

# INSCOOP

Intégration de Nanofils III-V sur SOI pour COnnexions Optiques sur Puce

Réunion T0+18, 9 Avril 2013  
LTM/CEA Grenoble

Tâche 2 : Nanowire nucleation and orientation  
*JB. Barakat-INL, M. Gendry-INL*



Arrêt des réacteurs: juin 2012

Remise en marche des réacteurs: décembre 2012- janvier 2013

Arrêt: février 2013

- T2.1 : Vertically standing InP NWs on Si(001)



Promote vertically standing NWs on Si (001) orientation using a thin SrTiO<sub>3</sub> oxide template

- T2.2: Alternative to Au-catalyst



Avoid Au contamination in silicon process lines using alternative catalyst or self-catalysed growth

- T2.3: Selective growth



Avoid the formation of a 2D/3D layer between the NWs by appropriate growth conditions and substrate masking

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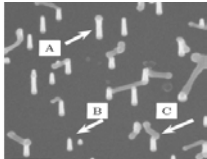
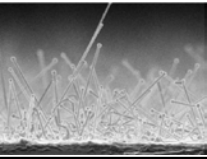
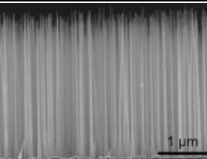
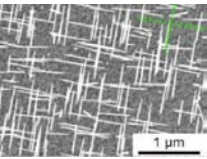
Avoid Au contamination in silicon process lines using alternative catalyst or self-catalysed growth ... **self catalysis with In droplets**

- T2.3: Selective growth

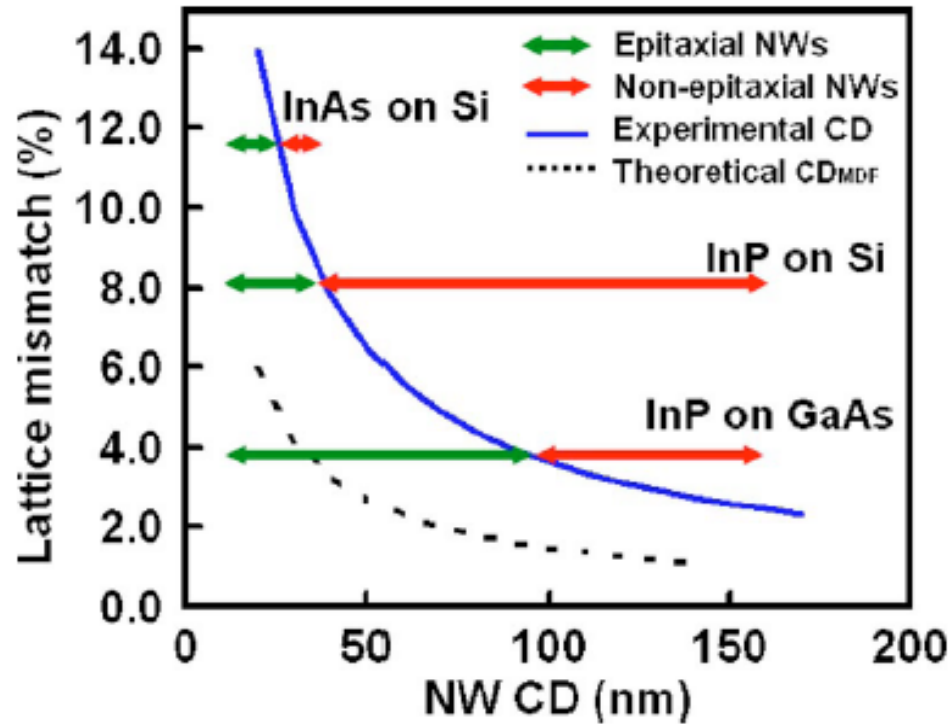


Avoid the formation of a 2D/3D layer between the NWs by appropriate growth conditions and substrate masking

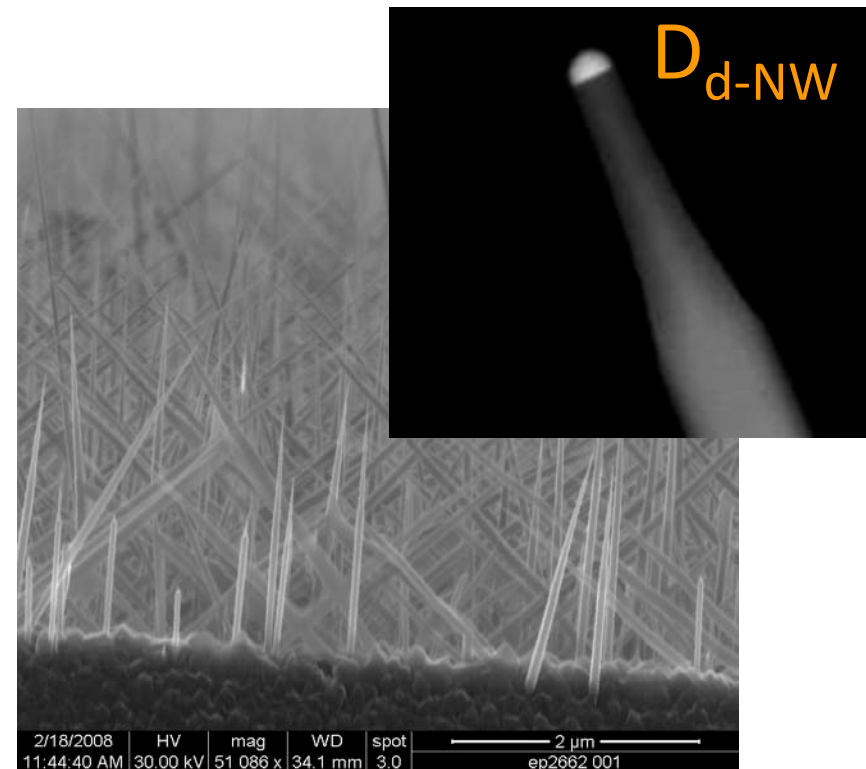
# State of the art on self-catalyzed InP NWs

Author	Technique	Substrate	Chemical Treatment	Orientation	Structure
<i>Mattila</i>	MOVPE - VLS	InP(111)	No (native oxide)		ZB
		InP(100)	No (native oxide)	No growth	∅
		Si(111)	No (native oxide)		WZ
<i>Gao</i>	MOVPE - VLS	Si(111)	Yes (oxide free)		ZB
		Si(001)	Yes (oxide free)		ZB
<i>Ivanov</i>	MBE-VLS (InAsP)	Si  InP	<i>Nanotechnology</i> , 24, 085707 (2013)		

# Which droplet diameter?



INL results for InP NWs/Si  
with Au-In droplets  
 $D_{d-NW} < 33$  nm



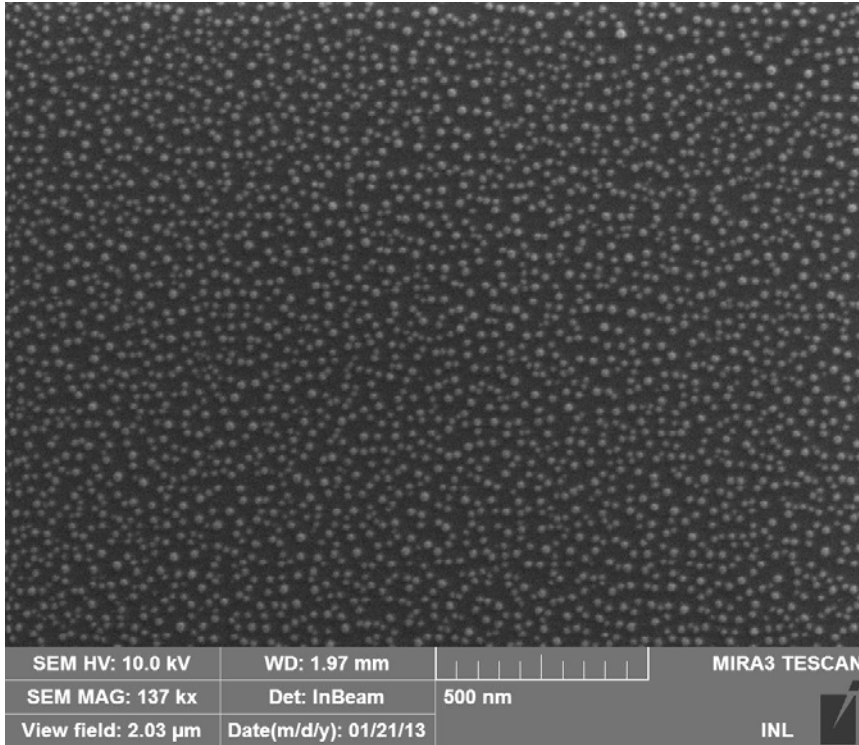
$CD_{NW} < 35$  nm for InP/Si

Chuang et al, Appl. Phys. Lett. 90, 043115 (2007)

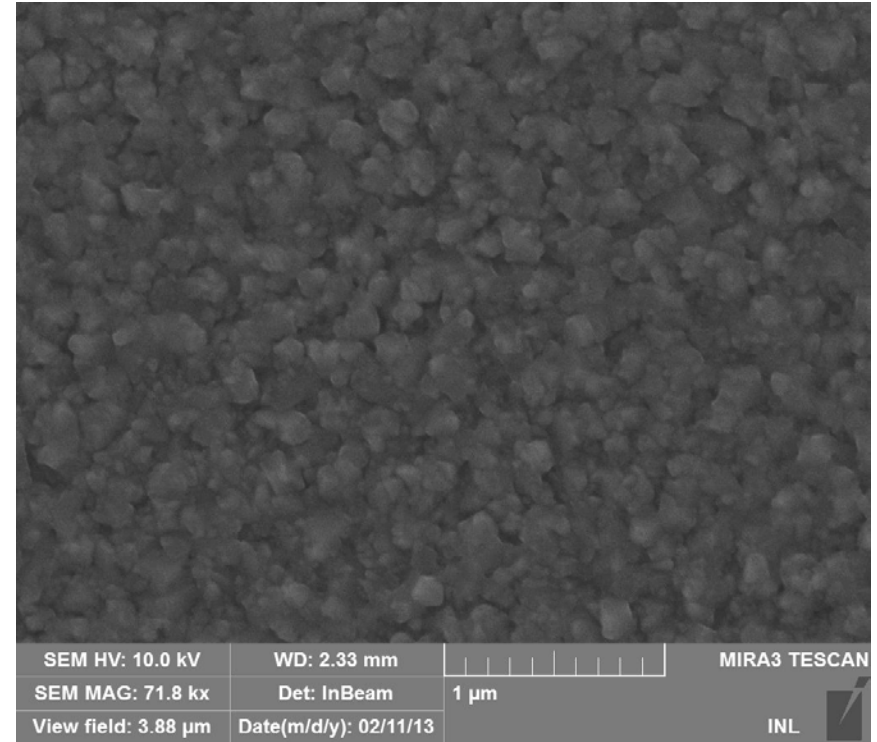
Naji et al, Appl. Phys. Lett. , to be published, 2013

