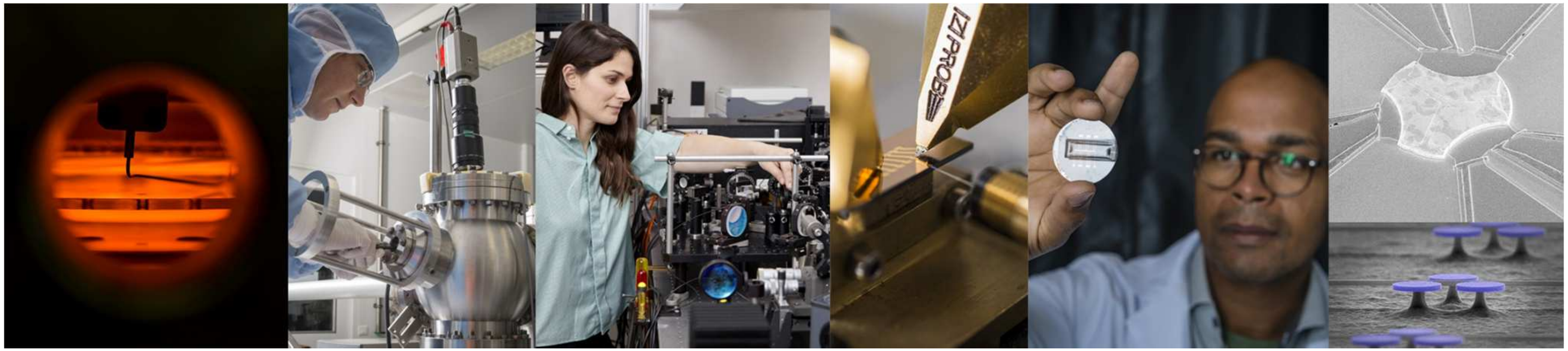
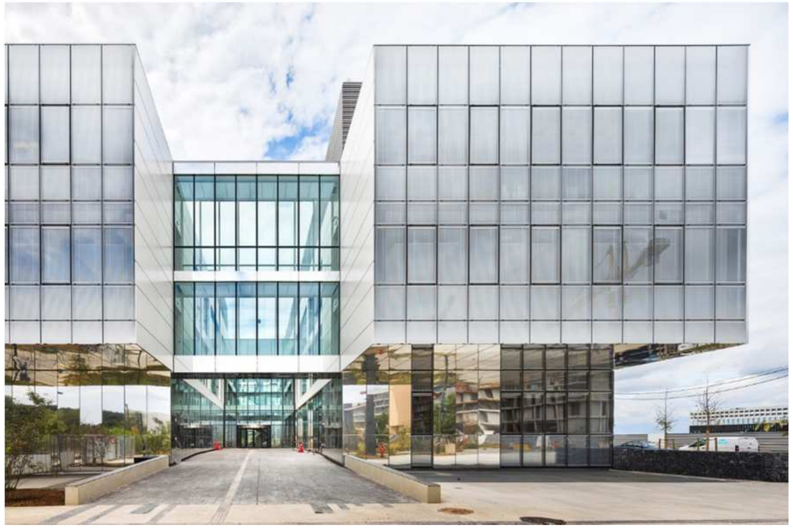
Centre de Nanosciences et de Nanotechnologies

Center for Nanoscience and Nanotechnology



A new research center for nanoscience

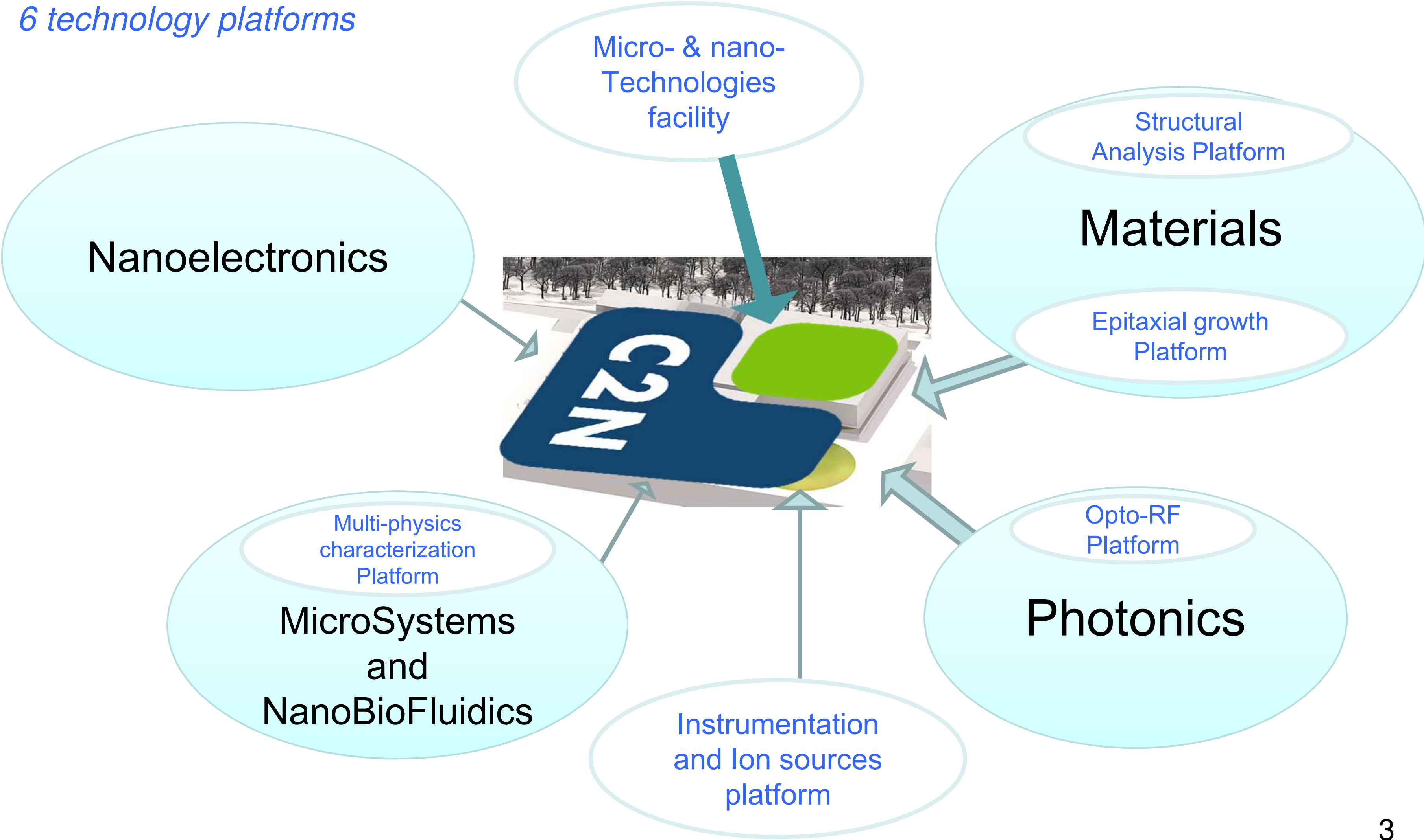
- Created in June 2016, merging of two labs IEF-upsud and LPN-cnrs
- 400 members:
  - 200 permanent researchers, engineers and admin staff
  - More than 100 PhD students and Post-docs
  - 37 nationalities
- 4 research departments
- 6 technology platforms
- A new building in the heart of Paris-Saclay
  - 18 000 m², including 2 900 m² high class cleanroom facilities



Photos: Sergio Grazia

C2N Research and Platforms

4 research departments
6 technology platforms



Photonique hybride III-V sur Si :

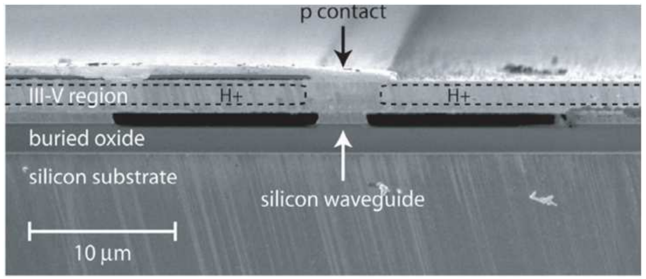
Caractérisation structurale, optique et électrique de l'interface

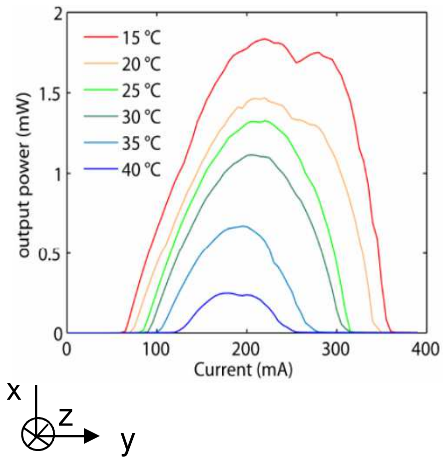
A.Talneau[(1)] , G.Beaudoin[(1)] , K.Pantzas[(1)] , F.Ducroquet[(2)] , D.Alamarguy[(3)] and G.Patriarche[(1)]

(1) C2N, Centre de Nanosciences et de Nanotechnologies 10 Boulevard Thomas Gobert, 91120 Palaiseau
(2) IMEP LAHC Univ. Grenoble Alpes F-38000 Grenoble
(3) GeEPs Group of Electrical engineering Centrale Supelec Univ Paris-Sud Plateau du Moulon F-91192 Gif sur Yvette

- Intégration hybride III-V sur Si : état de l'art
- Collage Oxide-free: caractérisation structurale et optique de l'interface
- Collage oxide @ 300°C : préparation des surfaces PEALD-SiO_2 et Ozone
- Caractérisation XPS des surfaces activées
- Mesure de l'énergie du joint de collage par nanoindentation
- Performances de dispositifs photoniques comportant un interface hybride
- Mesure du transport électrique à travers l'interface hybride
- Conclusion

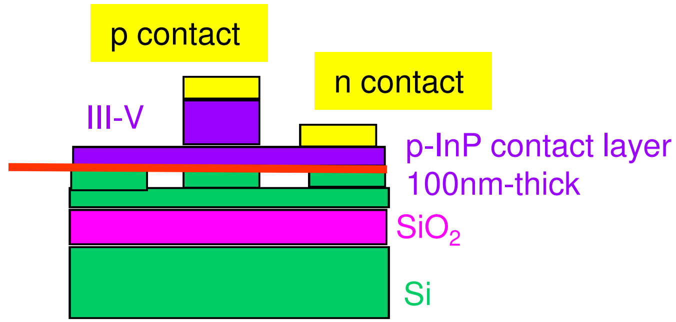
State of the art: Hybrid optoelectronic III-V/Si devices operating at 1.55µm including a Oxide-mediated bonded interface

