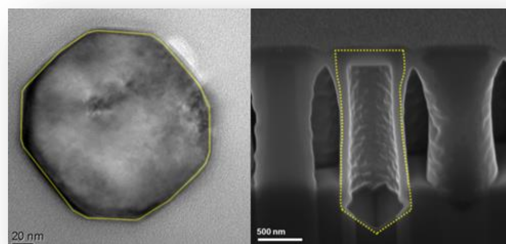
6-2014

Silicon Carbide-Based One-Dimensional Nanostructures Growth: Towards Electronics and Biology Perspectives

Laurence Latu-Romain

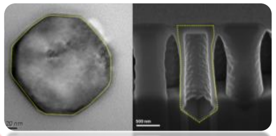
Silicon Carbide-based one-dimensional nanostructures growth: towards electronics and biology perspectives



Grenoble University
LTM-SIMaP

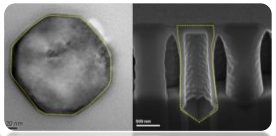
Laurence LATU-ROMAIN

DSL 2014 International Conference
PARIS • FRANCE
23 –27 June, 2014
10th International Conference on Diffusion in Solids and Liquids



Outline

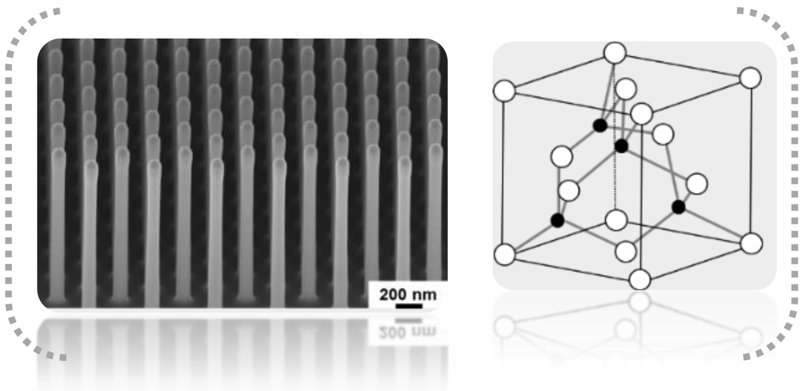
- Introduction
- State of the art
- Study of the Si nanowires carburization
  - Si-SiC core-shell nanowires
  - SiC nanotubes
  - Existence diagram
- Alternative approach
- Potential applications



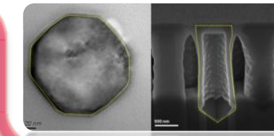
Introduction

Interest of SiC nanostructures:

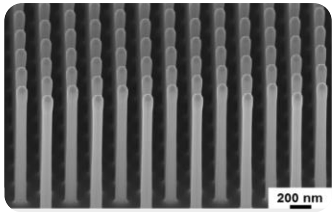
to associate a high S/V ratio and a material with exceptional properties

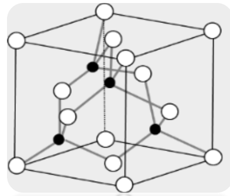


- SiC has excellent intrinsic properties:
  - Large bandgap semiconductor (2.2 eV, Cubic-SiC)
  - High electron mobility
  - High breakdown field: 15.10^{5} $V.cm^{-1}$ (10x sup. to Si)
  - High thermal conductivity: 450 $W.m^{-1}.K^{-1}$ (3x sup. to Si)



Introduction





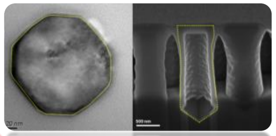
- SiC can be used in harsh and biological environments:
  - High operating temperature: 900°C
  - High chemical stability
  - Biocompatibility
    both hemo- and bio-compatible
  - One of the most abundant materials on earth and in the universe

"This material [SiC] system may prove to be the dream material for biomedical devices"

S. Saddow,
Silicon Carbide Biotechnology, A biocompatible semiconductor for advanced biomedical devices and applications, Elsevier, 2012

P. Merino,...,P. Soukiassian et al. , Graphene etching on SiC grains as a path to interstellar polycyclic aromatic hydrocarbons formation, Nature comm., 5, 3054, (2014)
and INVITED TALK

- Potential applications:
  - **Nano-electronics**: high power/high T°/high frequencies or/and in extreme conditions
  - **Bio-nanosensors**



State of the art

1) Quantum confinement effect in SiC:

Bohr exciton radius of 3C-SiC is very low ~ 2.7 nm

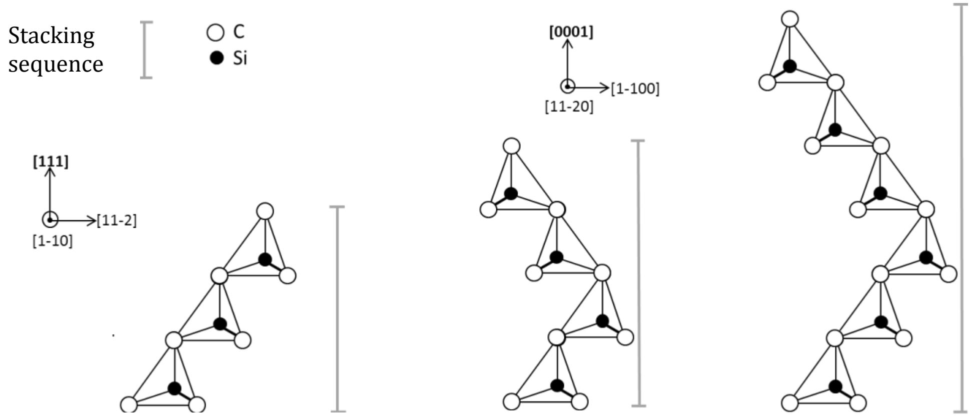
High sensitivity

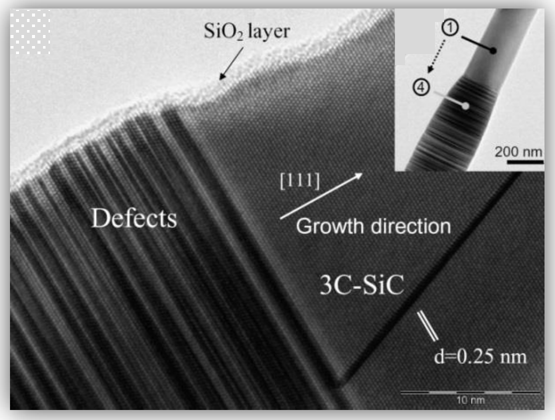
2) The great majority of the grown nanostructures is SiC nanowires (NWs).

Whatever the growth method, nanowires are :

- **cubic SiC NWs** because of the low growth temperature
- and contain a high density of structural defects, stacking faults

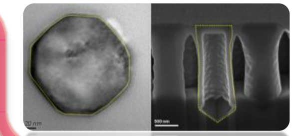
The doping is still not controlled.





TEM Image of a SiC NW (VS method) Bechelany M., Brioude A., Cornu D. et al. Advanced Functional Materials, 17(6), 939(2007)

Polytypism (more than 170 polytypes)



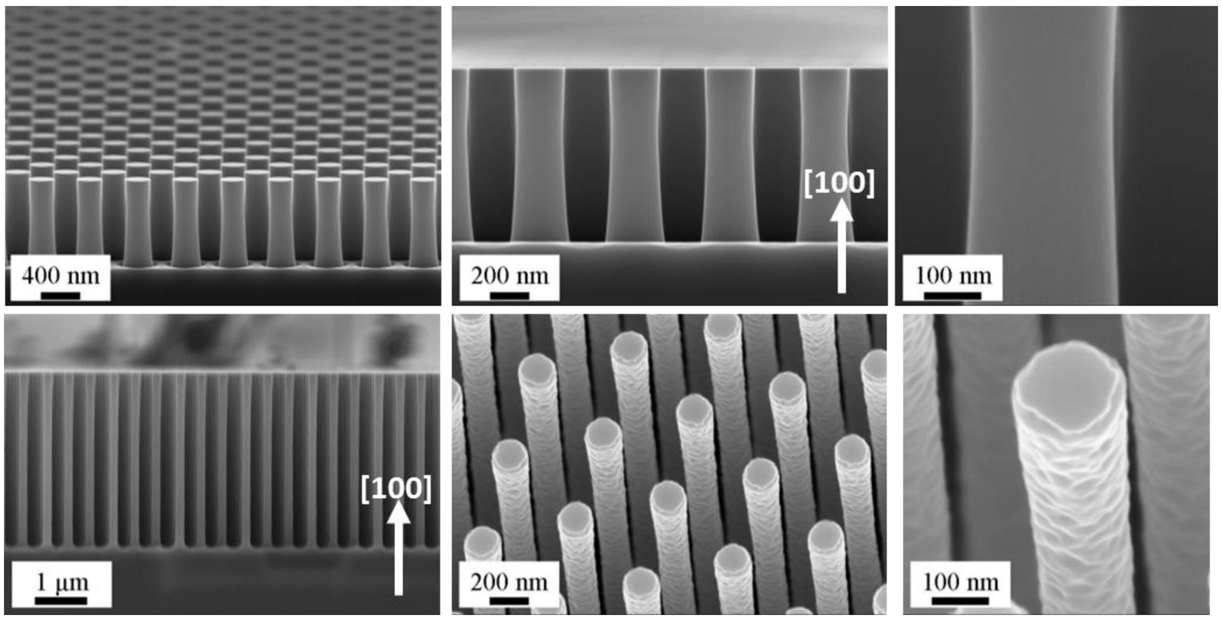
Si nanowires carburization

Very few previously studied

Advantage: Si nanowires obtained by plasma etching are an excellent basis

Diameter: 250 nm
Lenght: 1 µm

Diameter: 200 nm
Lenght: 3.4 µm



M. Martin, LTM

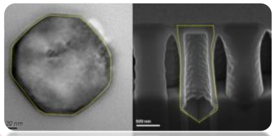
- Reproductibility
- Controlled doping
- No catalyst
- Single crystalline NW

Roughness on the sidewalls

In order to explore original nanostructures

like Si-SiC core-shell nanowires and SiC nanotubes

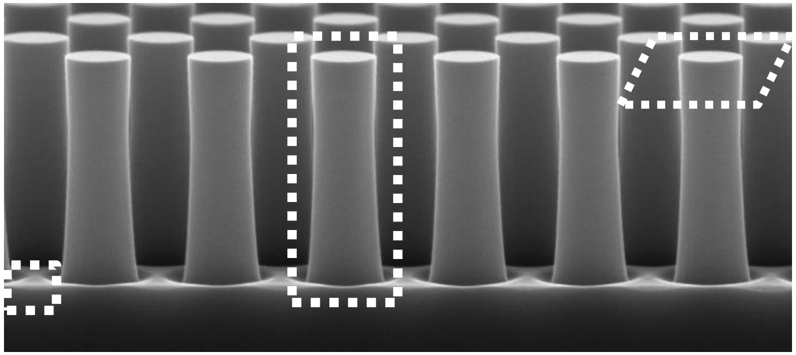
Characterization of choice

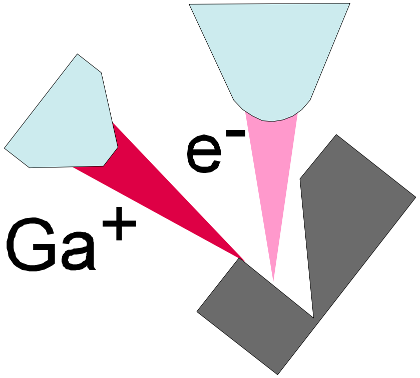


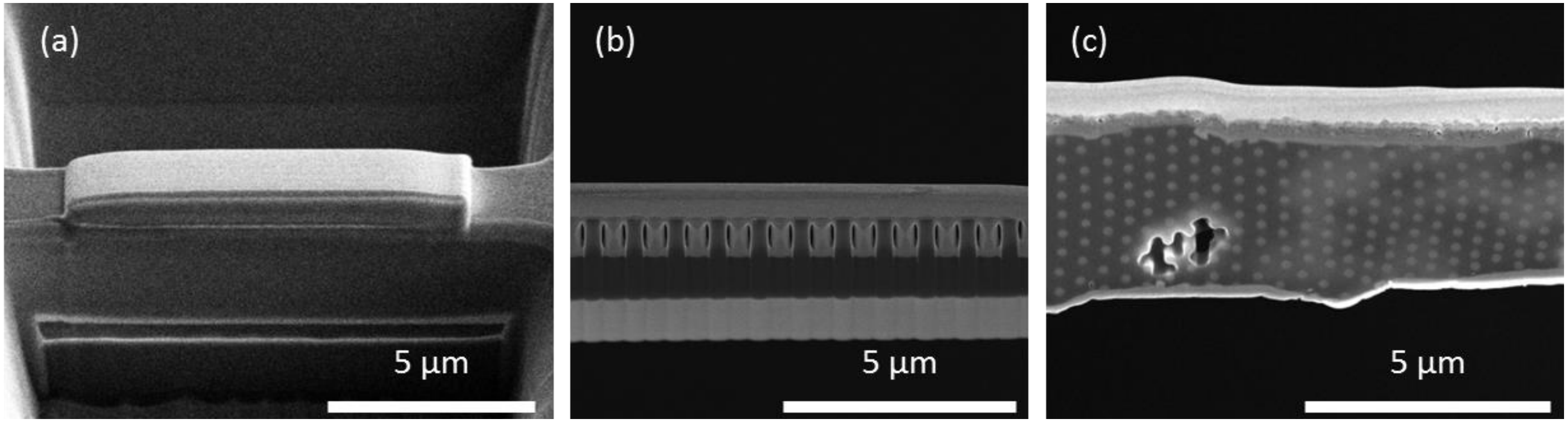
1) SEM observations and thin lamellas prepared by double column Focused Ion Beam-Scanning Electron Microscope (FIB-SEM):

Transversal cross section on the substrate (a) or along the nanostructure (b), or longitudinal cross section around the nanostructures (c)

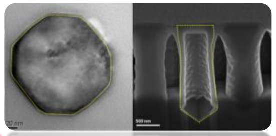
2) Characterization by Transmission Electron Microscopy



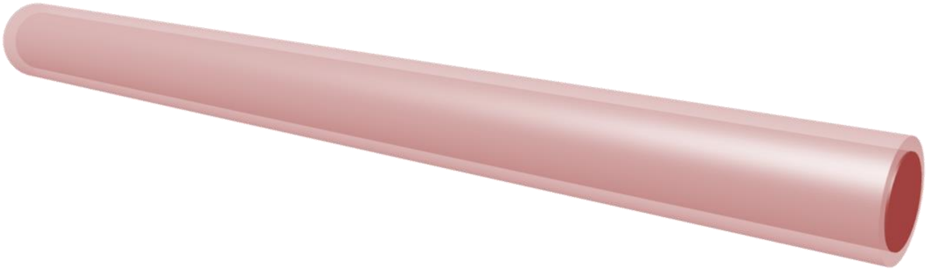


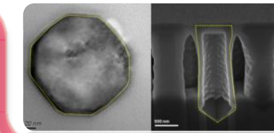


Si-SiC core-shell nanowires



- «Perfect » object for bio-sensing:
  - Electronic transport in the Si core
  - Biocompatibility and chemical inertness of the SiC shell (further functionalized)
  - NW-FET technology
- Objectives :
  - A protective and the best structural quality for the SiC shell
  - To keep intact the Si core

