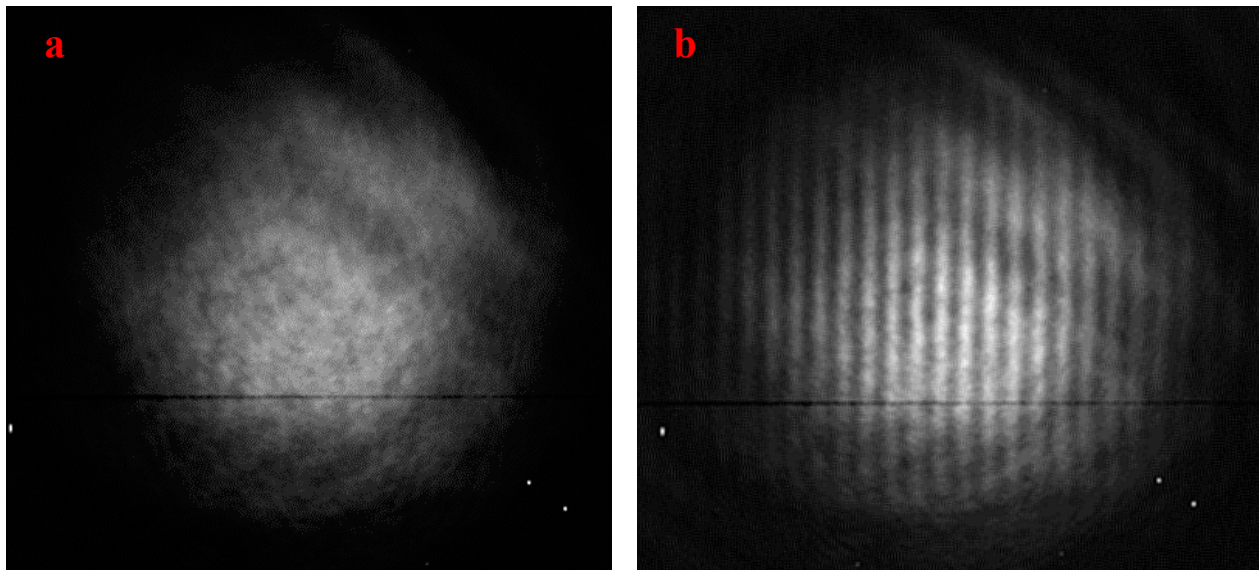
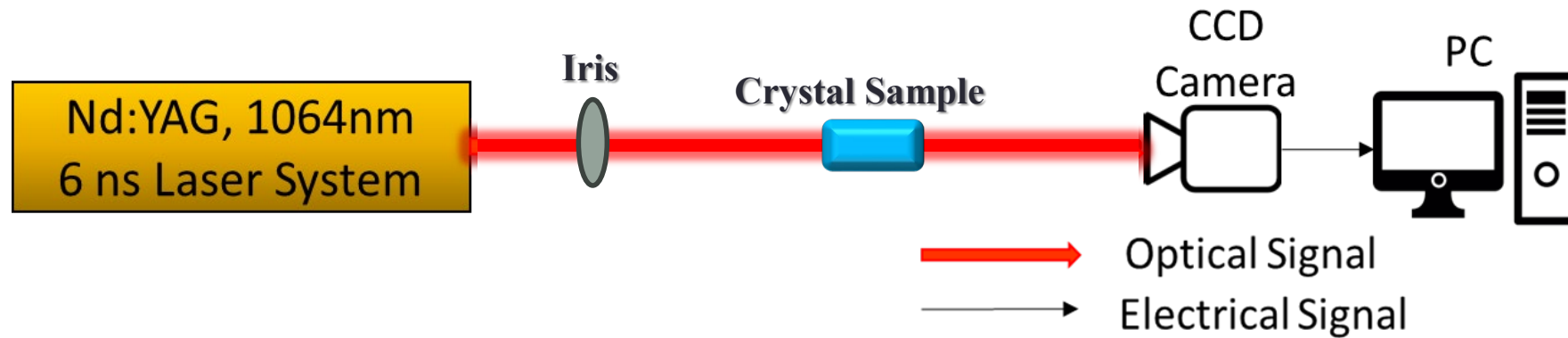
**Acoustic waves propagation inside the crystal →**