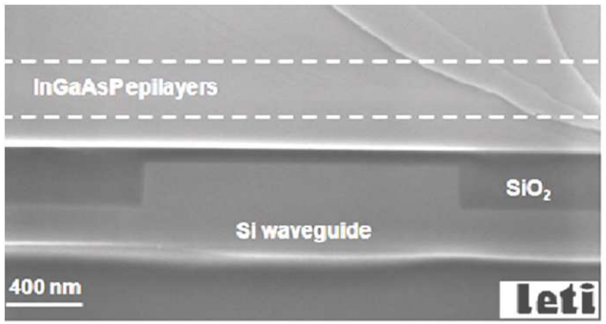


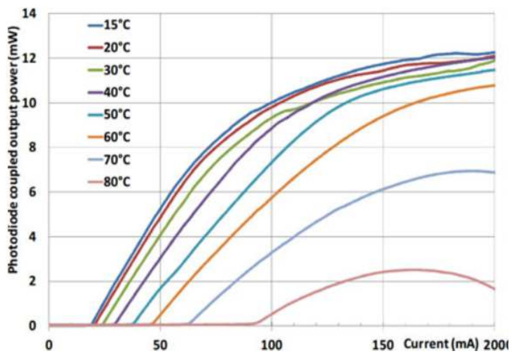


5nm-thick RIE SiO2 bonding layer

*A.W.Fang et al., MaterialsToday, **10**,28 (2007)*







50-100nm thick PECVD SiO2 layer
CMP planarized prior bonding

*B.BenBakir et al., Optics Express, **19**,10317 (2011)*

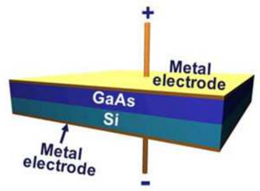
*G.H.Duan et al., J. Light. Technol, **33**, 976 (2015)*

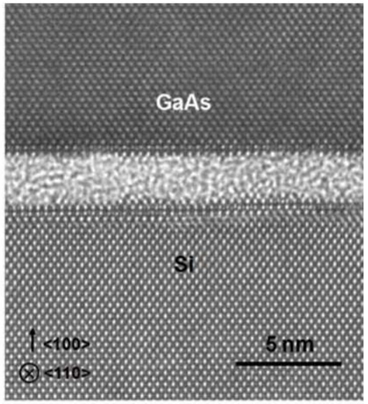
The oxide layer
- poor thermal conductivity
- low optical index material in the core of the device
- prevent electrical transport at the interface

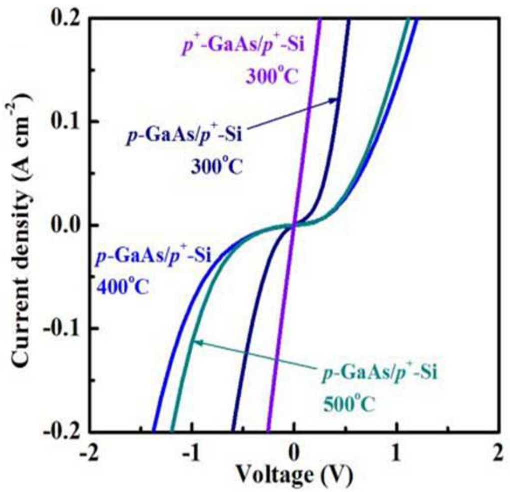
→ The thinnest layer will optimize the optical performances and allow electrical operation of the device through the hybrid interface

State of the art: Electrical injection through III-V / Si hybrid interface

➔ **GaAs /Si direct-bonded heterointerface**

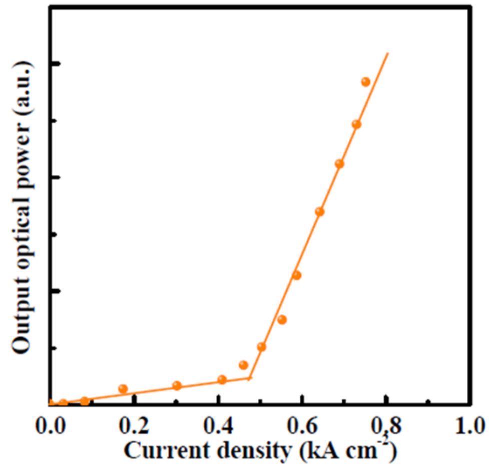


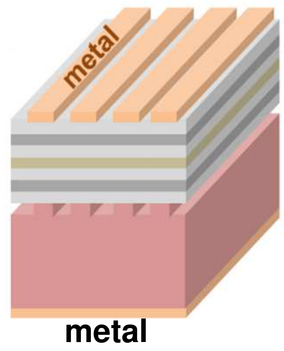




*K. Tanabe, K. Watanabe, and Y. Arakawa, Scientific Reports, **2**, 349 (2012)*

➔ InAs/GaAs QD lasers on Si rib waveguides with current injection through the heterointerface

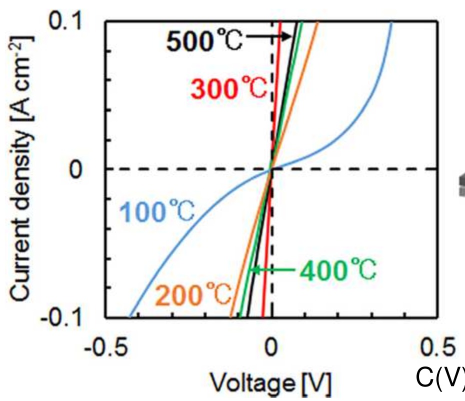


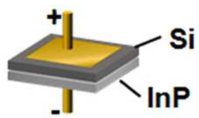


*K. Tanabe, K. Watanabe, and Y. Arakawa, Optics Express **20**,B315 (2012)*

➔ **Ohmic InP/Si direct-bonded heterointerface**

R.Inoue and K. Tanabe, Appl. Phys. Lett., 114,191101(2019)

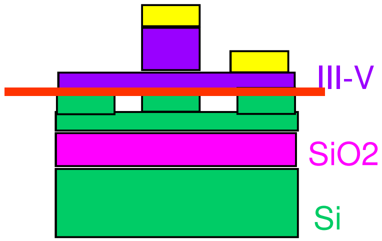


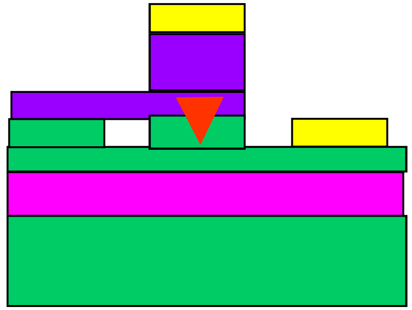


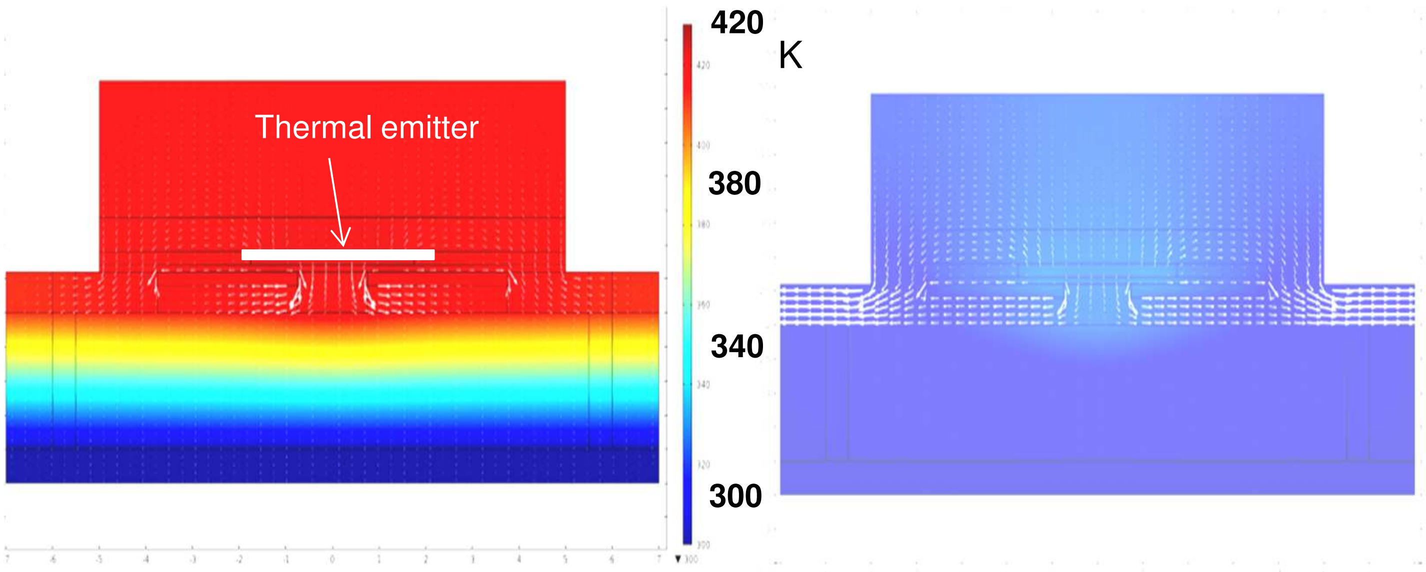
C(V) curves n-InP/p-Si bonded interfaces

Electrical operation through the interface : Improved thermal behavior

Simulation results







*M.N.Sysak et al., Optics Express, **15**,15041 (2007)*

COMSOL simulation, C2N, unpublished

Bonding equipement at C2N

Presse Instron + four SEMCO

custom-design

T up to 700°C
Perfect flatness on 2”
Bonding under vacuum

SB6e-Suss Microtech bonding machine

T up to 500°C
Flatness on 6”
Bonding under vacuum

Description des échantillons

- Membrane InP épitaxié (450nm) épitaxiée sur InGaAs/InP par MOVPE
- Retrait des oxydes natifs des surfaces de l'InP et du Si
- Collage
- Retrait chimique sélectif du substrat InP et de la couche d'arrêt InGaAs