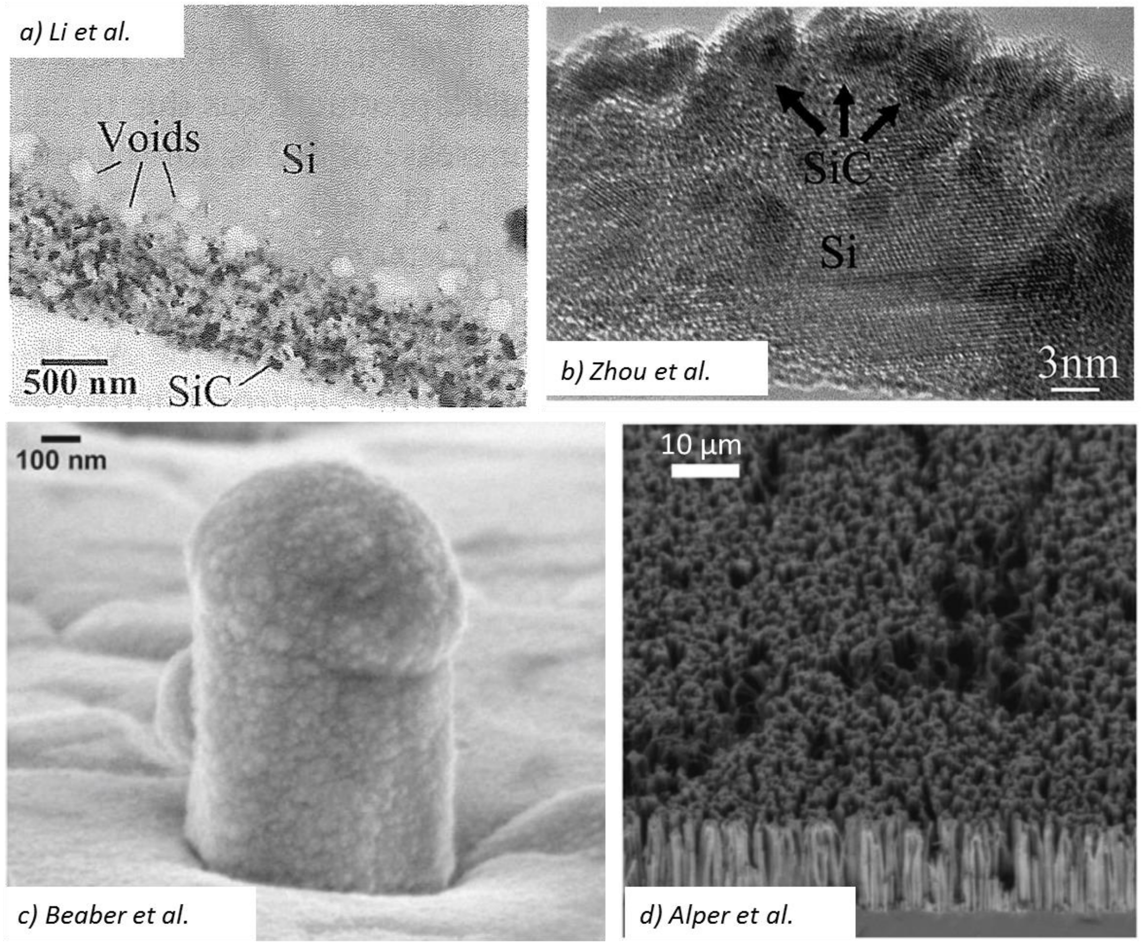




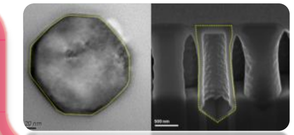
Si-SiC NWs: state of the art

Very few studies:

From:
a) Y.L. Li etal. Journal of the Ceramic Society of Japan, 115(1347):717–723, (2007)
b) X.T. Zhou, et al. Chemical Physics Letters, 332(3–4):215–218, (2000)
c) A.R. Beaber, et al.. International Journal of Fracture, 171:177–183, (2011)
d) J.P. Alper, et al. Applied Physics Letters, 100(16):163901, (2012)

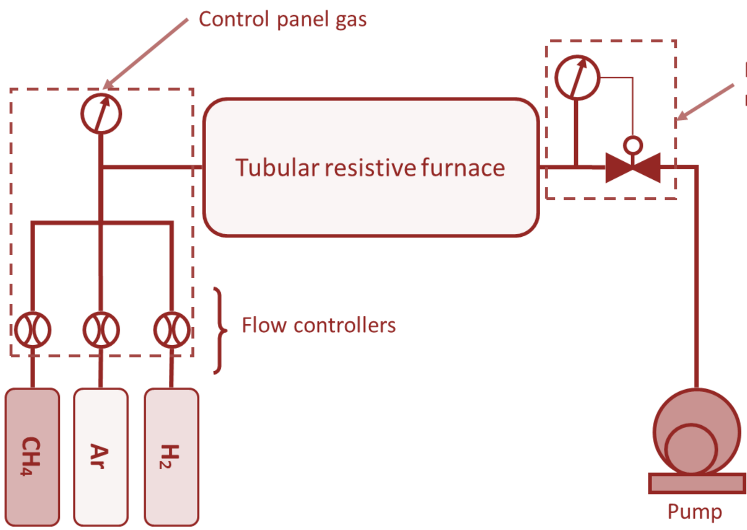


Various growth methods that lead to a discontinuous and a polycrystalline SiC deposit.

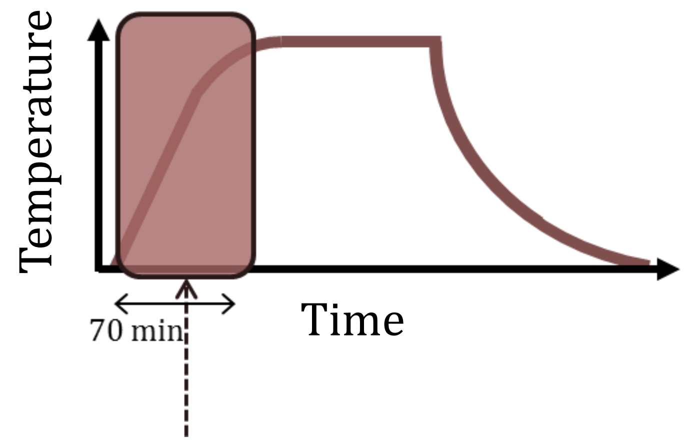


To keep intact the Si core ?

Carburization experiments:

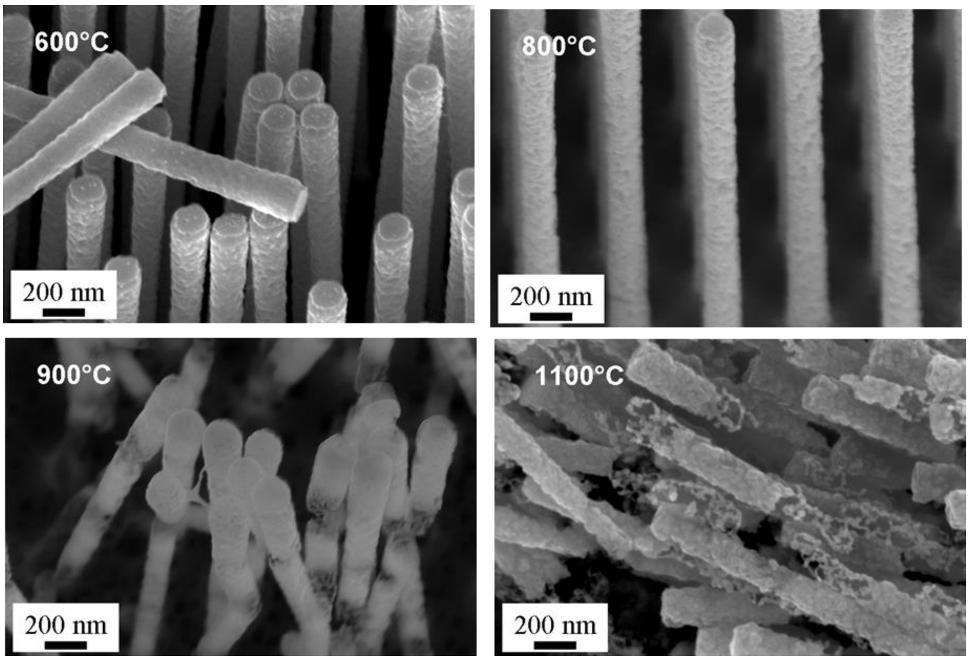


Horizontal tubular furnace:
Temperature: 20 à 1200°C
Pressure: 0,2 à 10^5 Pa
–Ar flow: 0 — 200 $mL.min^{-1}$,
–H_2 flow: 0 — 200 $mL.min^{-1}$,
–CH_4 flow: 0 — 4 $mL.min^{-1}$;

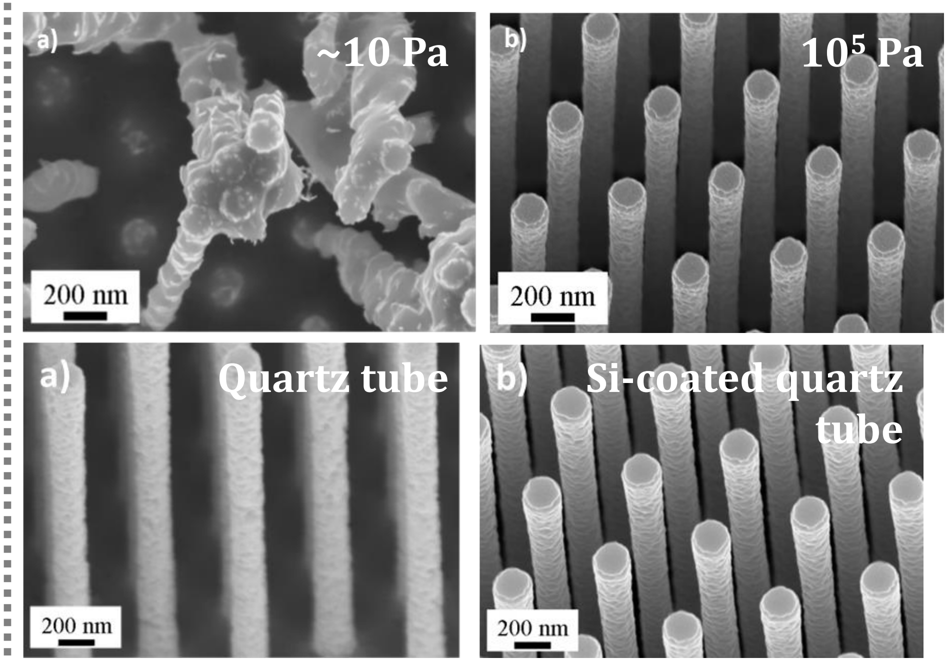


-Study of the heating temperature ramp
-Optimal carburization temperature

Si-NWs heating temperature



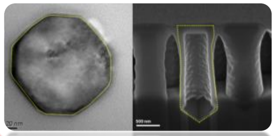
Heating temperature at ~10 Pa



Untill 900°C

- Si nanowires are damaged at high temperature
- Silicon sublimation is exacerbated at low pressure and with the presence of residual oxygen

Si-NWs heating temperature



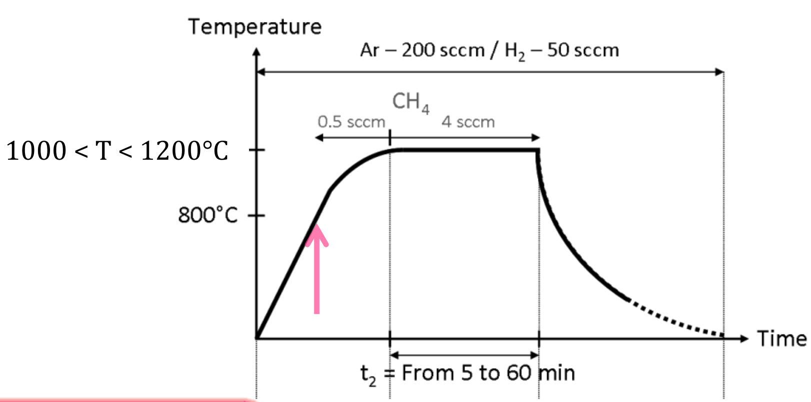
Supported by thermodynamical considerations (free enthalpy minimization, Factsage simulation)

Initial system	<Si>	$[H_2]$	<Si>	$[H_2]$	$[O_2]$
Initial quantities (moles)	2	1	20	10	1
Final system (1200°C)	<Si>	$[H_2]$	<Si>	$[H_2]$	Si vapor phase : [SiO]
Final quantities (moles) at P=10 Pa	$2\text{-}10^{-4}$	$1\text{-}10^{-3}$	18	$10\text{-}10^{-3}$	2
Final quantities (moles) at $P=10^5$ Pa	$2\text{-}10^{-5}$	$1\text{-}10^{-4}$	19,98	$10\text{-}10^{-4}$	0,02

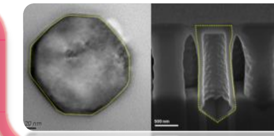
L. Latu-Romain et al., Journal of Nanoparticle Research 13, 5425-5433, 2011

FactSage

Solution: Introduction of methane at low T° (800°C) at atmospheric pressure in order to grow a protective SiC seed layer



Si-NWs heating temperature



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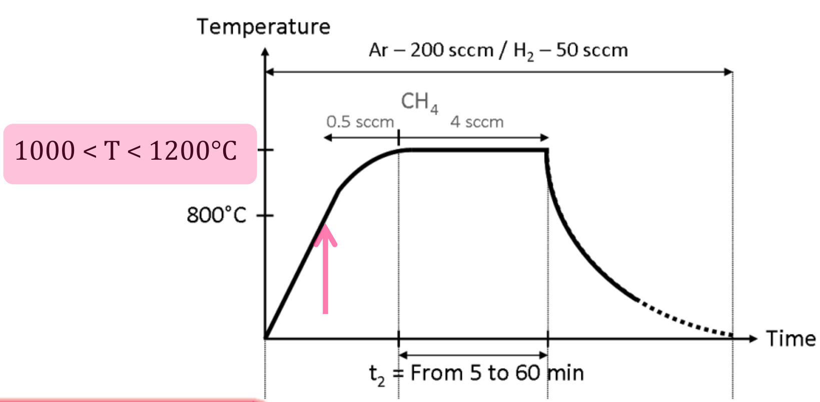
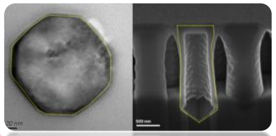
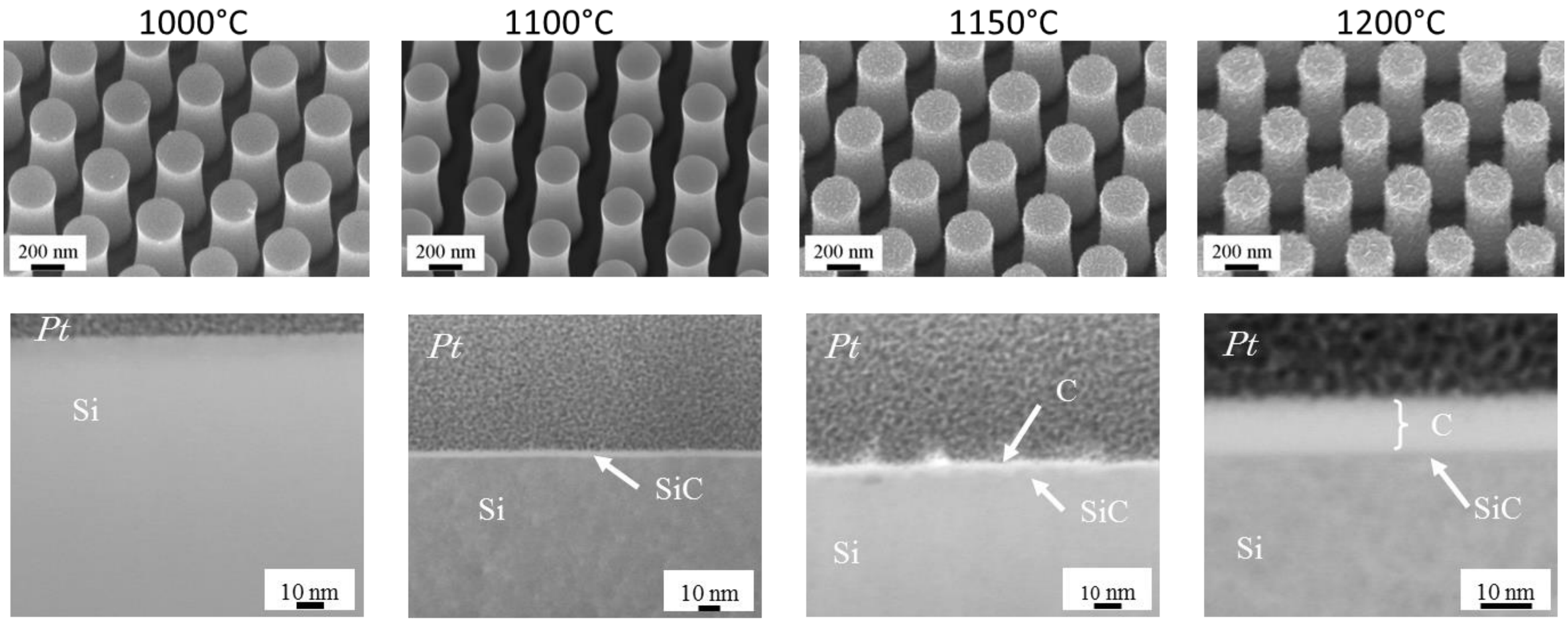