**Sinusoidal modulation of the refractive index perpendicularly to the probe beam propagation axis →**

**Modulation of the laser intensity distribution on the camera!**

# Nanosecond Refractive imaging of travelling AWs: Typical Experimental Results on thick Crystal

## Schematic diagram of the nanosecond refractive imaging technique setup

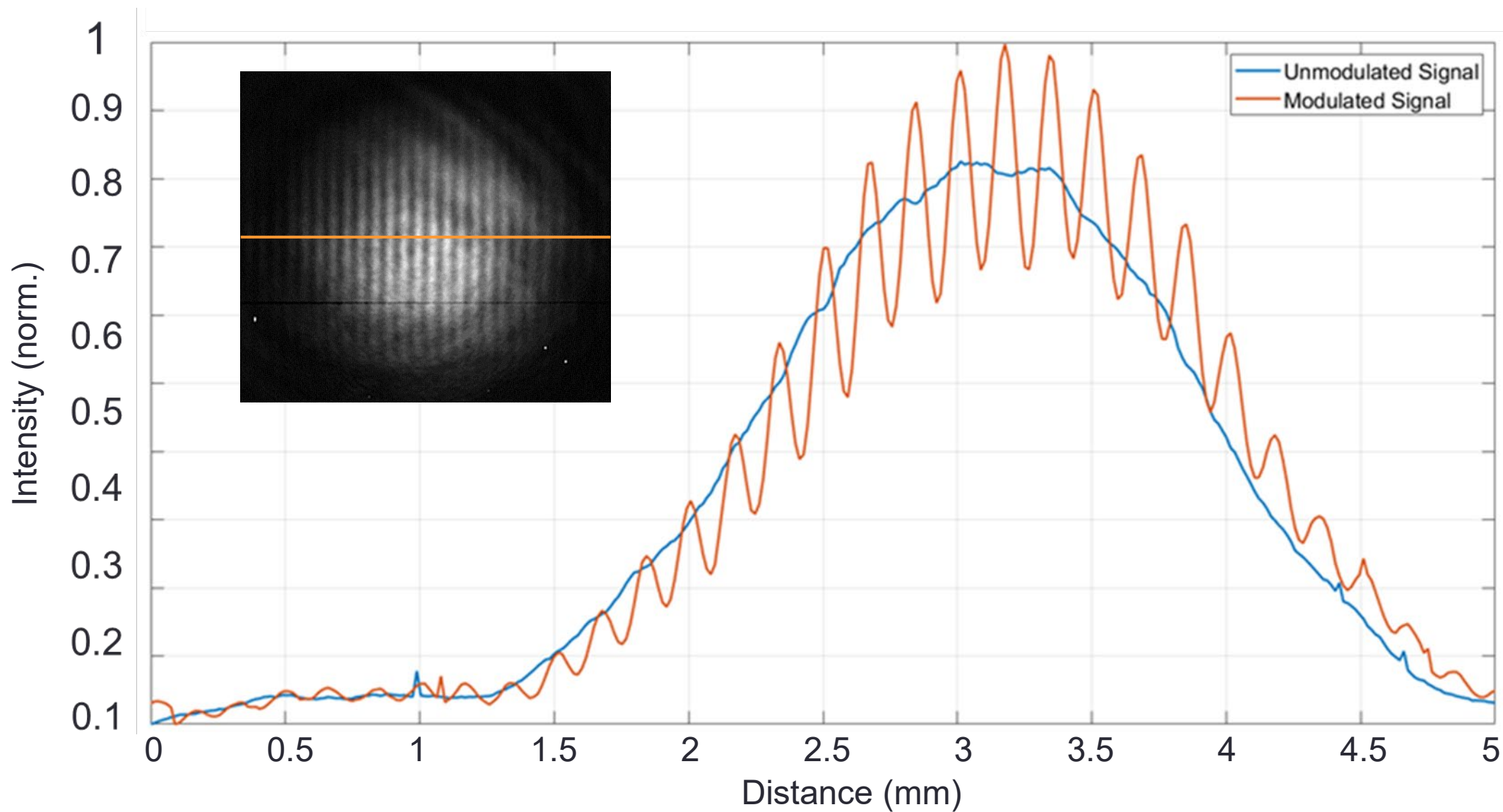


**Intensity distribution on imaging camera of**

***a) Unmodulated Quartz crystal***

***b) Acoustically modulated Quartz crystal***

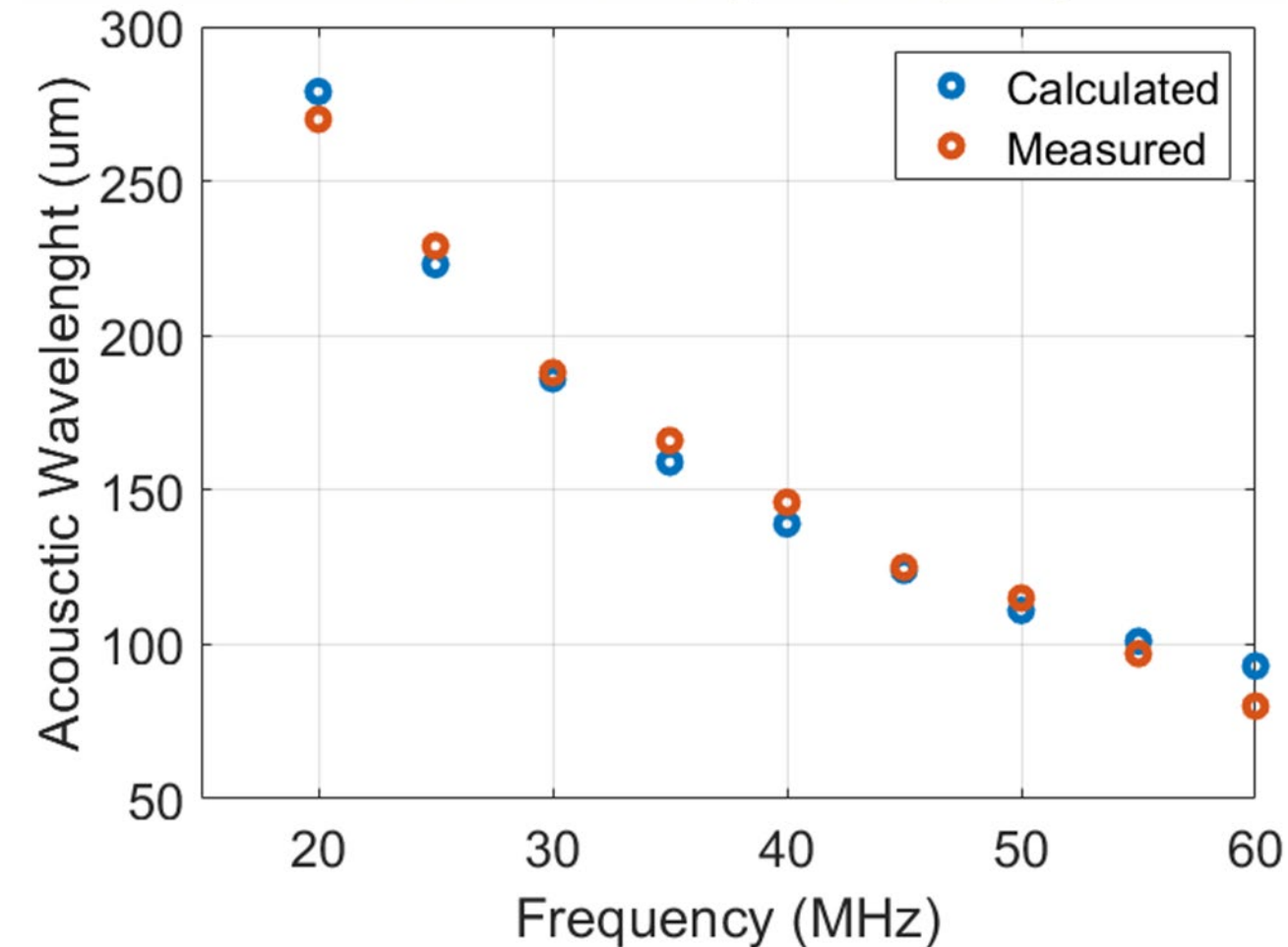
## Nanosecond Refractive imaging of travelling AWs: Typical Experimental Results analysis