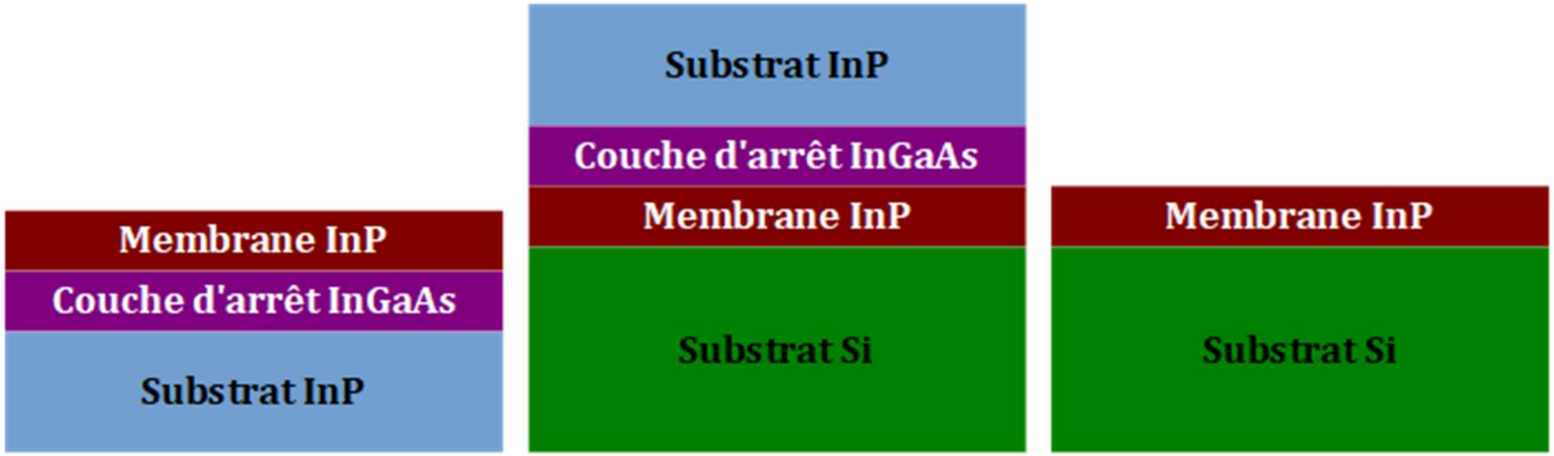


Fig. 2a : Structure épitaxiée

Fig. 2b: Structure après collage

Fig. 2c: Structure après retrait du substrat

InP / Si oxide-free bonding : experimental approach

1 - Both materials are grown, lattice matched, on their own substrate

$\Delta a/a = 8.10\%$

2 - Surfaces preparation, contact

3 – Annealing Vacuum T=550°C, 90mn Pressure : 0.2MPa

High pressure uniformity

Mandatory : smoothness, flatness, cleanliness, reactivity

Surface to be bonded

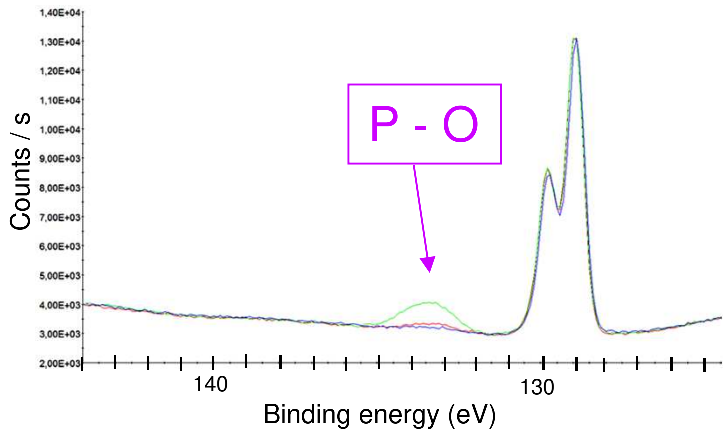
Quantum wells InP-based bonded stack

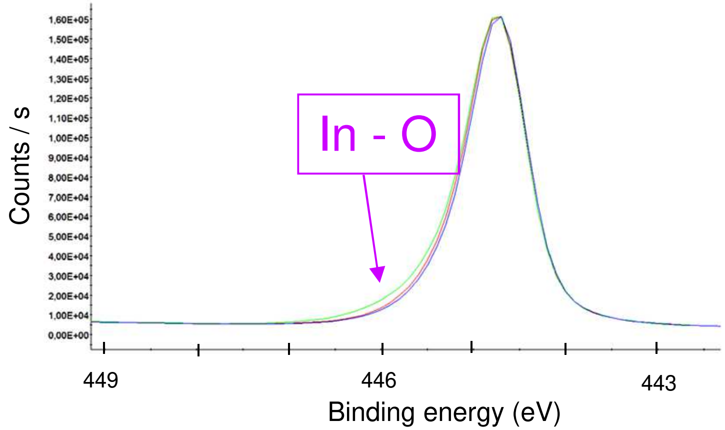
InP top layer	**71 nm**
5 InGaAsP barriers **4 InGaAs QW**	**16 nm** **10 nm**
InP	**71 nm**
InGaAs stop etch	**300nm**
InP buffer, InP substrate	**300µm**

♦ **Oxide-free surfaces** **Surface preparation:**

Si : RCA, HF 4% → hydrophobic, oxide-free –H terminated

InP : HF 40% → oxide-free





XPS of oxide-free InP surface

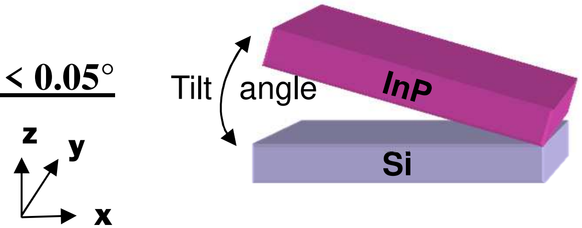
X-Ray characterization of the bonded membrane

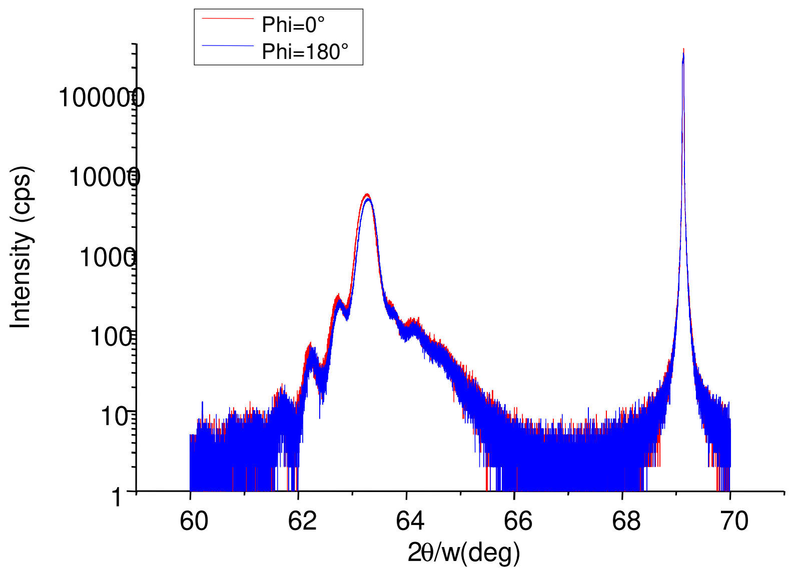
InP/Si $\Delta a/a = 8.10\%$

Perpendicular mismatch ~ **8.151%**

Parallel mismatch ~ **8.152%**

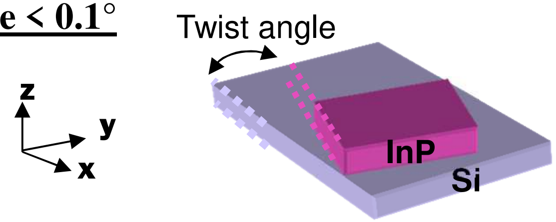
Tilt angle < 0.05°

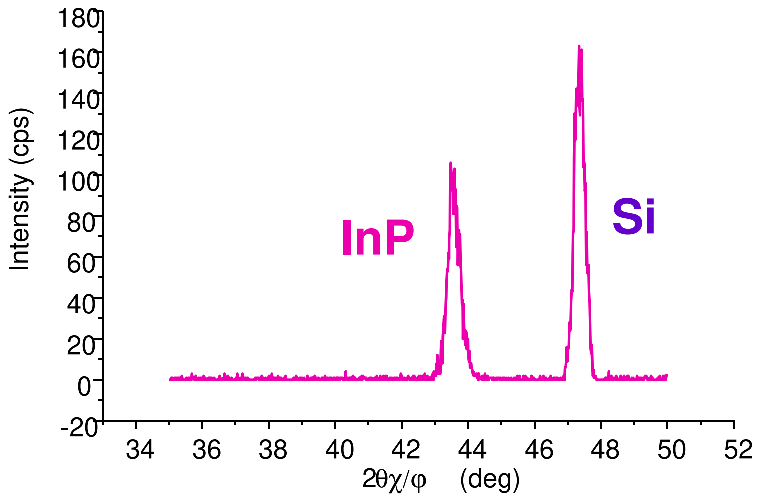




Diffraction peaks due to (004) planes, versus angular position of the detector

Twist angle < 0.1°





Diffraction peaks due to (220) planes normal to the surface

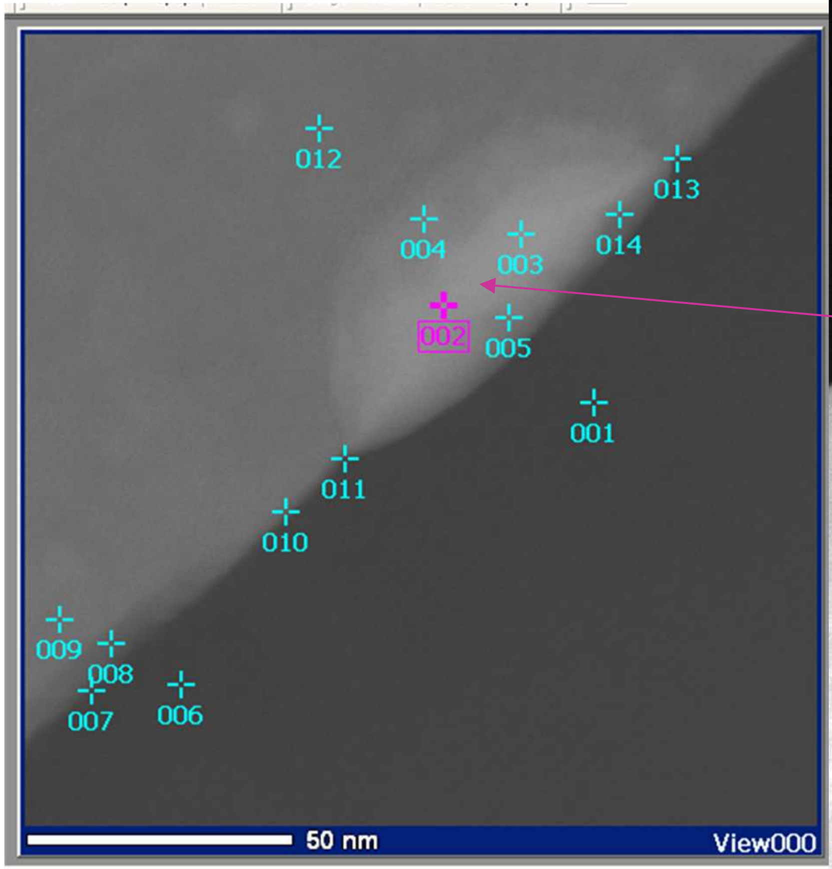
♦ Correct relative alignment of both crystals