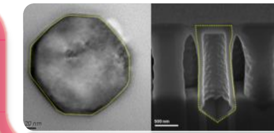


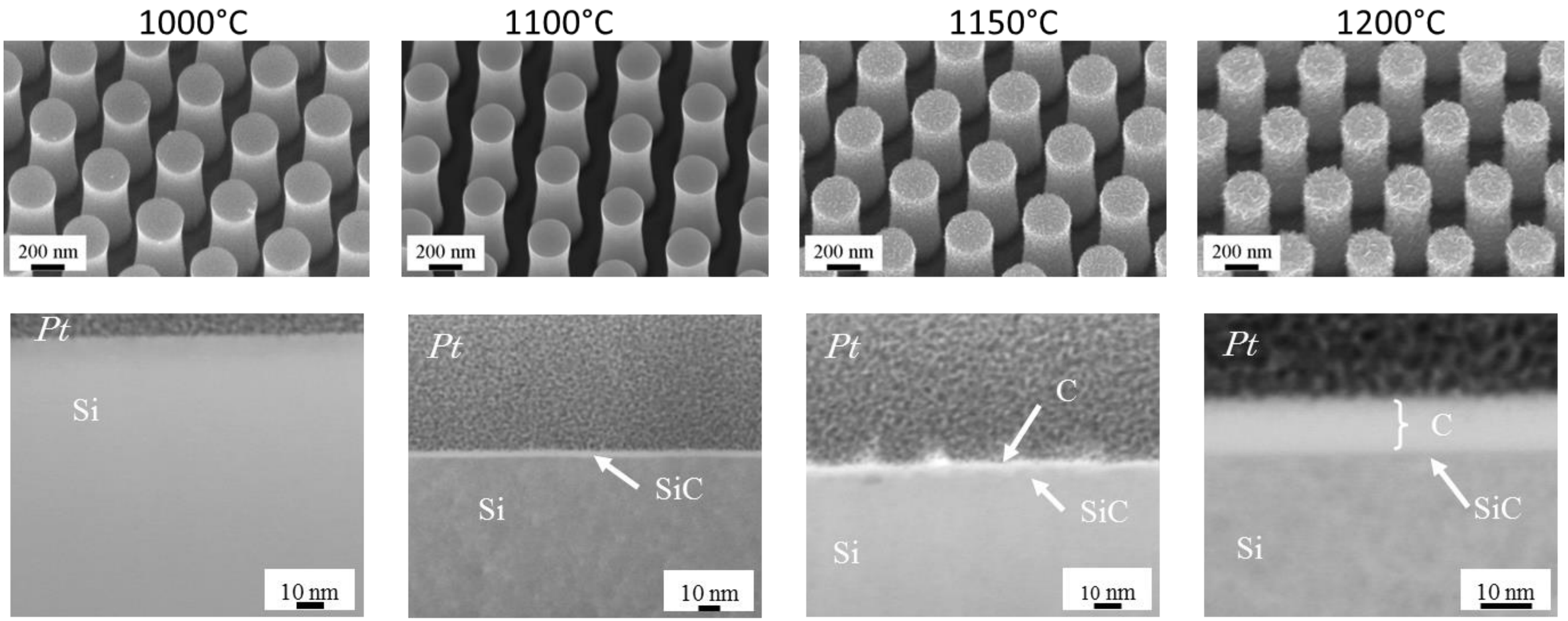
Carburization temperature



Nanowires SEM images
FIB-SEM *in-situ* STEM images (30 kV) of transversal cross section on (100) Si

Carburization temperature



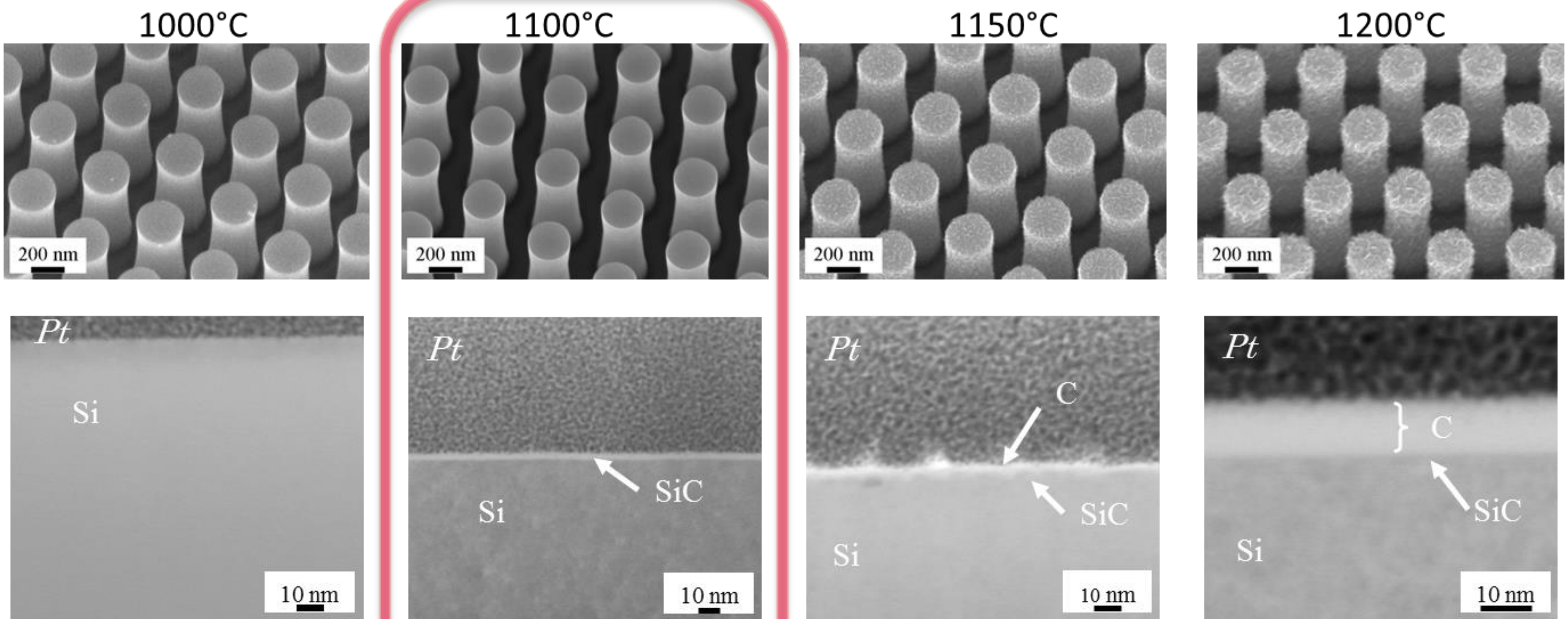


Silicon carbide cannot be observed

Thin SiC deposit on Si

SiC and amorphous C (Raman spectroscopy) deposits

Carburization temperature



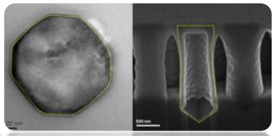
Silicon carbide cannot be observed

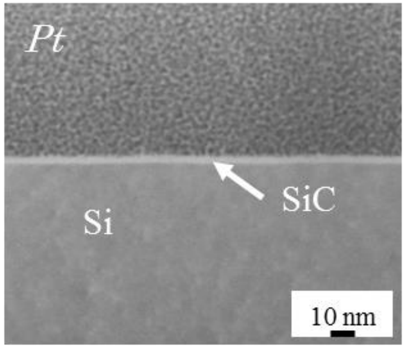
Thin SiC deposit on Si

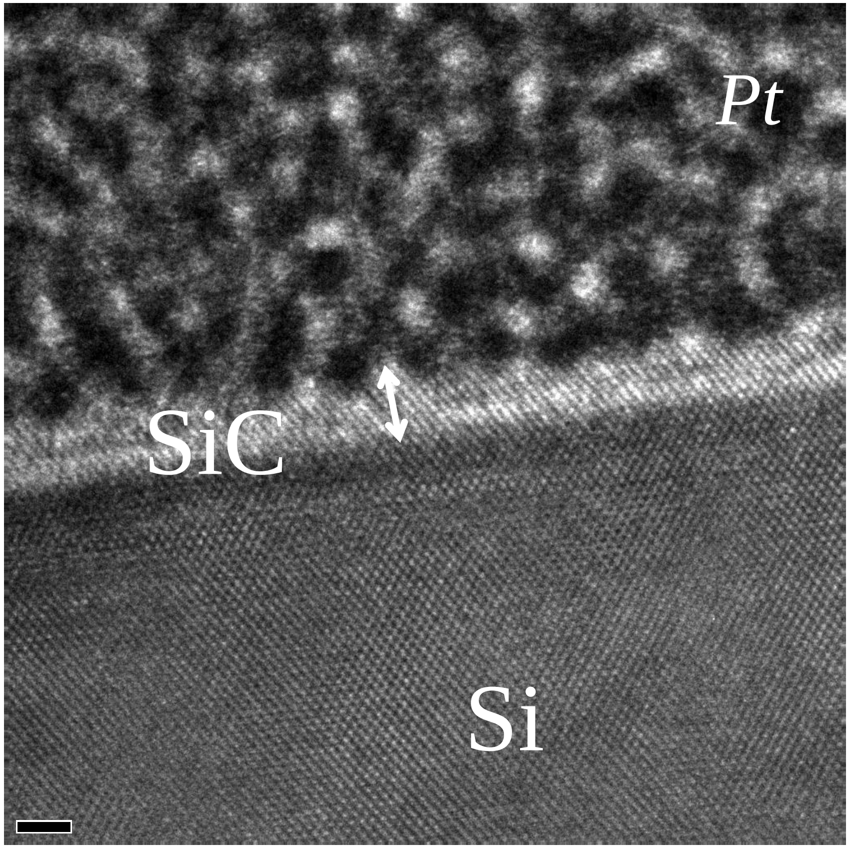
SiC and amorphous C (Raman spectroscopy) deposits

In these conditions, 1100°C seems to be the optimal carburization temperature.

Si-SiC core-shell nanowires



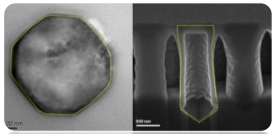


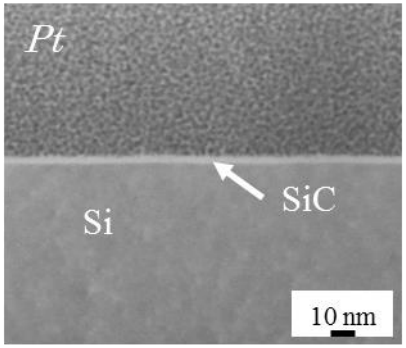


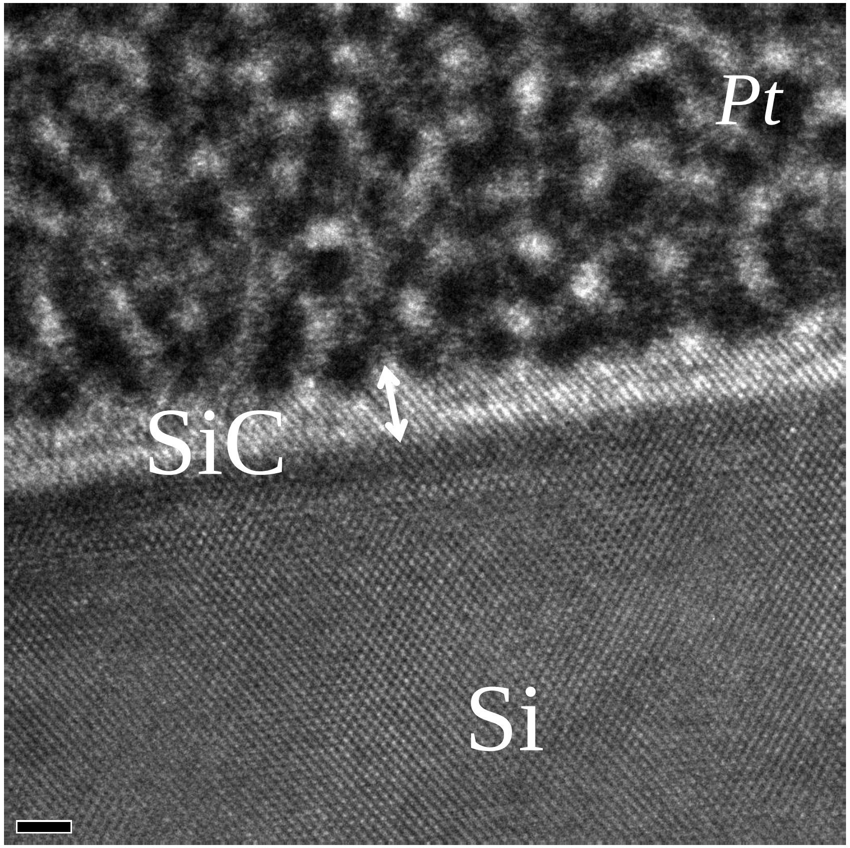
Δa/a = 20 %

HRTEM image (200 keV) of the **single crystalline cubic SiC** layer of a thickness of 2.8 nm on (100) Si by carburization at 1100°C.