# Aim: In $D_{\text{droplets}} < 30 \text{ nm}$

Formation of In droplets at 380°C



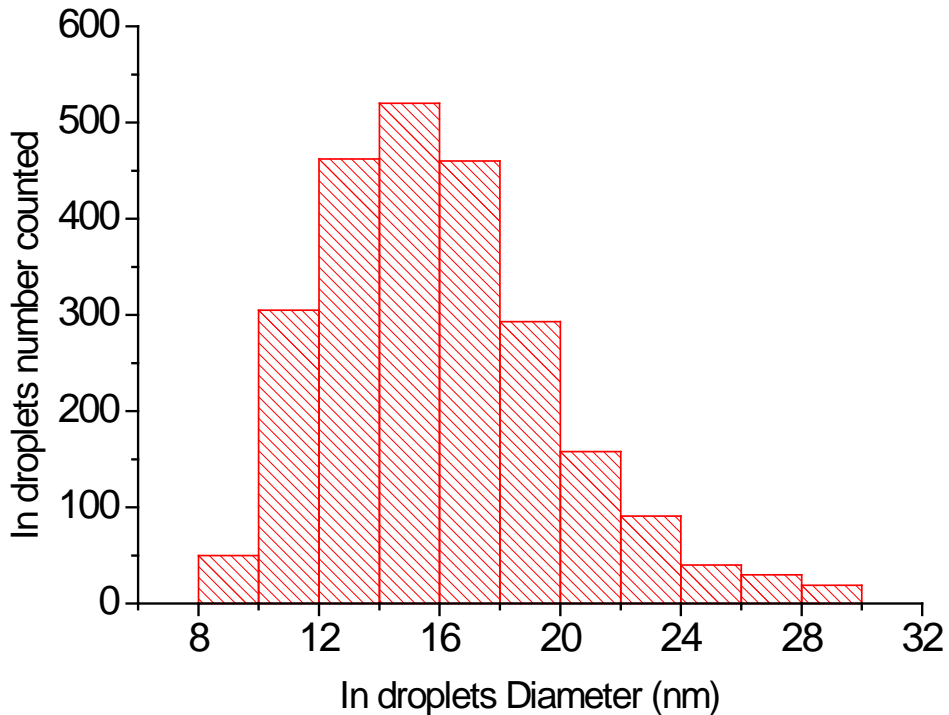
Growth of InP at 380°C



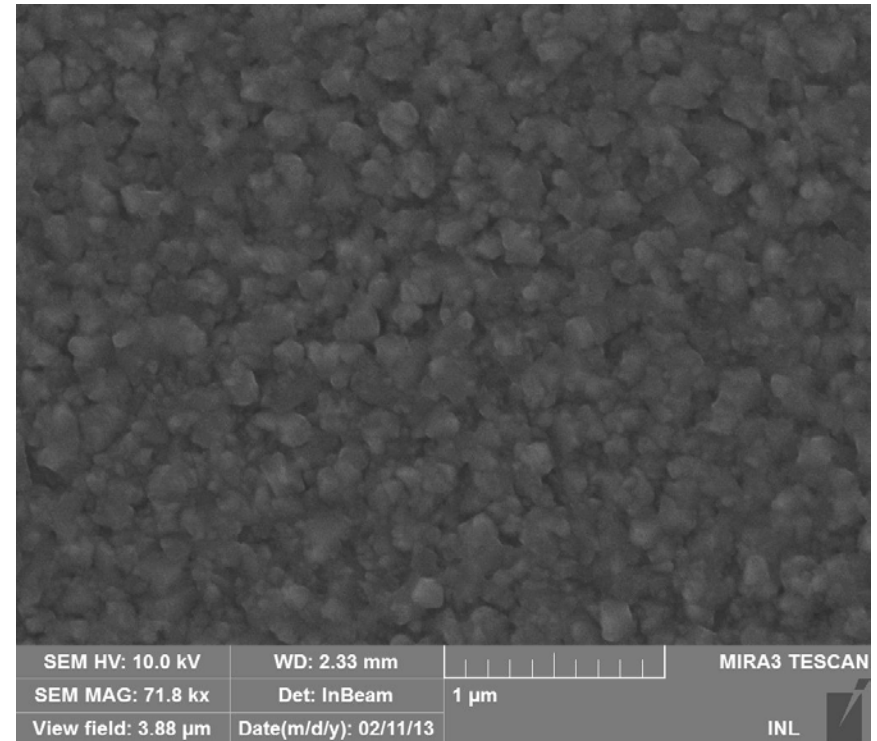
## No NWs Growth

# Aim: In D<sub>droplets</sub> < 30 nm

Formation of In droplets at 380°C



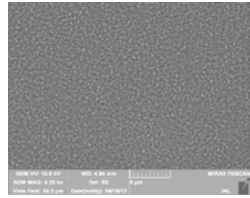
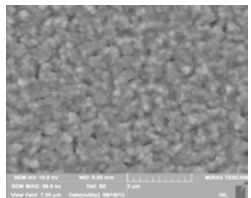
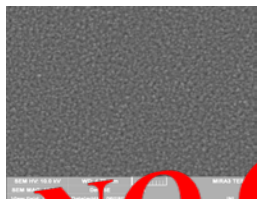
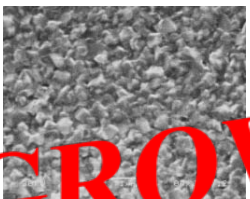
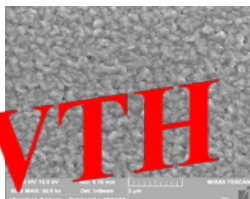
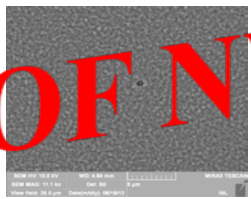
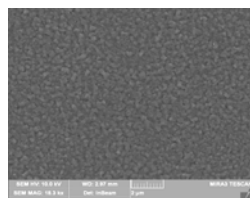
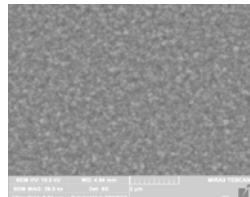
Growth of InP at 380°C



## No NWs Growth

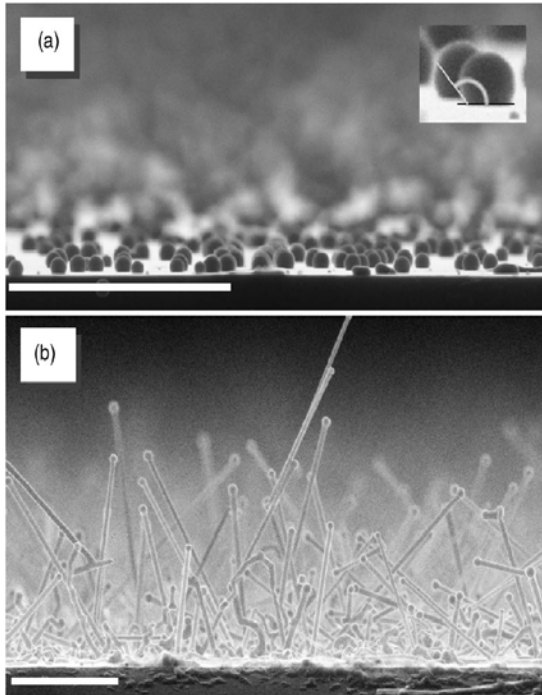


# Growth study with In $D_{\text{droplets}} < 30$ nm

$T_g$ V/III	300°C	340°C	380°C	420°C	460°C
30					
20					
10					
5					

**NO GROWTH OF NWS**

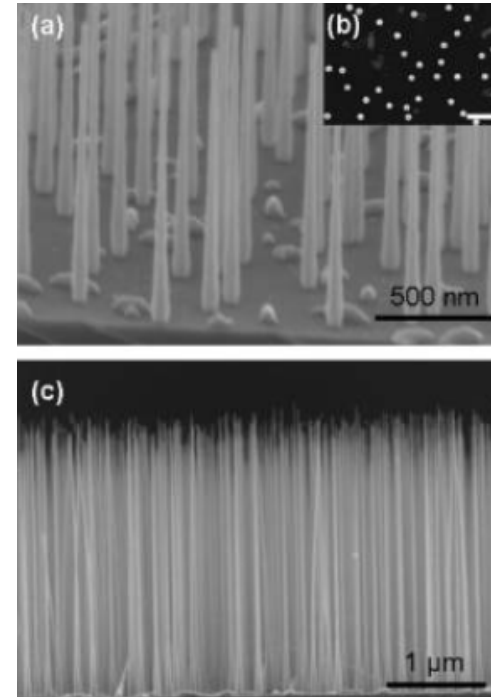
# Reported works: Focus on In droplet diameter



InP NWs on Si(111)

**D = 55-90 nm**

*Mattila et al, APL 89, 063119 (2006)*



InP NWs on Si(111)

**D = 25-50 nm**

*Gao et al, Nano Lett., 9, 2223 (2009)*

# Specific study on In droplet diameter

**What**

Aim to  $D_{\text{limit}} \geq 25 \text{ nm}$

**How**

$$D = f(v, N_{\text{ML}}, T)$$

In deposition  
rate (ML/s)

$N_{\text{ML}}$   
deposited In

Substrate  
temperature

**Which**

Substrates

Si(111)

SiO<sub>2</sub>  
Si(111)

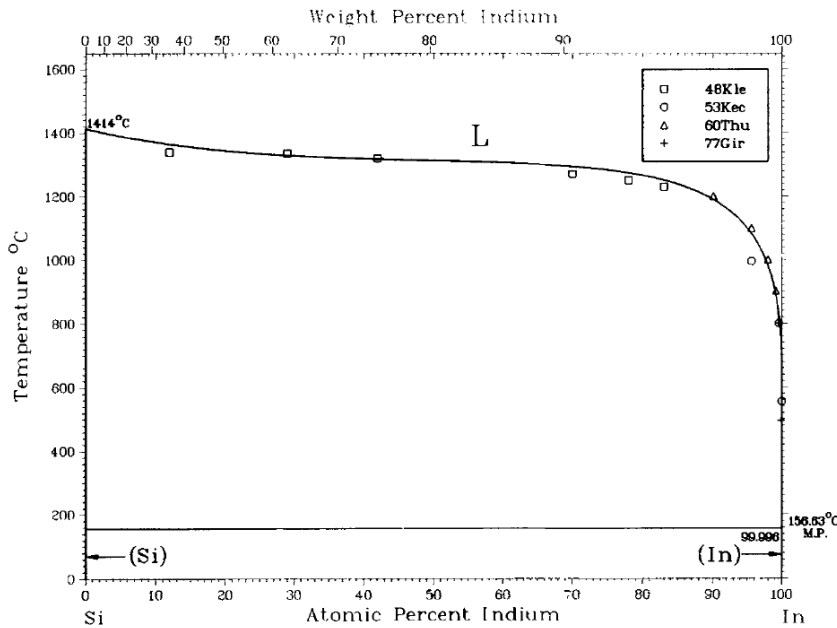
Si(001)

SiO<sub>2</sub>  
Si(001)

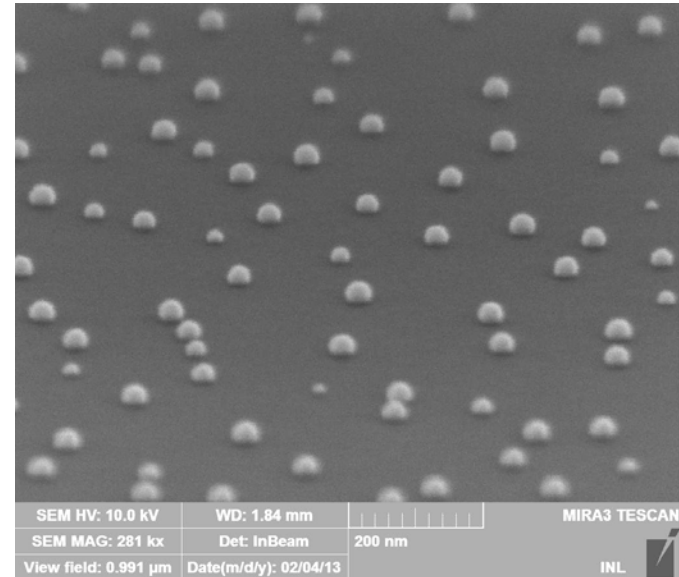
# Self-catalysis with In droplets

- Au-free
- Indium element of the InP nanowire
- Indium not miscible in Silicon

T=400°C  
0.2 ML/s  
6 In ML



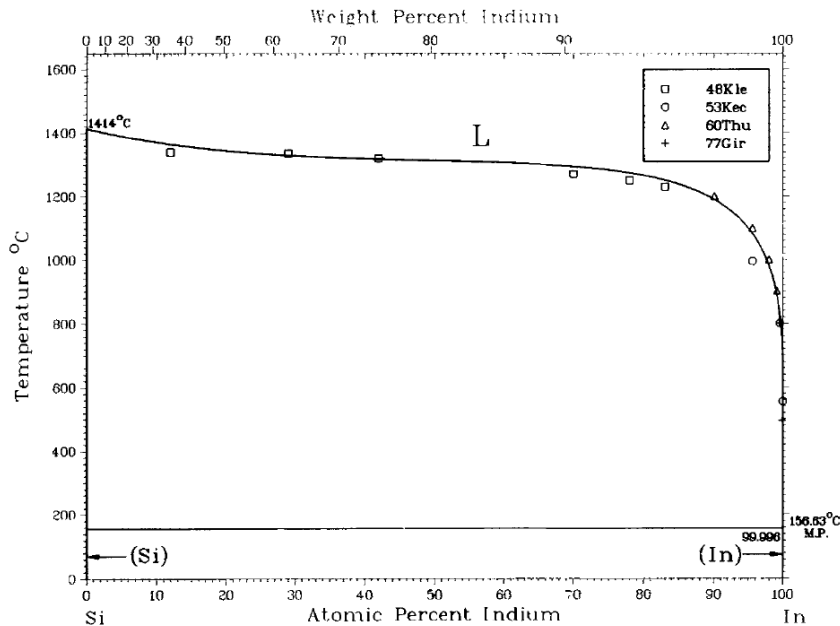
R. W. Olesinski, N. Kanani, and G. J. Abbaschian, 1985.



