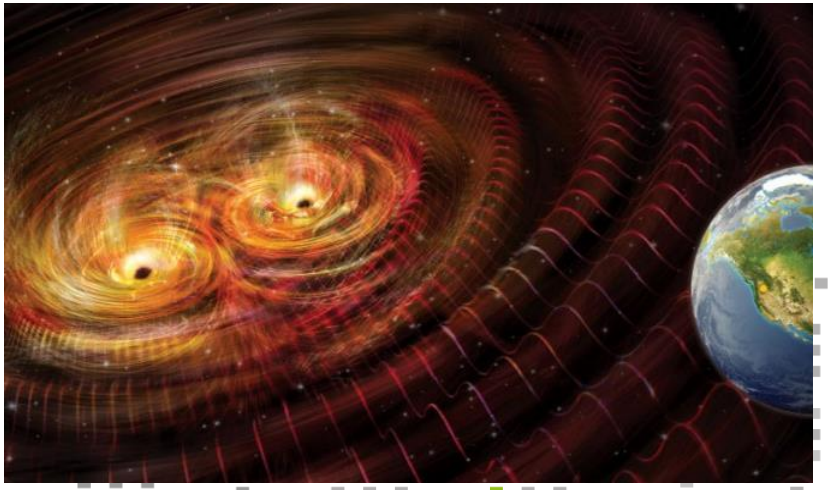


TRANSFER OF AN ALGAAS/GAAS MULTILAYER COATING ON A SILICA SUBSTRATE

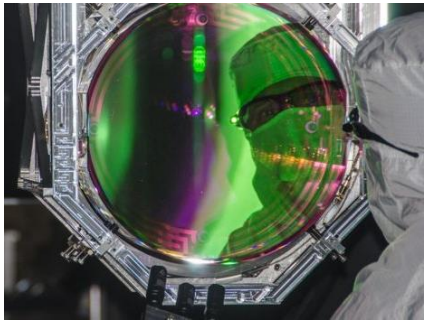
GdR Ondes Gravitationnelles| Hui Victor| 12/10/21

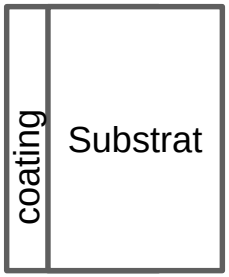
Supervisors :
Christophe Dubarry (CEA LETI)
Raffaele Flaminio (LAPP)

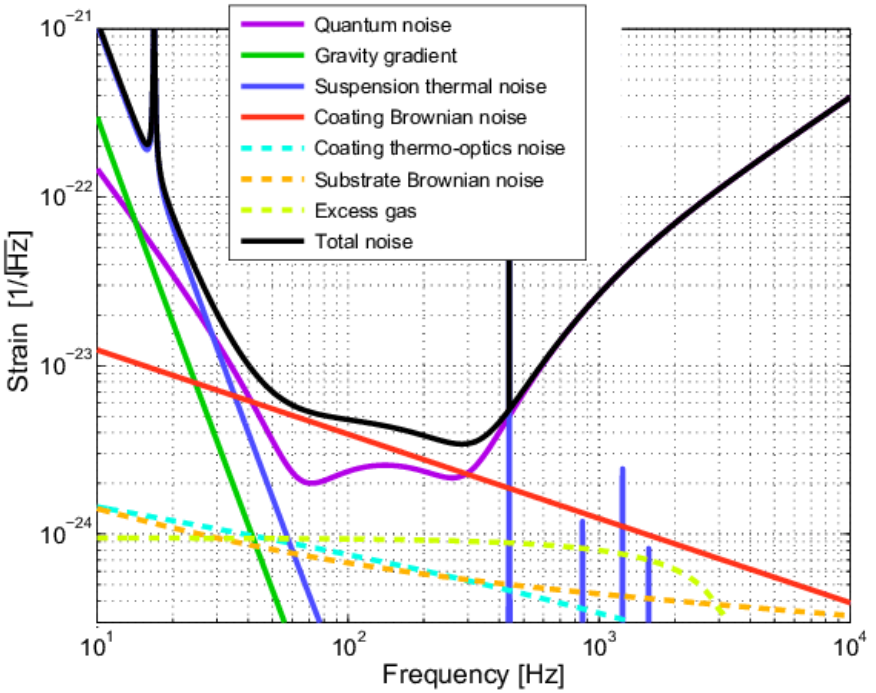
MOTIVATION

- **One of the main limitation of the sensitivity of GW detectors is the thermal noises on the interferometer's mirrors**

- **Two main thermal noises in the coating:**
  - Brownian noise
  - Thermo-optic noise

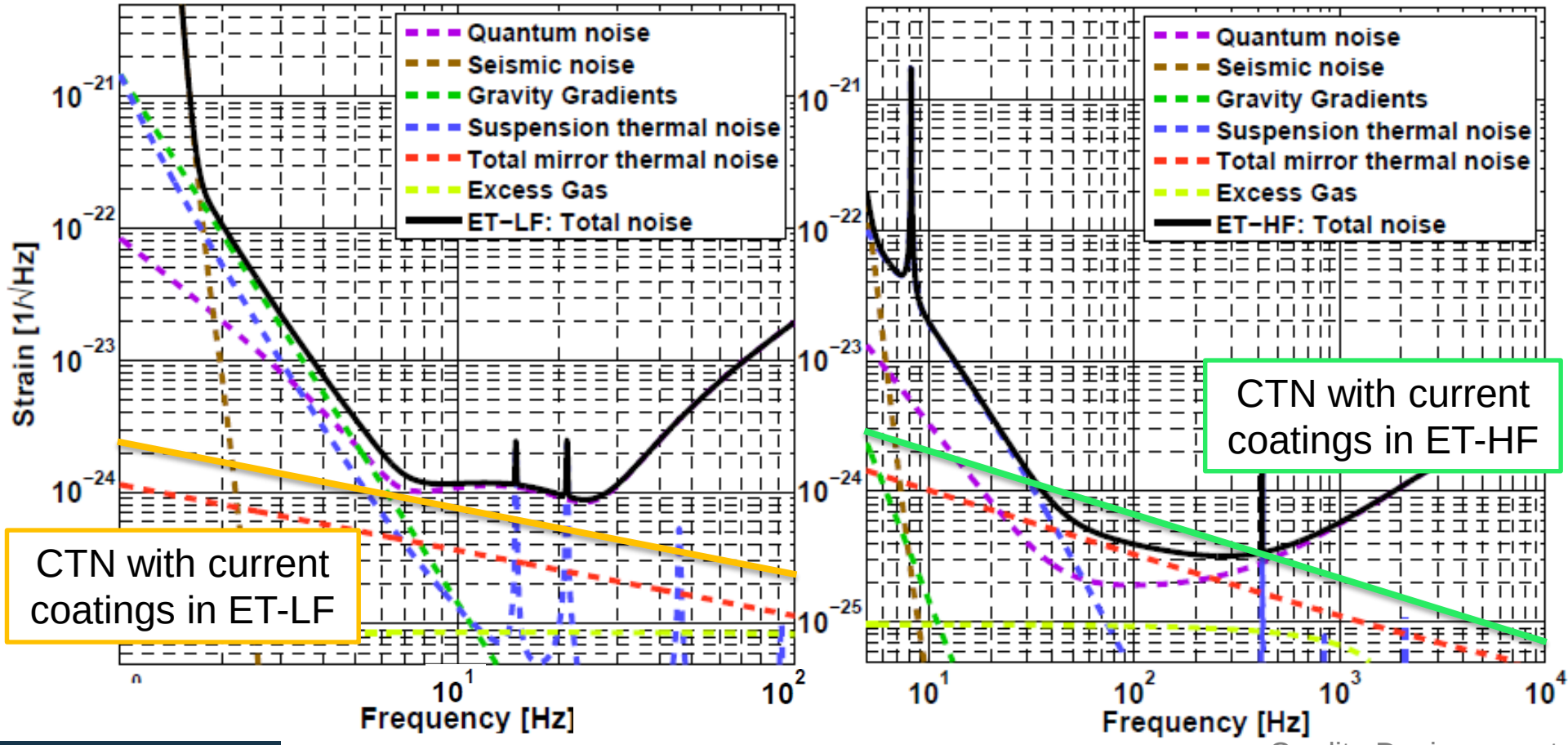






MOTIVATION

- **Comparison of coating thermal noise in ET with current coatings**



Credit : Design report update 2020 for ET

Need to reduce thermal noise to enhance sensitivity

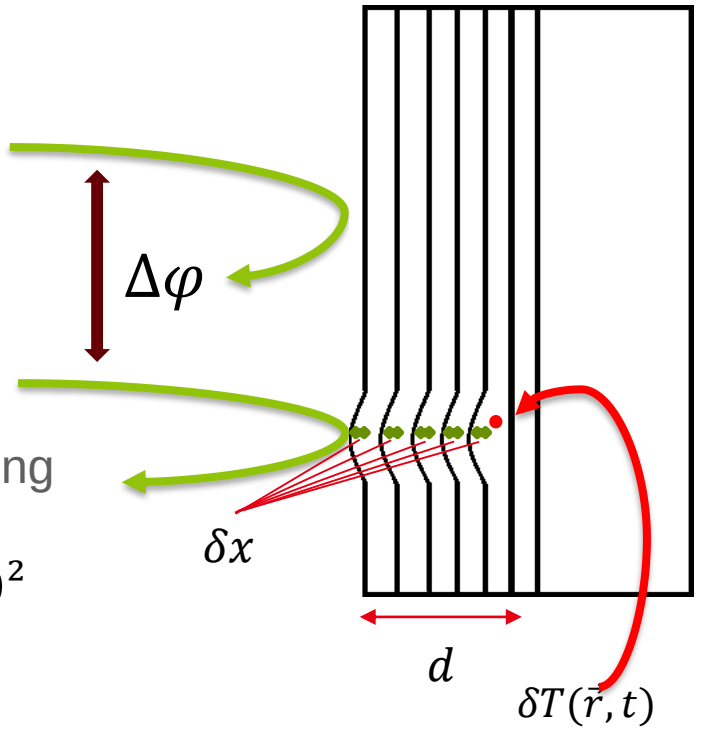
THERMAL NOISE

- **Brownian noise : due to mechanical damping of the coating layers driven by internal losses**

$$S_B(f,T) \propto \frac{k_B T}{\omega}\phi$$

Loss angle

- **Thermo-optic noise : contribution of Thermo-elastic noise and Thermorefractive noise**
  - Thermo-elastic noise : apparent expansion of the mirror coating causing phase shift in the reflected beam
  - Thermorefractive noise: physical change of the coating layers size and change in refractive index with temperature in the coating



$$S_{TE} \propto \frac{k_B T^2}{\sqrt{\omega}}(\alpha_c d)^2$$

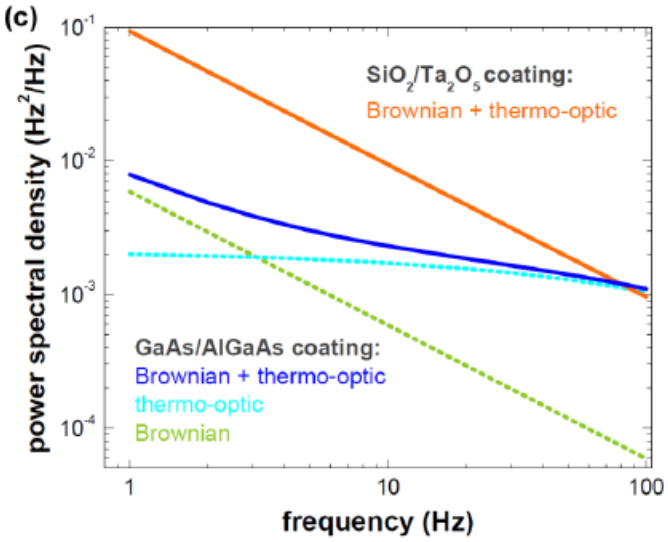
α_c : coating coefficient of thermal expansion

$$S_{TR} \propto \frac{k_B T^2}{\sqrt{\omega}}(\beta\lambda)^2$$

β: coefficient of thermorefraction

STATE OF ART THERMAL NOISE REDUCTION

- **Coatings used until now: amorphous**
  - Ta_2O_5/SiO_2
  - Made at LMA (Laboratoire des Matériaux Avancés)
- **Thermal noise reduction**
  - Cryogenic environment
  - Development of new coatings => Crystalline coatings