Si-SiC core-shell nanowires



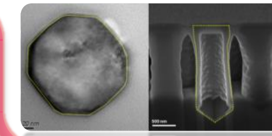




Δa/a = 20 %

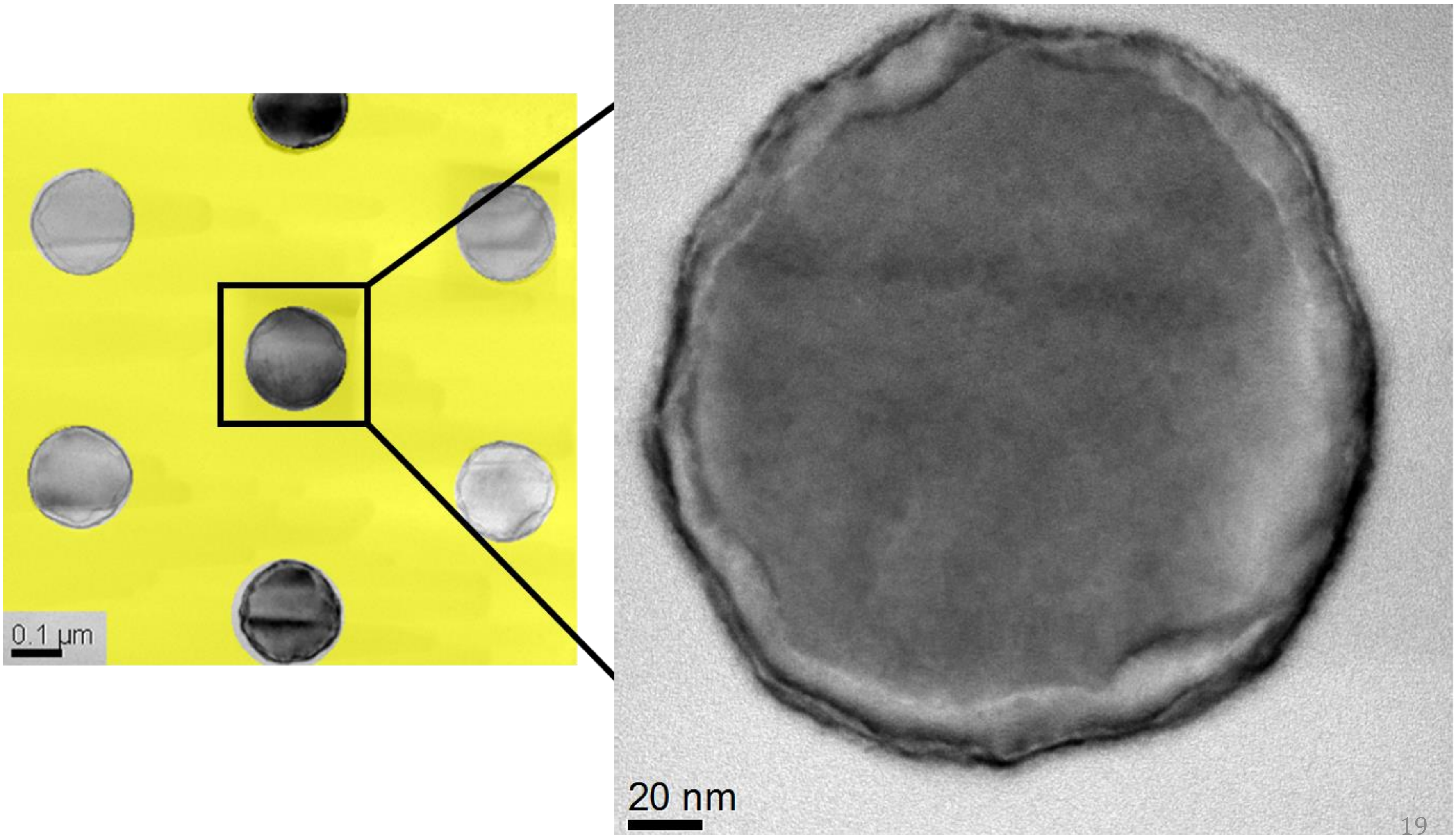
HRTEM image (200 keV)of the **single crystalline cubic SiC** layer of a thickness of 2,8 nm on (100) Si by carburization at 1100°C.

And what about the other surfaces, all around the nanowire?



SiC shell conformity (1100°C) ?

Longitudinal cross-section of Si-SiC NW, prepared by FIB-SEM, observed by TEM:



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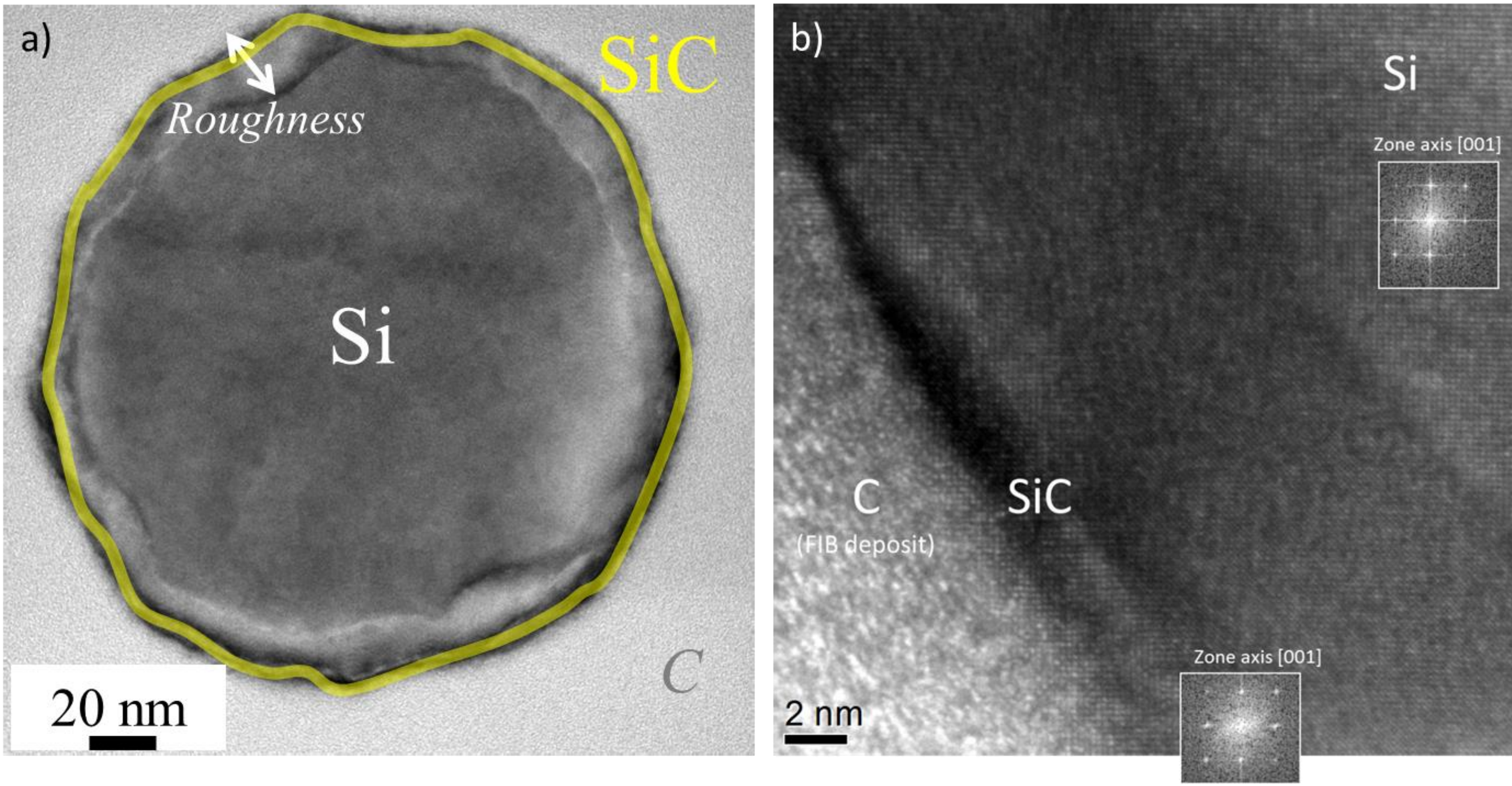
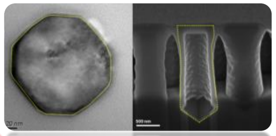
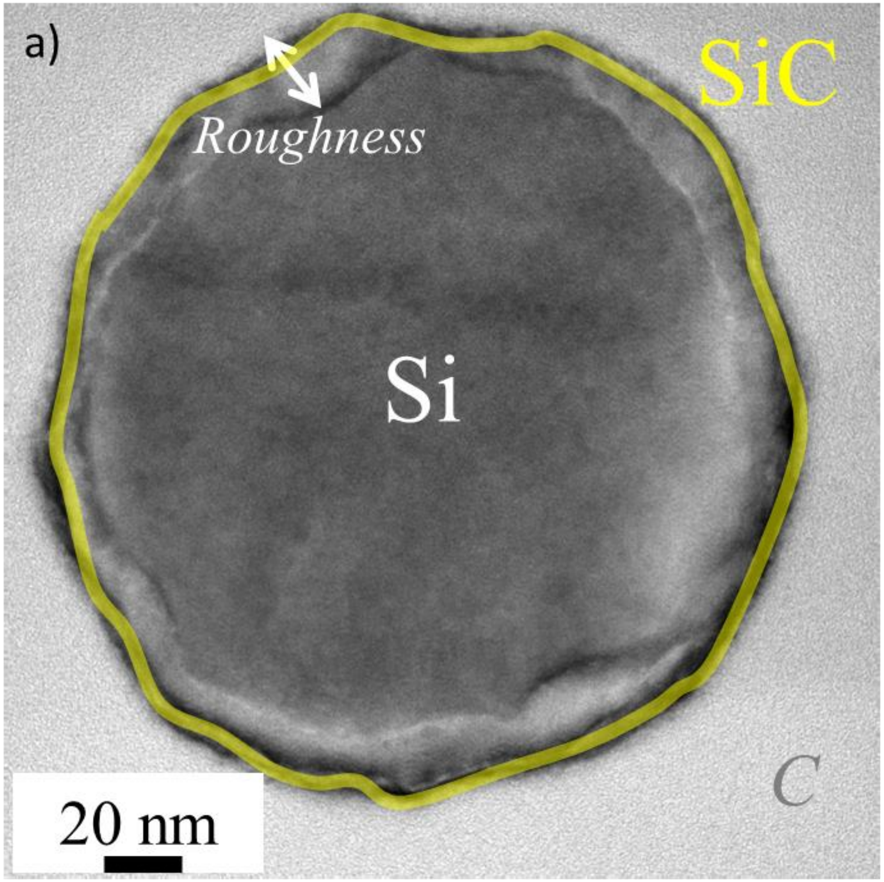
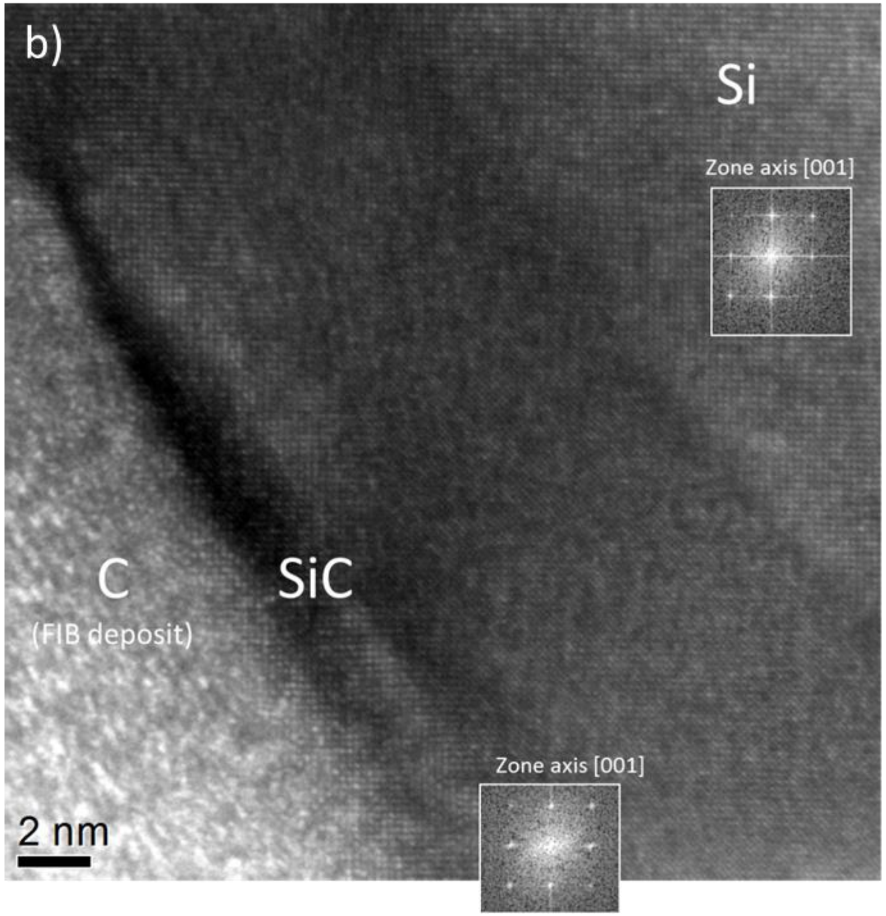


Si-SiC core-shell nanowire observed in [001] zone axis:
2.8 nm SiC on all the Si surfaces
Single crystalline SiC shell

SiC shell conformity (1100°C) ?



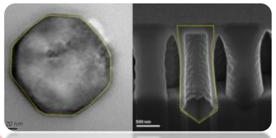


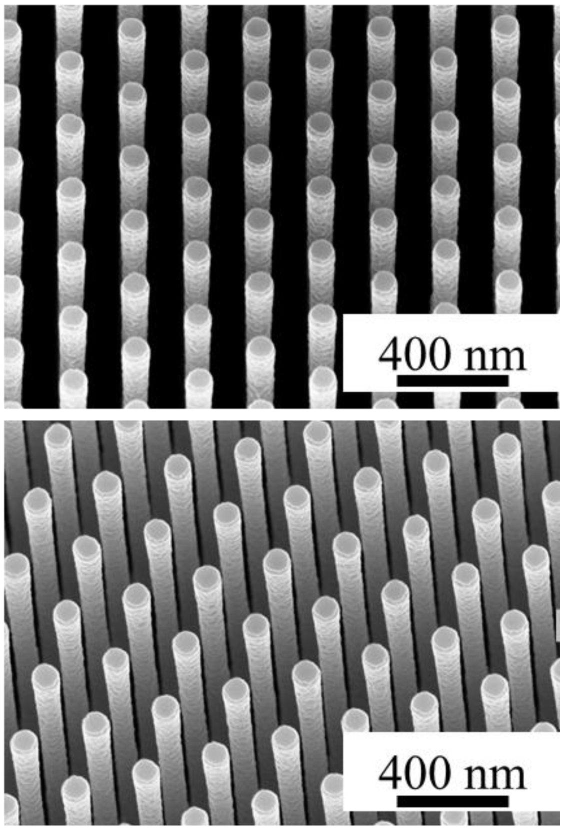


2.8 nm SiC on all the Si surfaces
Single crystalline SiC shell

→ Observation at a local scale

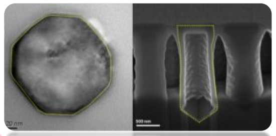
SiC shell conformity (1100°C) ?