No In-Si alloy

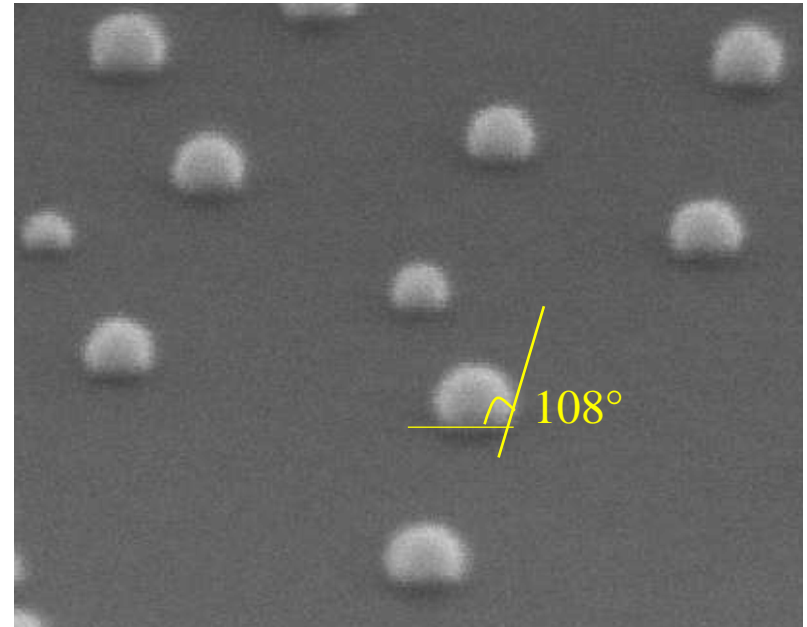
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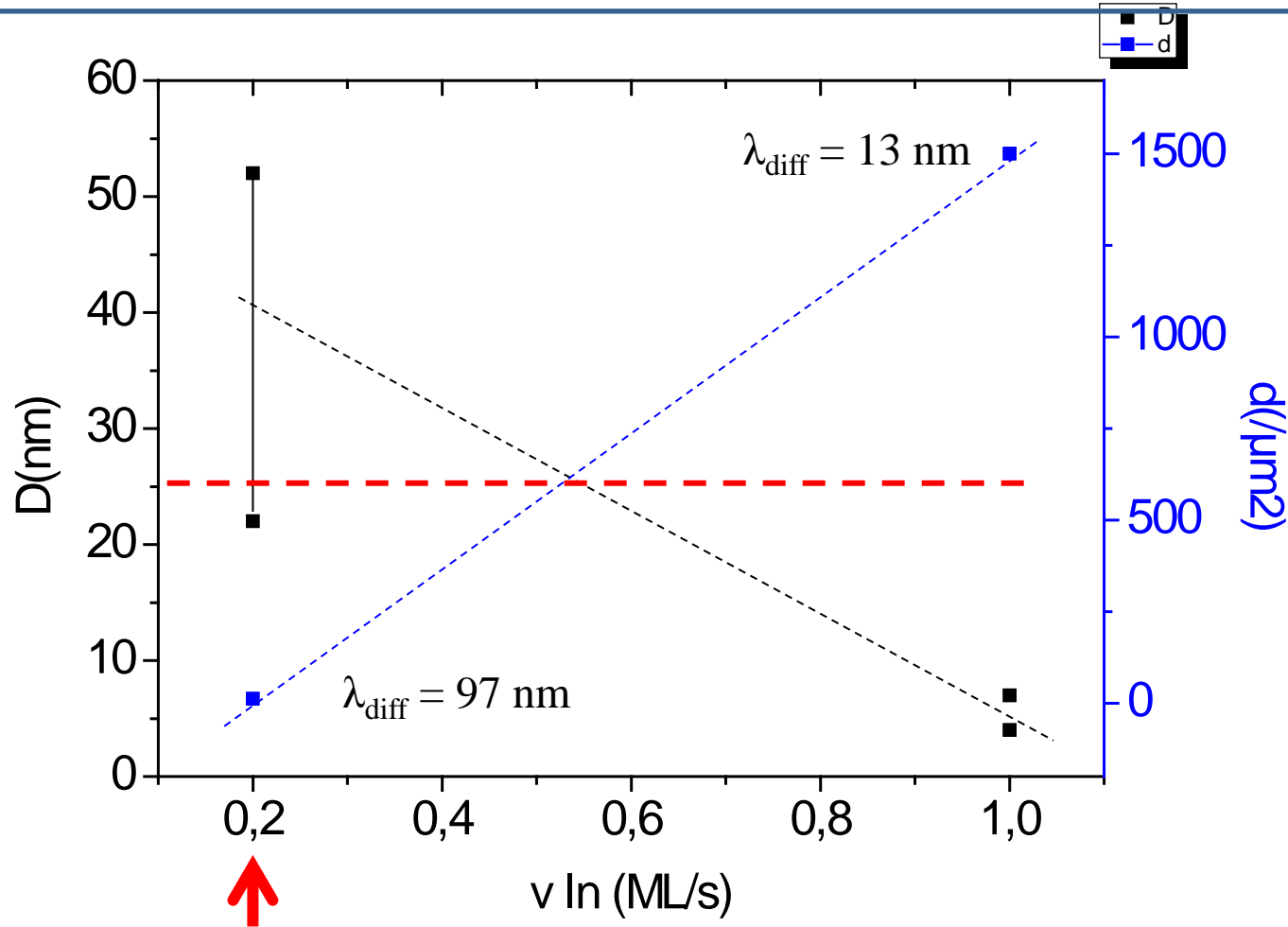
No In-Si alloy



Wetting angle :  $90^\circ < \theta < 130^\circ$

# Influence of In deposition rate $v$ (ML/s)

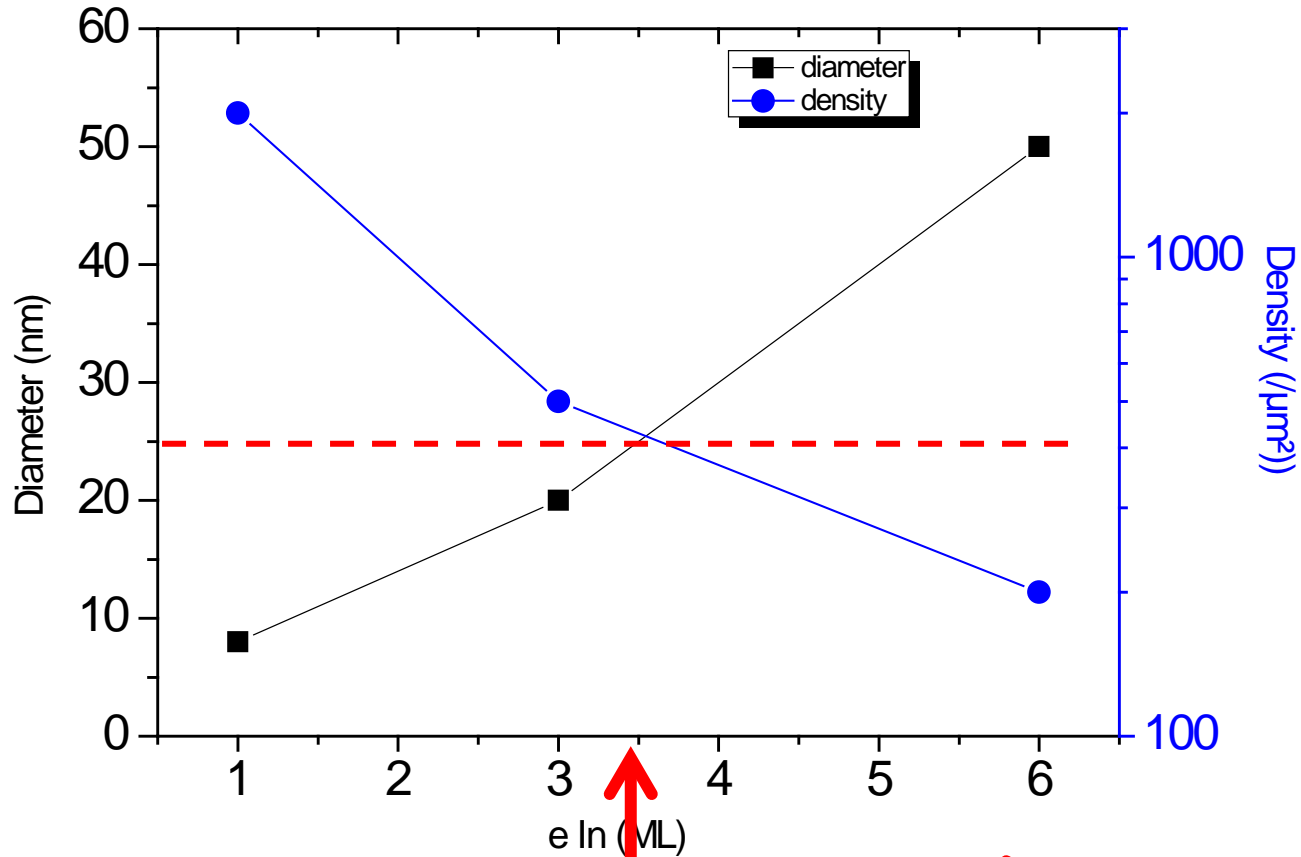
$T = 380^\circ\text{C}$ ,  $N = 6$  ML, on Si(111) oxide free



Low rate 0.2 ML/s

# Influence of $N_{ML}$ deposited In

$v = 0.2 \text{ ML/s}$  ,  $T = 380^\circ\text{C}$  on Si(111) oxide free

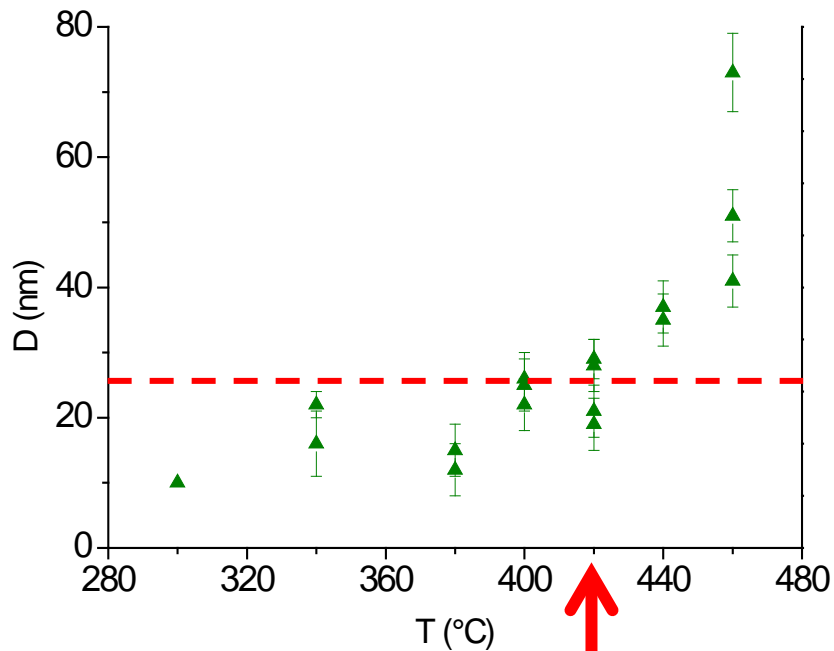


$N_{ML} \geq 4 \text{ ML}$

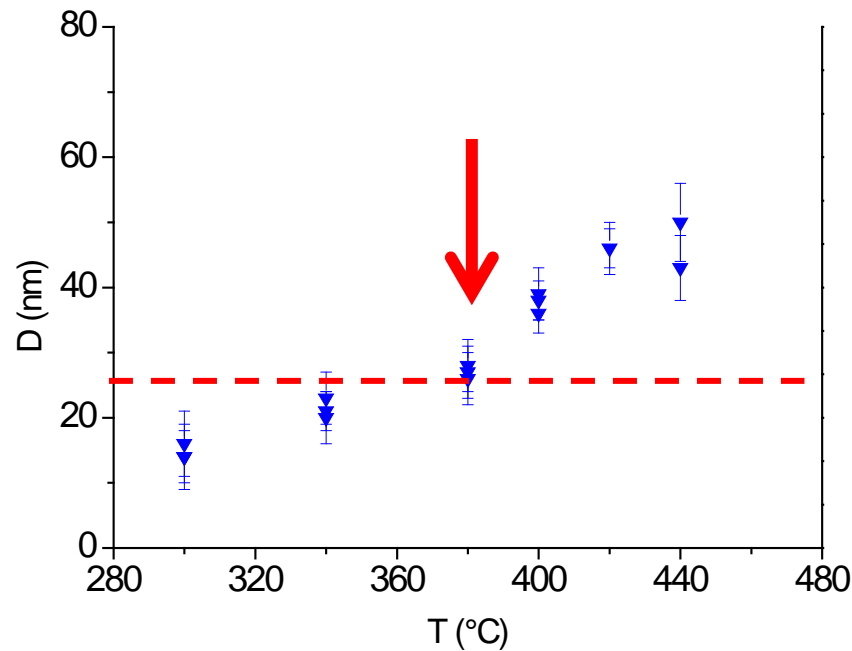
# Influence of substrate temperature $T$ ( $^{\circ}\text{C}$ ) on Si(111)

$$N_{\text{ML}} = 6 \text{ ML}, v = 0.2 \text{ ML/sec}$$

Si(111) oxide free



Si(111) with natural oxide



$T \geq 420^{\circ}\text{C}$  for  $D > 25$  nm

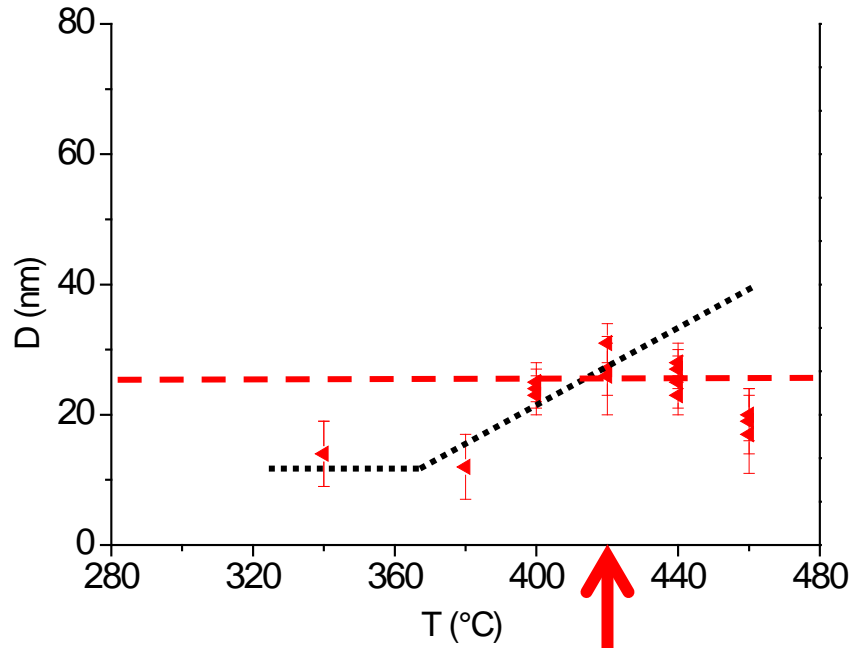
$T \geq 380^{\circ}\text{C}$   
Stage at  $T \geq 420^{\circ}\text{C}$