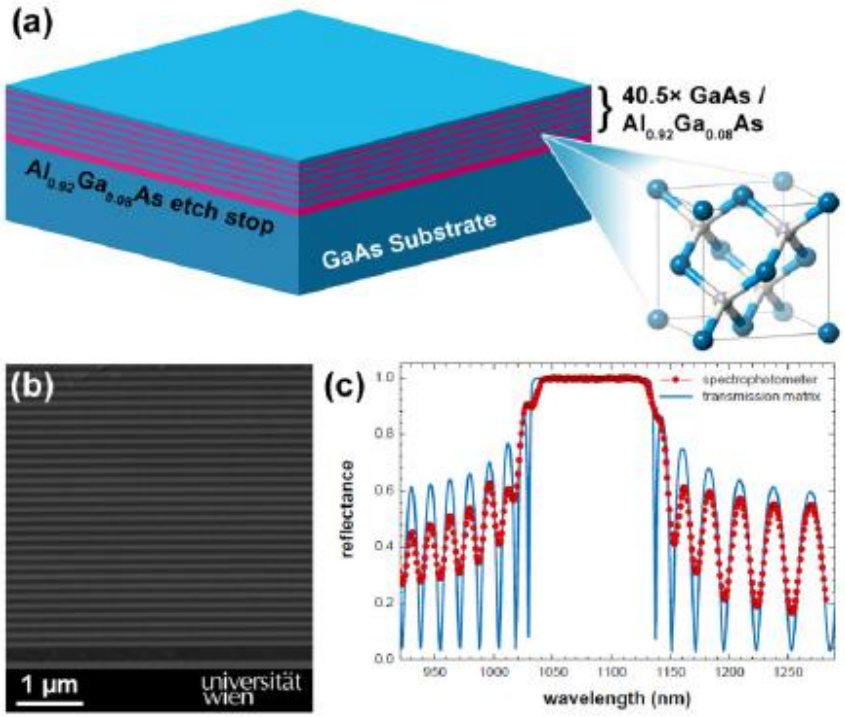


Crystalline coatings : Reduction x10 on Thermal noise

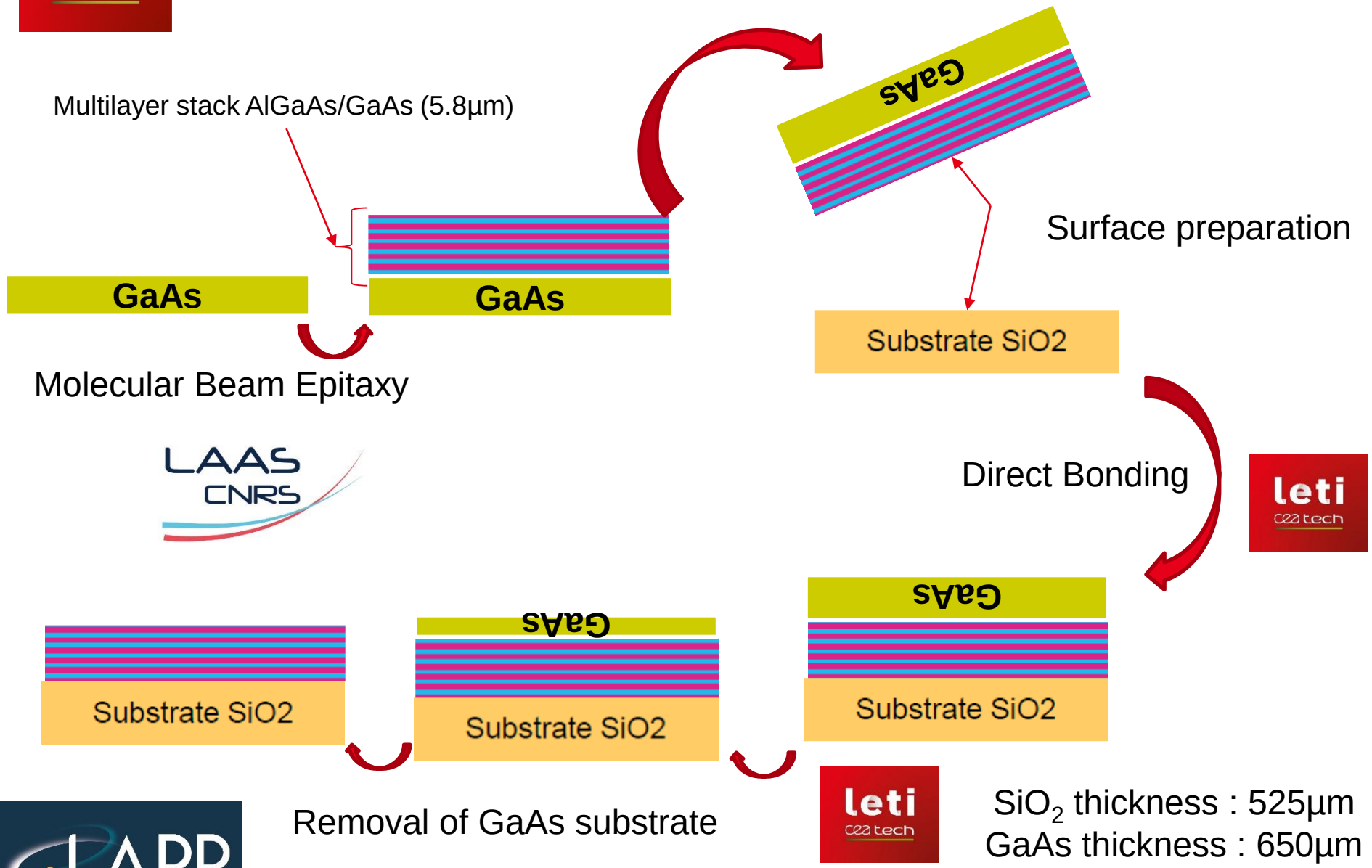
G. Cole et al. 2013

STATE OF ART THERMAL NOISE REDUCTION

- **G. Cole et al. 2013**
  Tenfold reduction of Brownian noise in optical interferometry
  - Crystalline Bragg mirror (AlGaAs/GaAs)
  - Excellent optical performances (R>0.9998, 4ppm diffusion)
  - Very low mechanical losses
    => Quality factor Q>4.10^4 (Q~1/ϕ)
    Q(Ta2O5/SIO2)~a few 10^3



GENERAL PROCESS

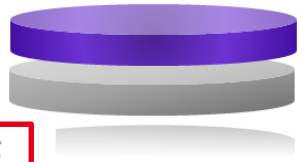


BONDING TECHNIQUE

Surface conditioning

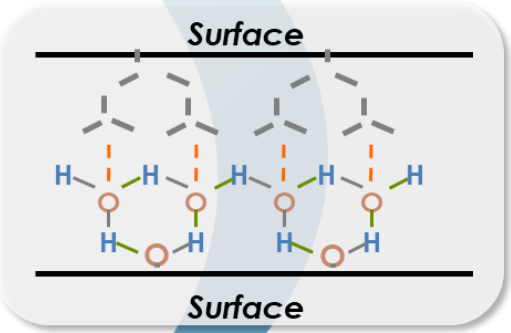
(cleaning, roughness, binding states, etc.)

- Roughness of ~0.3 nm RMS
- No particule
- No chemical contamination



Direct Bonding

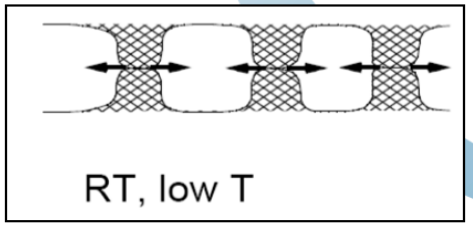
Room temperature and atmospheric pressure bonding:
Van de Waals adhesion



Annealing

Binding states evolution upon annealing: from Van der Waals to covalent bonding

Ziplock model



Fr. Rieutord et al., ECS 2006

MAIN CHALLENGES

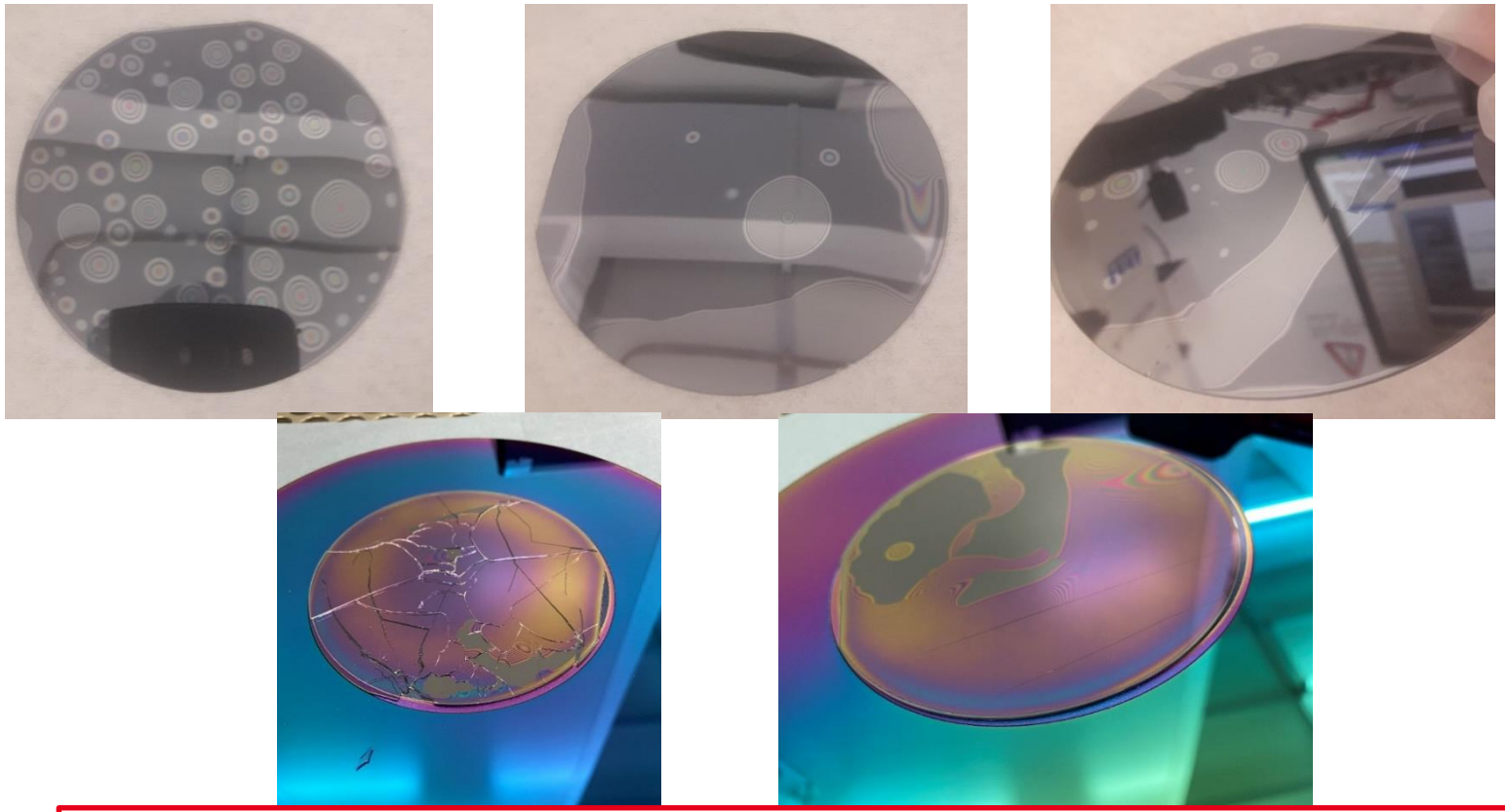
- **Heterostructure bonding**
  - GaAs naturally oxidize => native oxide layer GaAsO

  - Direct bonding difficult : Difference of themo-mechanic properties between GaAs and SiO_2 (large mismatch of Coefficient of Thermal expansion **CTE(GaAs)=5.3e-6; CTE(SiO_2)=5.1e-7**)

  - Thermal stress depends on wafer thickness (the more wafers are thick, the more stresses are important) => Difficult to anneal after direct bonding

MAIN CHALLENGES

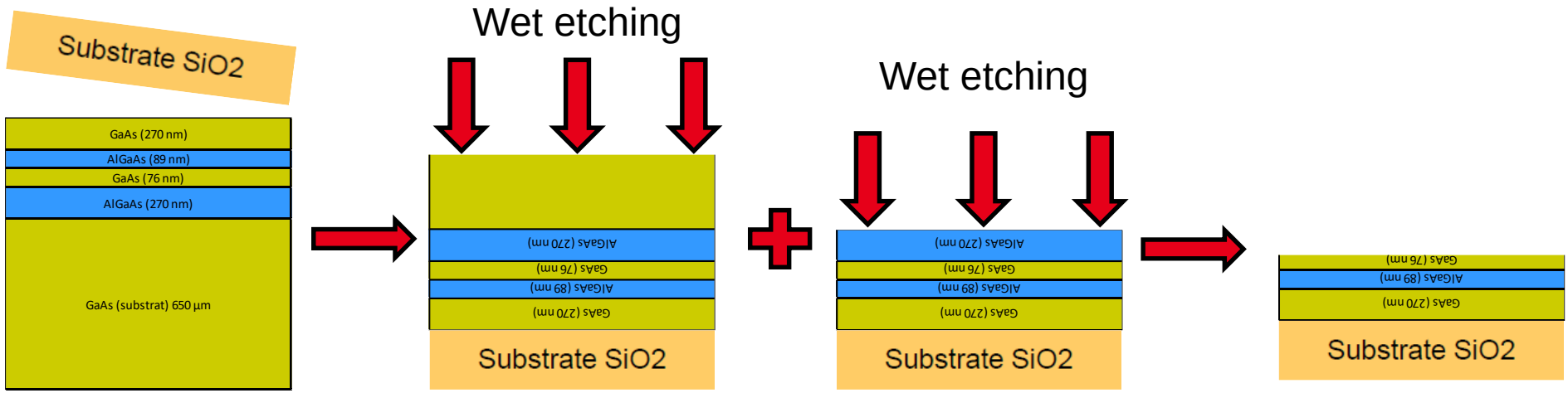
- **Hetereostructure bonding**
  - Results after direct bonding + annealing 100°C



Local debonding + breaking of the heterostructure due to material expansion (CTE(GaAs)=5.3e-6; CTE(SiO_2)=5.1e-7)

MAIN CHALLENGES

- **Removal of GaAs substrate**
  - Need to stop properly on the multilayer
  - Selective chemical solution that etches GaAs and not AlGaAs
  - Selective chemical solution that etches AlGaAs and not GaAs



SOLUTIONS?

- **Heterostructure bonding**
  - Wet cleaning : Removal of GaAs native oxide
    - Cleaning with NH4OH solution
  - Deposition SIO_2 by PECVD : Removal of GaAs native oxide + improvement of bonding quality (Oxide-Oxide direct bonding)
    - Preclean with plasma
    - Deposit of 500nm of SiO_2

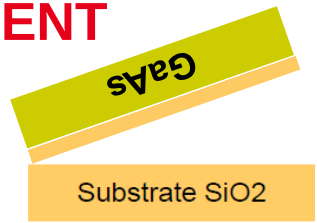
PECVD Direct bonding

  - CMP (Chemico-Mechanical Polishing) => improve roughness/micro-topography
  - Grinding of GaAs (physical thinning of GaAs before annealing)

PROCESS FLOW

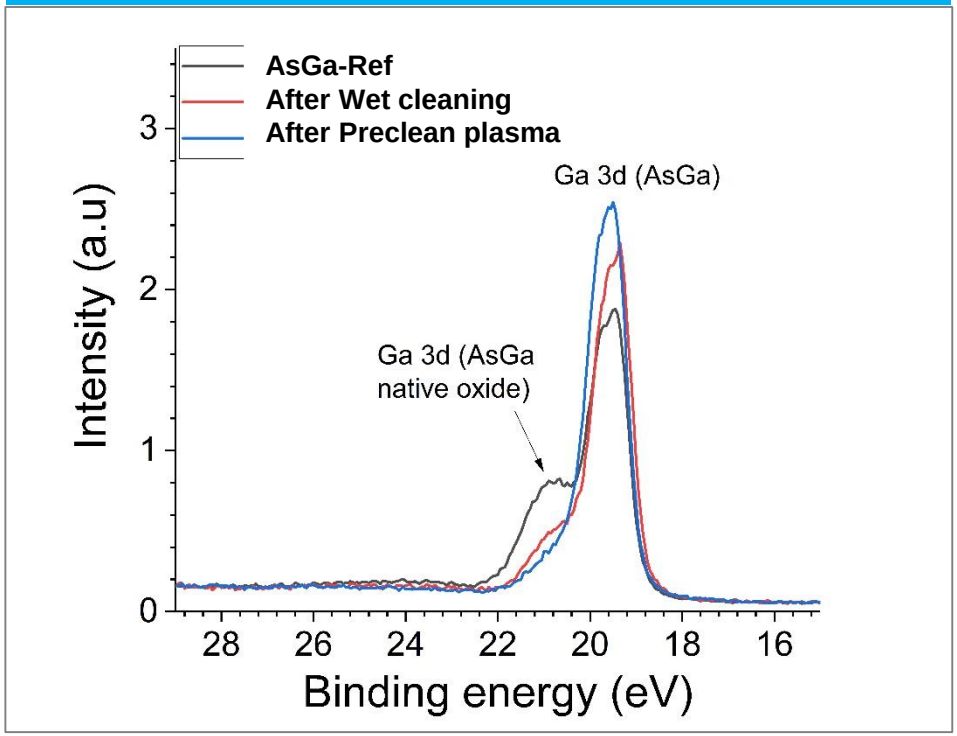
- **Surface treatment**
  - Wet Cleaning of GaAs wafers
  - Plasma treatment + SiO_2 deposition (500nm)
- **Direct bonding**
  - Chemical-Mechanical Polishing on SiO_2 wafers
  - Chemical-Mechanical Polishing on GaAs wafers after deposition
  - Direct bonding
- **Removal of GaAs**
  - Grinding of GaAs (until 100µm thickness)
  - Chemical wet etching of GaAs (until 20µm thickness)
- **Annealing 2h @100°C**