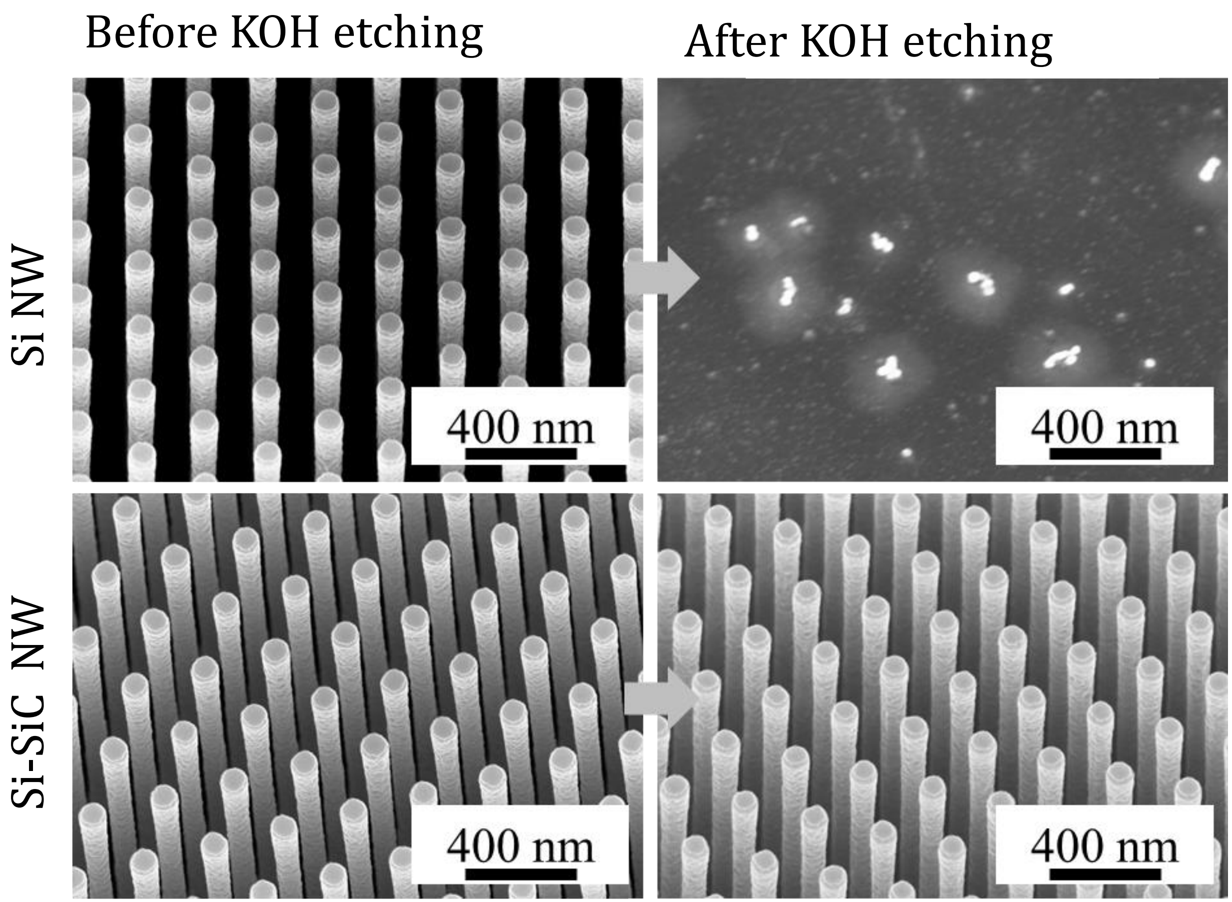




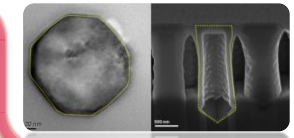
1) Identical morphology after carburization

SiC shell conformity (1100°C) ?

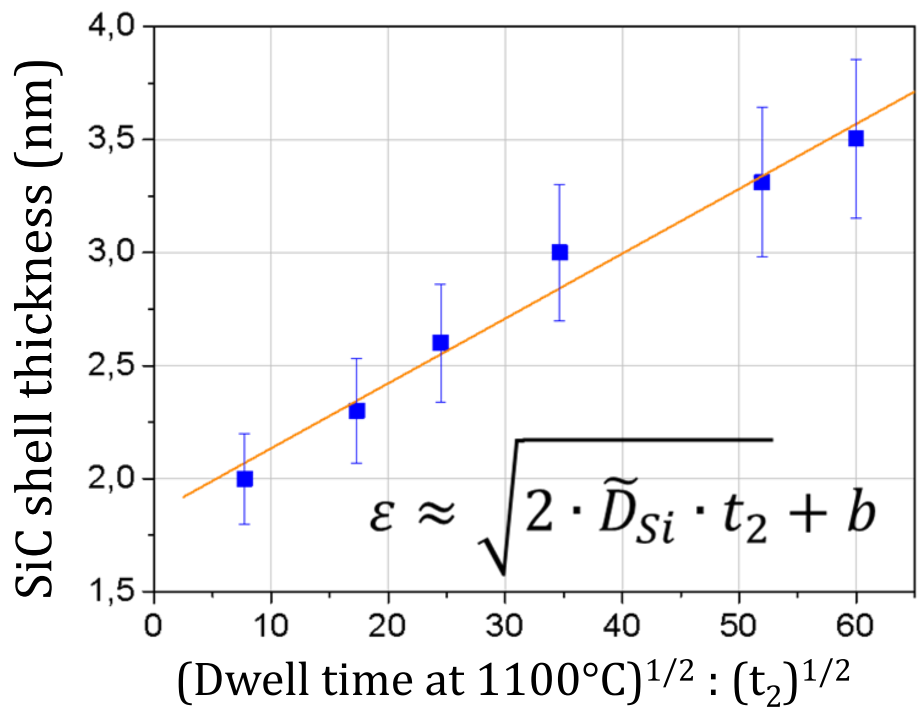




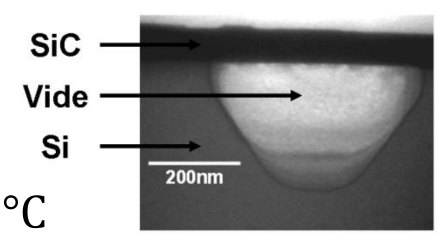
1) Identical morphology after carburization
2) The SiC shell is conform on all the surfaces



Kinetic study

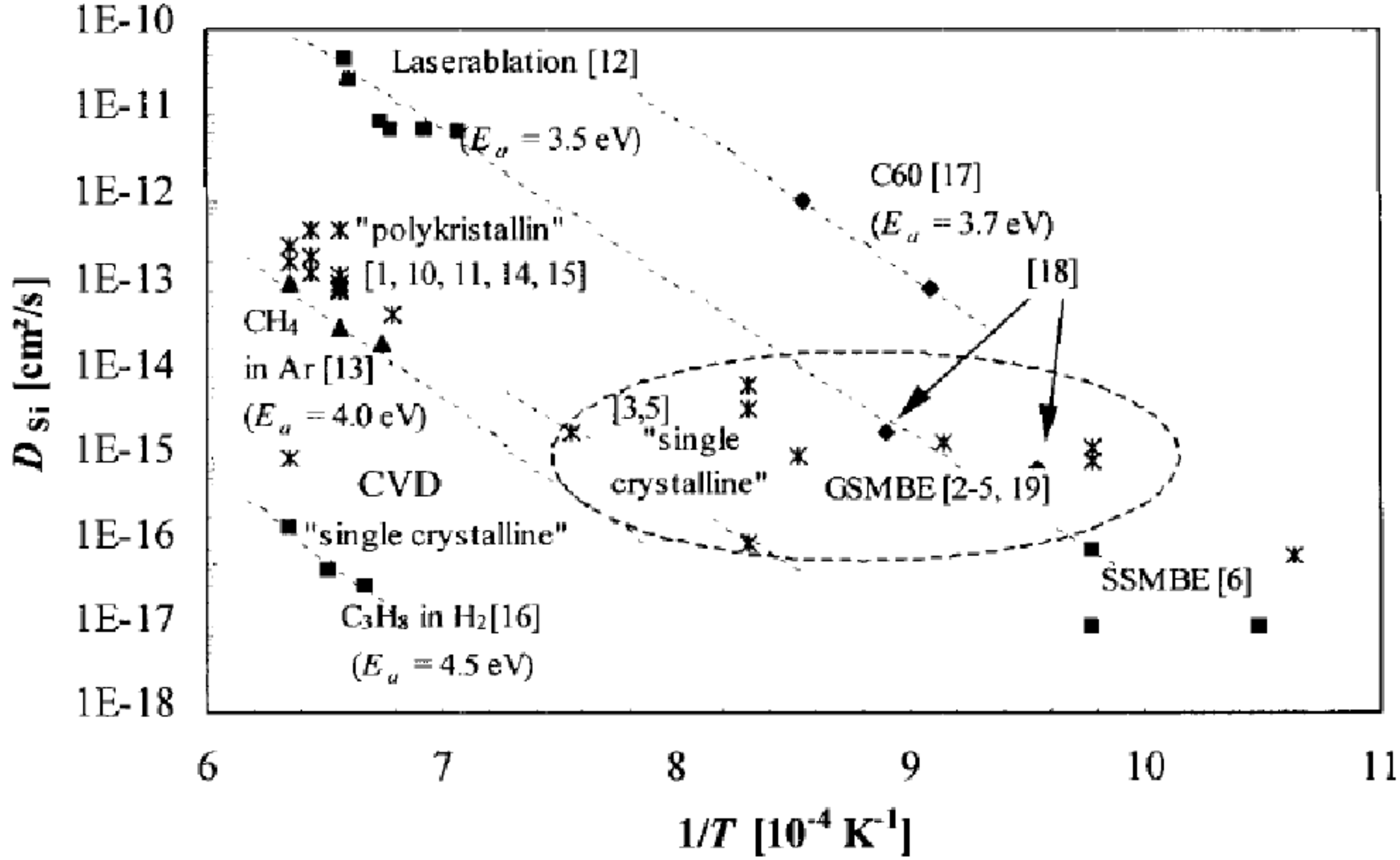


Einstein-Smoluchowski Relation: ε= $\sqrt{2Dt}$
The carburization is limited by the diffusion
Which diffusion ? Si out-diffusion through SiC*
D_{Si}= 3,7.10^{-18} cm^2.s^{-1} at 1100°C.
b~1,9 nm seed layer during methane injection from 800°C to 1100°C
At this nanometer scale, Fickian diffusion.

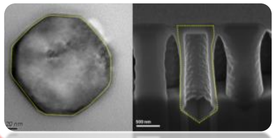


** G. Ferro HDR, Université Claude Bernard - 2006*

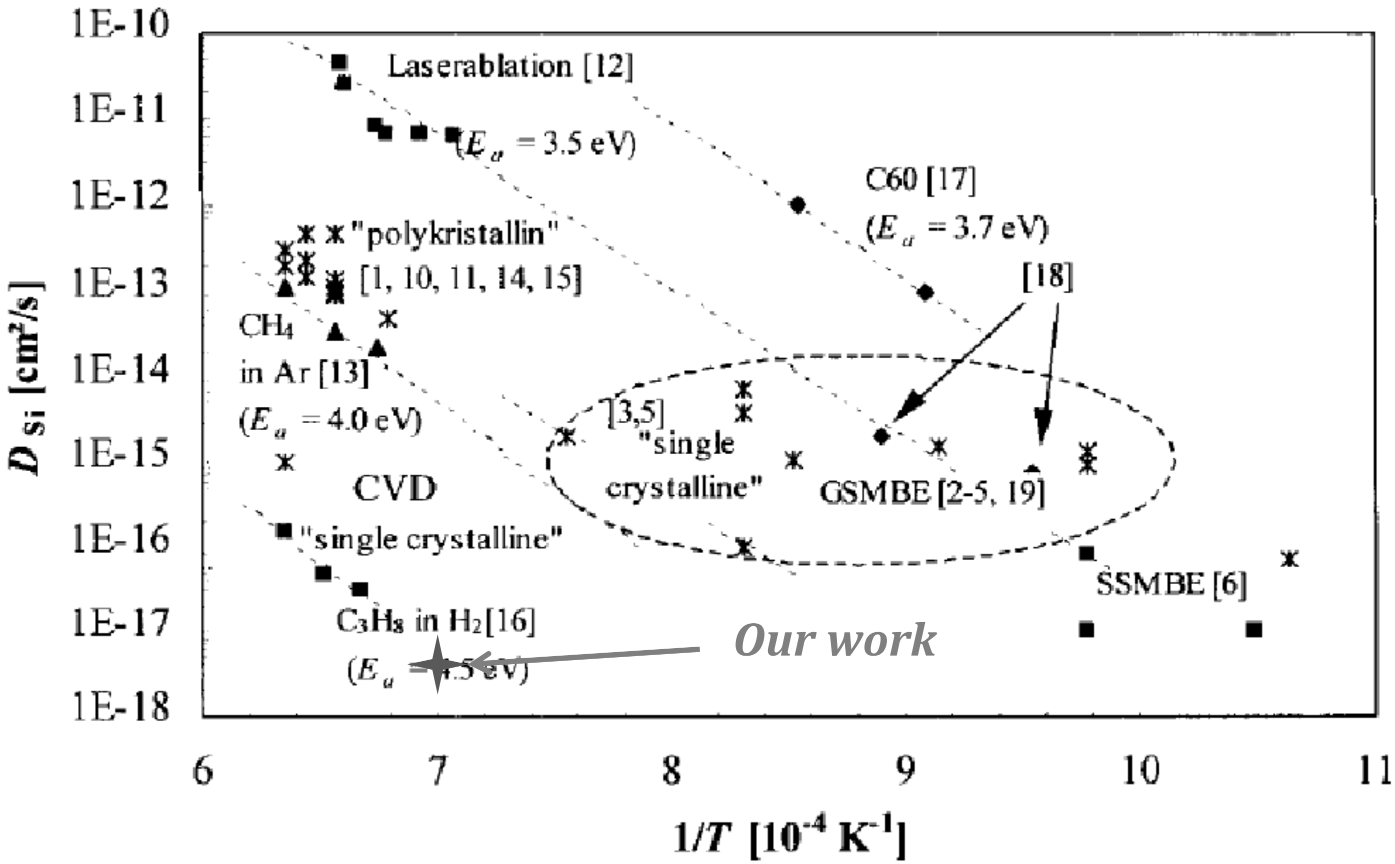
Kinetic study



From V. Cimalla et al. Materials Science Forum, Silicon carbide and related materials, 321–324, 2000