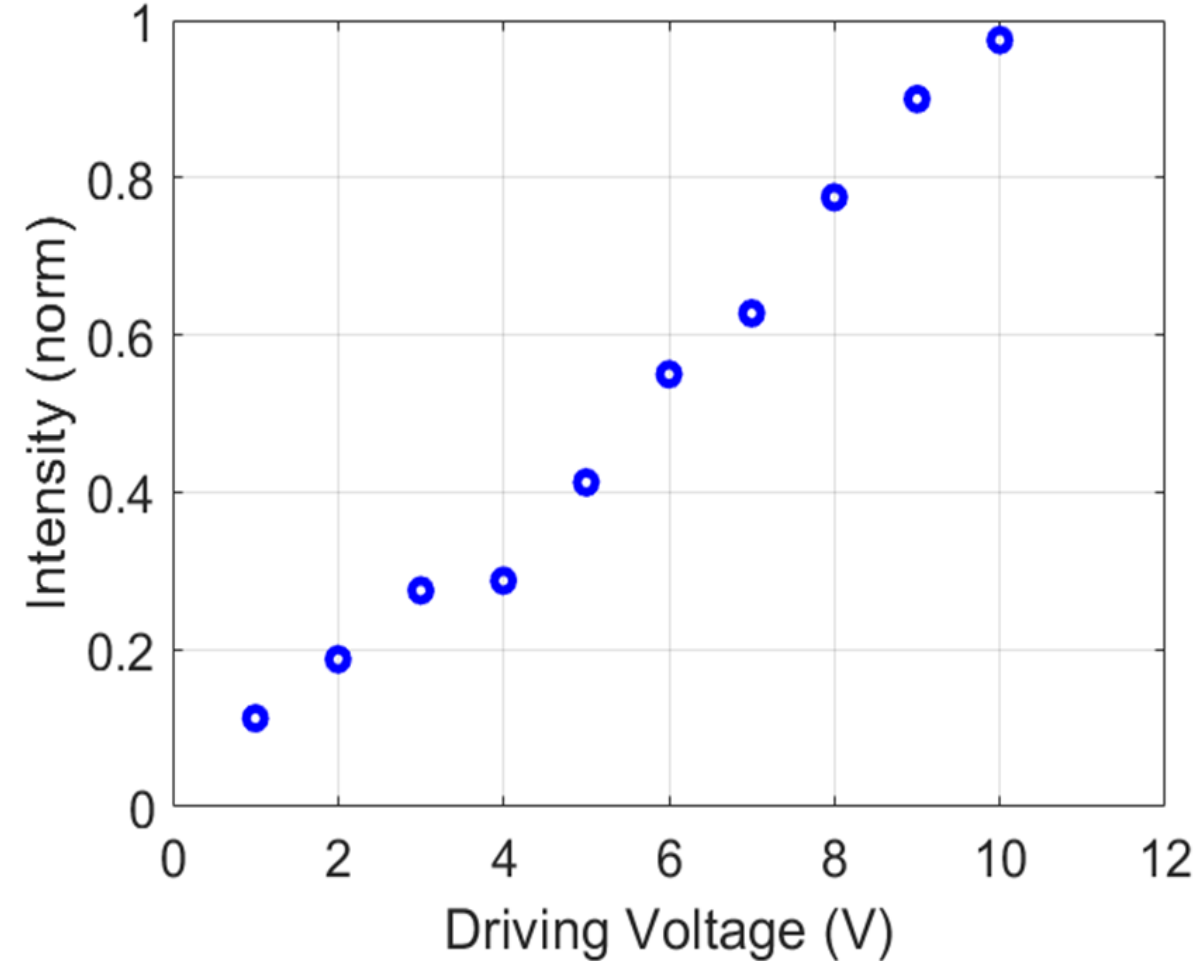


## Nanosecond Refractive imaging of travelling AWs: Typical Experimental parametric study

*Acoustic wavelength vs frequency*

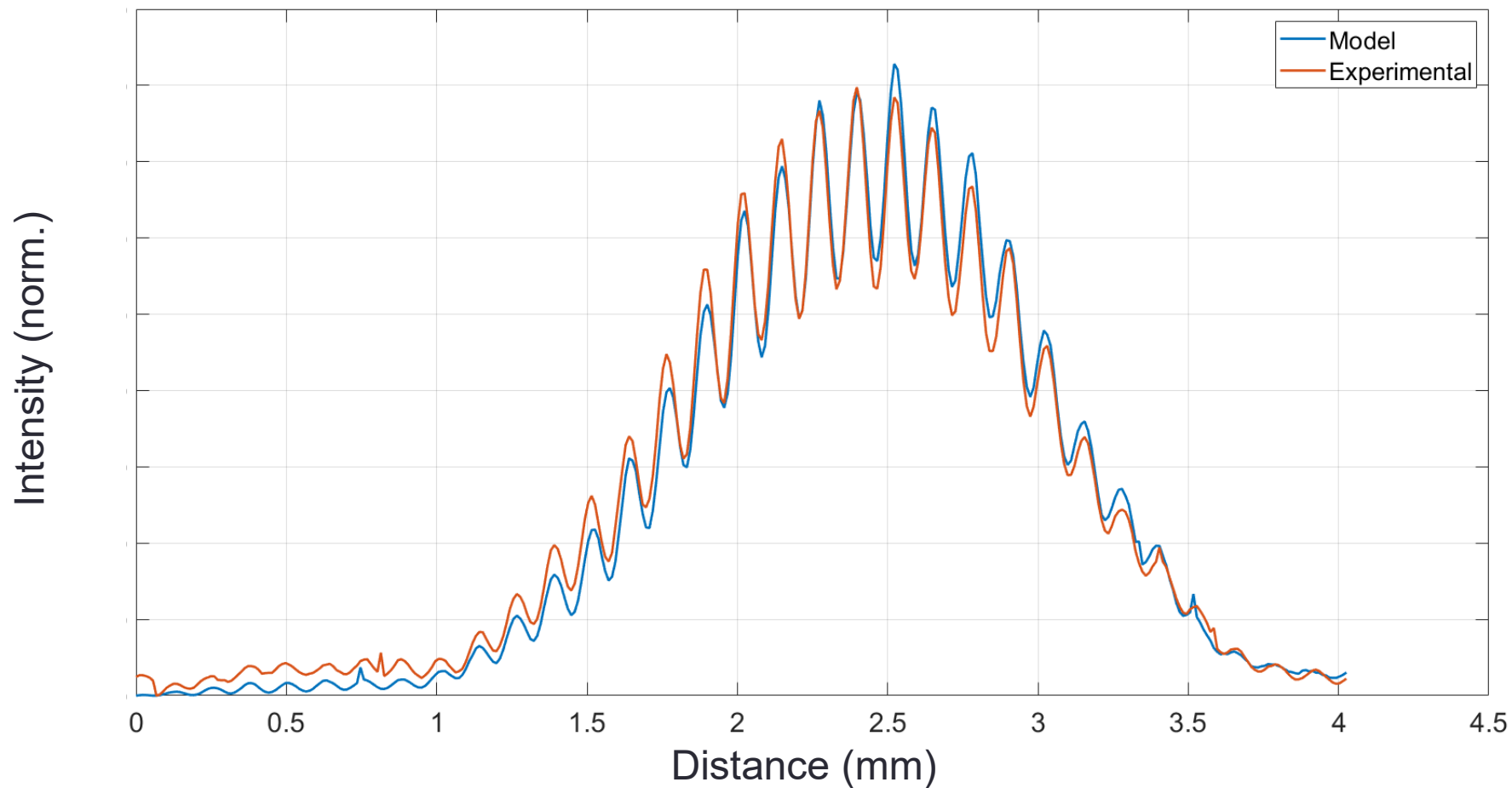
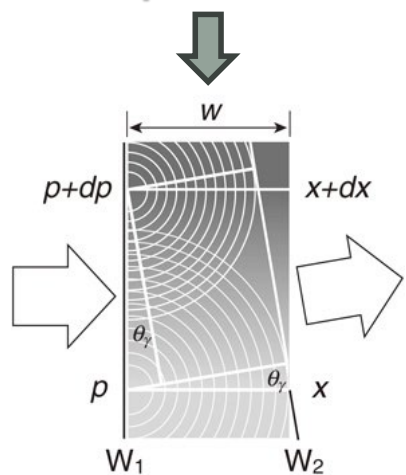


*Acoustic intensity vs driving voltage*



# Nanosecond Refractive imaging of travelling AWs: Experimental Results vs Computational Model

## Computational model Principle



# Summary of 2<sup>nd</sup> year activity for WP4

- Fabrication and characterization of first prototype of BC with tensile film of silicon nitride
- Parametric simulation of optimal pattern shape for silicon nitride PBCs optimized for positron with 10-20 GeV energies and started the fabrication of first prototypes of PBCs
  
- Fabrication of BC samples via PLM
- Set-up of lithography to produce PLM PBC-CU -> samples realized
- Progress in the optimization of patterning design in term of experimental constraints (stressor strength and lithography resolution)
  
- Started patenting procedure of the novel design, i.e., COS/LAT, for static PBC-CU
  
- Development of laser diagnostics systems for fast imaging of travelling AWs
- Proof-of-Principle characterization of travelling acoustic waves in Quartz crystal
- Parametric study of AW control via driving frequency and voltage
- Computational model development for AW pressure calculation in nanosecond laser refractive imaging

**JOINT ACTIVITY WITH WP2 for gamma-ray emission simulation**

# Prospect for next years

## O4.1 & O4.2

- Realize different **samples of static PBC** via Surface modification with Silicon Nitride deposition and Pulse Laser Melting with parameters **adapted for high-energy positron beams**  $> \text{GeV}$ .
- Select the best sample for possible experiments.
- Compare the two techniques for static PBC to **define the best method in terms of performance as CU**.

## O4.3

- Development of a **Ge-based A-CU** suitable for **high energy positron beams**  $\geq 20 \text{ GeV}$
- Development and characterization of a **Si- and Ge-based A-CU** for **lower-energy positron beams** ( $\sim 0.5 \text{ GeV}$ ) – Scheduling of experiments at MAMI

**Work in synergy with both WP2 and WP3**