CHARACTERIZATION OF NATIVE OXIDE TREATMENT

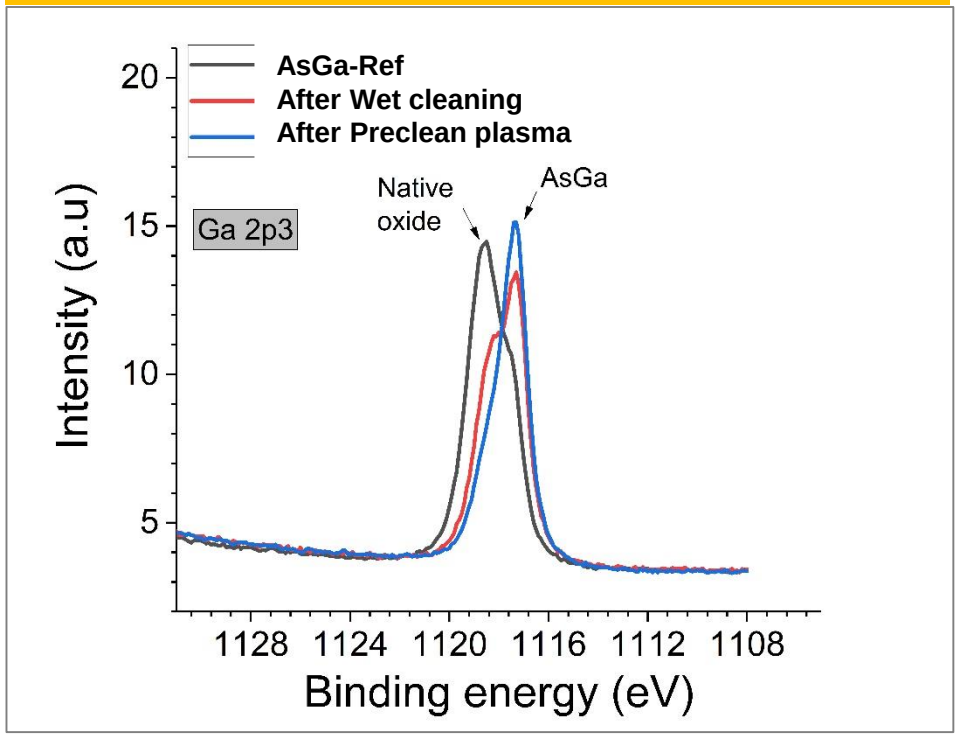


- X-Ray photoelectron spectrometry on GaAs

Ga 3d : depth of analysis ~ 7 nm



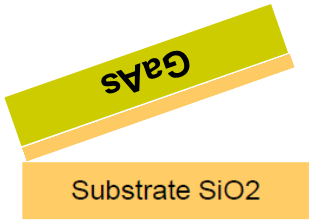
Ga 2p : depth of analysis ~ 2 nm



Treatment of native oxide validated

CHARACTERIZATION OF ROUGHNESS : POLISHING EFFECT

- **AFM (Atomic force microscopy) on substrate**



SIO2

		Before CMP 10µmX10µm	After CMP 10µmX10µm
centre	RMS, nm	0,622	0.26
Mi-rayon	RMS, nm	0,852	0.26
bord	RMS, nm	0,737	0.28

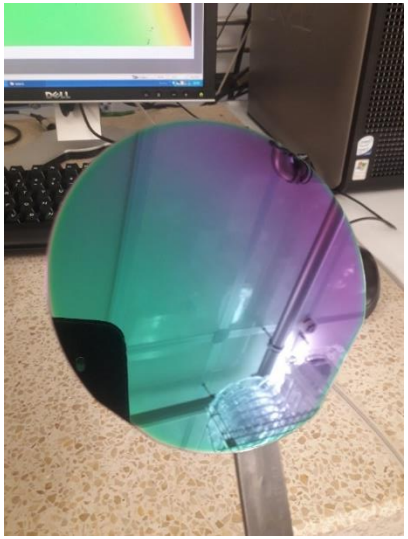
→ Ok for direct bonding

GaAs with SiO2 deposit

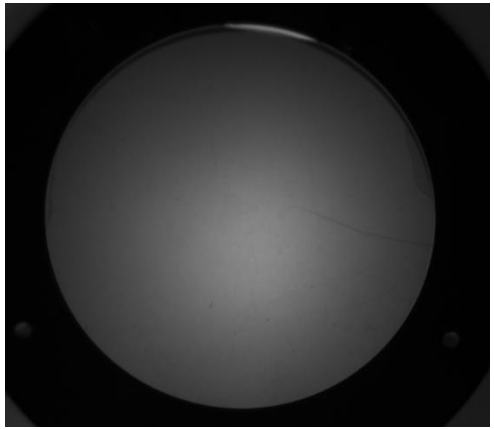
		Before CMP 10µmX10µm	After CMP 10µmX10µm
Centre	RMS,nm	0,521	0,215
Mi-rayon	RMS,nm	0,600	0,212
Bord	RMS,nm	0,579	0,198

→ Ok for direct bonding

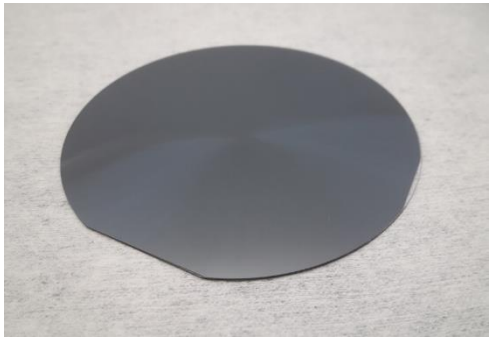
MAIN RESULTS



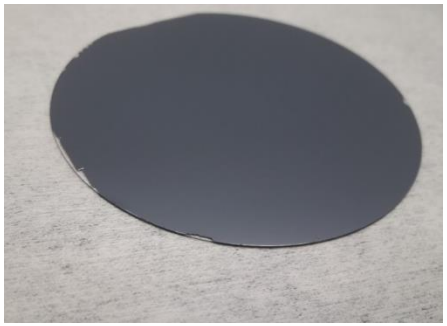
Preclean + SIO2 deposit



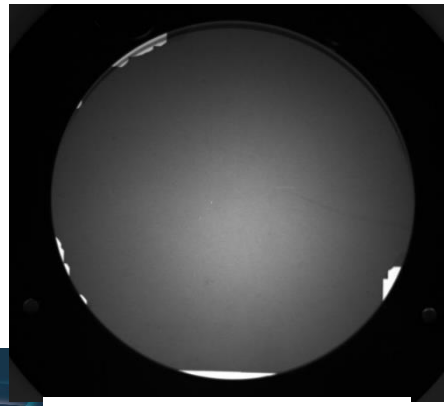
Direct bonding



Grinding GaAs (650µ->100µ)

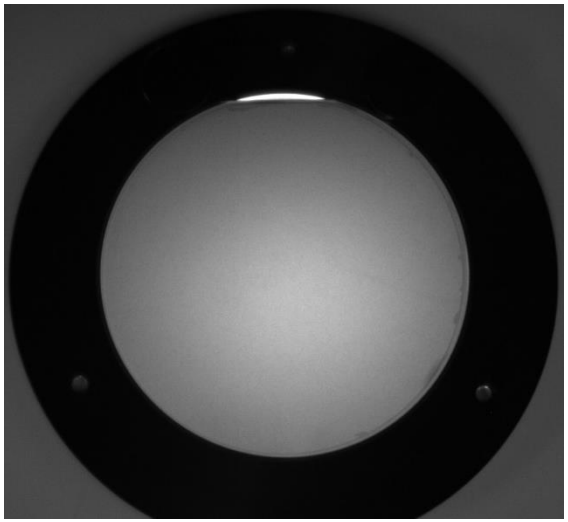


Etching GaAs (100µ->20µ)

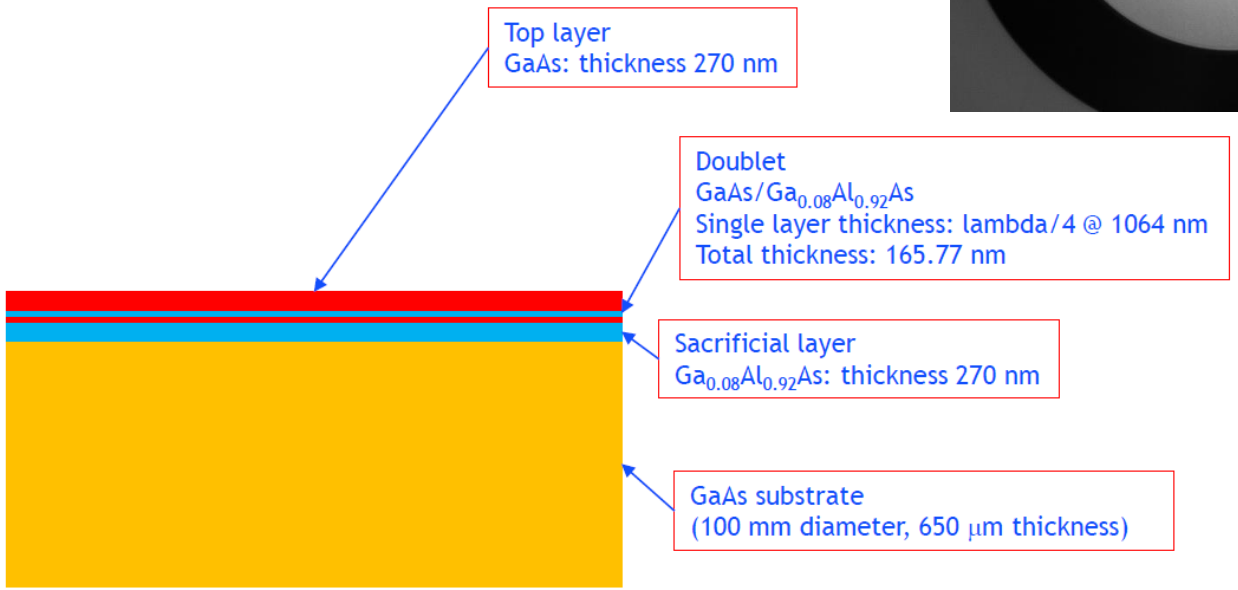


Final annealing

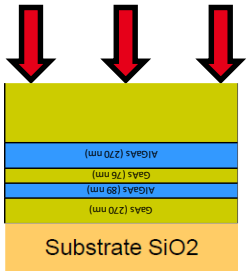
TRANSFER OF A DOUBLET



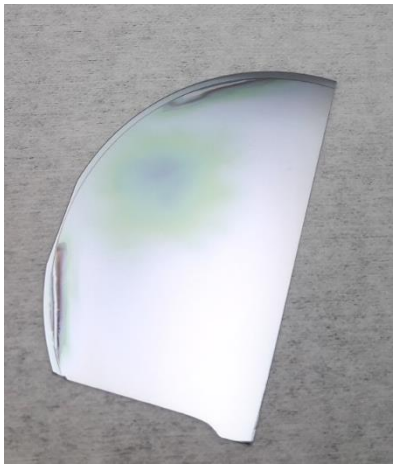
- **Process applied to the doublet**
  - Cleaning, PECVD SIO2, CMP, Bonding



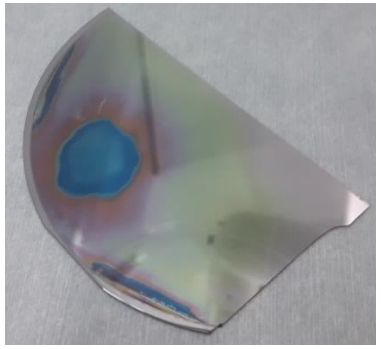
HARD POINT : WET ETCHING



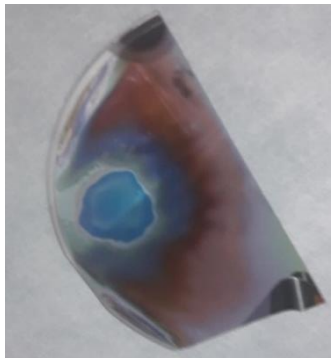
- **Removal of GaAs substrate with NH4OH/H2O2/H2O**
  - Results after wet etching tests



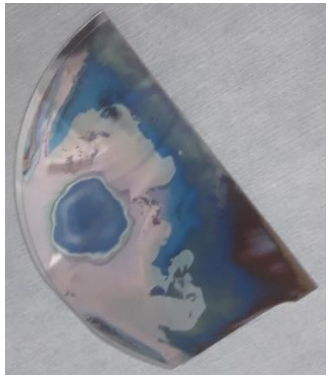
Etching t+75s



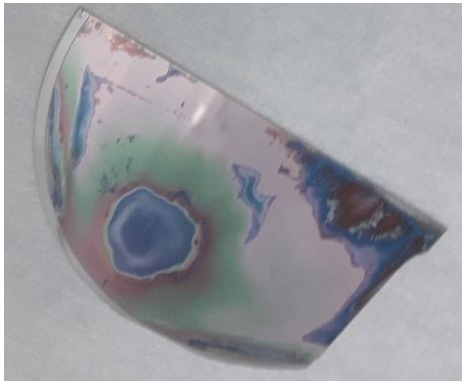
Etching t+90s



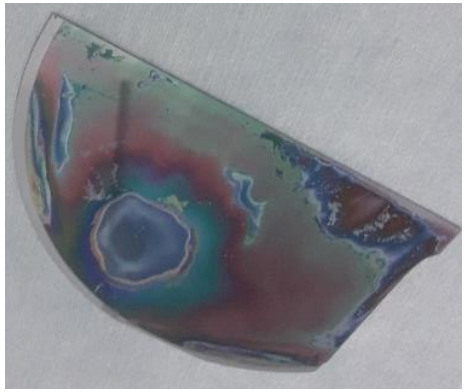
Etching t+105s



Etching t+120s



Etching t+135s

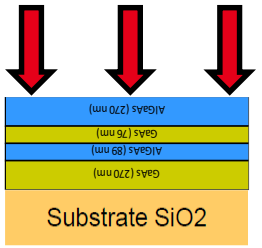


Etching t+150s

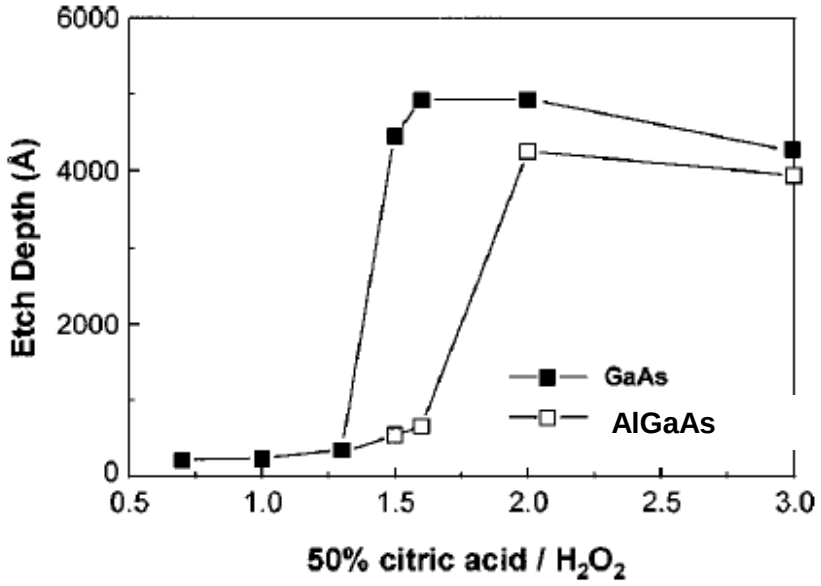
Etching t+195s

Non homogeneous etching : Etching chemistry non selective on GaAs over AlGaAs

WET ECTHING GAAS/ALGAAS



- **NH4OH/H2O2 not selective for wet etching of GaAs over AlGaAs**
- **Possible solution :**
  - Citric Acid/H2O2



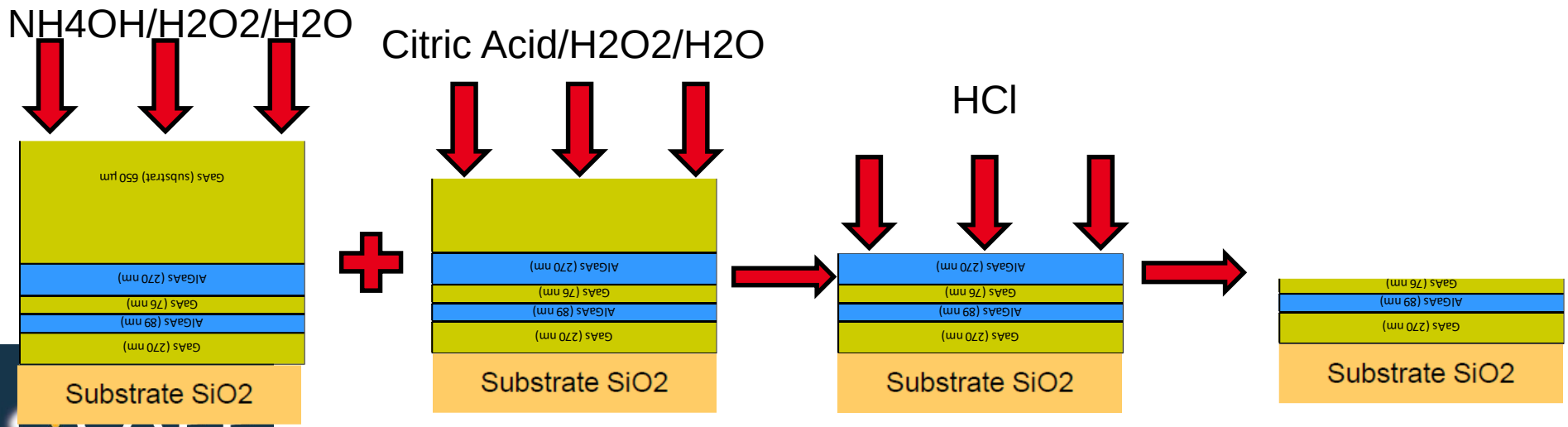
Eun-A Moon et al. (1998)

doi.org/10.1063/1.368571

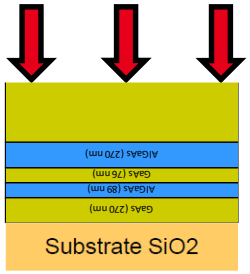
WET ECTHING GAAS/ALGAAS

- **Process**
  - HCl etching (de-oxidation)
  - NH4OH/H2O2/H2O etching
  - Citric Acid/H2O2/H2O etching
  - HCl etching