Kinetic study



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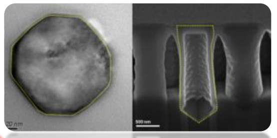
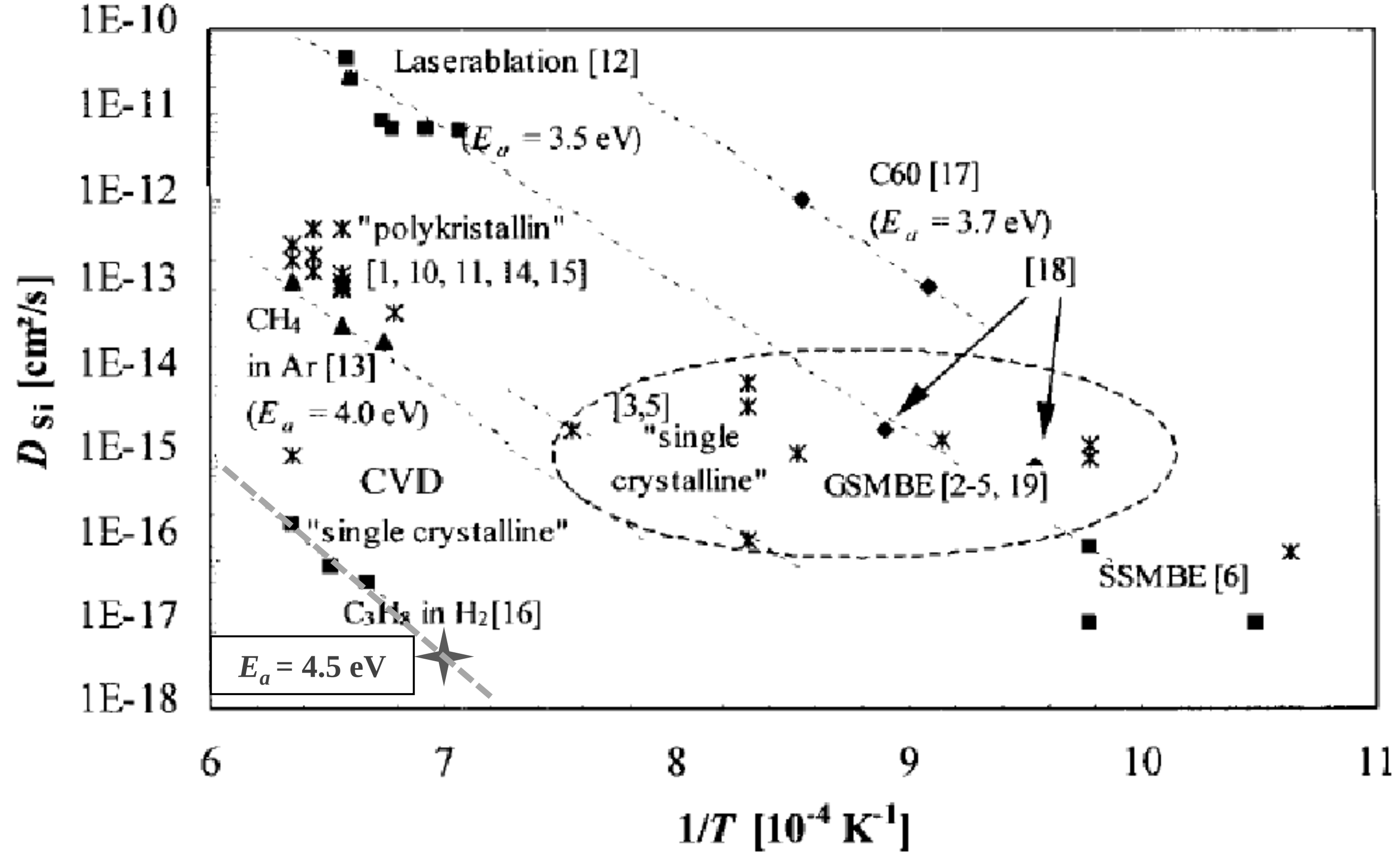
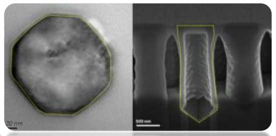




From V. Cimalla et al. Materials Science Forum, Silicon carbide and related materials, 321–324, 2000

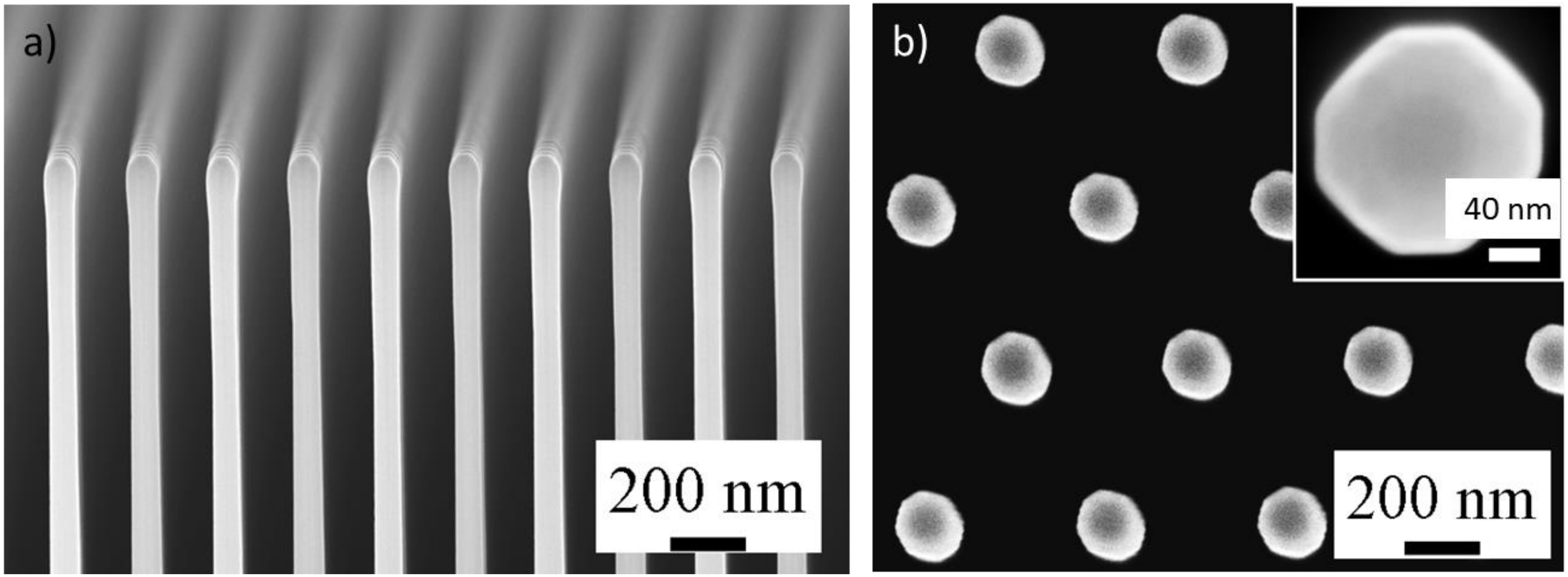
Si out-diffusion in a CVD growth regime of a single crystalline SiC deposit

Si-SiC core-shell nanowires



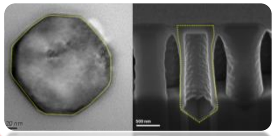
High purity carburization and epitaxy set-up
Collaboration with G. Ferro and V. Soulière (LMI, Lyon)

After heating-up (2°C.s^{-1}, D_{H_2}~ 16L.min^{-1}), Si nanowires are smooth and facetted,

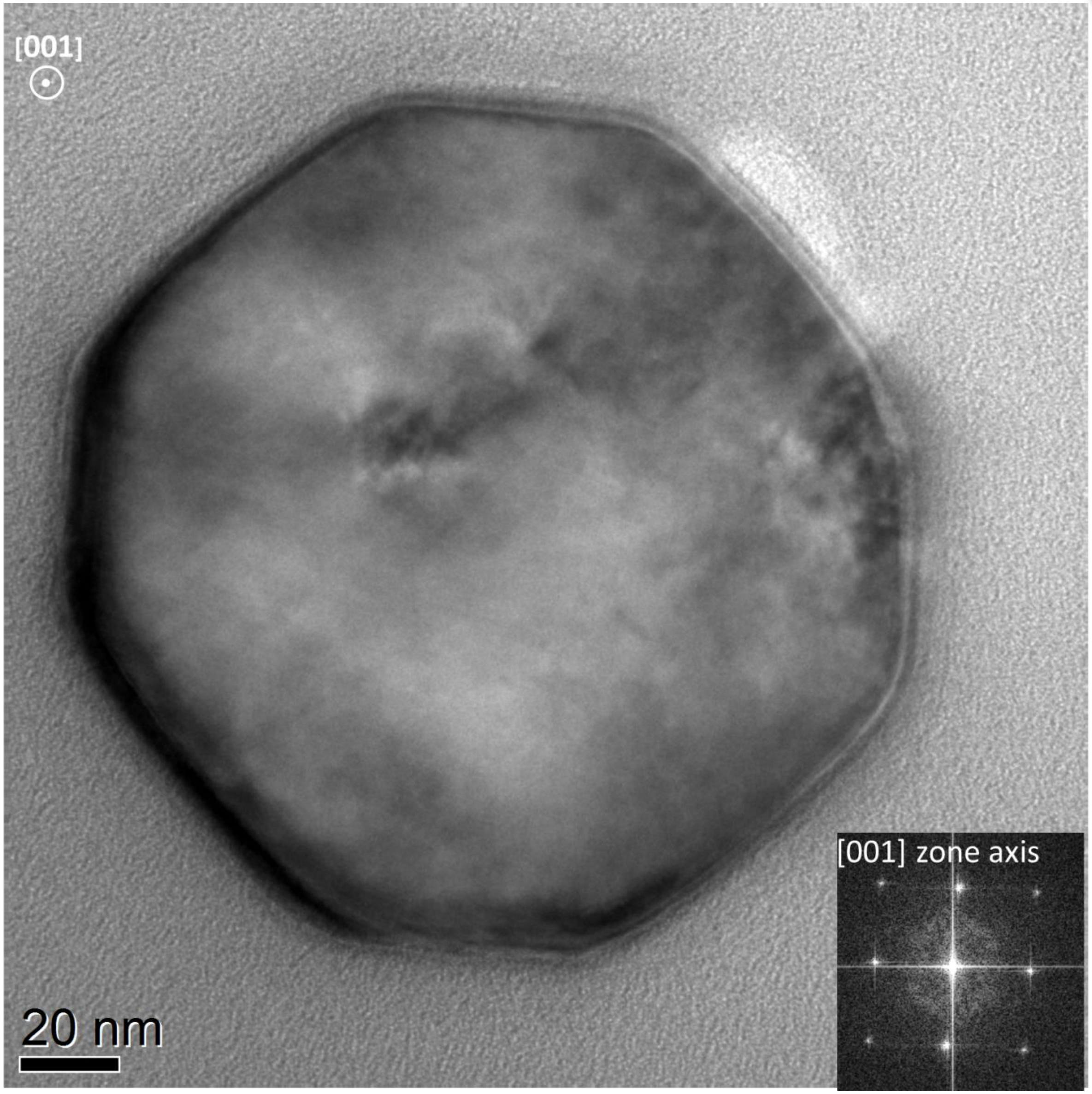


and makes appear an octogonal cross section.

Si-SiC core-shell nanowires



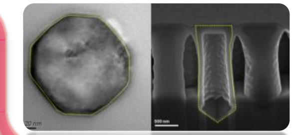
After carburization:



TEM image of a longitudinal cross section observed in [001] zone axis

Single crystalline SiC shell of 2 nm

Si-SiC core-shell nanowires



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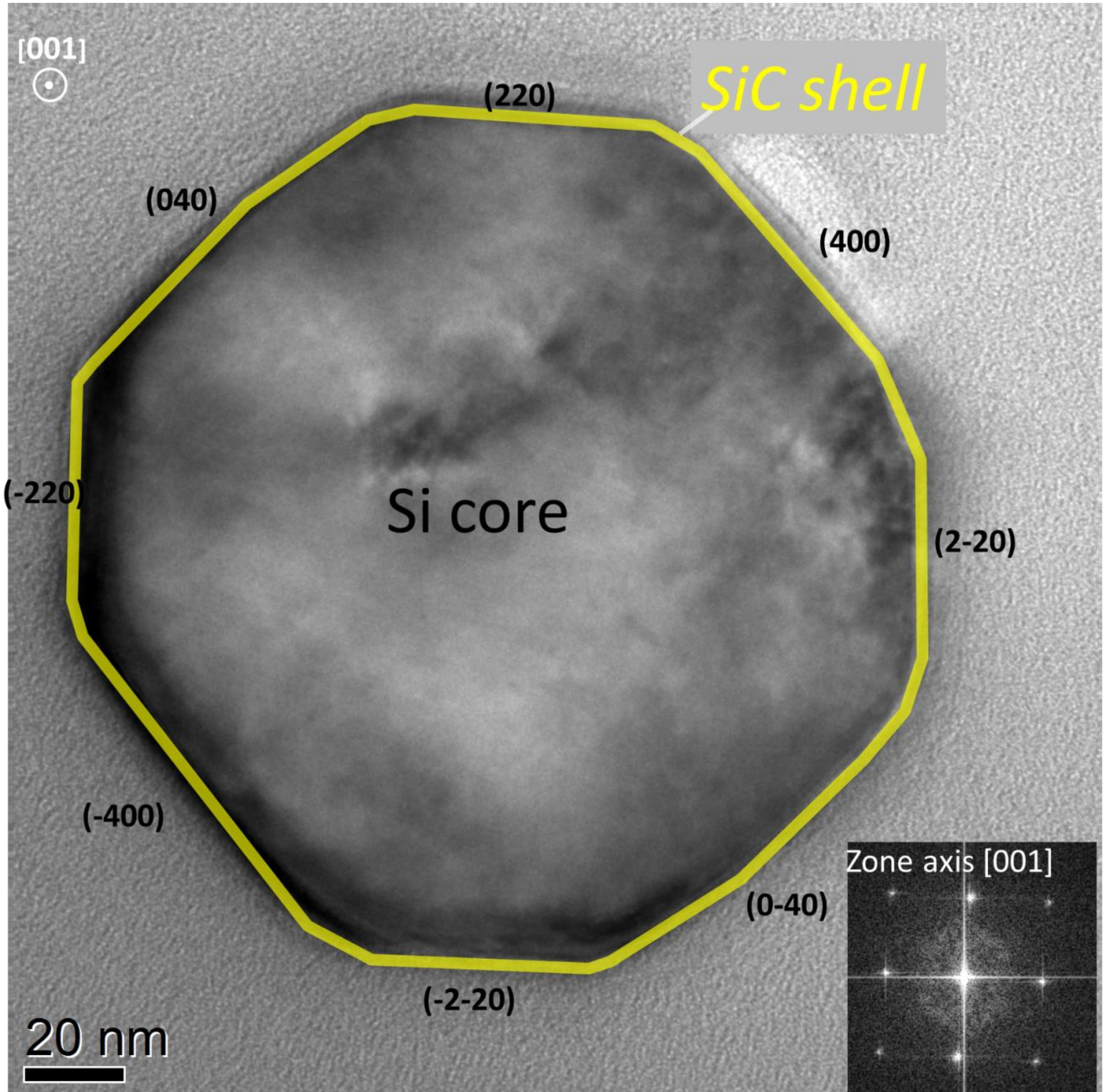
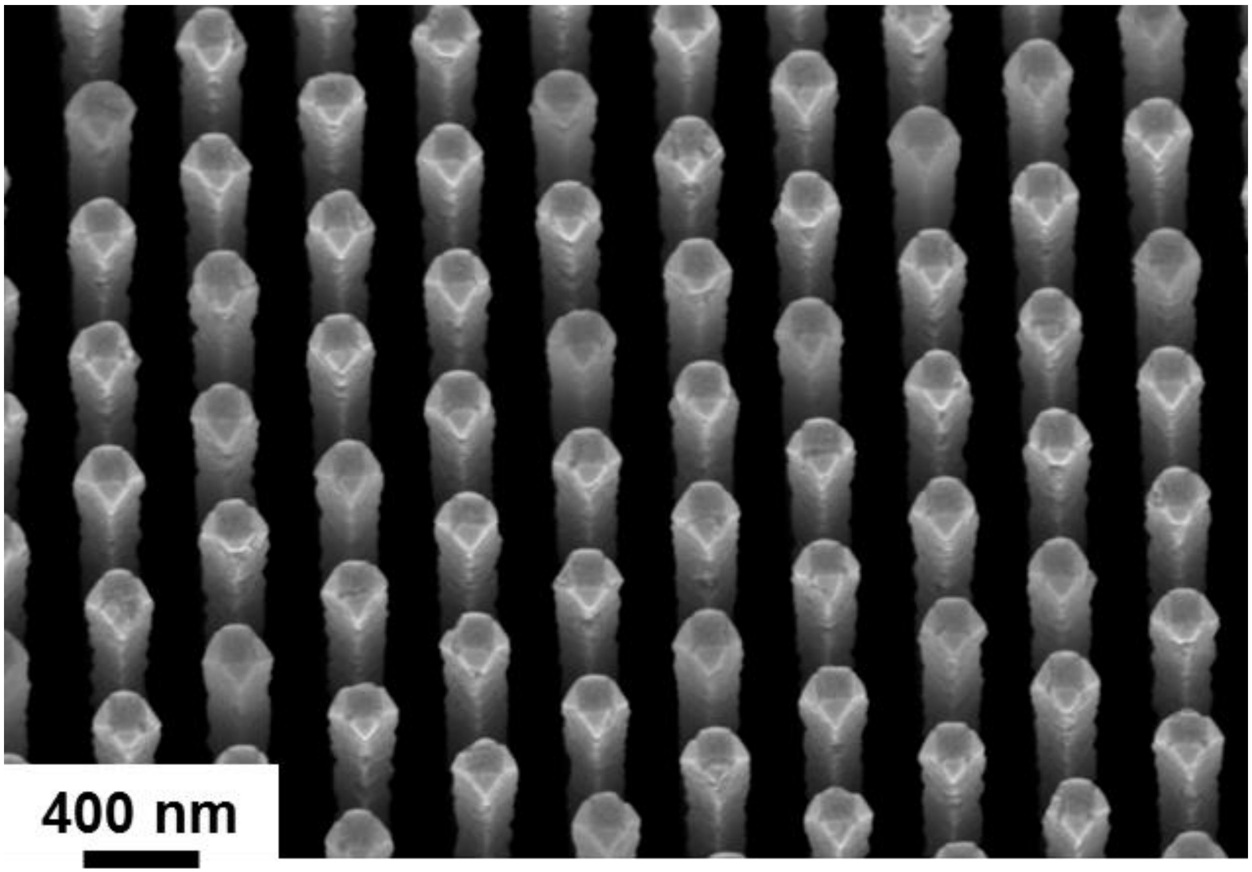