# Influence of substrate temperature $T$ ( $^{\circ}\text{C}$ ) on Si(001)

$$N_{\text{ML}} = 6 \text{ ML}, v = 0.2 \text{ ML/sec}$$

Si(001) oxide free

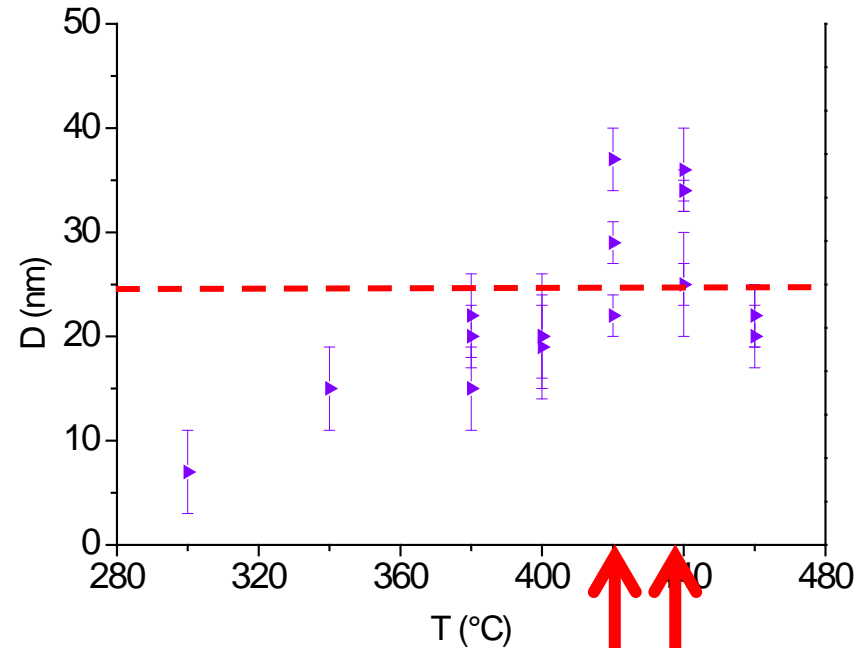


$T = 420^{\circ}\text{C}$

D decrease at  $T > 420^{\circ}\text{C}$

Desorption of In

Si(001) with natural oxide

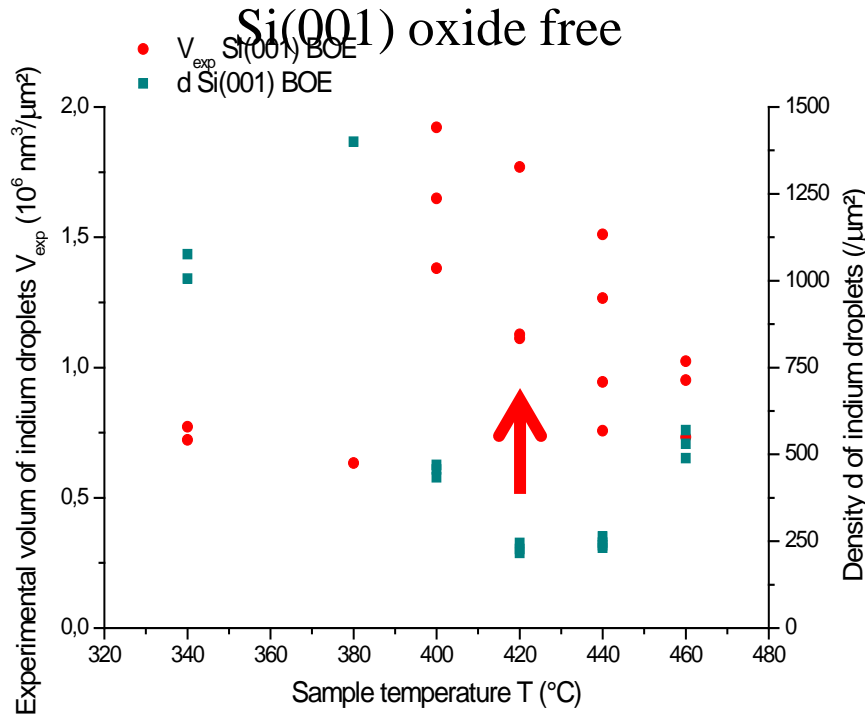


$T \geq 420\text{-}440^{\circ}\text{C}$

Same behaviour at  $T > 440^{\circ}\text{C}$

# Influence of substrate temperature $T$ ( $^{\circ}\text{C}$ ) on Si(001)

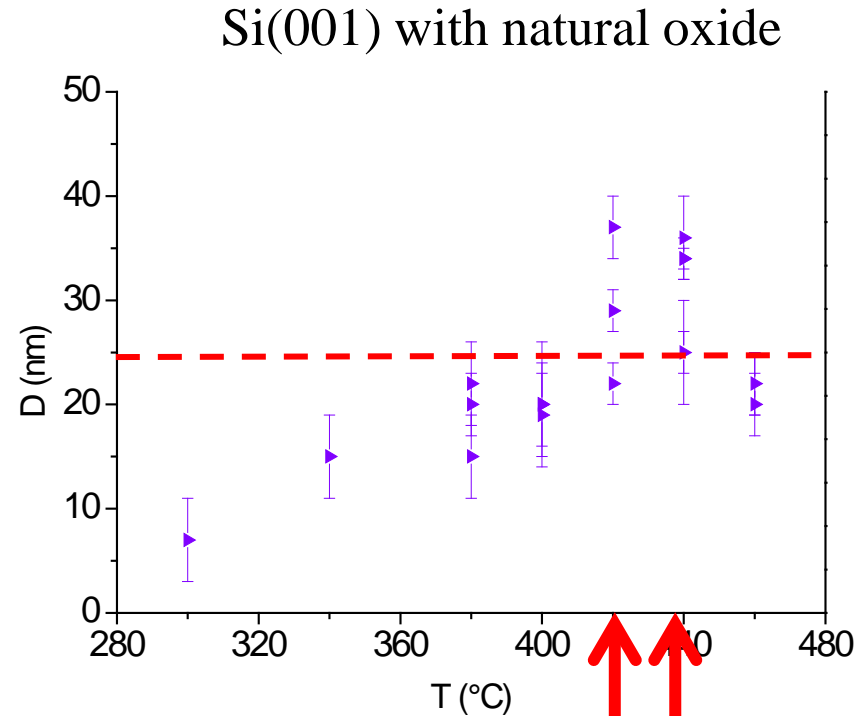
$$N_{\text{ML}} = 6 \text{ ML}, v = 0.2 \text{ ML/sec}$$



$T = 420^{\circ}\text{C}$

D decrease at  $T > 420^{\circ}\text{C}$

Desorption of In



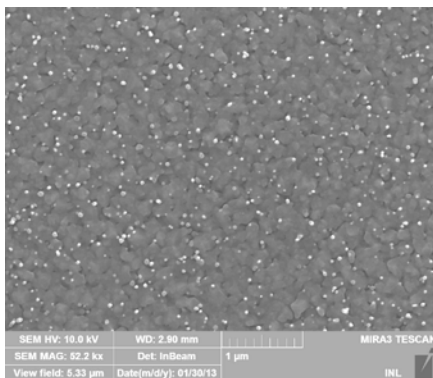
$T \geq 420\text{-}440^{\circ}\text{C}$

Same behaviour at  $T > 440^{\circ}\text{C}$

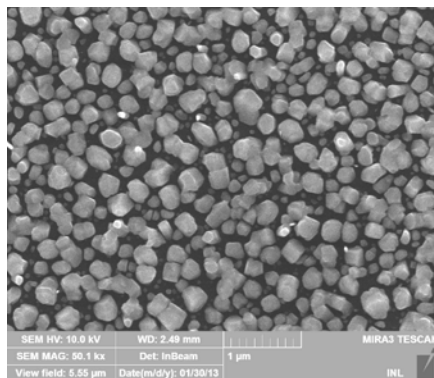
# NWs Growth with $D_{\text{droplets}} \geq 25 \text{ nm}$

$T_g = 380^\circ\text{C}$ ,  $V/\text{III} = 20$ ,  $t_g = 5 \text{ min}$

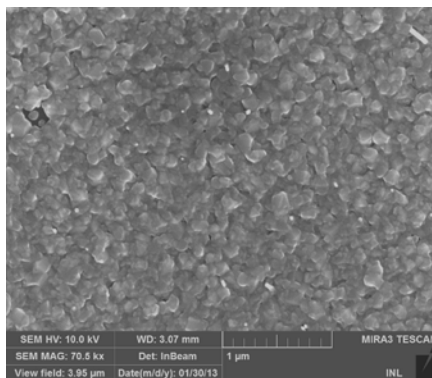
$T_{\text{droplets}} = 400^\circ\text{C}$



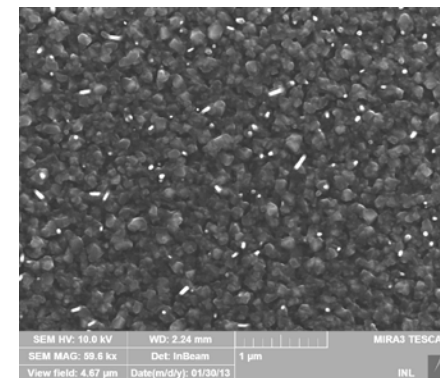
Si(111) oxide free  
 $d \sim 8\text{-}30/\mu\text{m}^2$



Si(111) nat. oxide  
 $d \sim 0.34\text{-}1.6/\mu\text{m}^2$

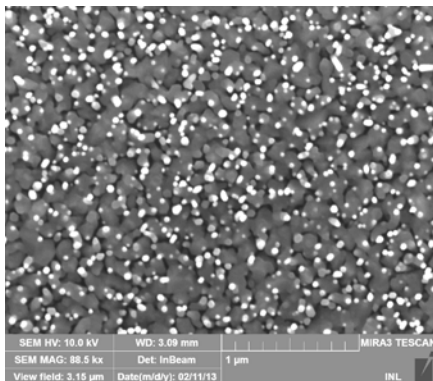


Si(001) oxide free  
 $d \sim 1.9\text{-}6/\mu\text{m}^2$

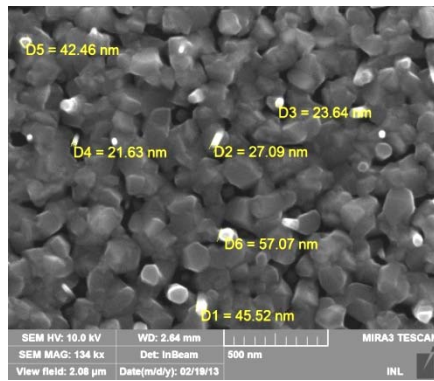


Si(001) nat. oxide  
 $d \sim 1.37\text{-}2.17/\mu\text{m}^2$

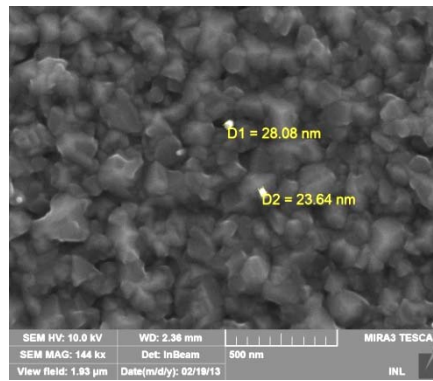
$T_{\text{droplets}} = 420^\circ\text{C}$



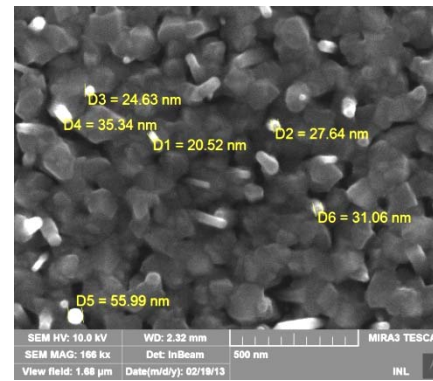
Si(111) oxide free  
 $d \sim 100/\mu\text{m}^2$