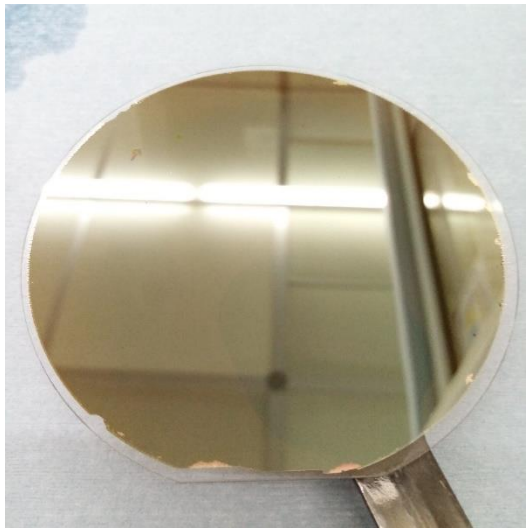
Courtesy to Isabelle Sagnes for improvement process compatible with multilayer design



ELLIPSOMETRY CHARACTERIZATION



- **Wet etch AC/H2O2**



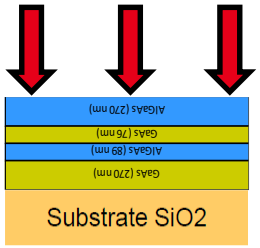
Theoretical stack

AlGaAs	270 nm
GaAs	76,4 nm
AlGaAs	89,3 nm
GaAs	270 nm
SiO2	525 µm

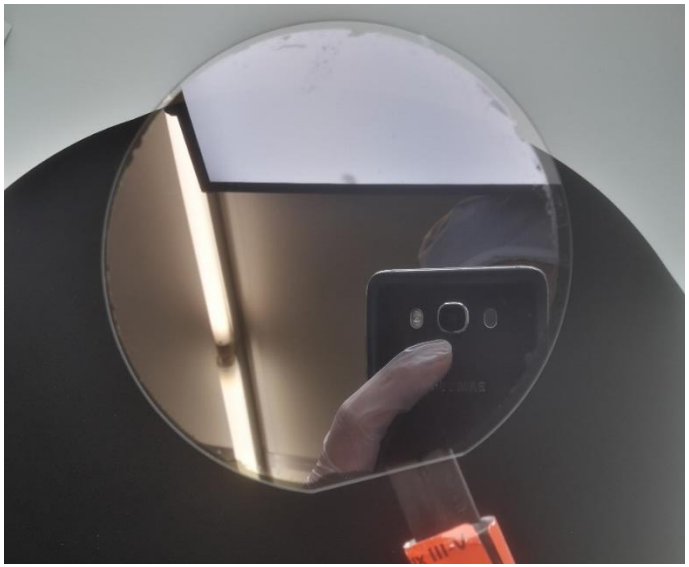
Measured stack

AlGaAs : 257,391 nm
GaAs : 78,284 nm
AlGaAs : 89,657 nm
GaAs : 273.888 nm
SiO2: 0.52 µm

ELLIPSOMETRY CHARACTERIZATION



- **Wet etch HCl 10%**



Doublet transfer successful!

Theroretical stack

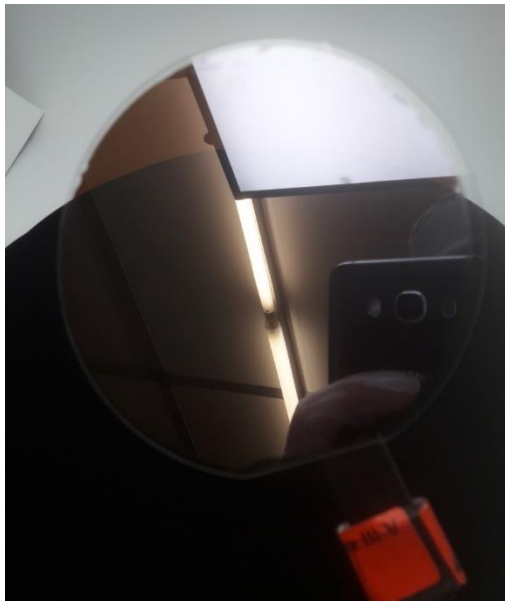
GaAs	76,4 nm
AlGaAs	89,3 nm
GaAs	270 nm
SiO2	525 µm

Measured stack

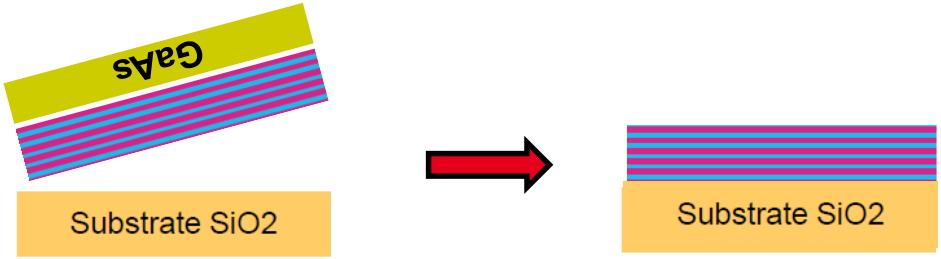
GaAs : 77.863nm
GaAs : 89,083 nm
GaAs : 274.402 nm
SiO2 : 0.52 µm

CONCLUSION

- Development of a process to transfer GaAs on SiO2 for 100mm substrates
- Complete validation of each step of the process
- Doublet successfully transferred on SiO2

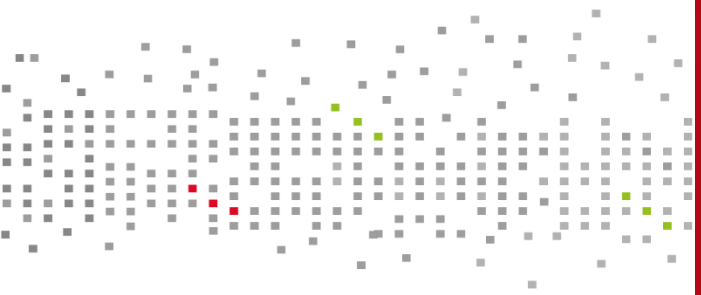


- Next step : Applying the process to the transfer of the final multilayer



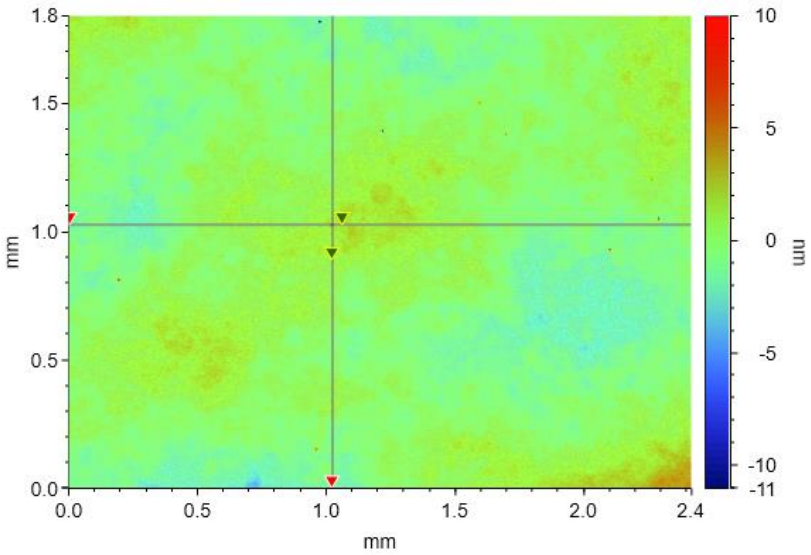
MERCI POUR VOTRE ATTENTION!

THANK YOU FOR YOUR ATTENTION!

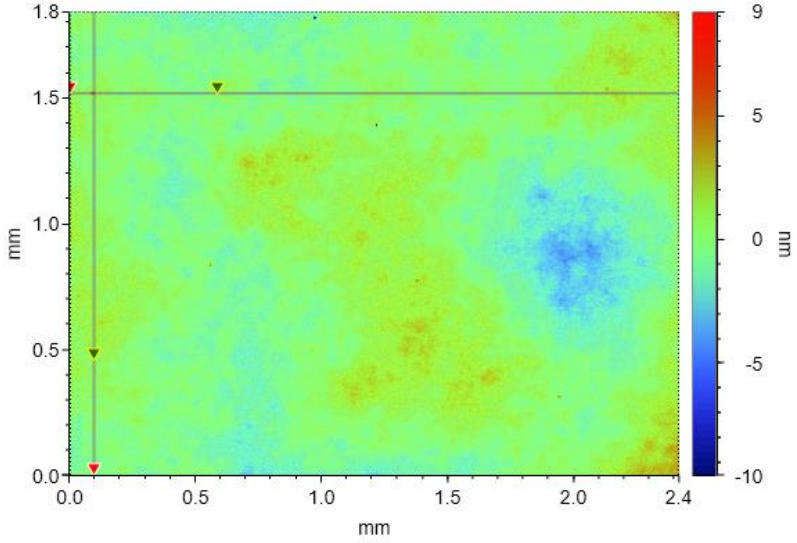


INPUT CHARACTERIZATION ON MULTILAYERS AT : INTERFEROMETRIC MICROSCOPY

- **Current Multilayers**



A1195



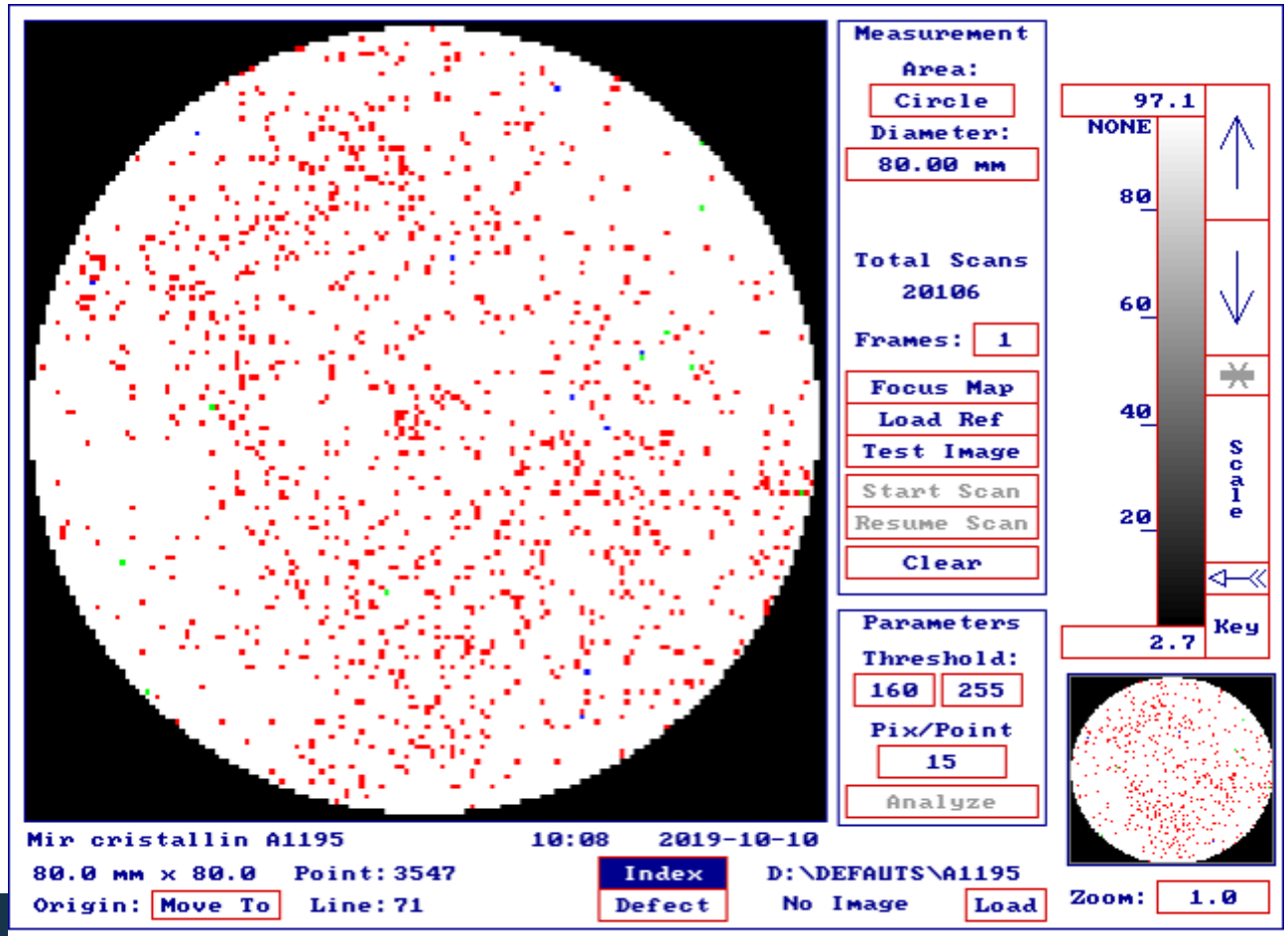
A1197

	A1195	A1197
RMS,nm	1,03	1,1
Nb defects	~8	~6
Nb defects/mm²	1,8	1,4

Better surface quality than in 2018 but planarization necessary for direct bonding (RMS>0.2nm) => CMP