Single crystalline SiC shell of 2 nm on {400} and {220} planes.
{400} planes are longer than {220} planes.

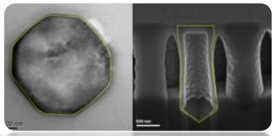
Si-SiC core-shell nanowires

After 3C-SiC epitaxy:

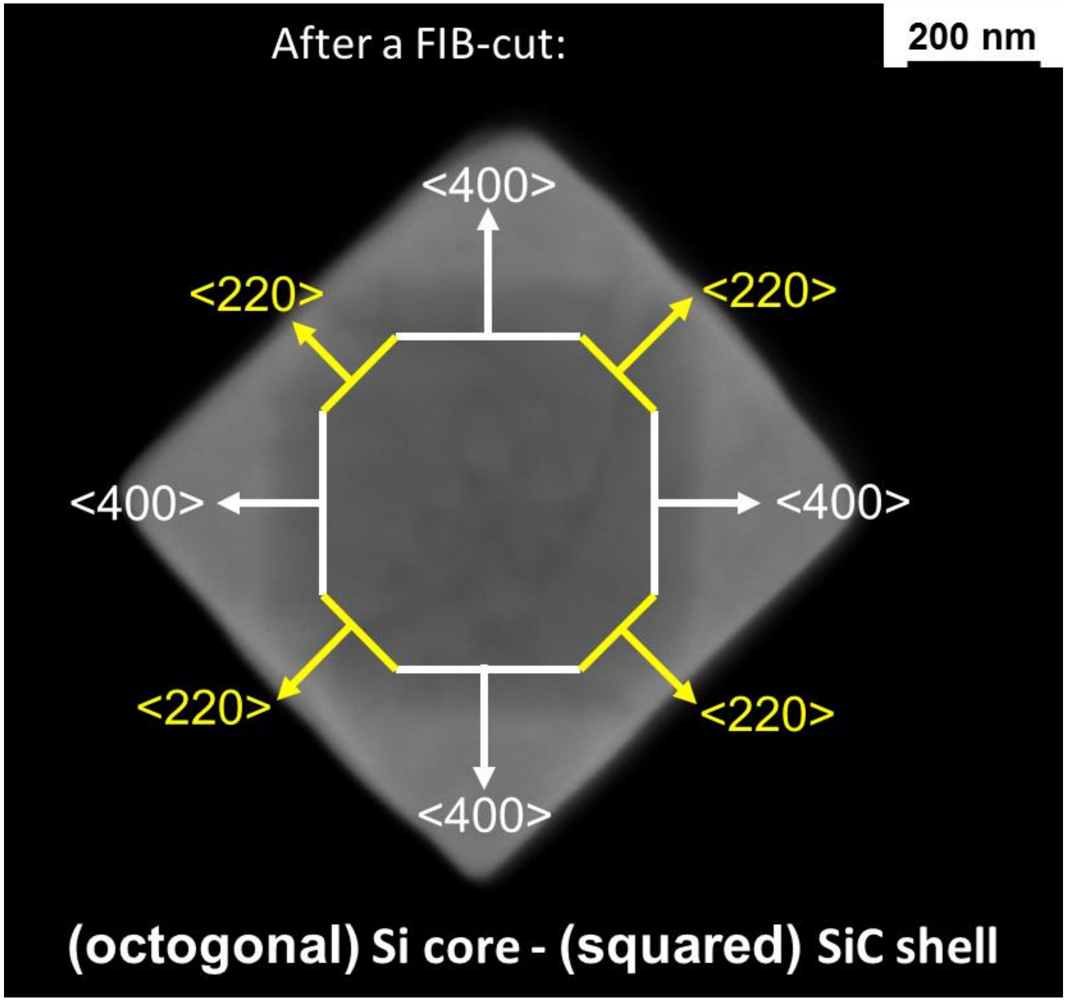


SEM image of Si-SiC nanowires

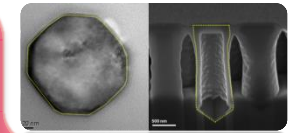
Si-SiC core-shell nanowires



After 3C-SiC epitaxy:



Growth of 3C–SiC on carburized Si NWs is favored toward <400> directions at the expense of the <220> ones.
SiC lateral growth anisotropy of 6 between these two directions.

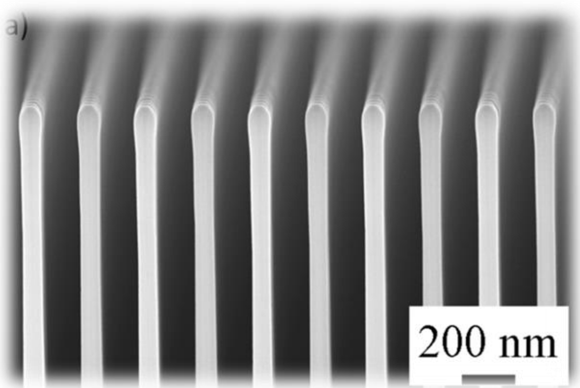


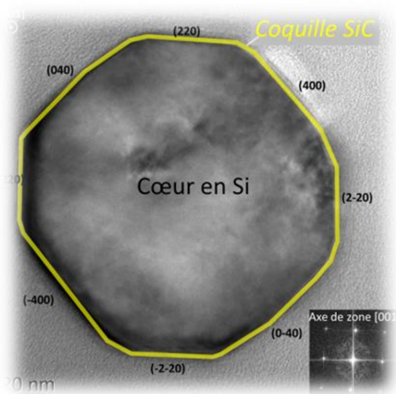
Si-SiC core-shell nanowires

Carburization of Si NWs priory obtained by plasma etching allows to demonstrate:

- Smooth and facetted sidewalls (H_2 fast heating-up)
- Si-SiC nanowires with a crystalline quality never observed before:
  single crystalline core / single crystalline shell
- Moreover, the doping of the Si core is controlled.

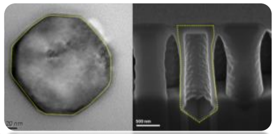
An interesting object for the bio-detection…





M. Ollivier, L. Latu-Romain et al. Journal of Crystal Growth 363, 158-163, (2013)

SiC nanotubes



SiC nanotubes are very interesting…

T.A. Hilder et al. Silicon Carbide Nanotube as a Chloride-Selective Channel.
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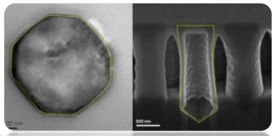

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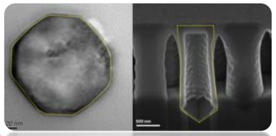
By increasing Si out-diffusion trough SiC (Kirkendall effect).



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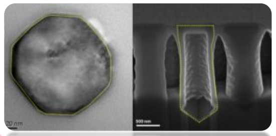

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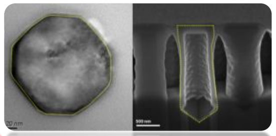
Idea: Via carburization pressure

SiC nanotubes



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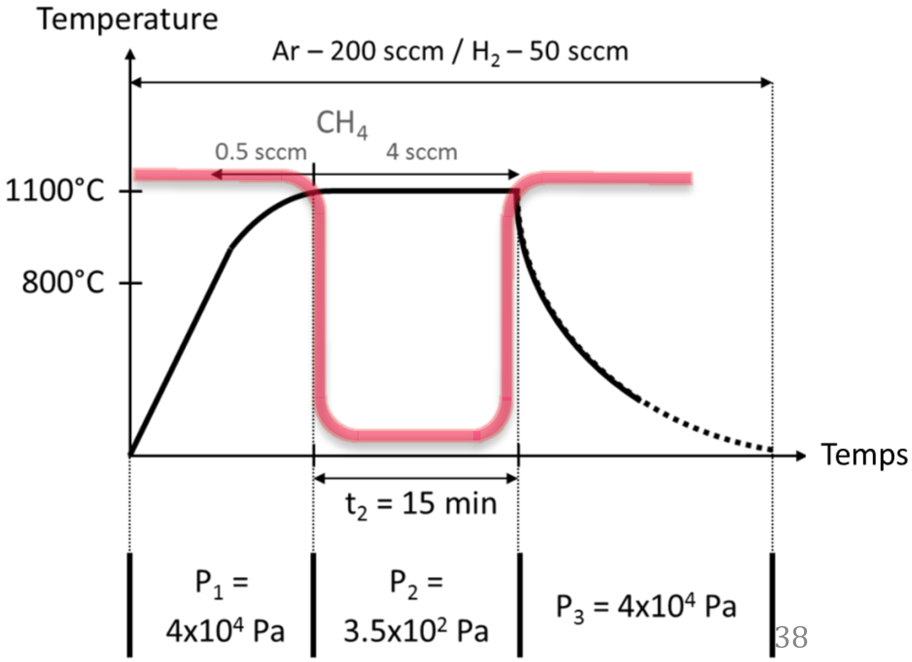
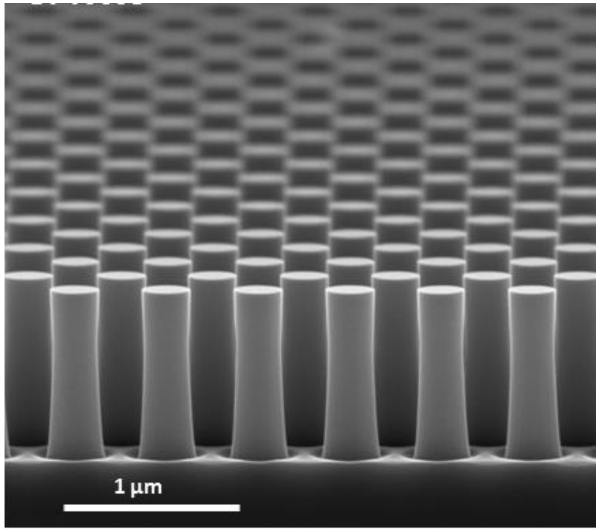
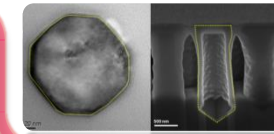
Carburization experiment in three parts:



SiC nanotubes

Si nanowires
Initial diameter: 280 nm





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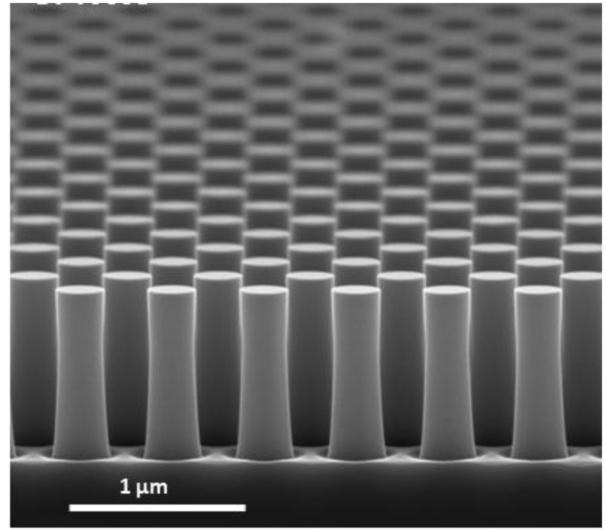
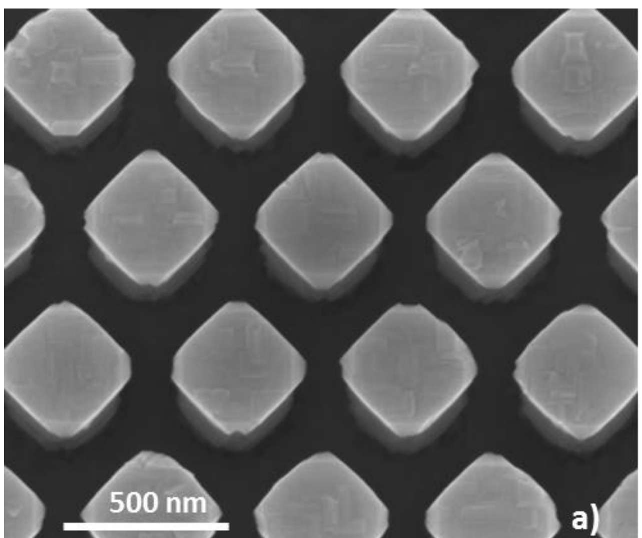
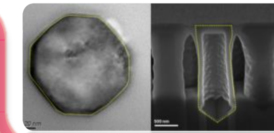


After carburization :



Nanostructures with a squared cross-section



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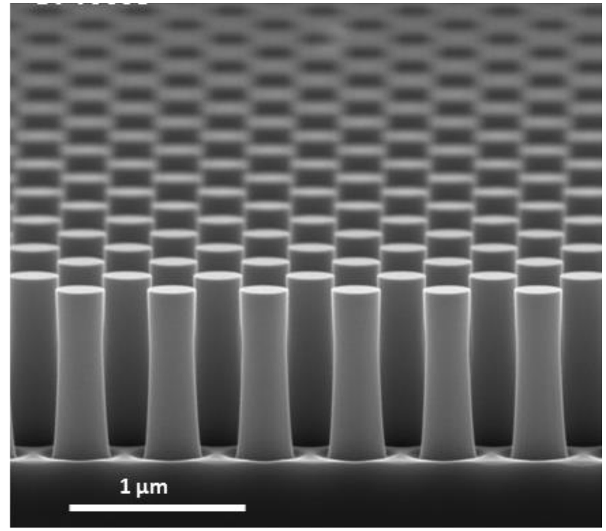