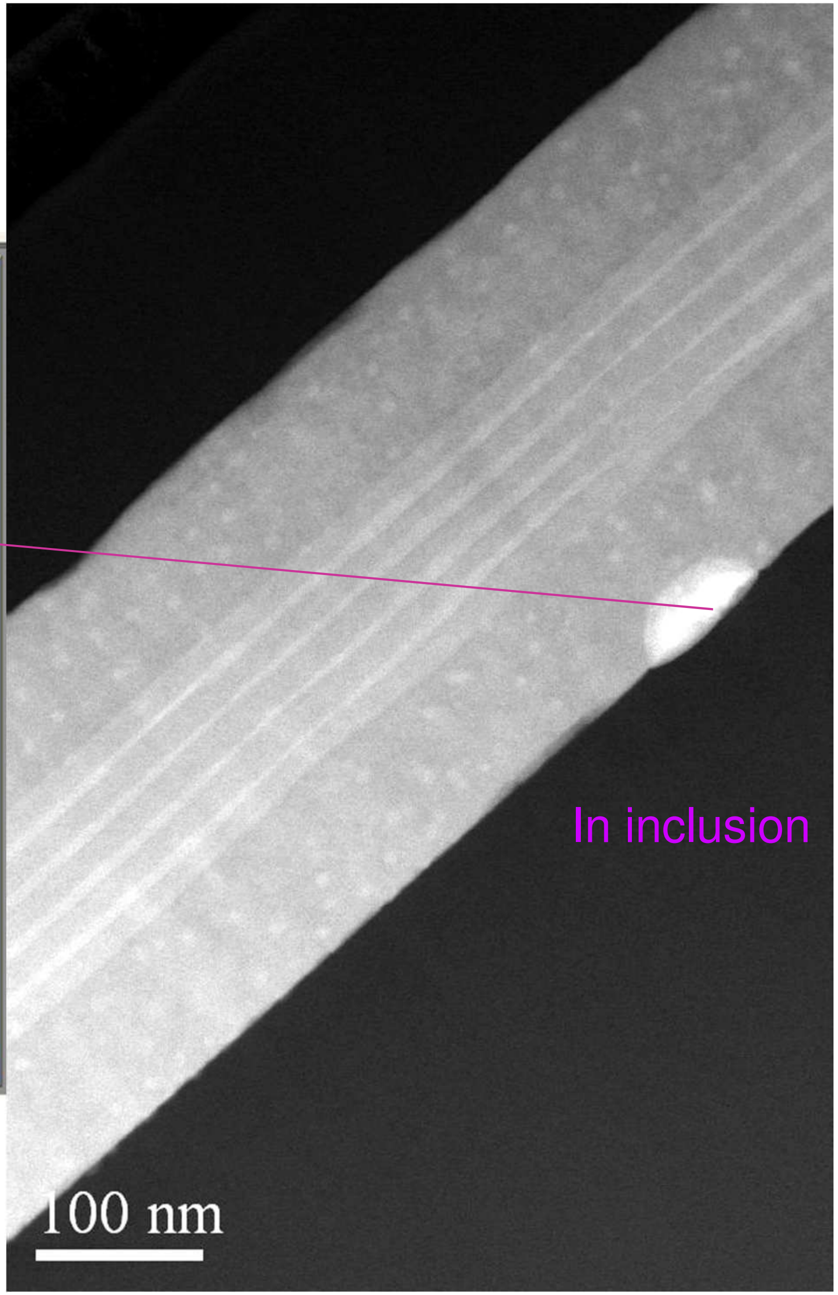
Tilt angle < 0.05°

Twist angle < 0.1°

TEM characterization of the bonded interface

Bright field STEM micrograph





- No voids at the bonded interface

TEM characterization of the bonded interface

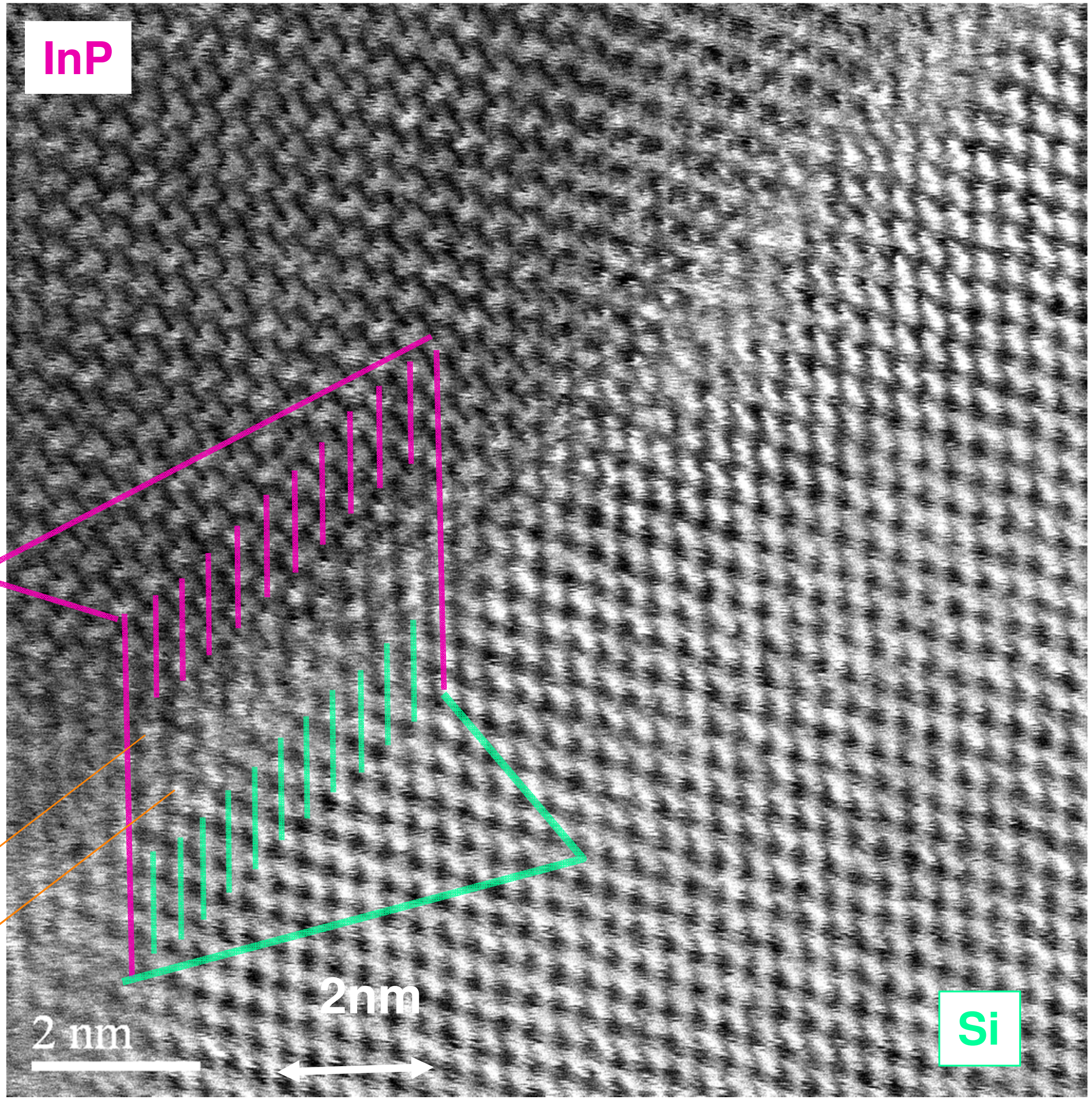
a(InP)= 5.87Å

a(Si)=5.43Å

(111) planes

11×(111)

12×(111)