Si(111) nat. oxide  
 $d \sim 3\text{-}7/\mu\text{m}^2$



Si(001) oxide free  
 $d \sim 0.73\text{-}1.14/\mu\text{m}^2$



Si(001) nat. oxide  
 $d \sim 9\text{-}11/\mu\text{m}^2$

# InP NWs on Si(111) oxide free at $T_g=380^\circ\text{C}$

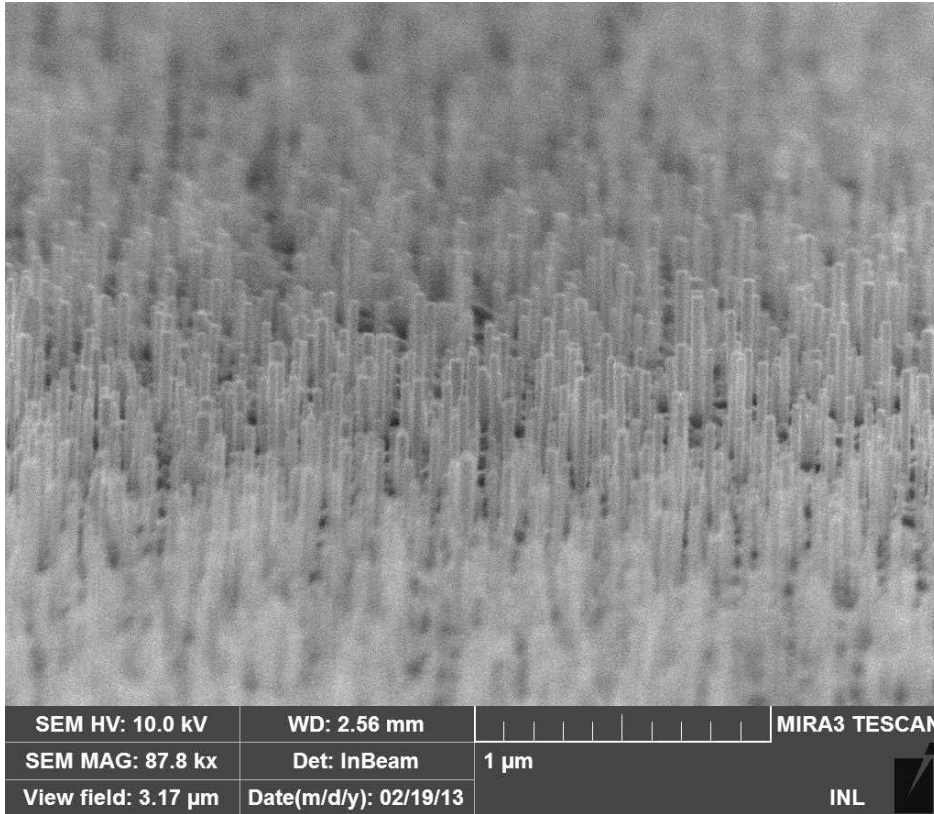
$T_{\text{droplets}} = 420^\circ\text{C}$ ,  $T_g=380^\circ\text{C}$ ,  $V/\text{III}=20$ ,  $t_g = 5 \text{ min}$

$d_{\text{NWs}} \sim 100 / \mu\text{m}^2$

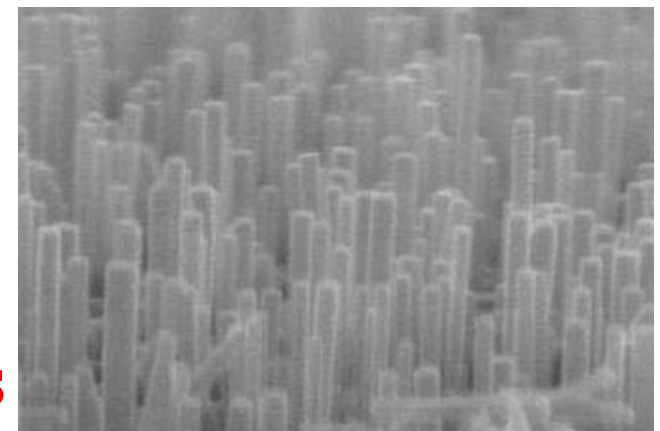
Very uniform in D

$L < 300 \text{ nm}$

$dL/dt = 50 \text{ nm/min}$



**No droplets at the tip**

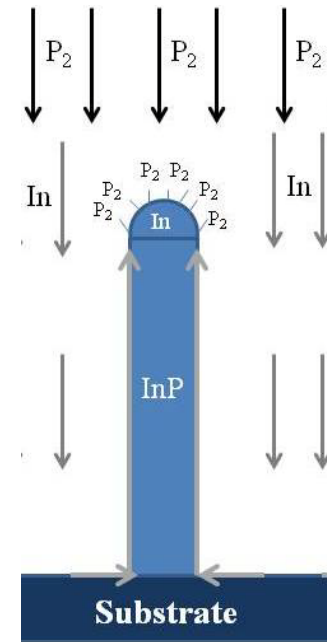
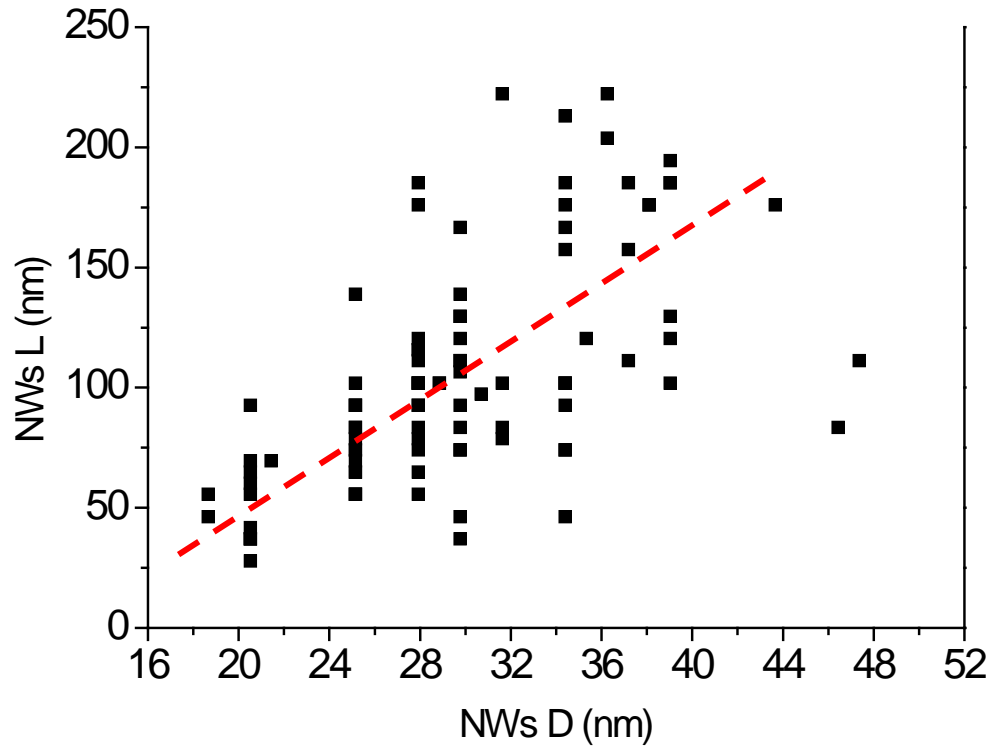


**Consumption of the In droplets  
Due to cooling under phosphorus**

# InP NWs on Si(111) oxide free at $T_g=380^\circ\text{C}$

$T_{\text{droplets}} = 420^\circ\text{C}$

$T_g=380^\circ\text{C}$ ,  $V/\text{III}=20$ ,  $t_g = 5 \text{ min}$



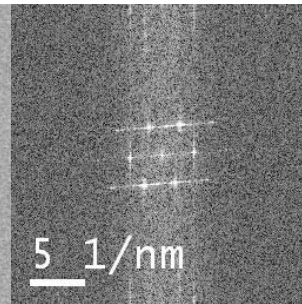
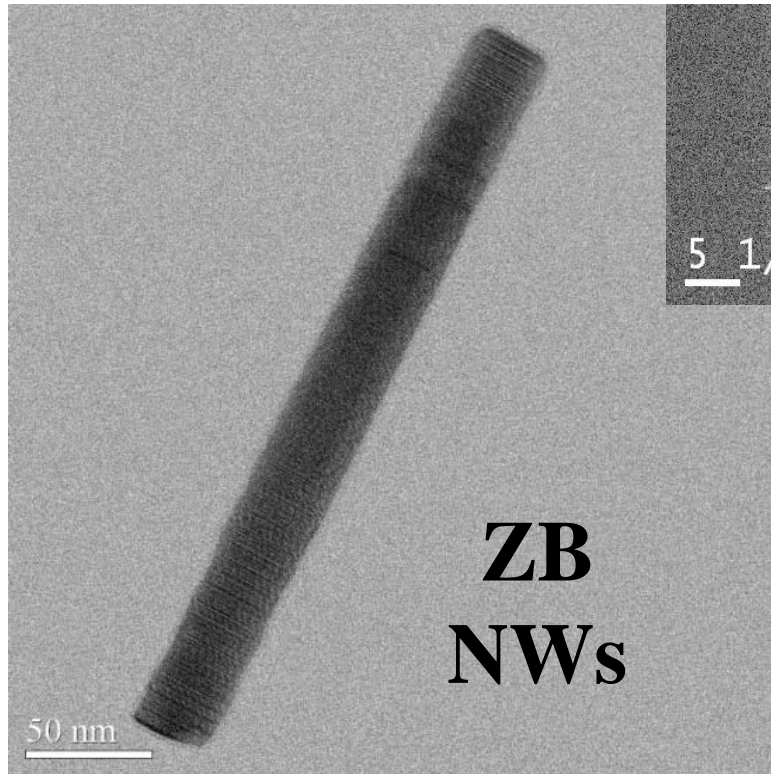
$L_{\text{NW}} \uparrow$       $D_{\text{NW}} \uparrow$

NW growth non-induced by surface diffusion as for VLS-CVD

# Structural properties of InP/Si(111) NWs at $T_g = 380^\circ\text{C}$

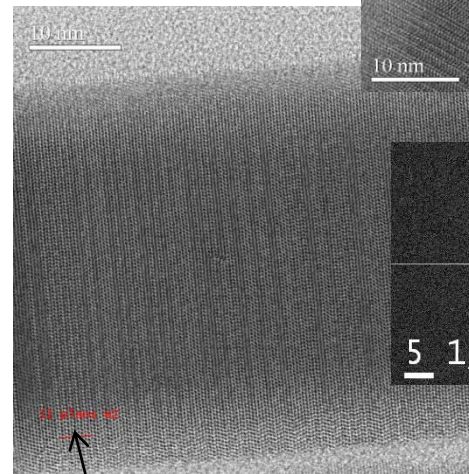
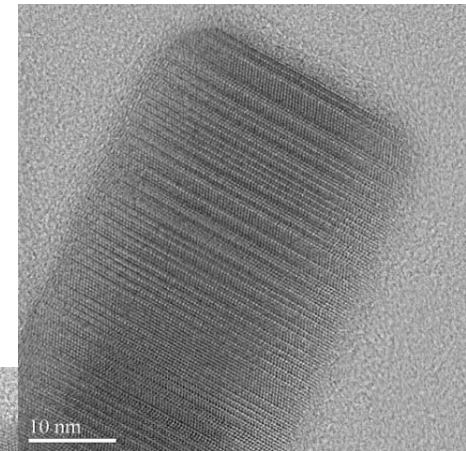
$T_{\text{droplets}} = 420^\circ\text{C}$