INPUT CHARACTERIZATION ON MULTILAYERS AT LMA : DEFECTS MAP

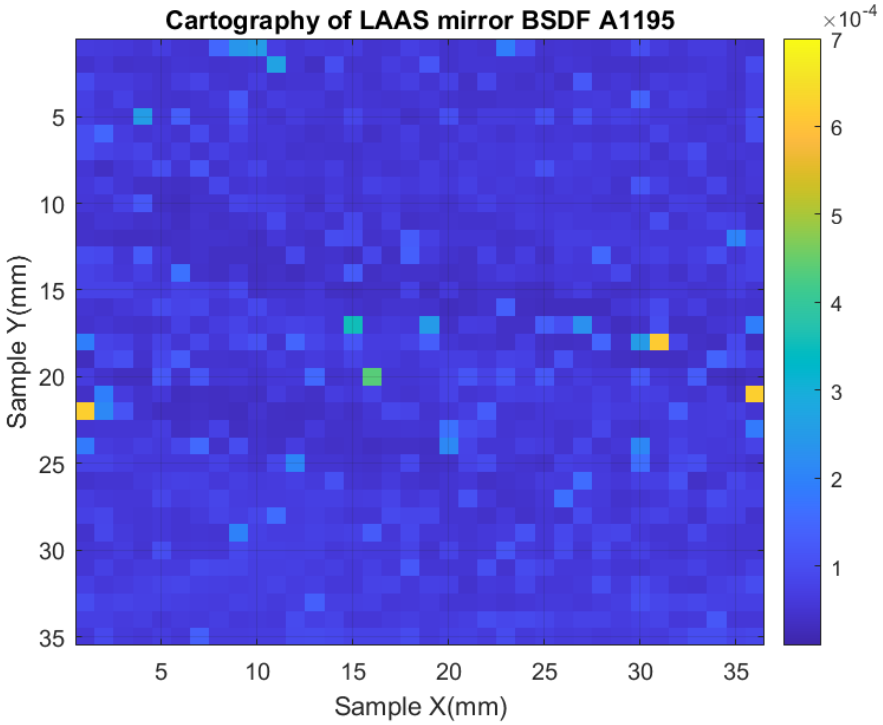
- Defects map of A1195



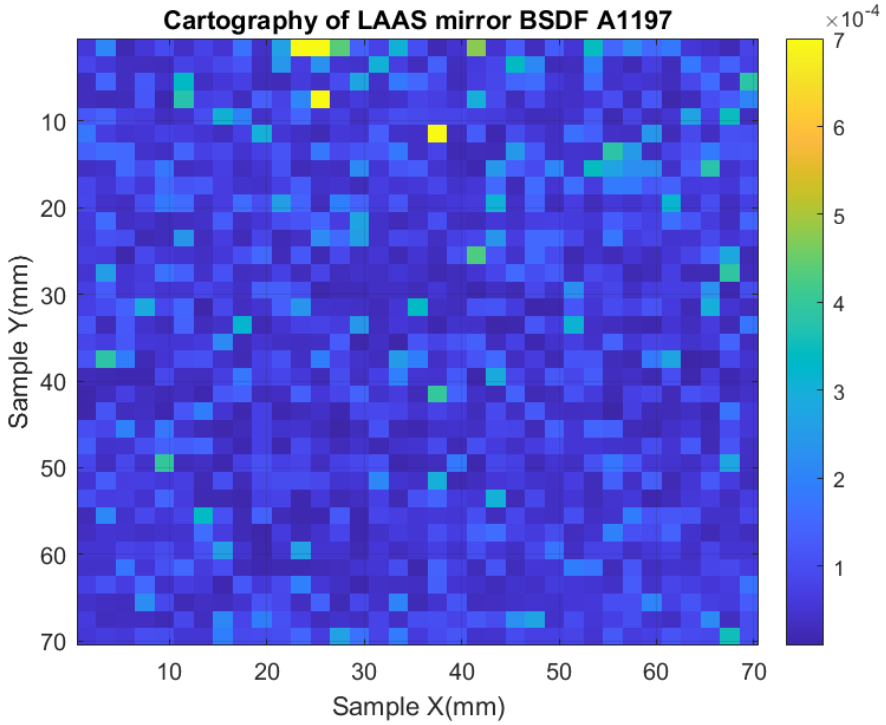
Nb tot of defects :	3618
Nb defects/mm²	1,4

Match characterization with interferometric microscopy

INPUT CHARACTERIZATION ON MULTILAYERS AT LMA : DIFFUSION MAP



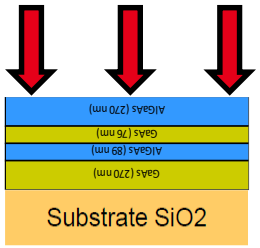
Mean diffusion : 45 ppm



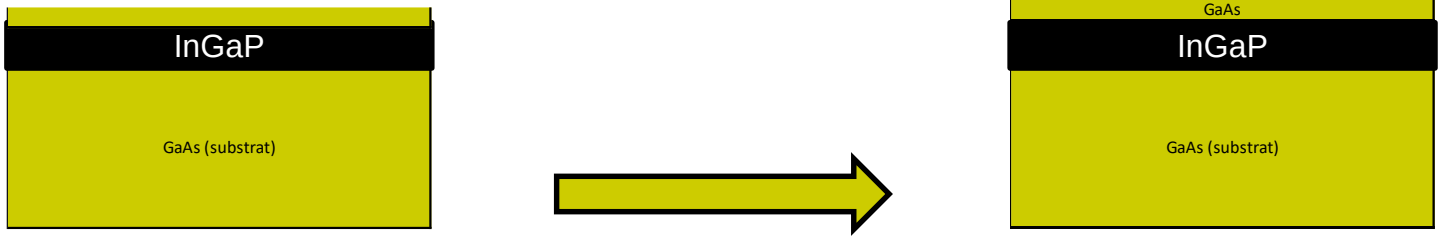
Mean diffusion : 60 ppm

Virgo mirrors : 5-10 ppm

WET ECTHING GAAS/ALGAAS

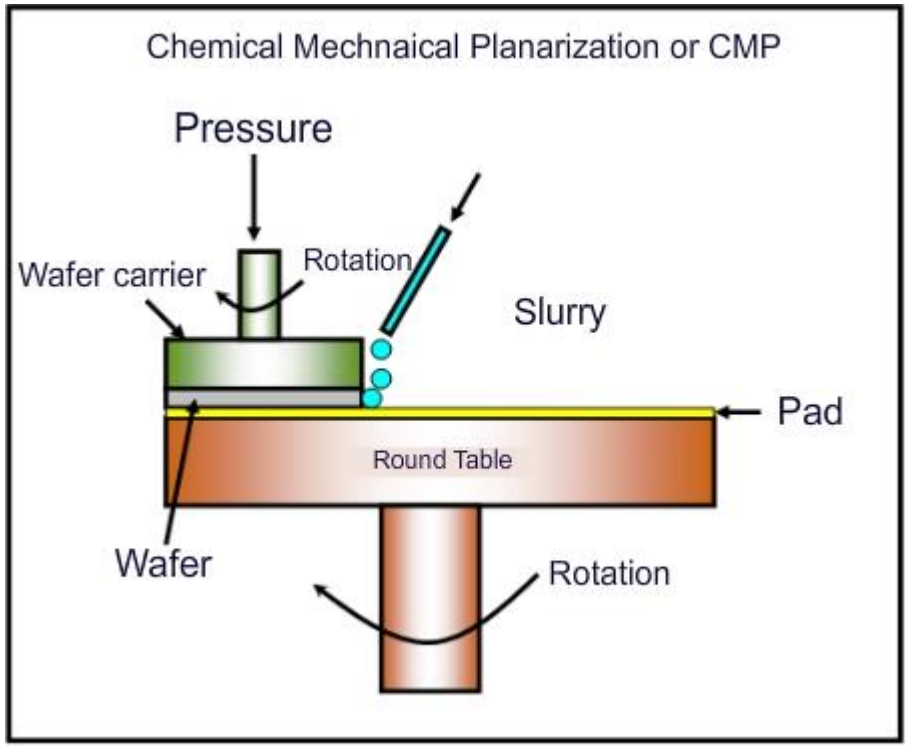


- **NH4OH/H2O2 not selective for wet etching of GaAs over AlGaAs**
- **Possible solutions**
  - Indium based etch stop layer (InGaP)
  - Selective etching of GaAs over InGaP well known at CEA (H3PO4/HCl)

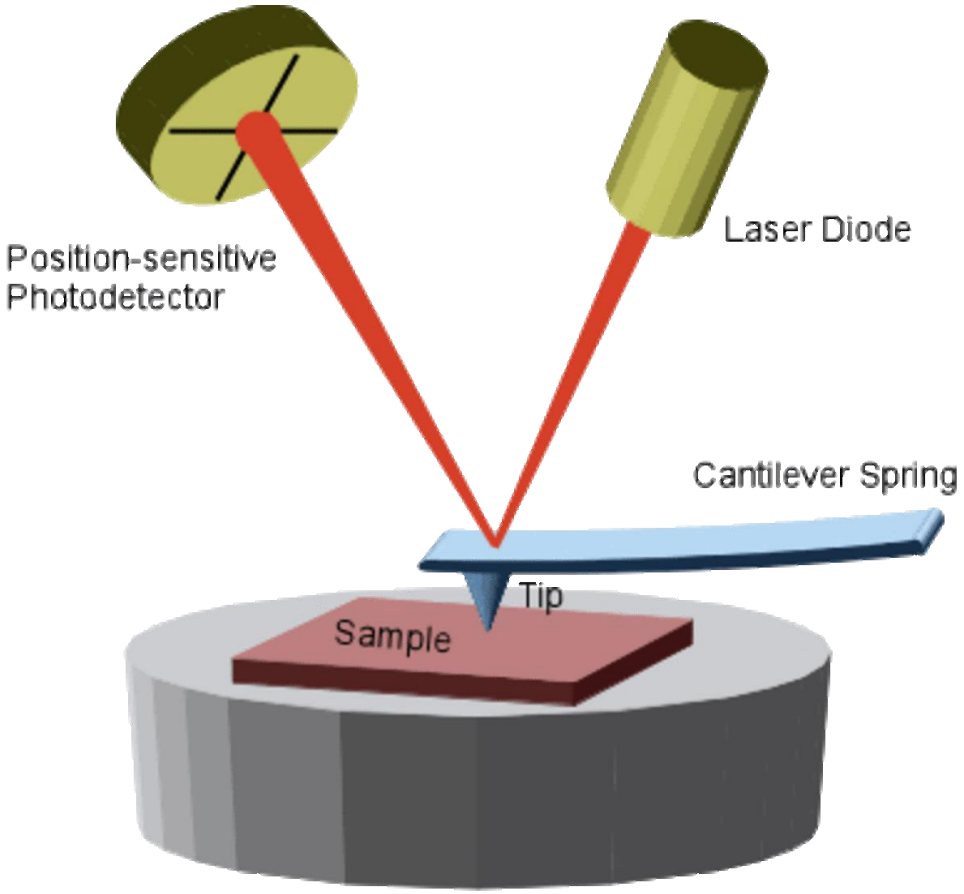


InGaP layer (70nm + GaAs cap layer (20nm)

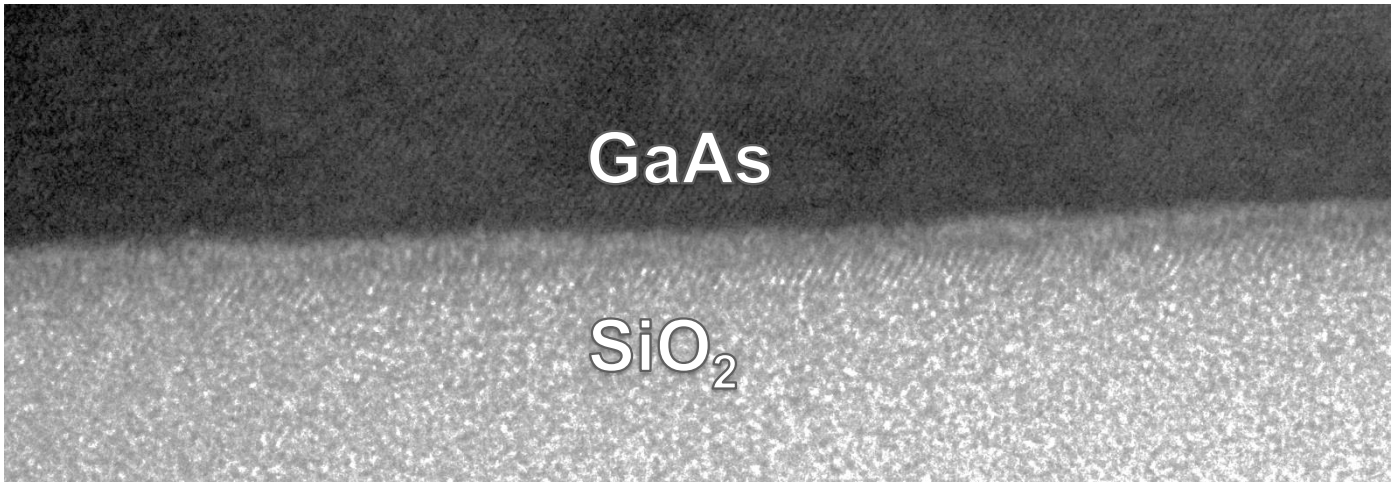
CMP

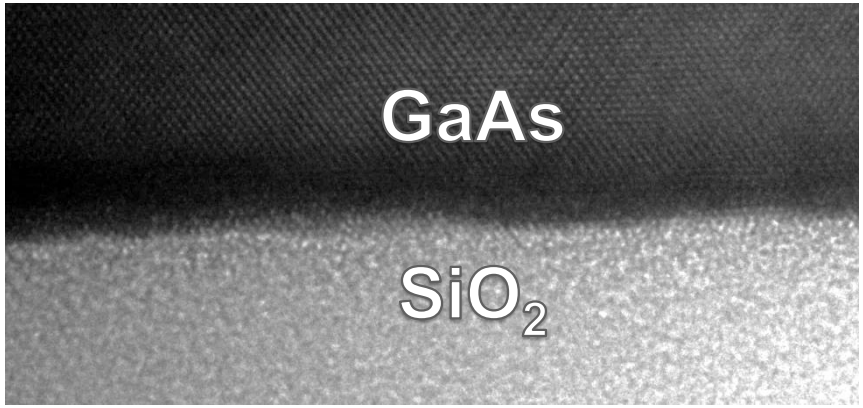

Chemical Mechnaical Planarization or CMP
Pressure
Wafer carrier
Rotation
Slurry
Pad
Round Table
Wafer
Rotation

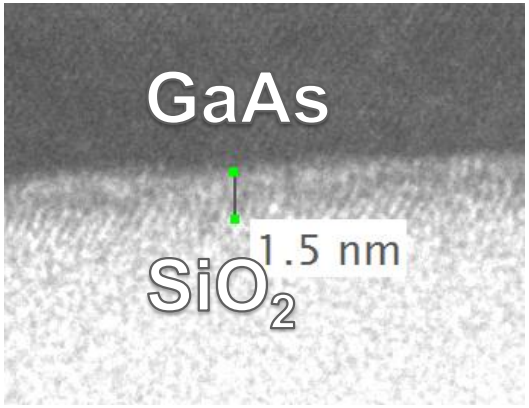
AFM


Position-sensitive
Photodetector
Laser Diode
Cantilever Spring
Tip
Sample

TEM CHARACTERIZATION OF BONDING INTERFACE



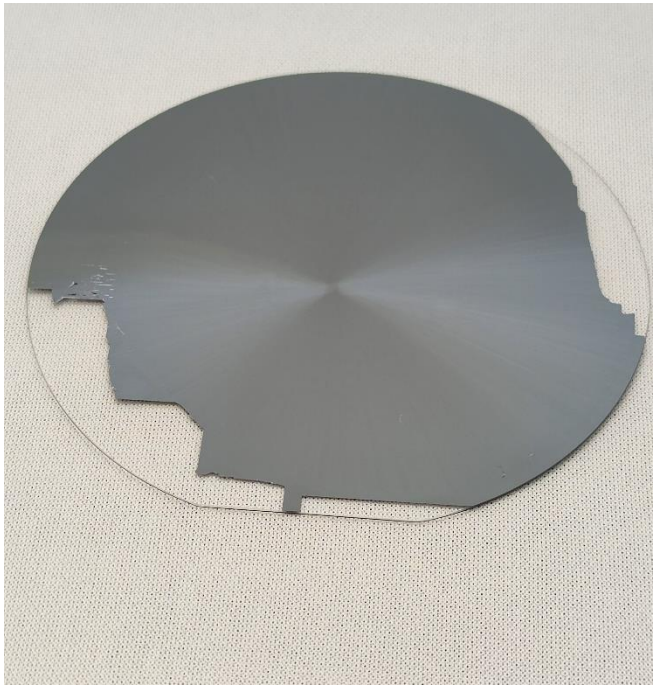




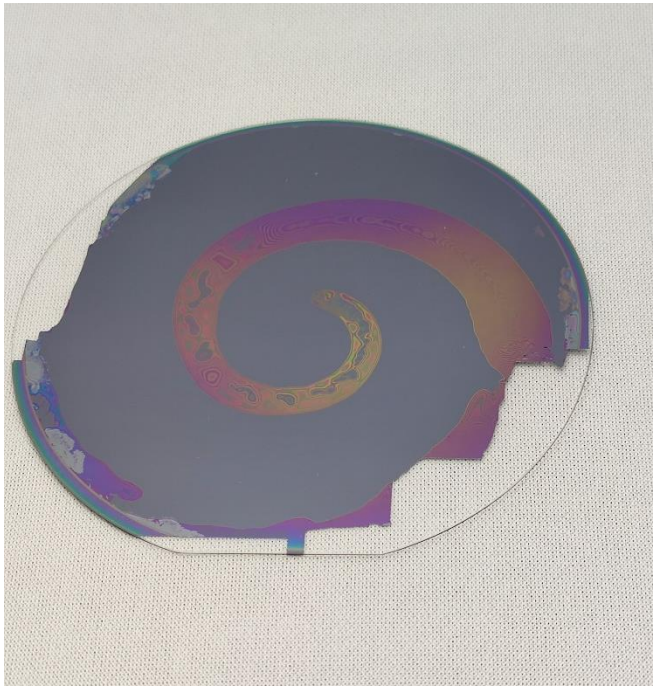
Heterostructure reconstruction OK

GRINDING

- **Grinding multilayer (A1195)**



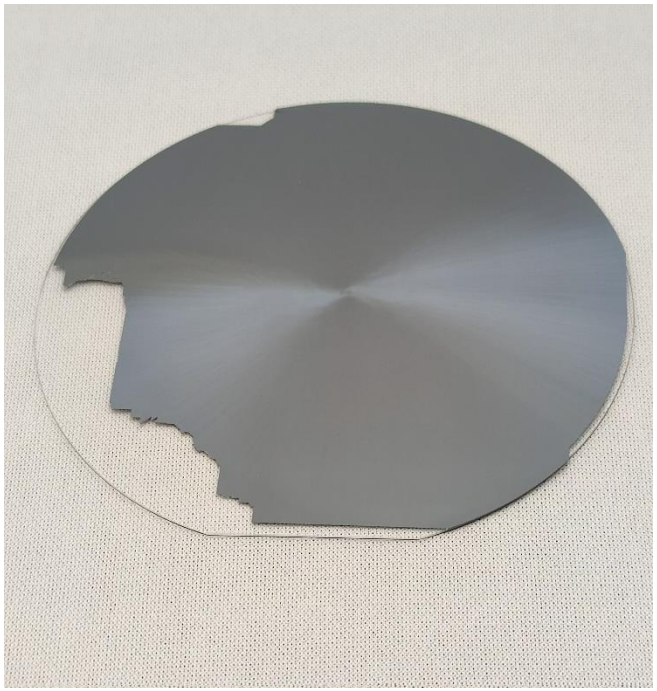
P01 Front face (GaAs)