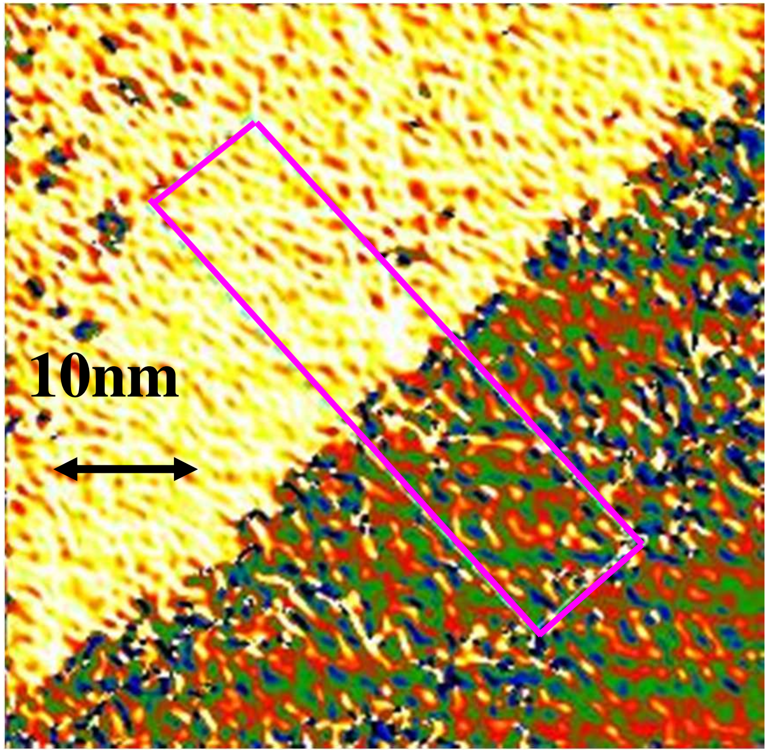


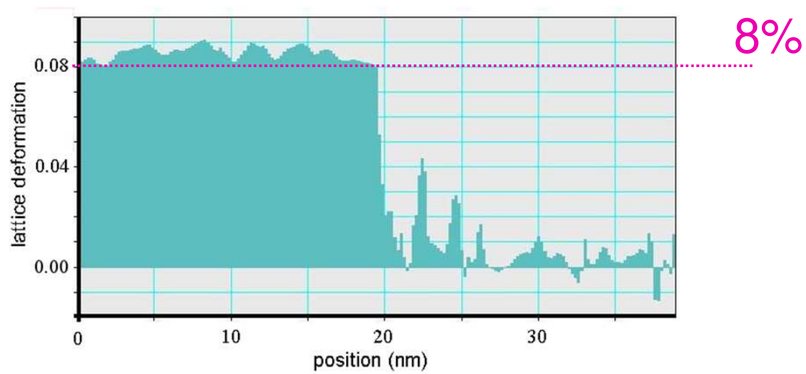
Bright field STEM micrograph

- Atomic-plane-thick reconstruction across the interface

Geometrical Phase Analysis cartography

Local analysis of lattice parameter





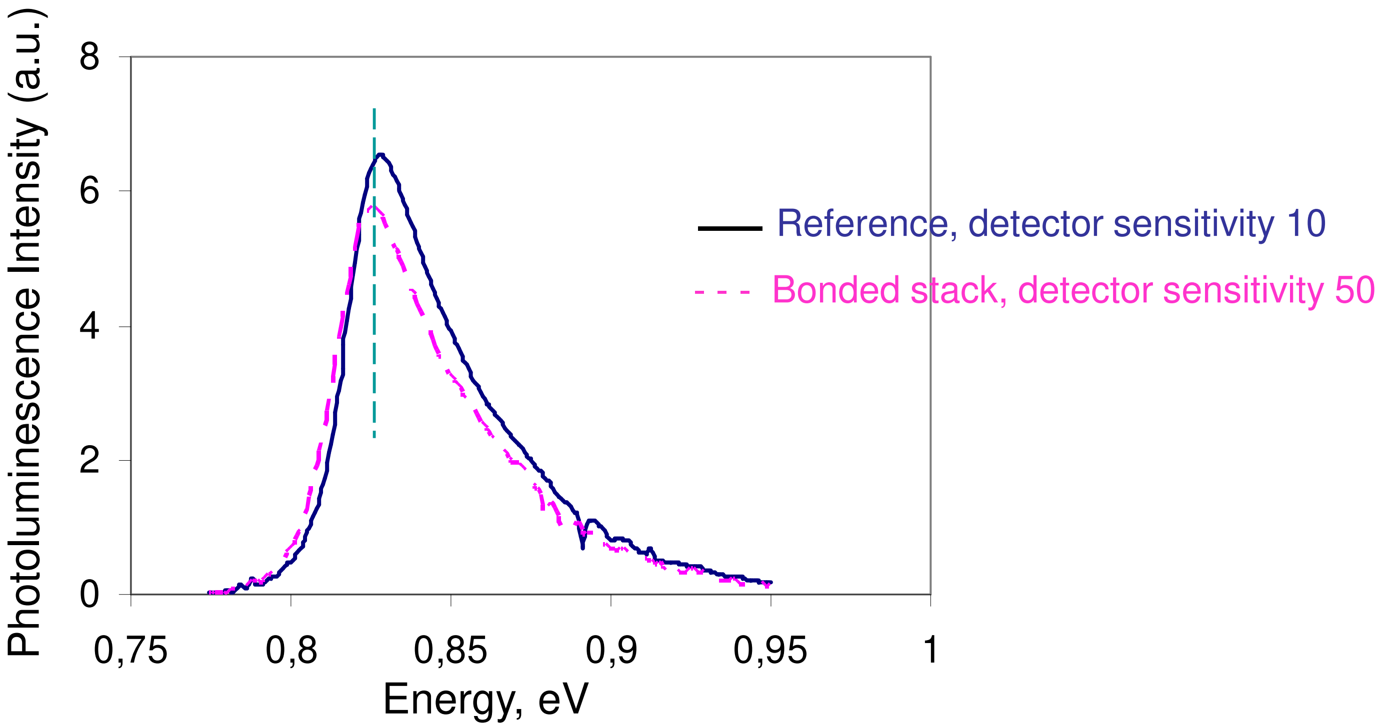
- Abrupt interface transition

Room Temp. Photoluminescence of bonded QW

Bonding operating conditions

Oxide-free : vacuum
T=550°C , 90mn
Low Pressure : 0.2MPa
High pressure uniformity

InP top layer	71 nm
5 InGaAsP barriers 4 InGaAs QW	16 nm 10 nm
InP	71 nm



PL shift < 3meV

♦ No strain in the bonded QW

*A.Talneau et al., Appl. Phys. Lett., **102,** 212101 (2013)*

Oxide-mediated bonding mechanism of III-V materials on Si

State of the art

Both surfaces are cleaned and desoxidized

♦ **3 steps process**

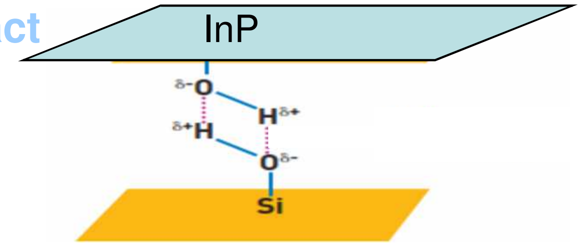
1st : Oxide layer Elaboration

Several techniques are used:
- SiO_2 PECVD
- SiO_2 Sputtering
- O_2 plasma CCP- RIE
- O_2 plasma ICP- RIE
- Thermal oxidation, for the Si surface

2nd : Oxide layer Activation

- O_2 plasma RIE-CCP
- O_3 *A.Itawi et al., JVSTB, **21**, 3784 (2013)*

3rd : Contact



and Thermal annealing

$$InP\text{-}OH + HO\text{-}Si \rightarrow InP\text{-}O\text{-}Si + H_2O_{(g)}$$
$$Si + xH_2O \rightarrow SiO_2 + xH_{2(g)}$$

SiO_2 PE-ALD layer

+ The thinnest layer ~ 1 MonoLayer

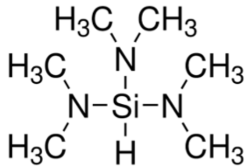
+ Conformal layer when structured surfaces are concerned

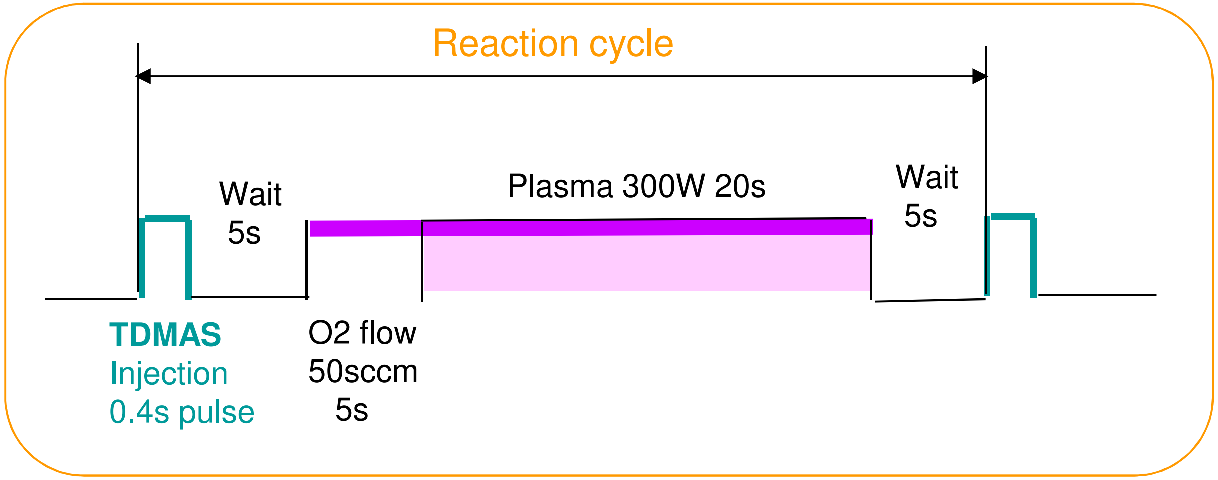
➡ Oxide Deposition **and** Surface Activation in a **single processing step**

SiO_2 Plasma Enhanced ALD cycle

<u>Equipment</u>: Fiji 200
Ultratech–Annealsys

<u>Operating conditions</u>
Precursor : TDMAS
Oxidant: O2 Gaz
Plasma Assisted
T=250°C





SiO_2 ALD layers

@ 0.6nm/cycle

Fabrication
2 cycles
4 cycles
30 cycles

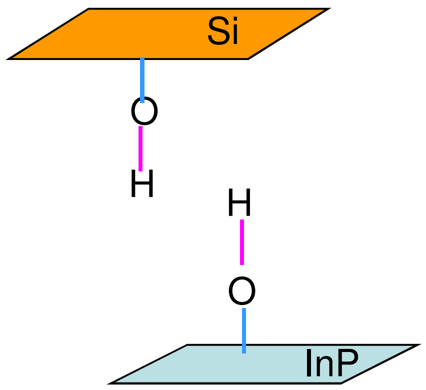
Ozone activation of both InP and Si surfaces

- De-oxidized surfaces,   HF last

- No oxide deposition

- O_3 activation of both surfaces

  → Hydroxyl groups on both surfaces



Condensation under annealing $InP\text{-}OH + HO\text{-}Si \rightarrow InP\text{-}O\text{-}Si + H_2O_{(g)}$

Should provide → Single oxygen atomic plane at the interface

Equipment: UV cleaner, Jelight
Operating conditions
- High intensity low pressure mercury vapor UV grid lamp (λ=184.9nm & 253.7nm)
- Air atmosphere, no Oxygen flow
- 30s

Surface characterization by X-ray Photoelectron Spectroscopy

→ Measure the Si–O links

Equipment : PHI 5000 VersaProbe
Scanning XPS Microprobe

Operating conditions
Source X : Al K_α monochromatic
($h\nu$=1486.6 eV), 15kV, 39.3W
Beam diameter: 200 µm
Emergence angle: 45°
Mean free path: ~ 4nm

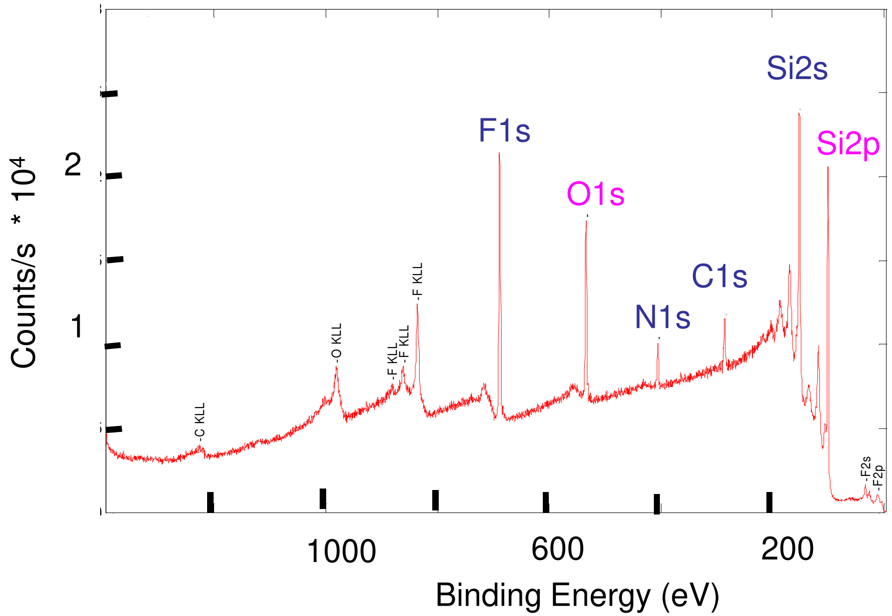
General spectra
Pass Energy: 187.85 eV
Step: 0.4 eV

Measured samples

- Si HF last, hybrophobe } references
- SiO_2 PECVD 5nm thick } references

- 4 cycles SiO2- PE-ALD layer
- O2 plasma RIE low bias 50V on Si
- O2 plasma ICP no bias on Si
- O3-activated Si surface