$V/\text{III}=20, t_g = 5 \text{ min}$

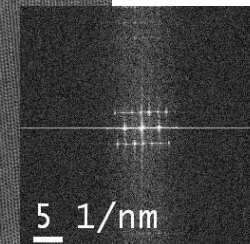


*FFT*

**High twin density**

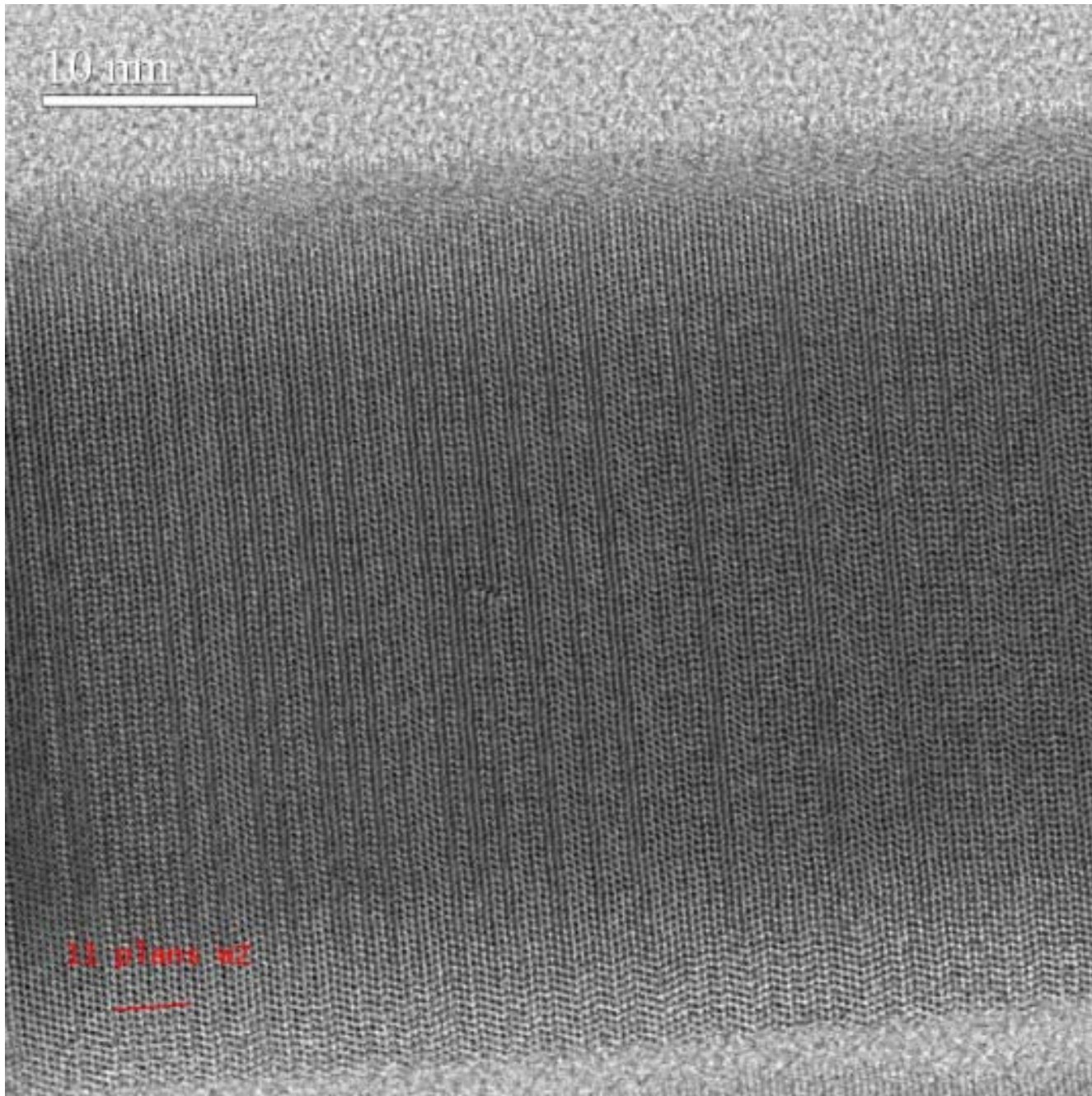


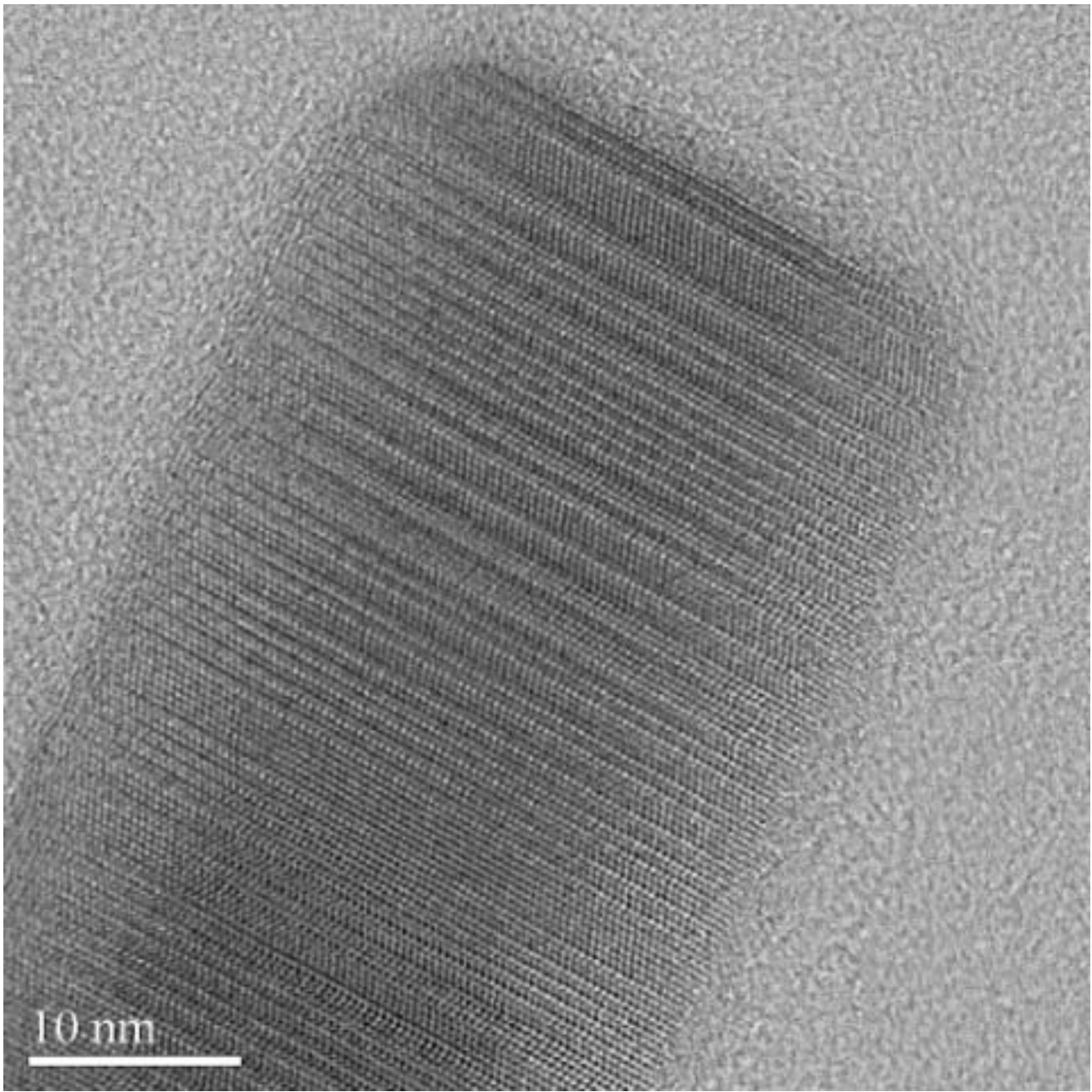
*Wz segment*



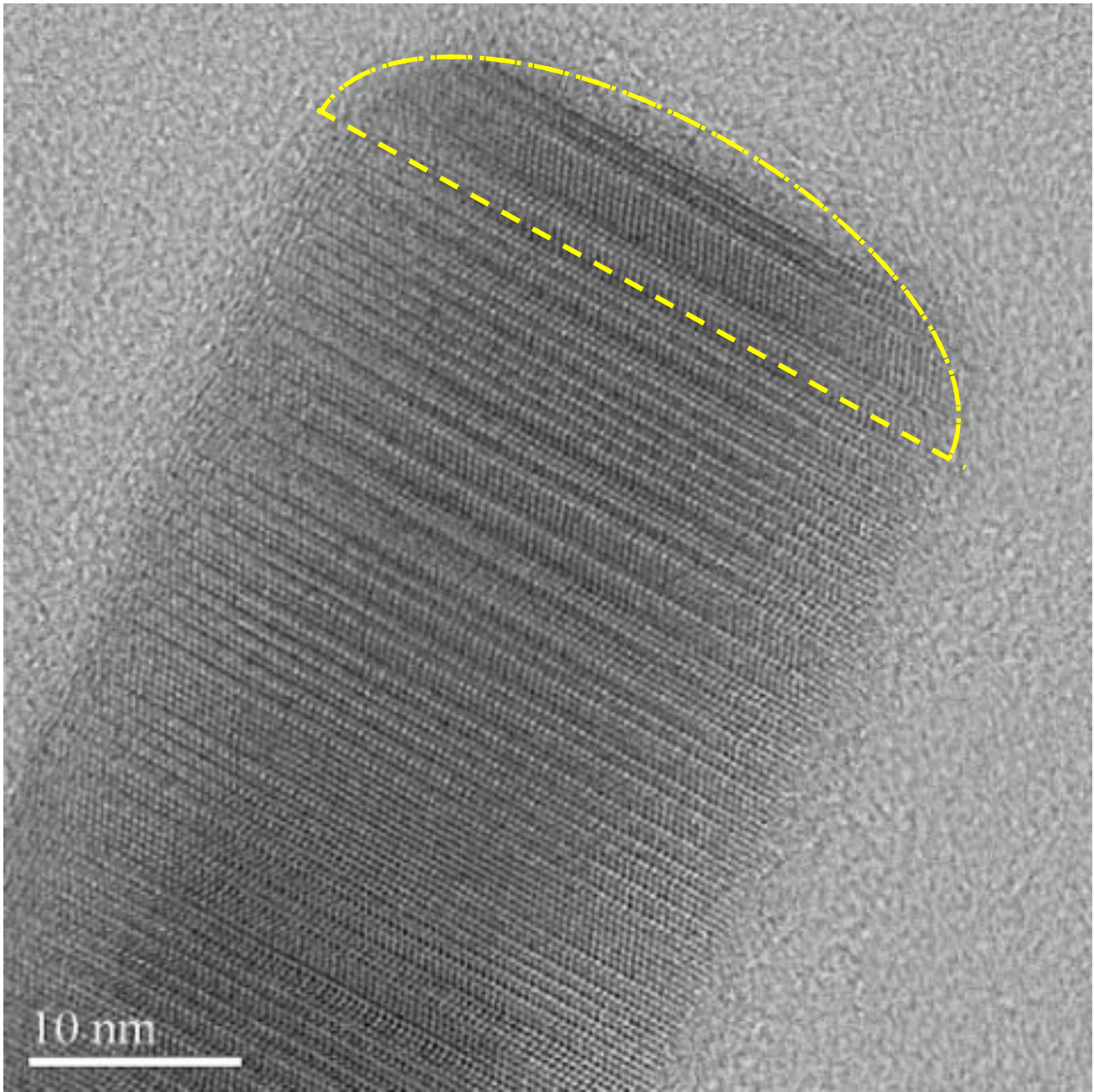
*FFT*

*TEM: G. Patriarche-LPN*

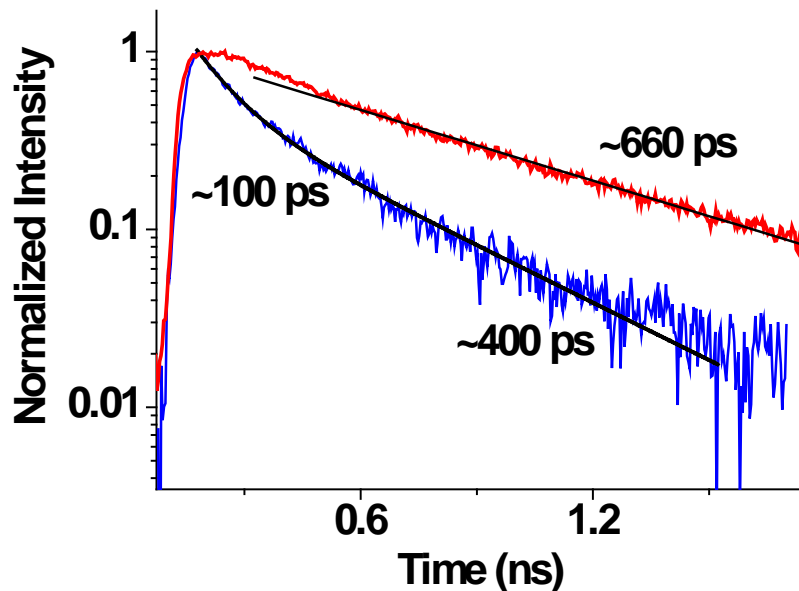
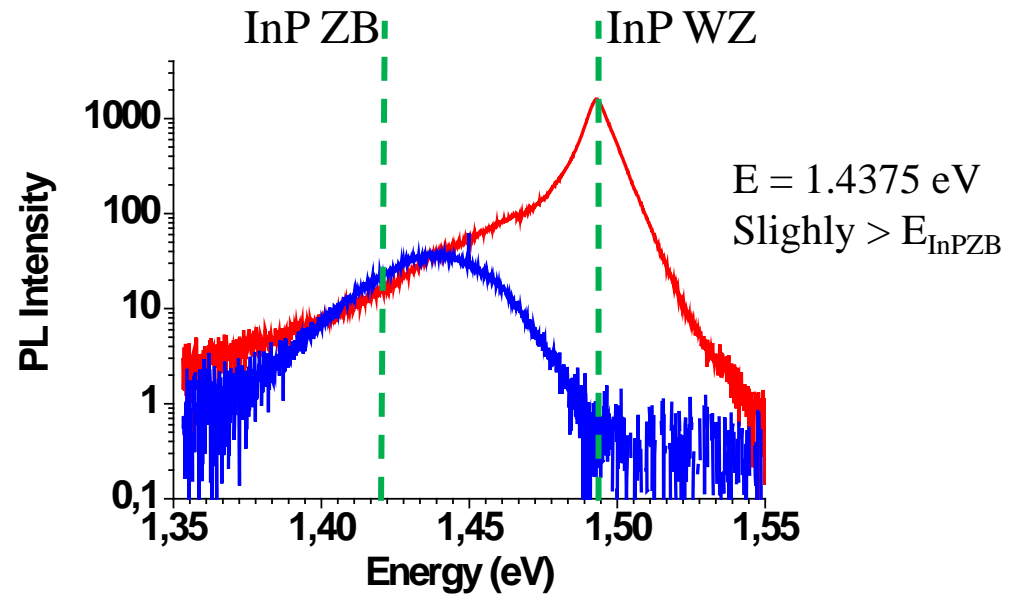
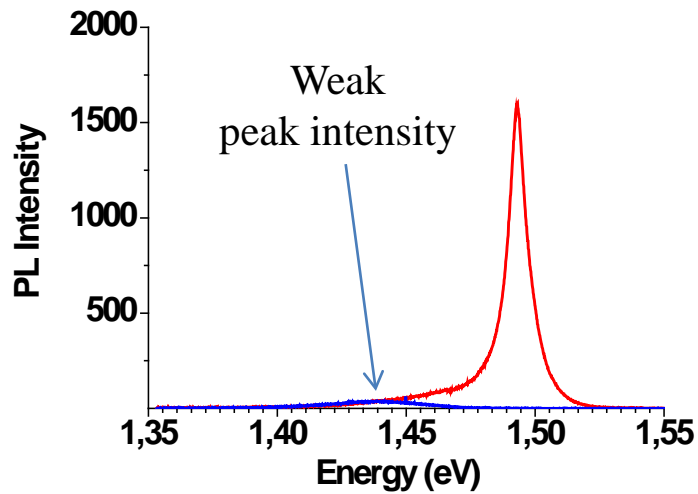








# Optical properties of InP/Si(111) NWs at $T_g = 380^\circ\text{C}$



400 ps: short decay time for InP ZB ?  
100 ps: Non-radiative recombination  
Surface recombination due to surface defect?