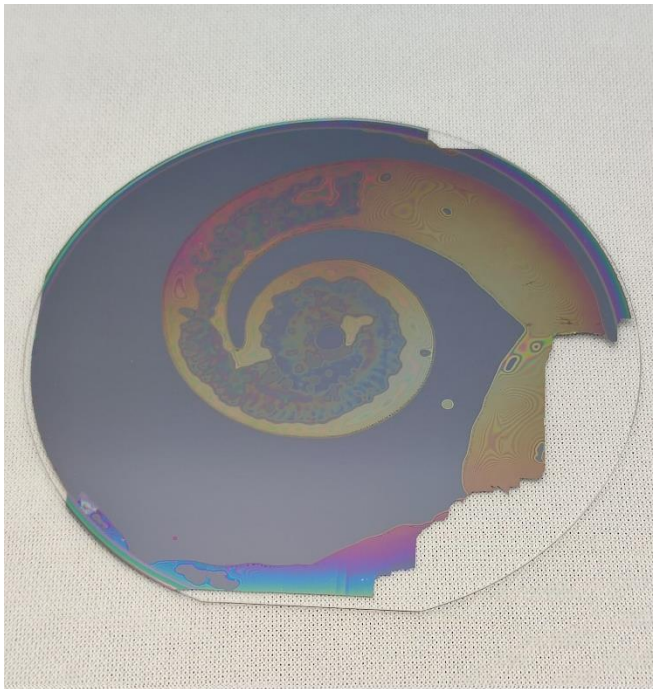
P01 Back face (SiO2)

GRINDING

- **Grinding multilayer (A1197)**

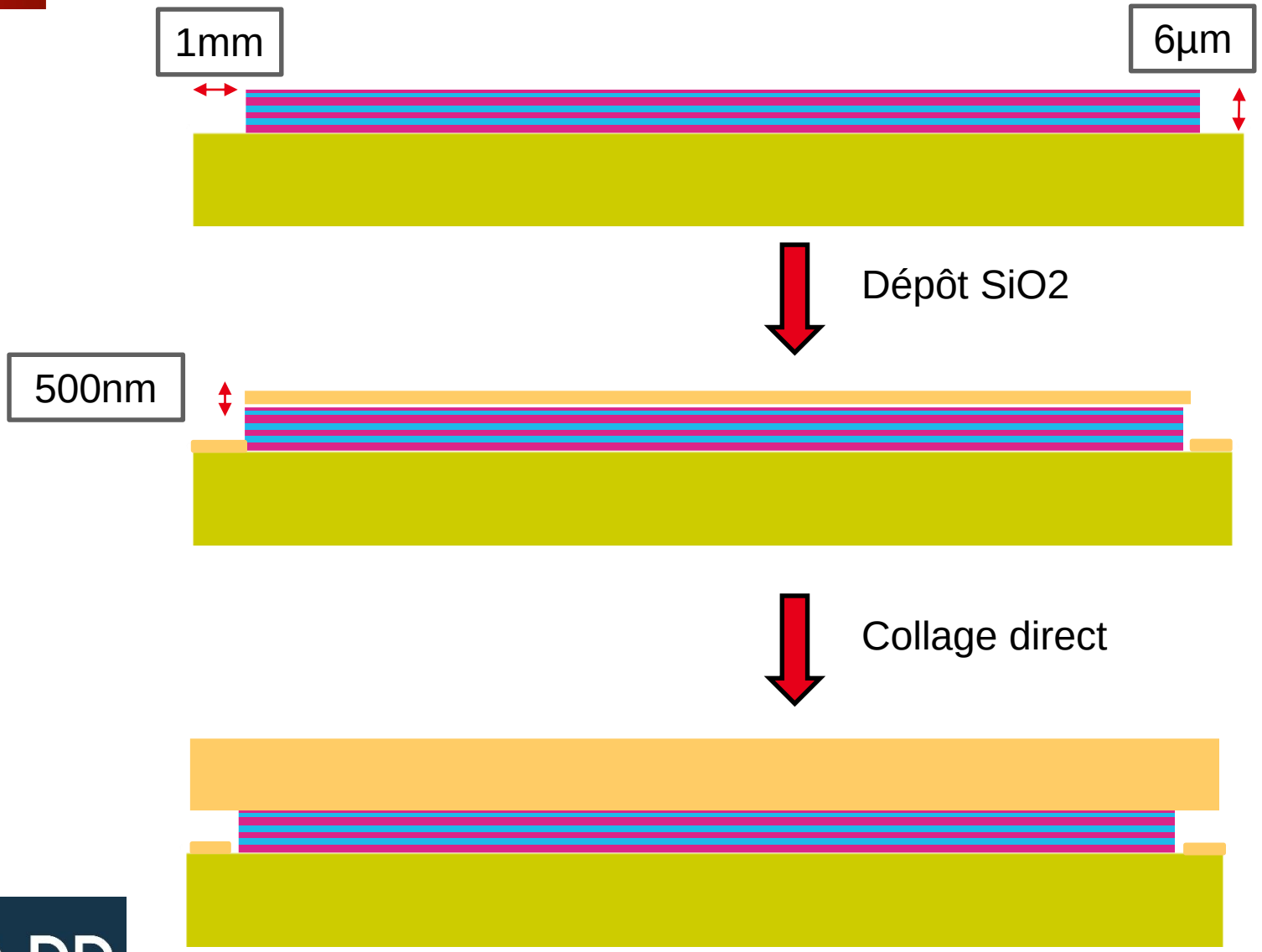


P02 Face avant (côté substrat GaAs)



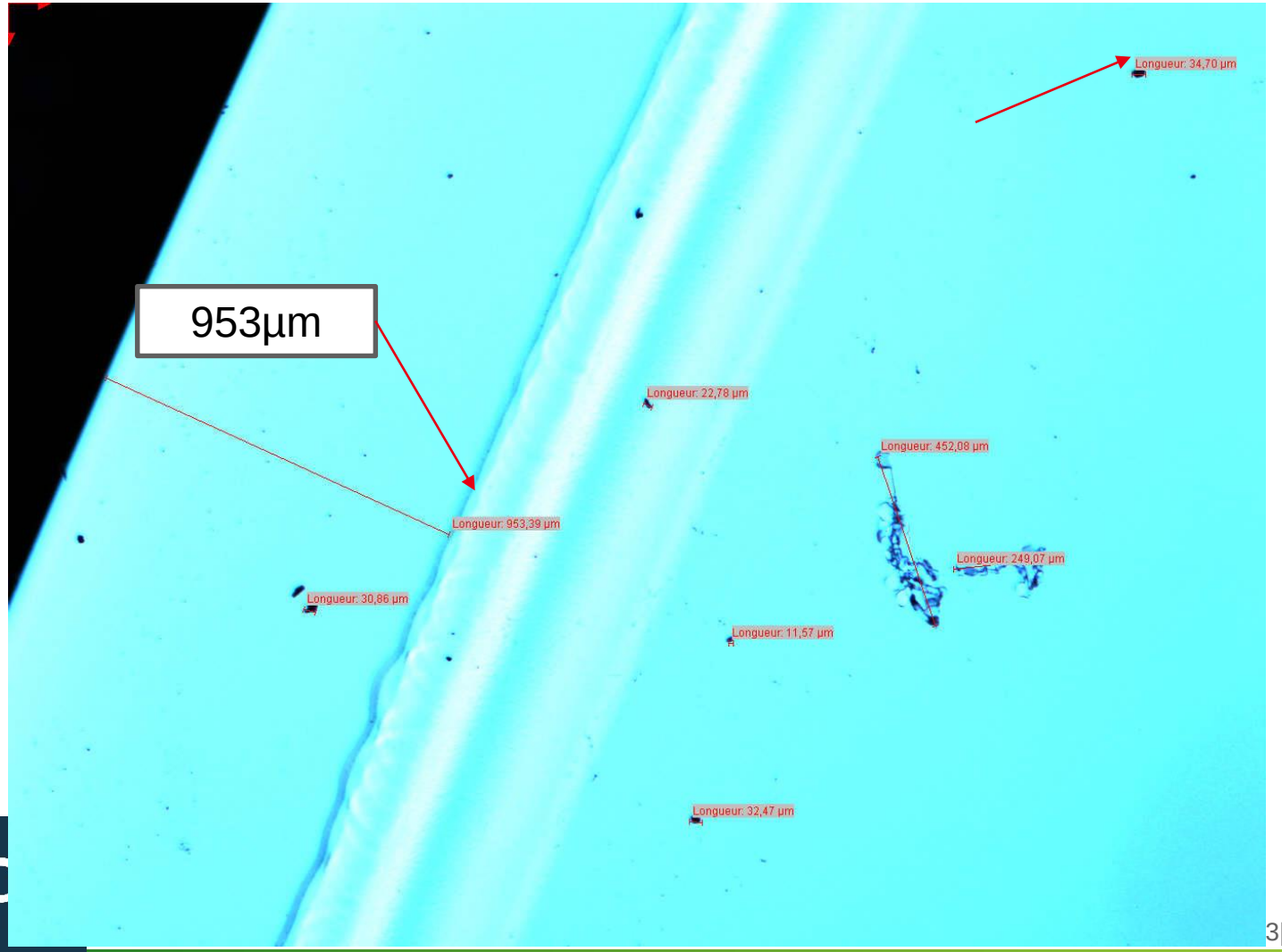
P02 Face Arrière (côté SiO2)