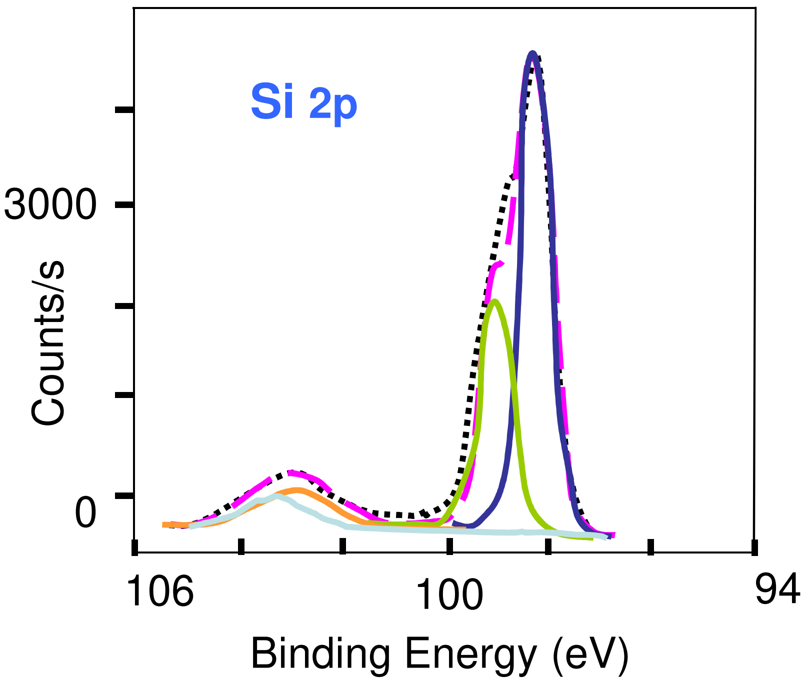
General spectrum

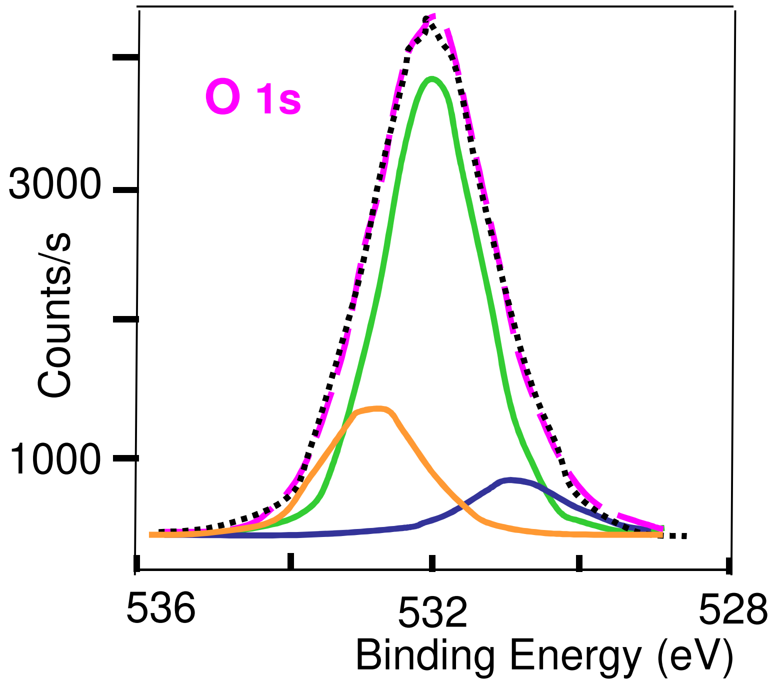


O3 activation   XPS surface characterization : Detailed spectra

Pass energy 23.5 eV
Step : 0.1 eV

Ozone-activated Si surface





- **99.24 & 99.89 eV   @ Si substrate**
- **103.07 & 103.72 eV @ SiO_2 and SiOH**

- **531.38 eV @ SiOH**
- **532.47 eV @ SiO_2**
- **533.21 eV @ H_2O**

Each fit gives the atomic proportion of the related chemical bond to the measured intensity

O3 activation XPS surface characterization : chemical bonds

Si-O $\frac{[SiOH]+[SiO2]}{[Si\ OH] + [SiO2]} = \frac{[O1s]}{[Si\ 2p]}$ SiO_2 only $\frac{[O1s]}{[Si\ 2p]} = 2$ SiOH only $\frac{[O1s]}{[Si\ 2p]} = 4$

$\frac{[SiO2]+[SiOH]}{[Si\ substrate]}$ is the signature of the thickness of the SiO2 layer

Mean free path: ~ 4nm

O2 Plasma-based

Surface Preparation	SiO2 sput.	SiO2 PE-ALD 4 cycles	Si RIE CCP 50V	Si RIE ICP no bias	Si O3
$\frac{[O1s]}{[Si\ 2p]}$	2.23	2.46	2.48	2.68	2.78
$\frac{[SiO2]+[SiOH]}{[Si\ substrate]}$	25.9	0.48	2.22	0.61	0.2

Si-O chemical bonds ← $\frac{[O1s]}{[Si\ 2p]}$

Signature of the thickness of the SiO_2 layer ← $\frac{[SiO2]+[SiOH]}{[Si\ substrate]}$

PE-ALD oxide layer bonding : 2.9nm oxide interface

<u>Annealing conditions</u>

- Vacuum
- T=300°C , 3 h
- Low Pressure : 0.2MPa
- High pressure uniformity

InP-OH + HO-Si → **InP-O-Si** + $H_2O_{(g)}$
Si + xH_2O → SiO_2 + $xH_{2(g)}$

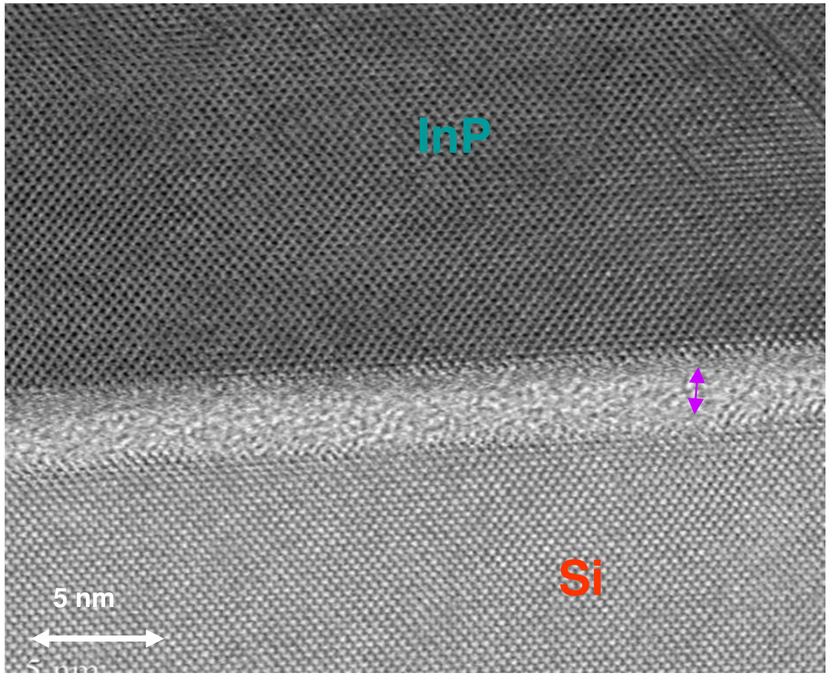
400nm thick InP membrane bonded on Si

after selective chemical removal of the substrate and the etch stop layer

4 cycles ALD layer

Hybrid interface TEM charcaterization

Atomic-resolution HAADF Bright Field STEM image of the hybrid interface between InP and Si



Oxide layer thickness

~2.9nm

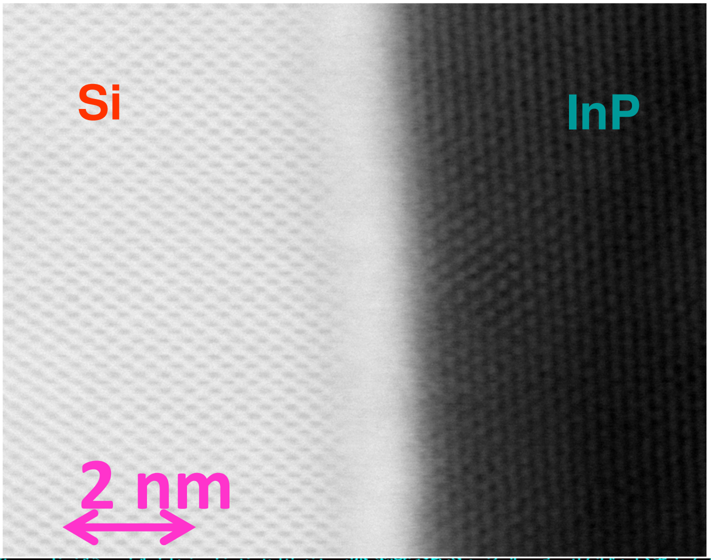
A.Talneau et al., Microelectronic Engineering, (2016), pp. 40-44, DOI: 10.1016/j.mee.2016.05.001

Structural and Chemical analysis of the Ozone Bonded Interface : 1.2 nm-thick oxide layer

Annealing conditions
- Vacuum
- **T=300°C** , 3 h
- Low Pressure : 0.2MPa
- High pressure uniformity