*R. Anufriev and N. Chauvin*

# Conclusion and Perspective

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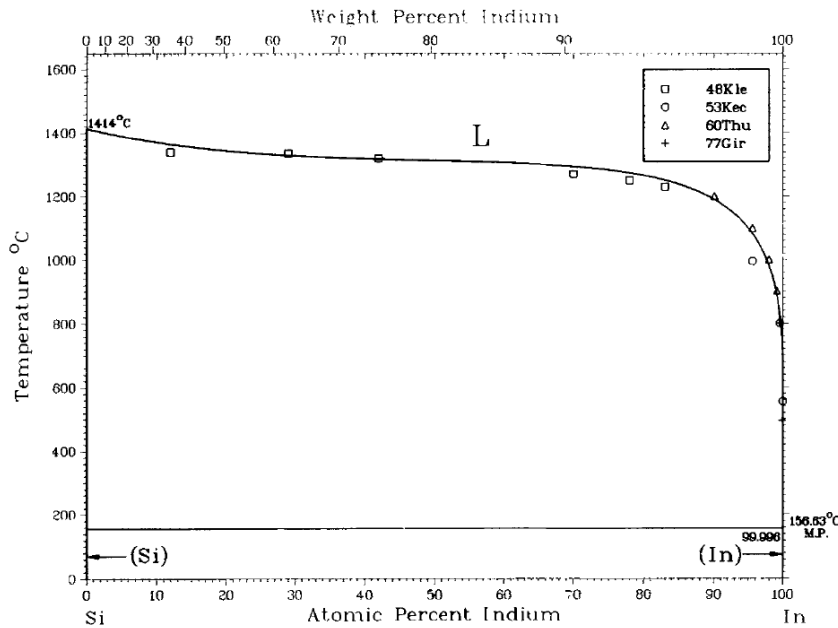
- **We know how to Self-catalyzed growth of InP/Si(111) by MBE with well adapted indium droplets diameter**

## Perspectives:

- Optimization of growth conditions
- Pure ZB InP NWs
- Verticality on Si(001)
- Selective growth on structured Si surface

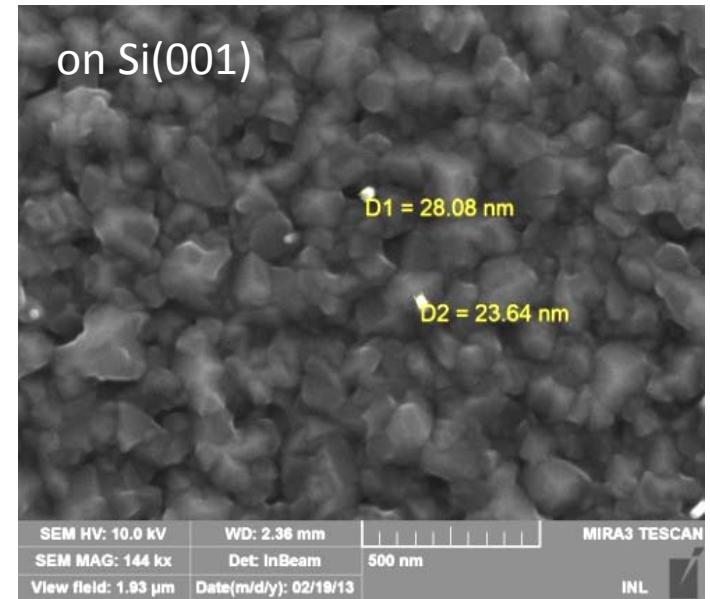
# Self-catalysis with In droplets

- Au-free
- Indium element of the InP NWs
- Indium not miscible in Silicon



R. W. Olesinski, N. Kanani, and G. J. Abbaschian, 1985.

No In-Si alloy



No digging of the Si substrate:  
**Verticality on Si001?**

## Position-controlled [100] InP nanowire arrays

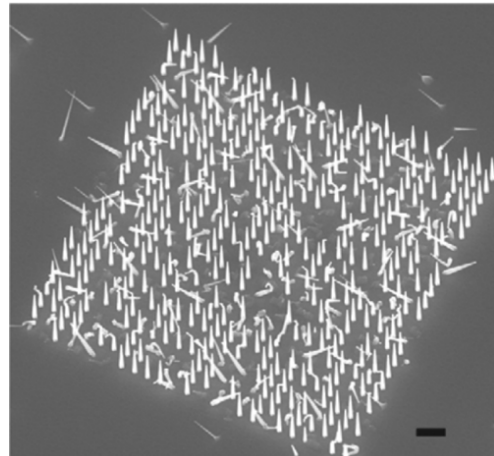
Jia Wang,<sup>1</sup> Sébastien Plissard,<sup>1</sup> Mojira Hocevar,<sup>2</sup> Thuy T. T. Vu,<sup>1</sup> Tilman Zehender,<sup>1</sup>  
George G. W. Immink,<sup>3</sup> Marcel A. Verheijen,<sup>1,3</sup> Jos Haverkort,<sup>1</sup>  
and Erik P. A. M. Bakkers<sup>1,2,a</sup>)

<sup>1</sup>Department of Applied Physics, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands

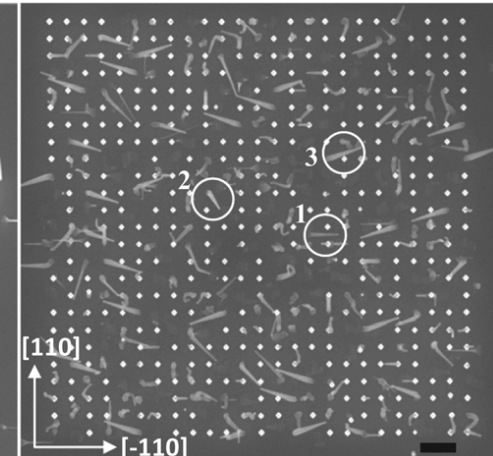
<sup>2</sup>Kavli Institute of Nanoscience, Delft University of Technology, 2628CJ Delft, The Netherlands

<sup>3</sup>Philips Innovation Services Eindhoven, High Tech Campus 11, 5656AE Eindhoven, The Netherlands

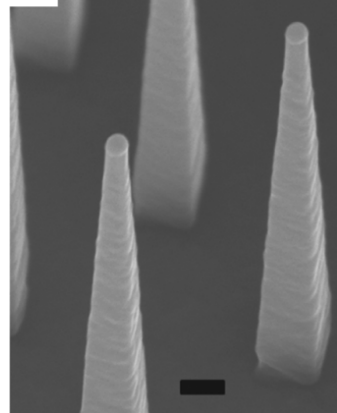
(a)



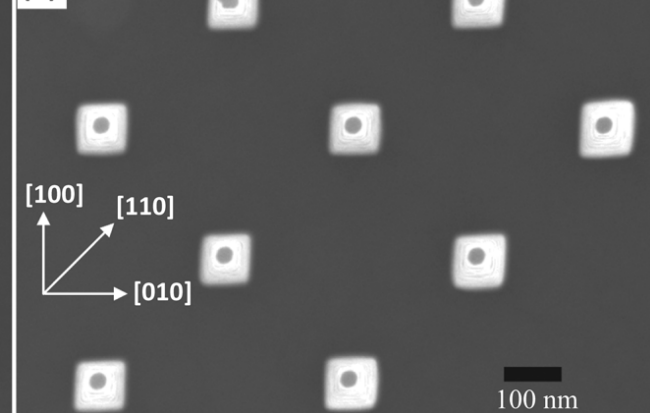
(b)



(c)



(d)



In this work, we report on the growth of [100] ZB InP NWs by the vapor-liquid-solid (VLS) growth mechanism<sup>21</sup> catalyzed by Au and using low-pressure (50 mbar) metalorganic vapor phase epitaxy (MOVPE) on Sn-doped InP (100) substrates;

À faire sur Si(001)  
Pour l'INL

SEM images of as-grown InP NW arrays on a InP (100) substrate with Au diameters (DAu)=50 nm, pitch=500 nm,

Feuille de route projet INSCOOP- Michel Gendry

Echantillon : INSCOOP06022013-1

Substrat Si(001)

Résine PMMA 4%

Litho électronique : réseaux de trous dans la résine (masque 30-50-70 nm) + dépôt 10 nm d'au + lift-off.

Masque utilisé :



+ résine de découpe AZ1512 ..... NB : réseaux de plots dans les coins des échantillons

- à enlever avec acétone/alcool/soufflette N2g .....puis plasma O2

NB : comme Pb de SB à l'INL, wafer retourné à Bassem le 19-02-2013 pour qu'il fasse cette manip !

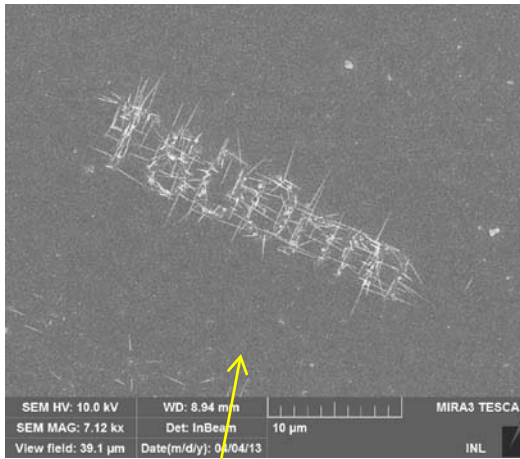
Retour le 13-03-2013 : 10 échantillons nettoyés

BOE/H2O/N2g

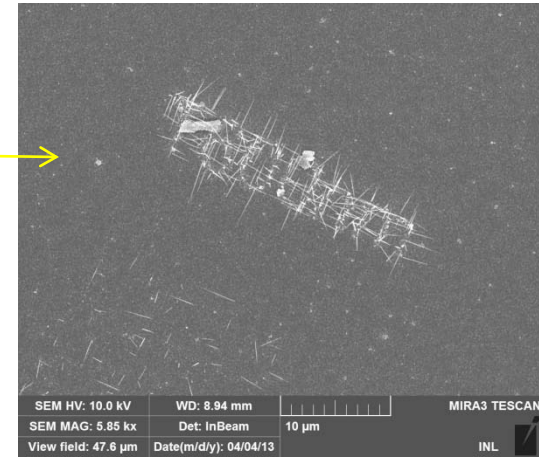
TT 600°C 5 mn + flux d'In (6ML)

Croissance NFs à 380°C, V/III=20

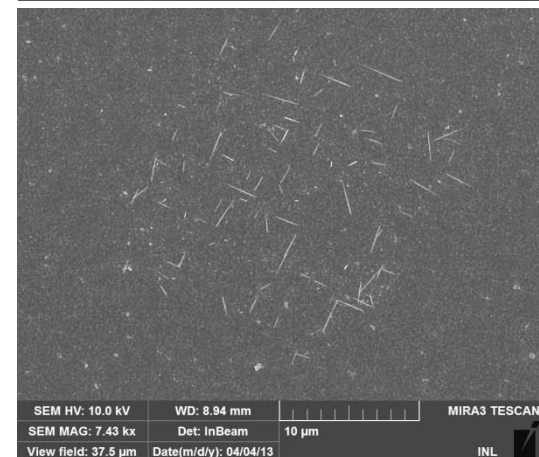
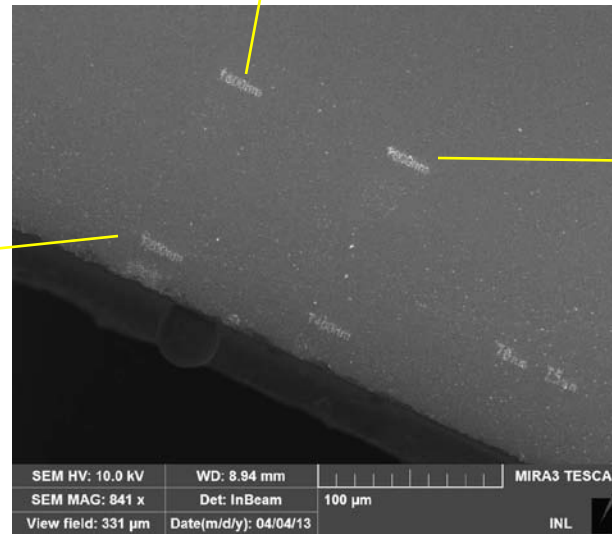
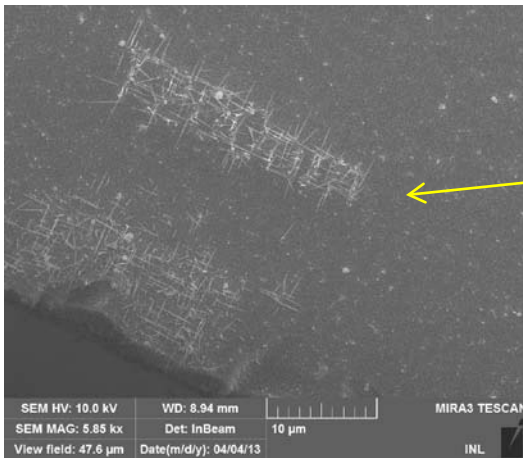
T=600 nm?