EXPLAINATION : EDGE TOPOLOGY

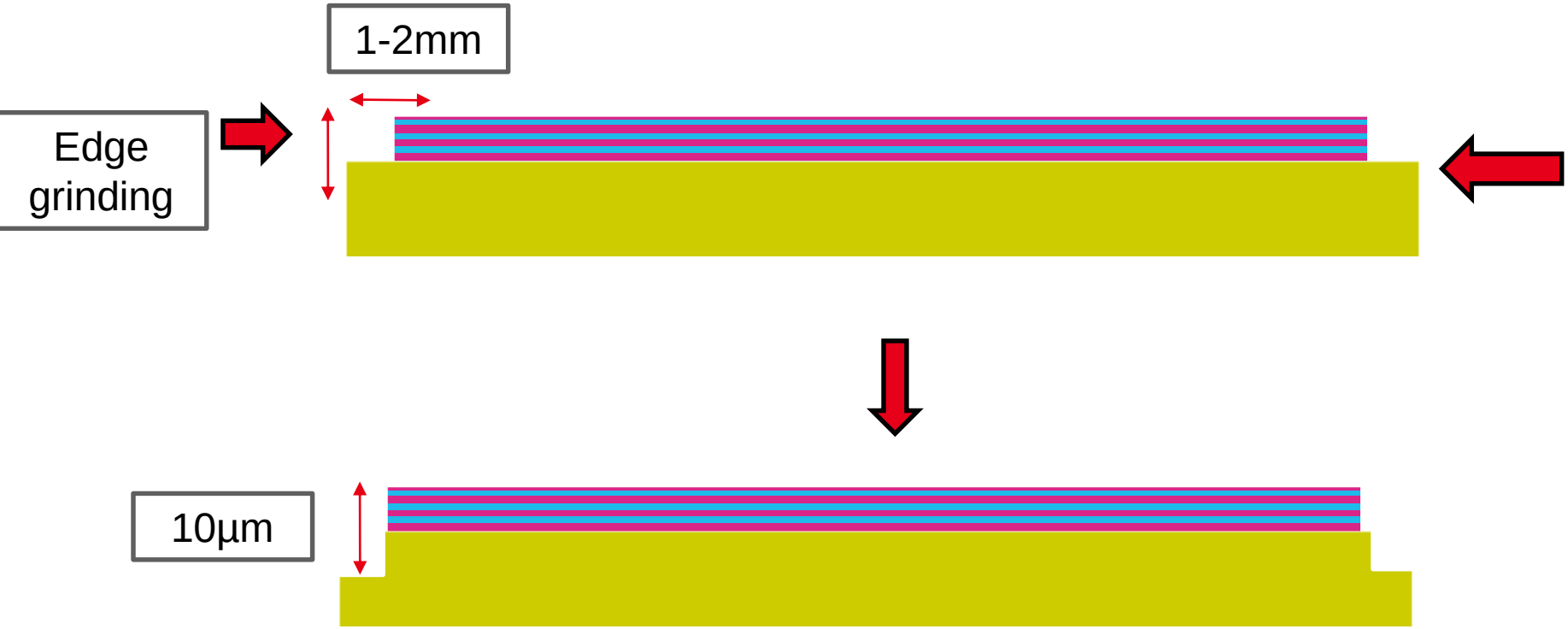


EDGE TOPOLOGY

- A1195 bord x2,5

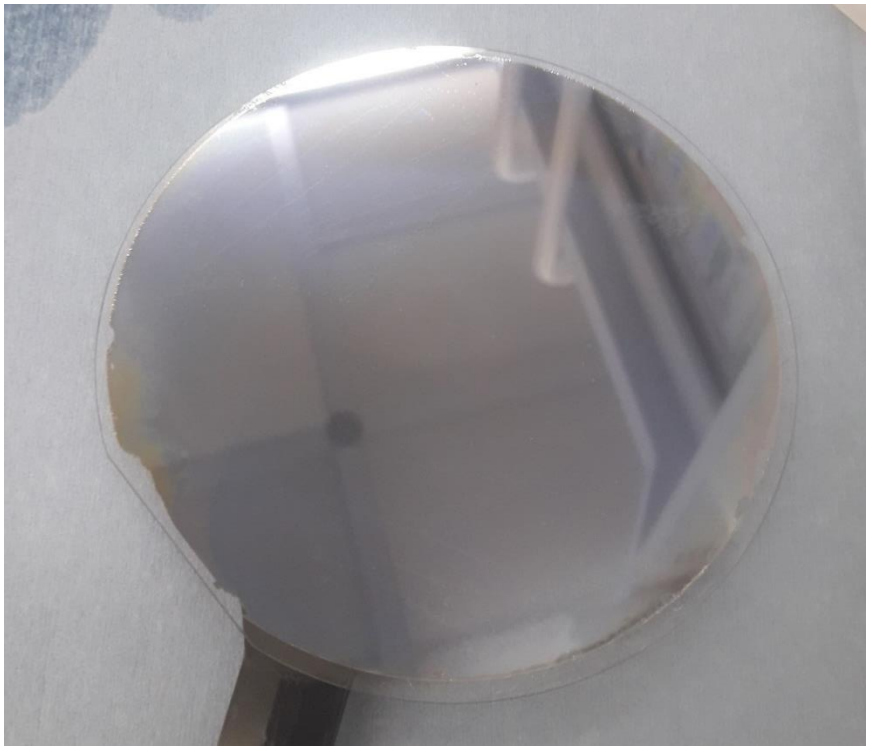


SOLUTION : EDGE GRINDING

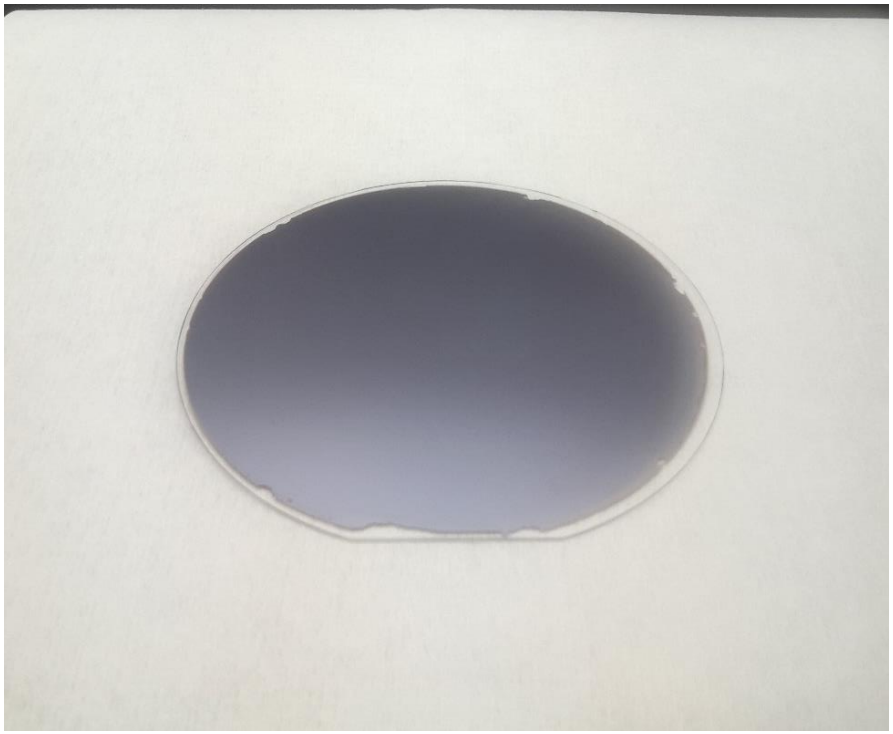


GRAVURE HCL10%

- **Gravure 2x30s**



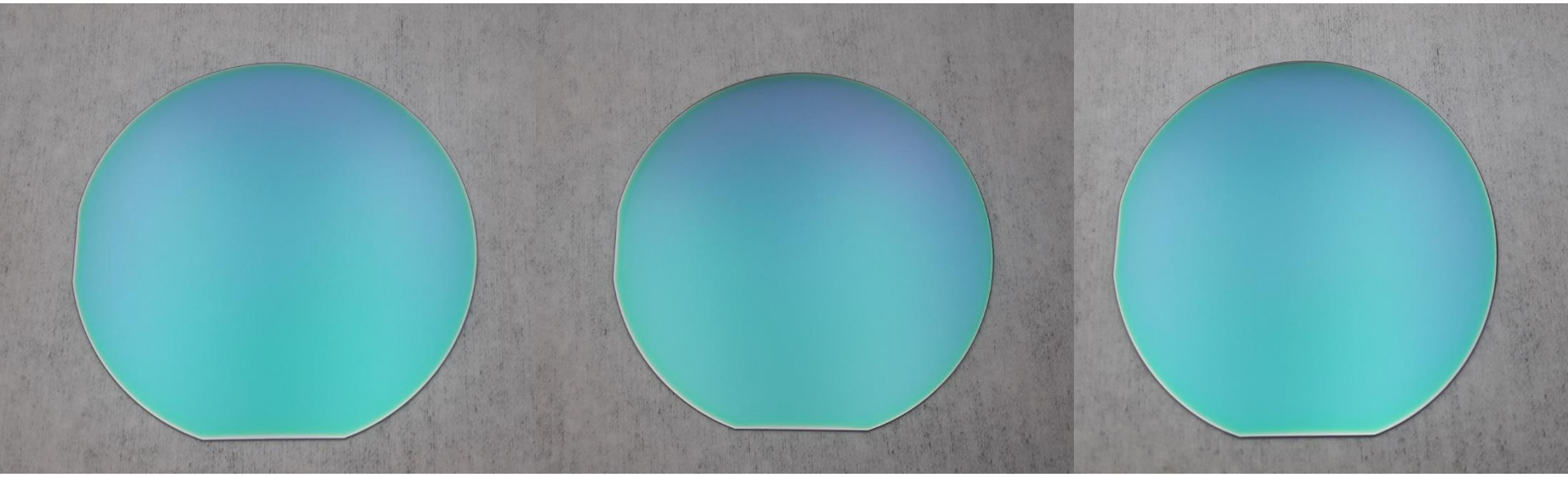
Après 30s de gravure



Après 60s de gravure

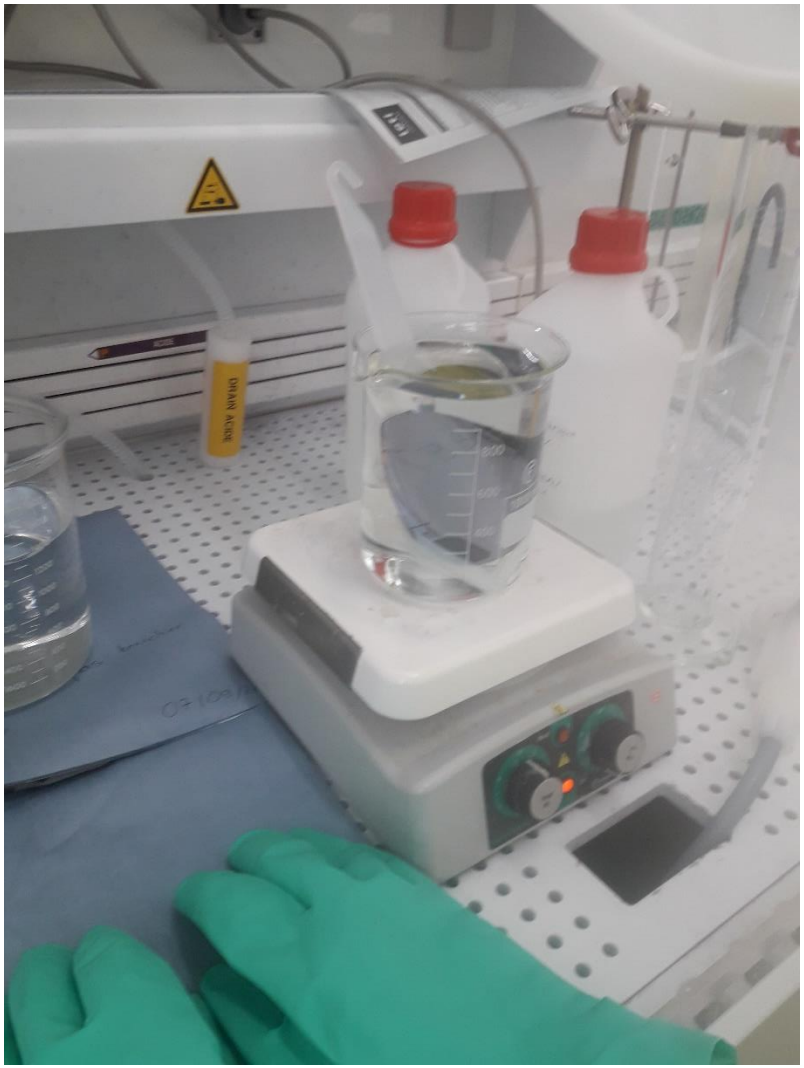
DÉPÔT SIO2 SUR ASGA

- **Photos lumière rasante**

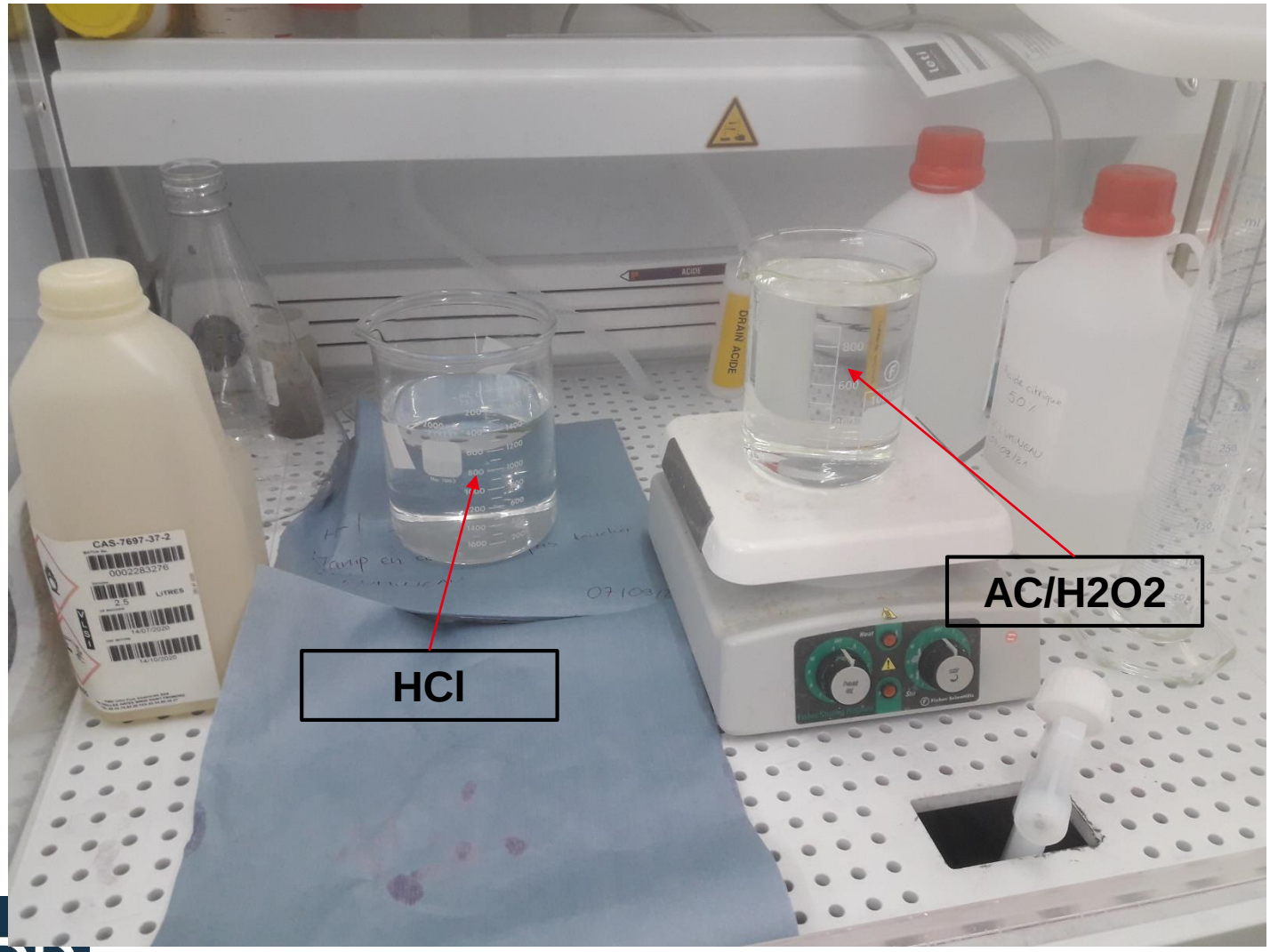


P01 P04 P05

GRAVURE CHIMIQUE



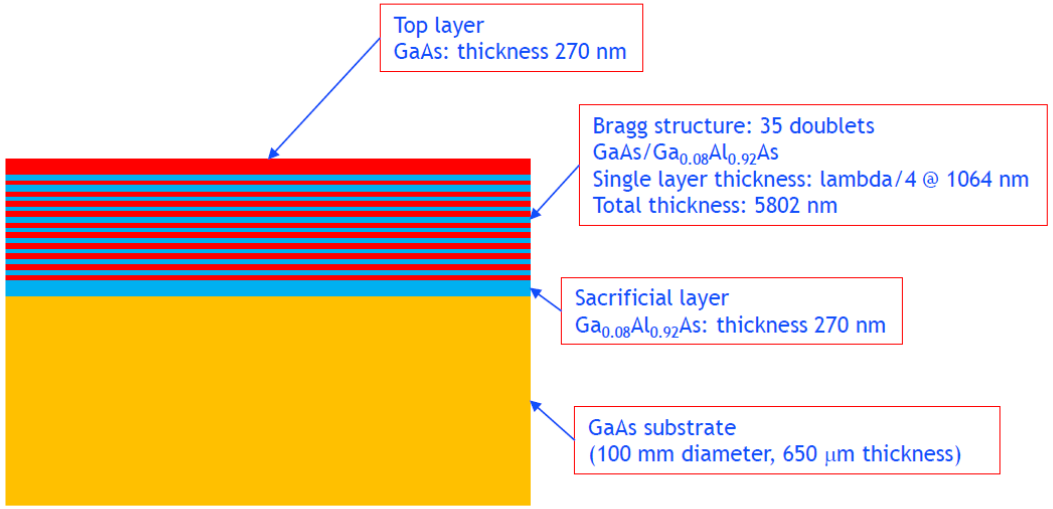
GRAVURE CHIMIQUE



GRINDING

MULTICOUCHE

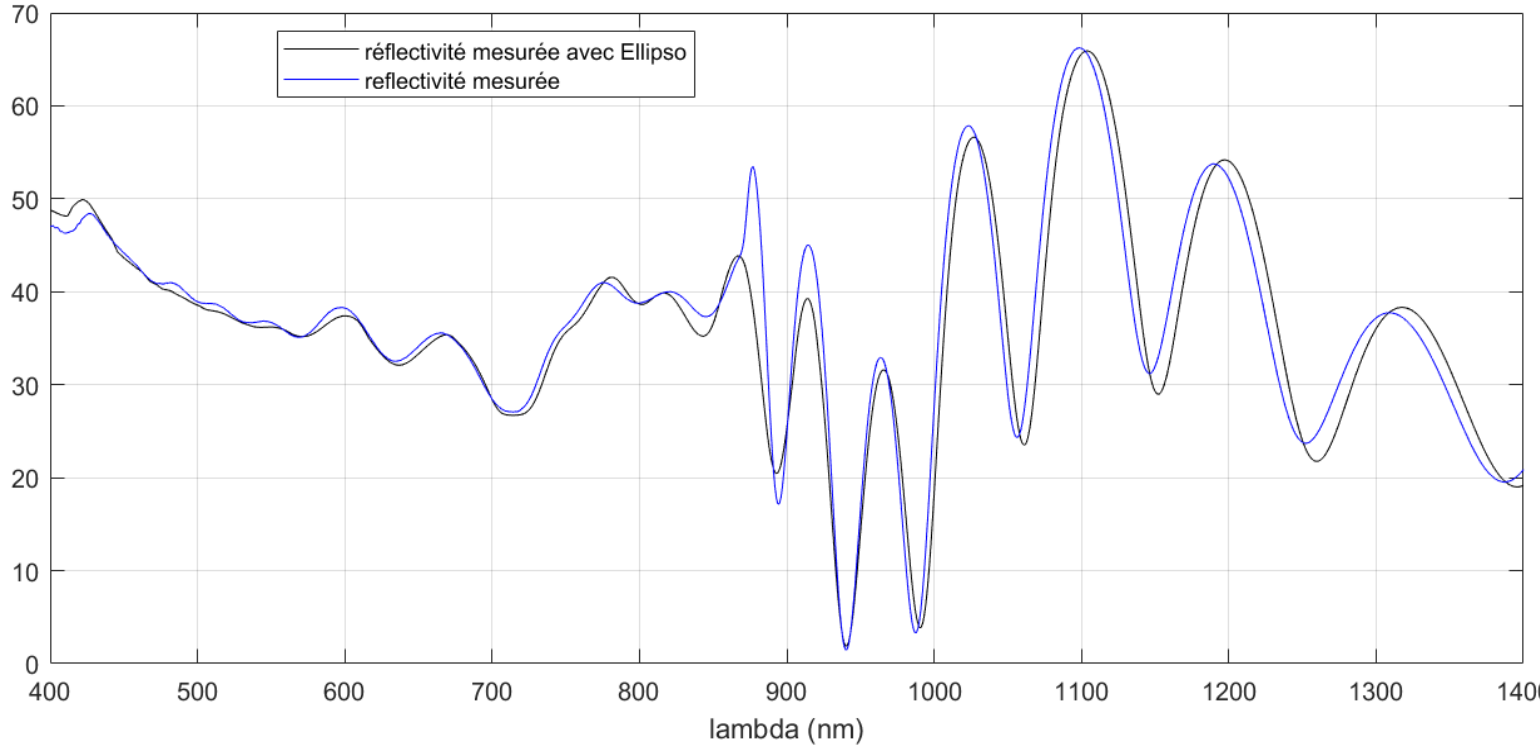
Multicouche GaAs/GaAlAs



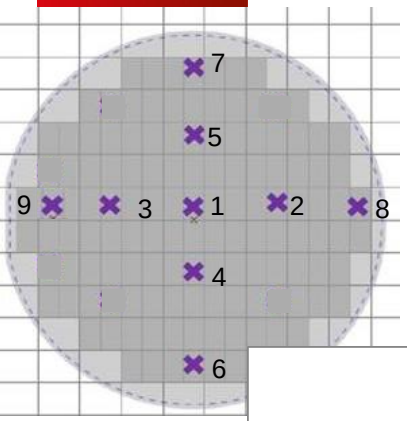
COMPARAISON SPECTRE DE REFLEXION AVEC ÉPAISSEURS MESURÉS AU CEA ET AU LMA