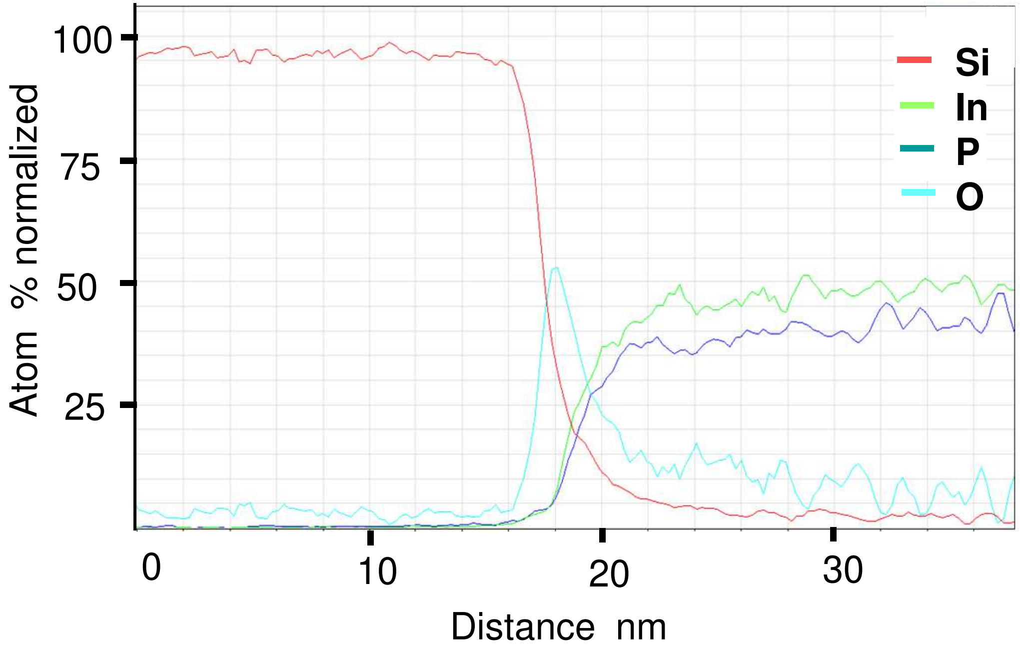
400nm thick InP membrane bonded on Si

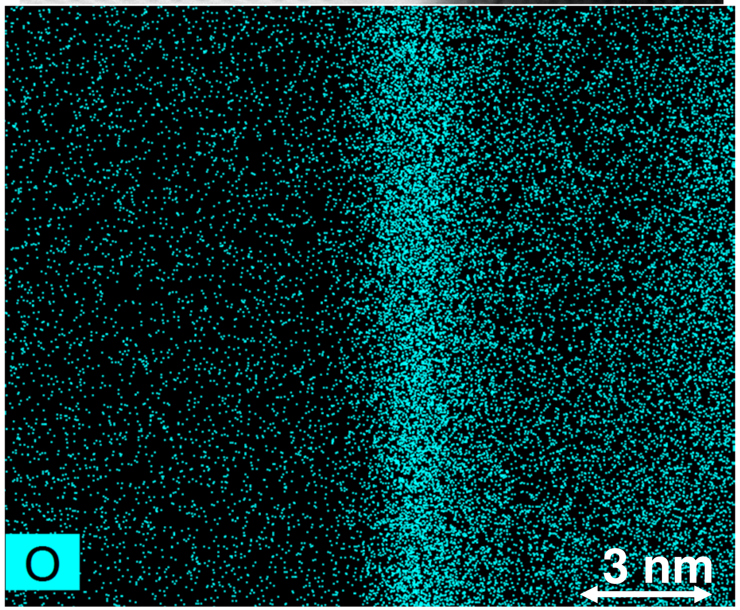
Bright field STEM of the interface



EDX measurement

Concentration of Si – In – P - O element all along the interface



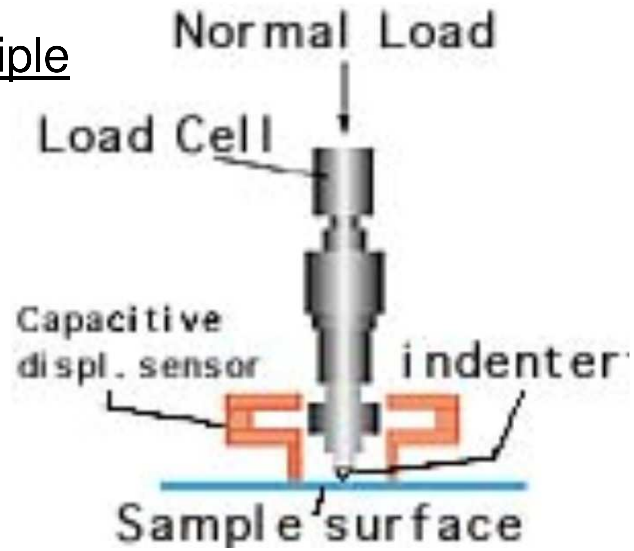


Cartography of O distribution at the interface

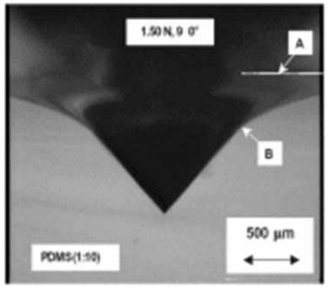
A.Talneau et al., Microelectronic Engineering, (2018), https://doi.org/10.1016/j.mee.2018.02.007

Bonding energy measurement by instrumented Nanoindentation

<u>Principle</u>



Berkovitch tip



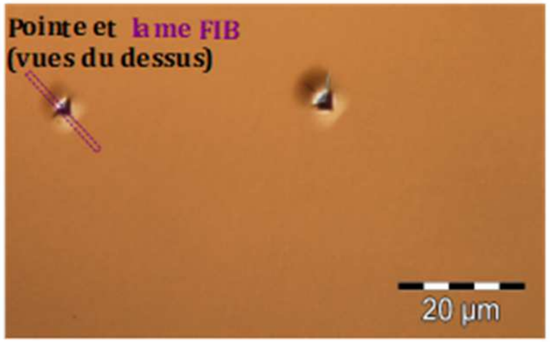
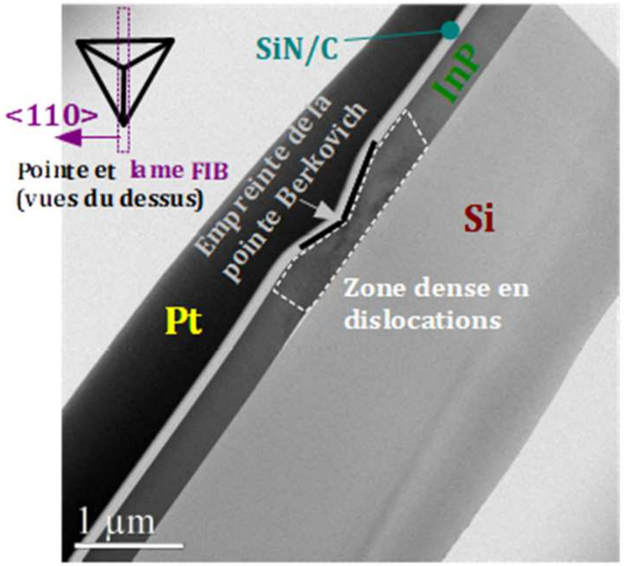


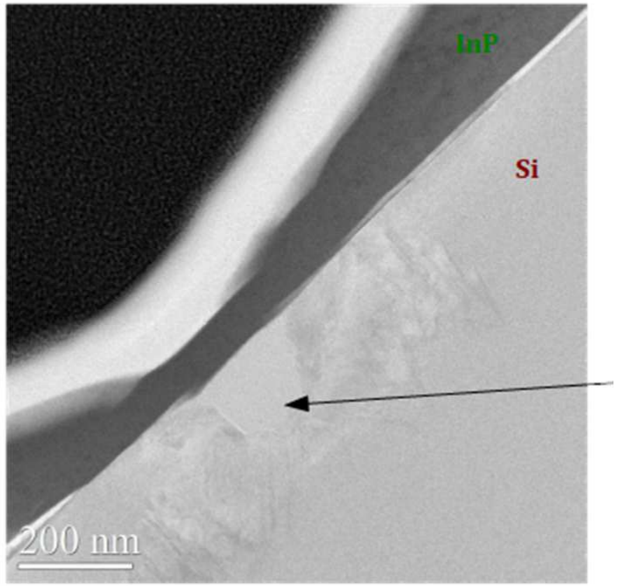
Fig. 5a : Image Nomarsky de cloques sur la membrane InP après indentation à 10 et 20 mN

Collaboration with
Eric LeBourhis, Institut P' Poitiers

Indentation à 20mN

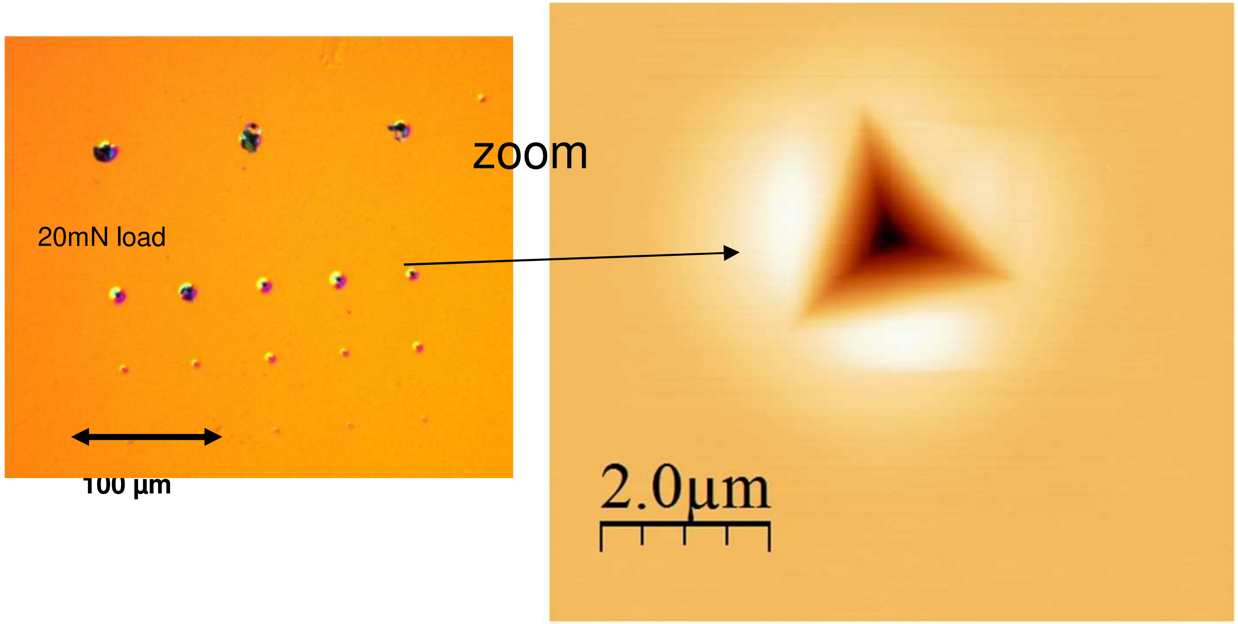


Aux fortes charges, Si est amorphisé sous la pointe



Bonding energy measurement by instrumented Nanoindentation

AFM Image of a blister



The bonding energy $$\gamma = \frac{3E_{InP}t_{InP}^3 t_b^2}{16L^4}$$

L and *tb* are the blister length and height

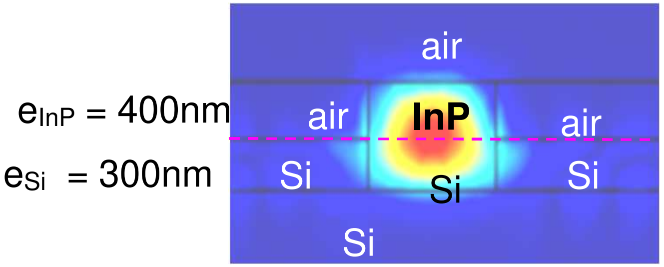
E_{InP} and t_{InP} are the Young modulus and the thickness of the InP membrane

K.Pantzas et al., Appl. Phys. Lett., **103**, 081901 (2013)