T=800 nm?



T=200 nm



# Annexes

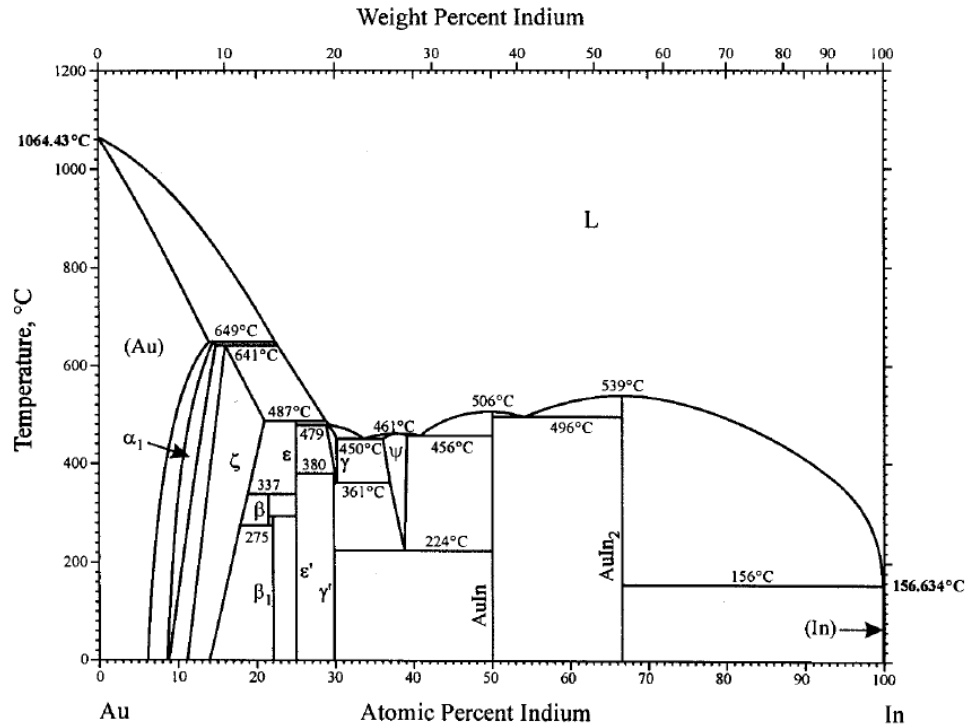
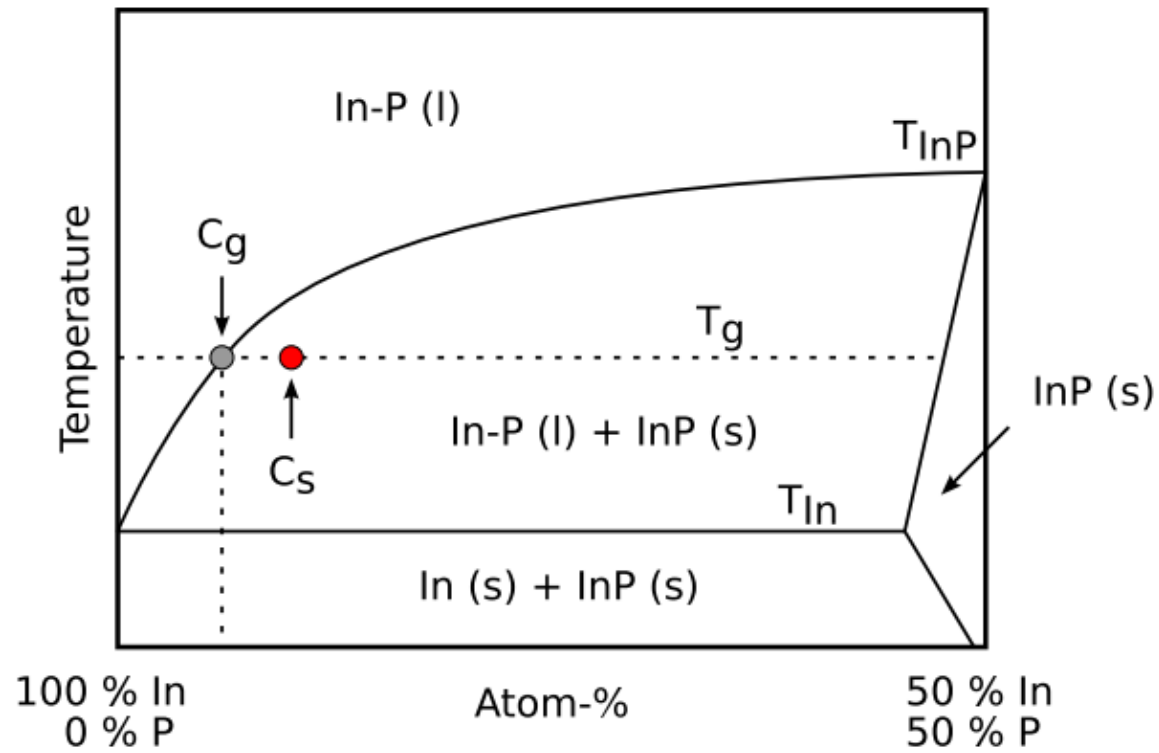
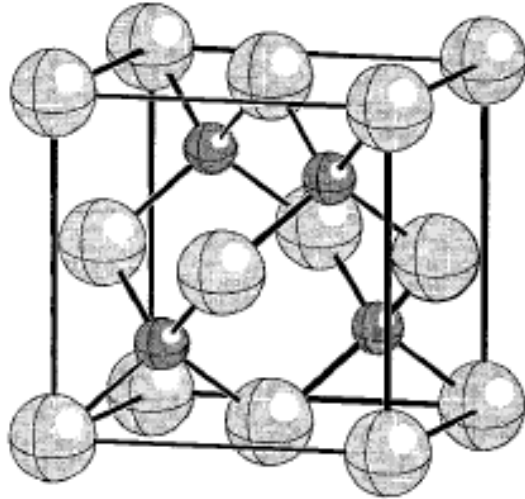


Fig. 1 Au-In phase diagram calculated by [2003Liu]



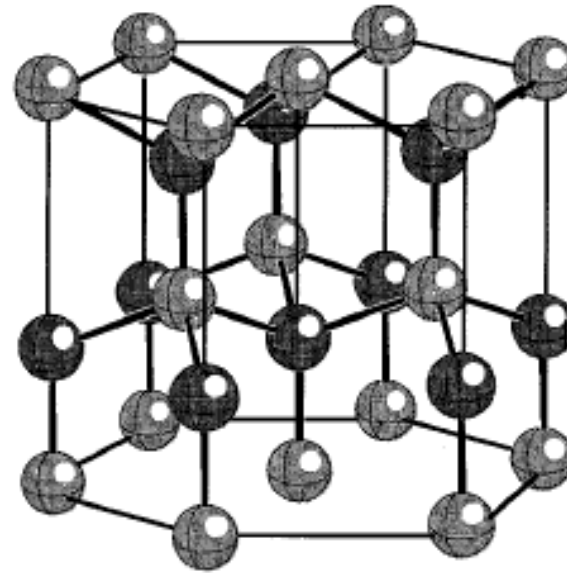


# Structure of III-V



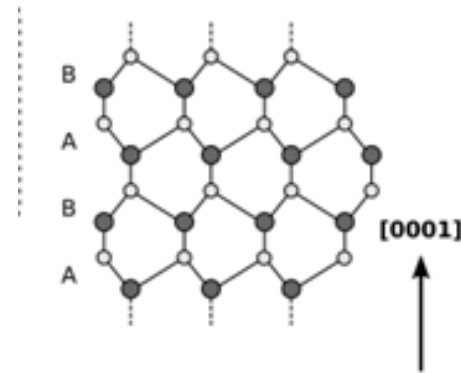
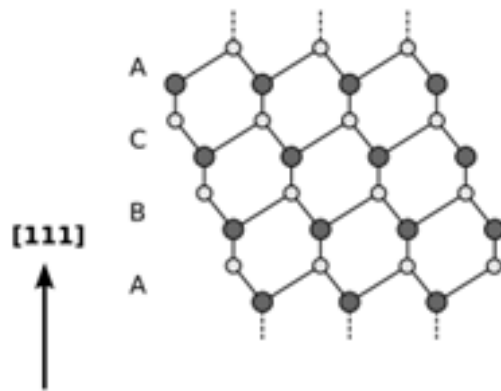
Zinc-blende

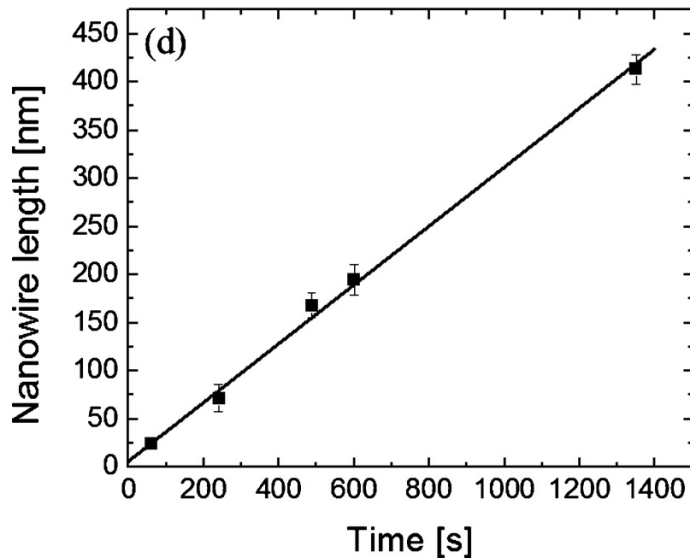
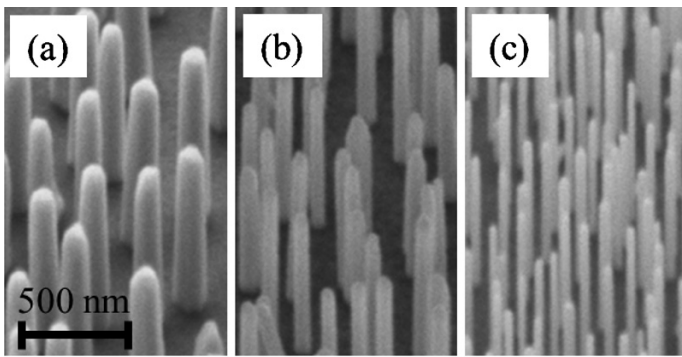
$a_{\text{Si}}=5.43 \text{ \AA}$   
 $a_{\text{InP}}=5.86 \text{ \AA}$   
 $E_{\text{InP}}=1.42 \text{ eV}$   
 $E_{\text{Si}}=1.08 \text{ eV}$



Wurtzite

$a_{\text{InP}}=4.054 \text{ \AA}$   
 $c_{\text{InP}}=6.625 \text{ \AA}$   
 $E_{\text{InP}}=1.49 \text{ eV}$





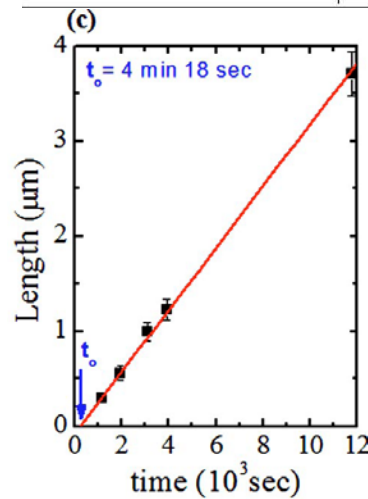
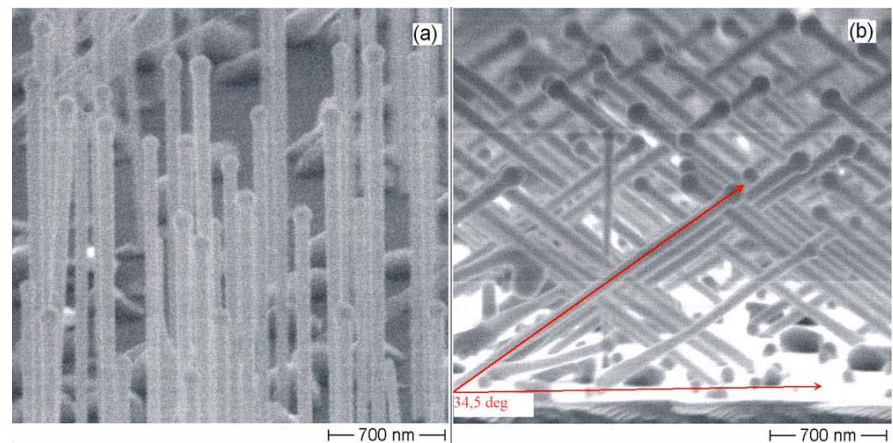
### InAs NWs MBE

No predeposition and no VLS

SiO<sub>x</sub>/Si(111) with openings

*Hirtenberger, APL, 98,*

*1231114 (2011)*



### GaAs NWs MBE

No predeposition and no VLS

20 nm-100 nm SiO<sub>2</sub>/GaAs

*Fontcuberta, APL, 92, 063112*

*(2008)*