Mesures LMA

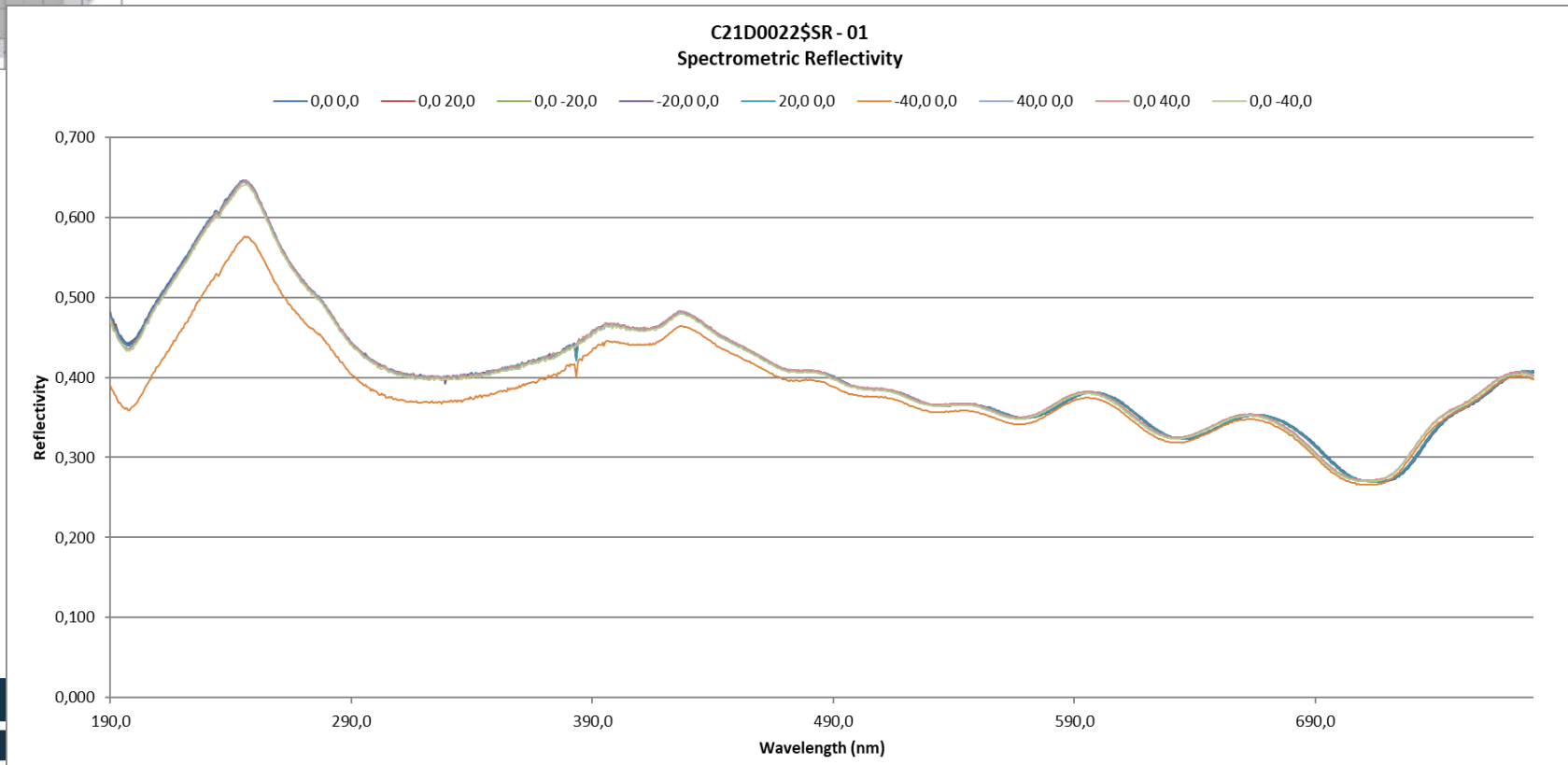
Simu avec mesures CEA



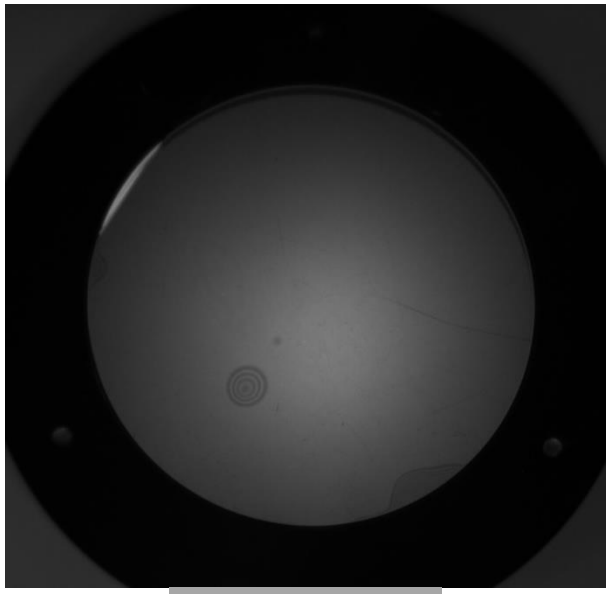
CARTO REFLEXION SUR NANOMETRICS



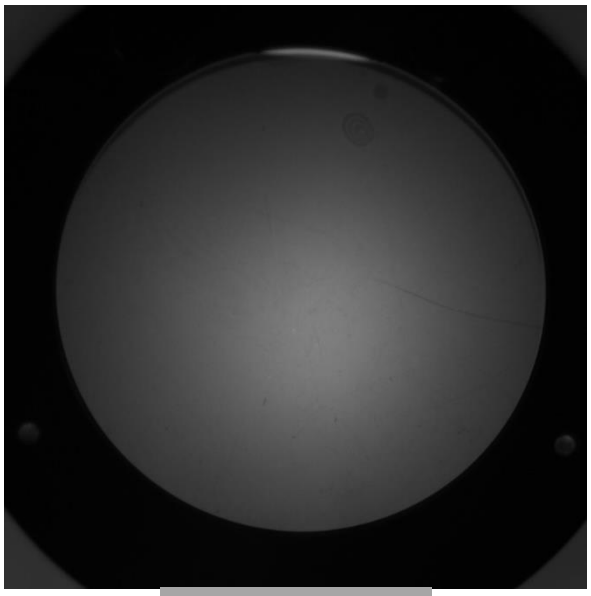
- Différence sur l'amplitude pour X=-40mm et Y=0mm
  Différence majoritairement dans l'UV => variation sur l'oxydation/état de surface



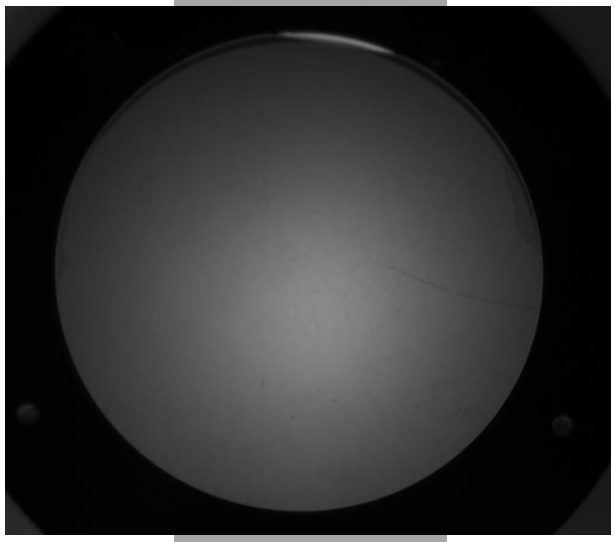
DIRECT BONDING GAAS/SIO2



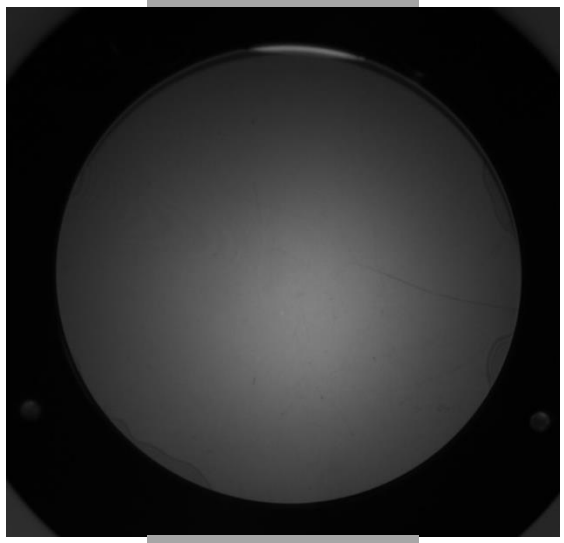
P01/P11



P02/P12



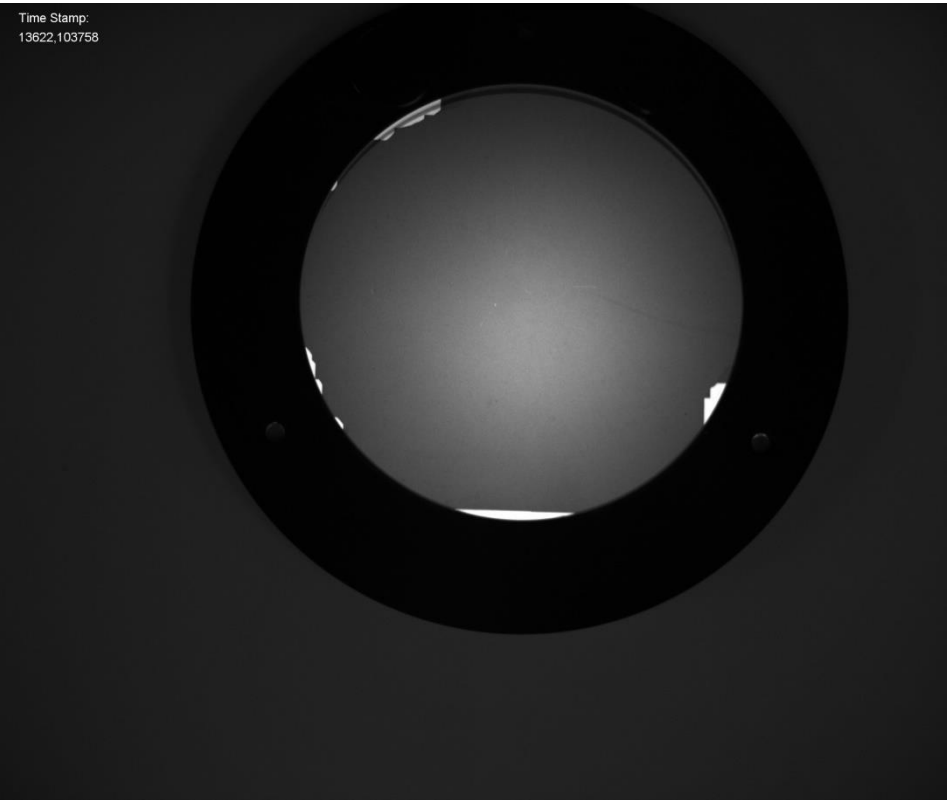
P03/P13



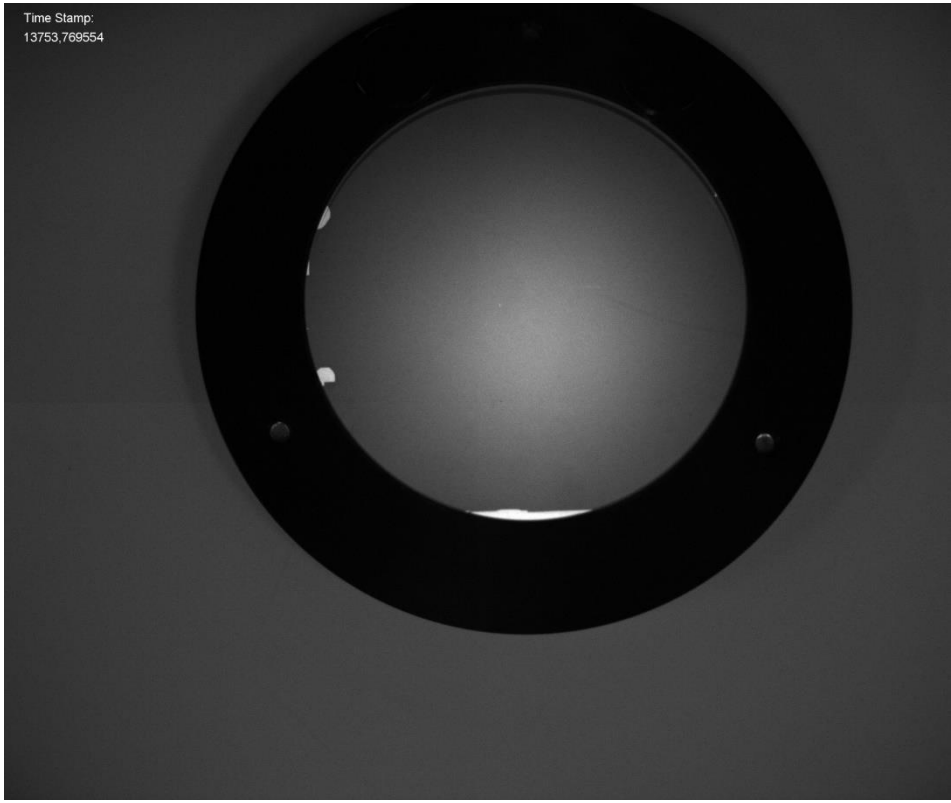
P04/P14

PHOTOS IR P12 AND P14

- P12 : grinding @100µm + etching @20µm+ recuit à 100°C sous N2
- P14 : grinding @100µm + annealing 100°C sous N2



P12



P14

WAFERS AFTER DELAMINATION (C20D0054)