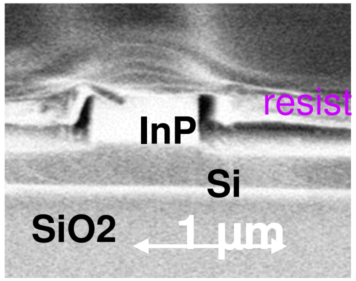
Photonic devices including an hybrid interface

Shallow ridge waveguide

Design cross view



TE mode
Cartography of E Intensity
(Lumerical simulation)



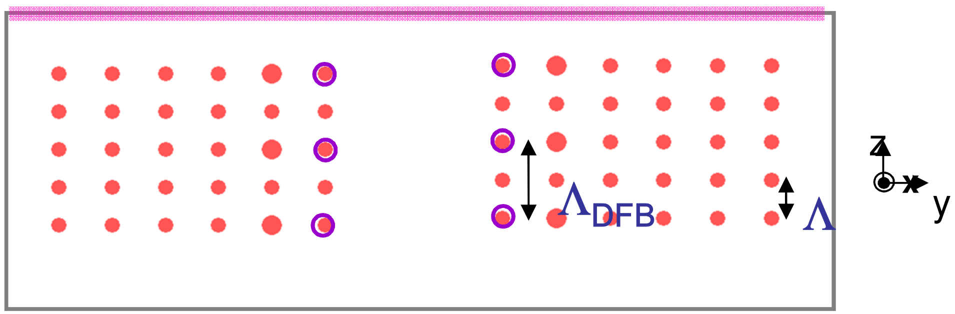
♦ **Technology:**

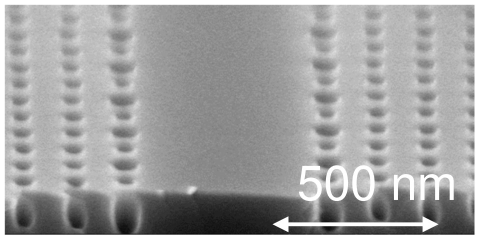
- HSQ e-beam lithography
- Cl_2-based ICP etching

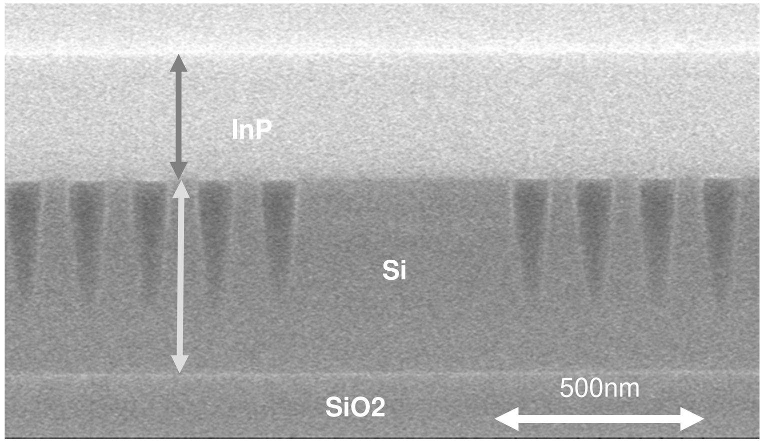
Hybrid cleaved facet

Sub-λ metamaterial + super periodicity waveguide

Design top view



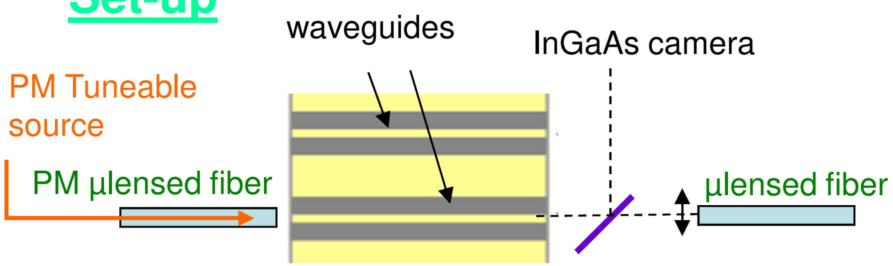




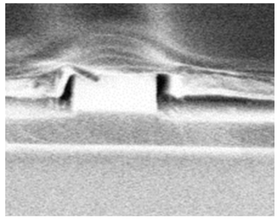
Hybrid cleaved facet

Optical performances of the photonic devices

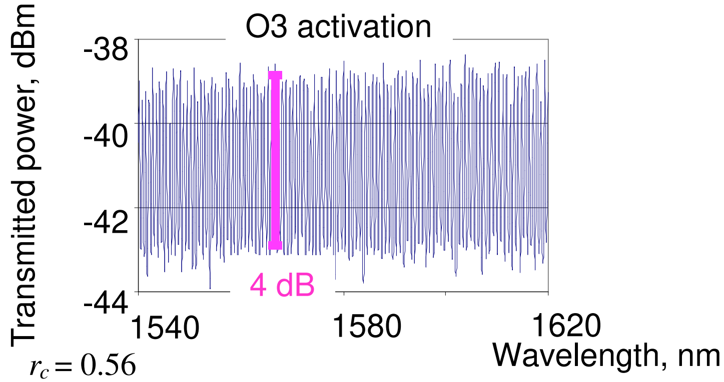
Set-up



♦ Shallow ride waveguide Losses measurement:

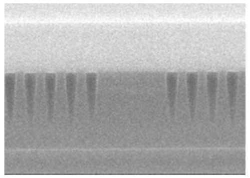


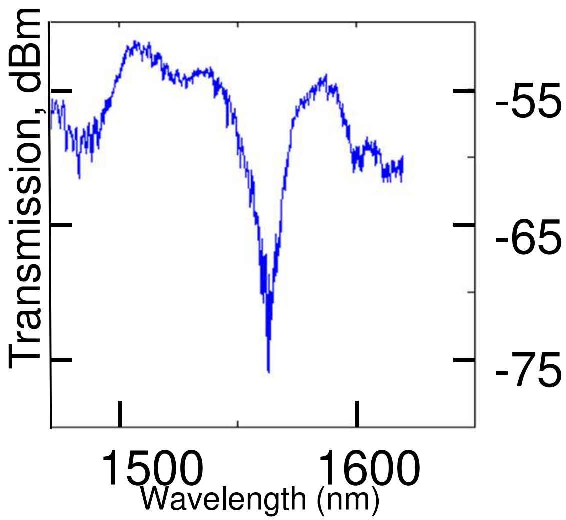
Fabry-Perot resonance method : Propagation losses are calculated from the fringe contrast



➔ **Propagation losses** $\alpha = 5.0\ cm^{-1}$

♦ Wavelength selective waveguide

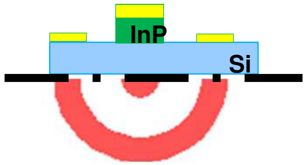




*A.Talneau et al., Optics Letters, **40**, 5148 (2015)*

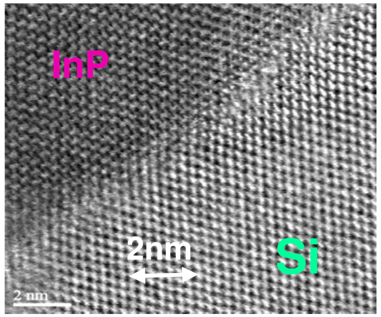
Electrical transport through the interface

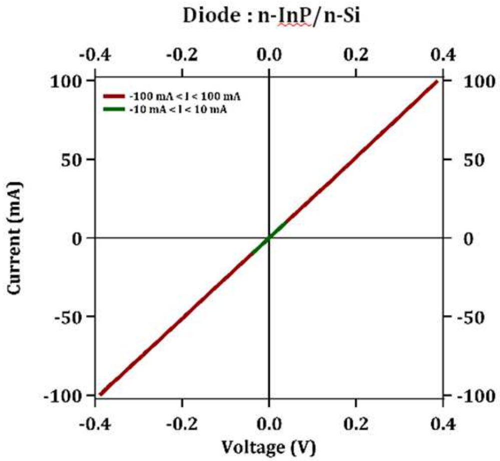
Contact geometry



n-doped (S 10^{19}) 500nm thick InP membrane
bonded on a n-doped 10^{19} Si substrate

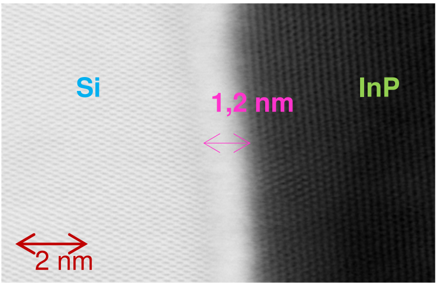
InP/Si oxide free interface

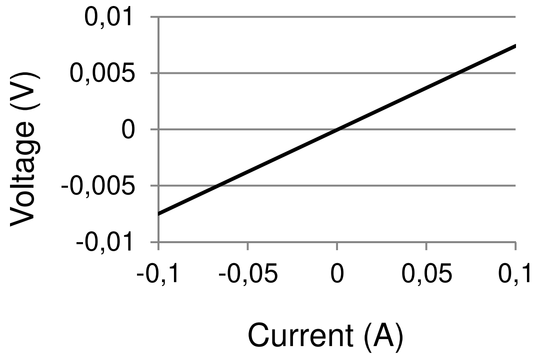




K. Pantzas et al., IPRM 2014

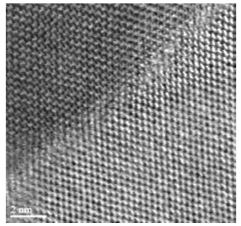
1,2nm oxide InP/Si interface





A.Talneau et al., MNE , 2018

Conclusion



Wafer fusion of III-V on Silicon or SOI for hybrid photonic devices

Bonding processes:

- ♦ Oxide-free @ 500°C
- ♦ Oxide-mediated (SiO_2 1.2nm) @ 300°C

→ High Optical Quality of the InP/Si Interface

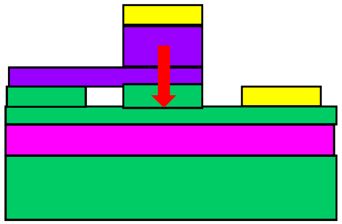
→ Efficient Electrical transport through the interface

Hybrid photonic devices

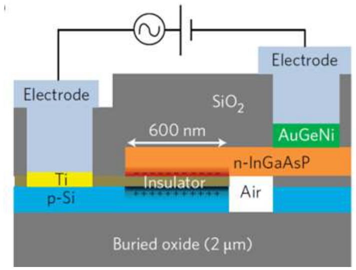
♦ shallow ridge waveguide

♦ Wavelength selective waveguide

➡ Hybrid III-V /Si laser polarized through the interface



➡ Hybrid III-V /Si MOS capacitor Mach-Zehnder modulator



*T. Hiraki et al., Nature Photonics, **11**,482 (2017)*