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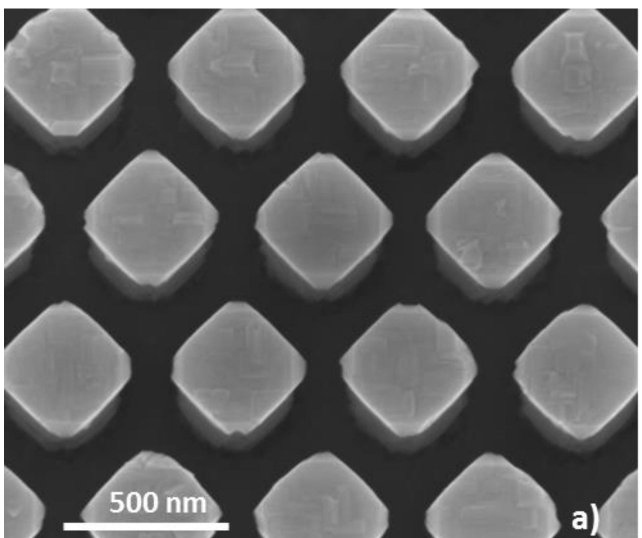
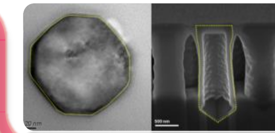


Cut with the FIB-SEM

Nanostructures with a squared cross-section



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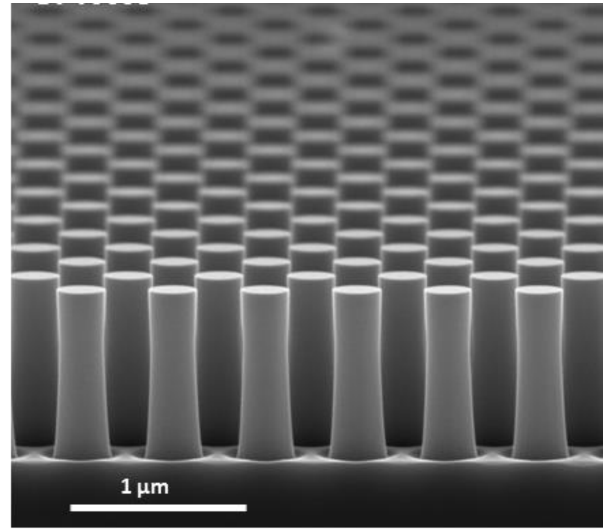
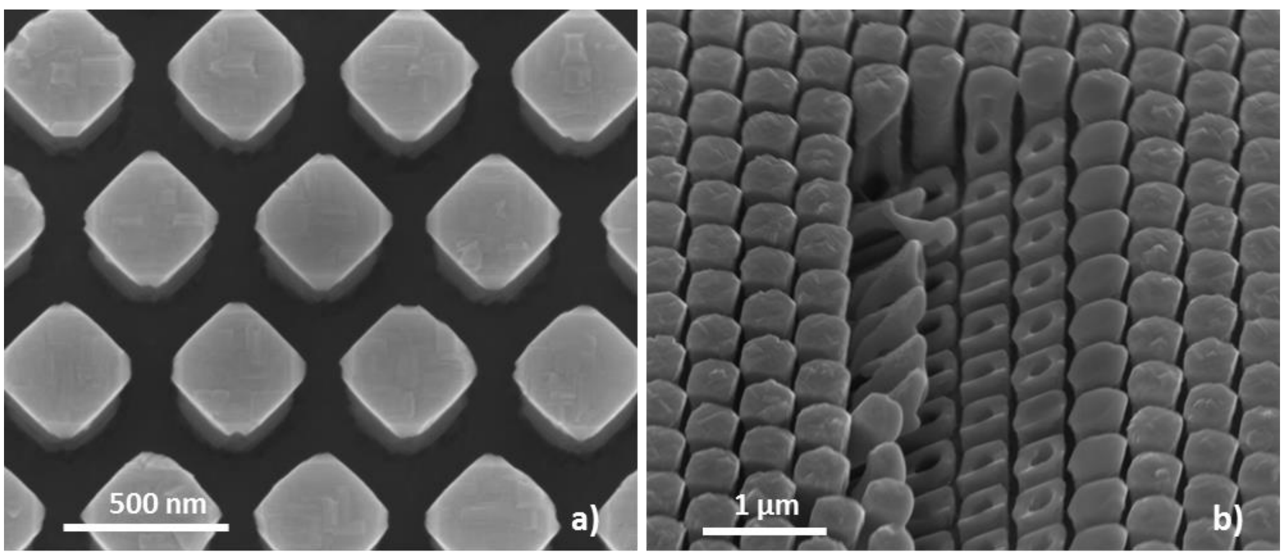


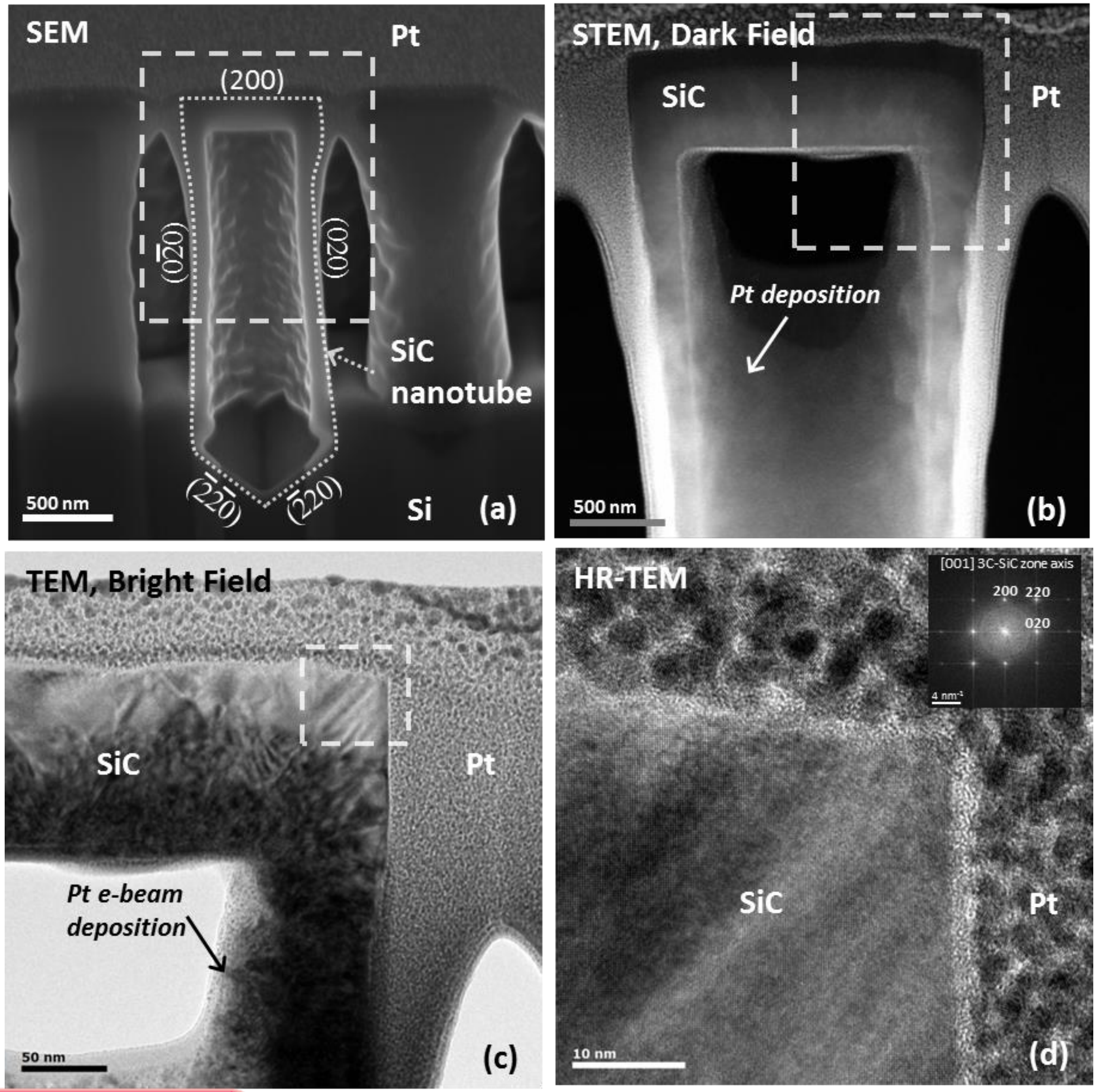
After carburization :



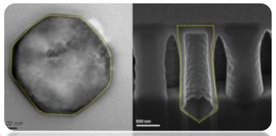
Empty nanostructures with a squared cross-section!

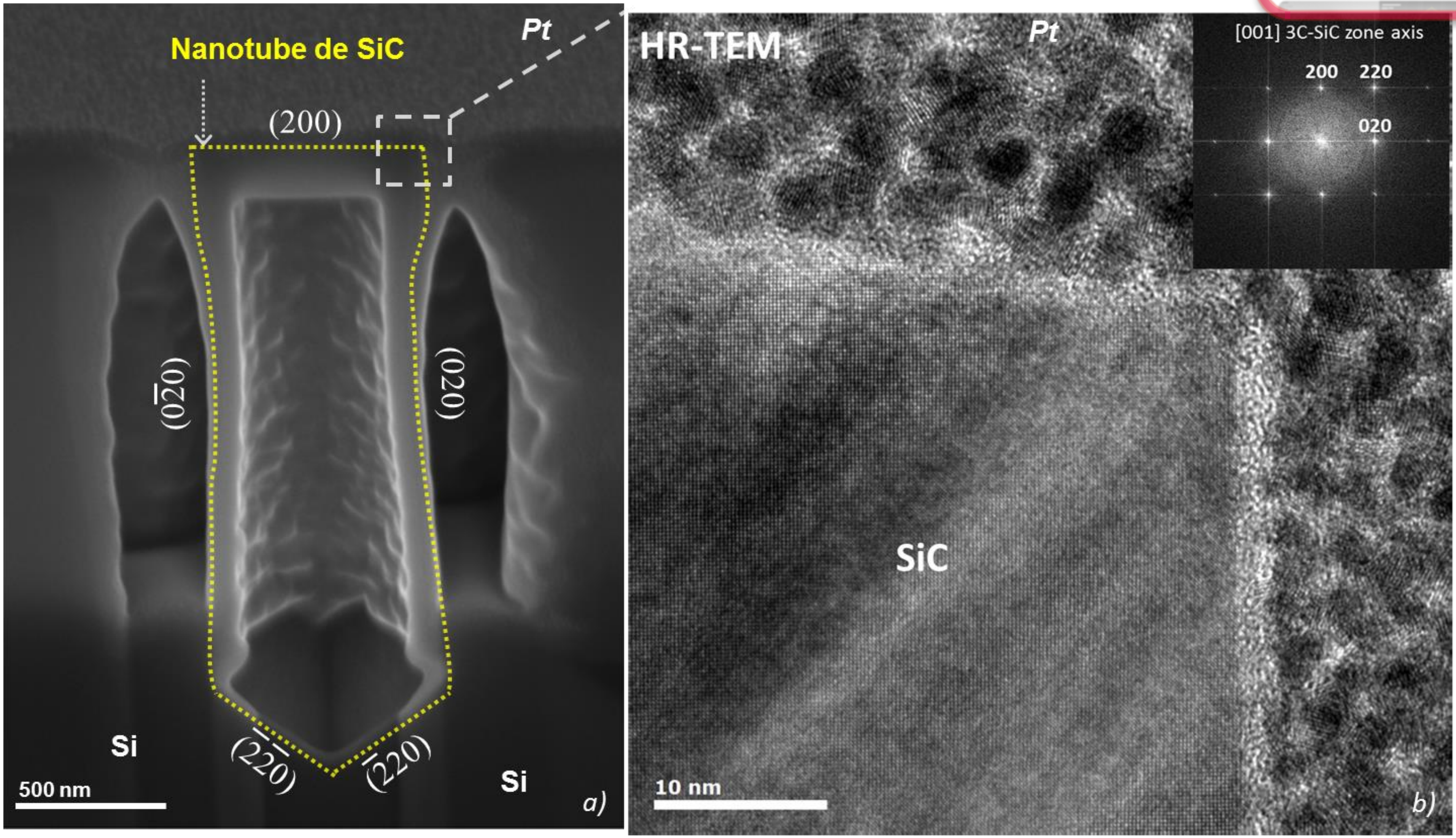
SiC nanotubes

TEM observation of transversal cross section of the nanotube:



SiC nanotubes

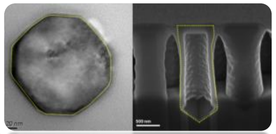


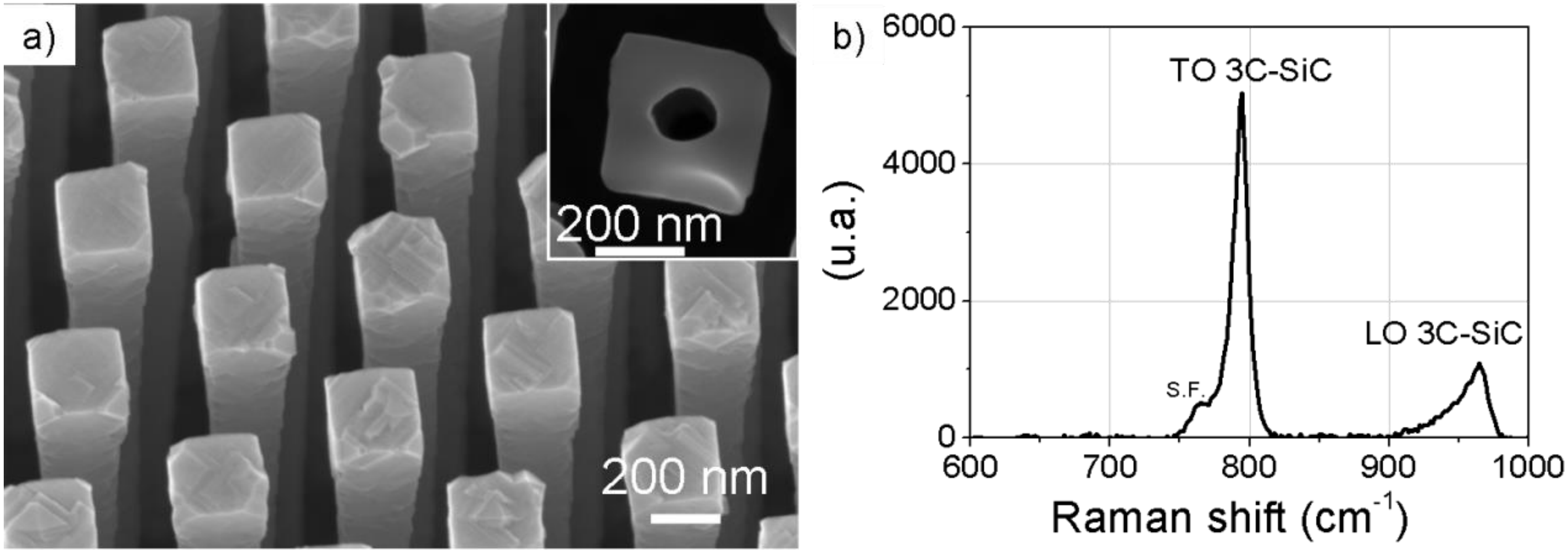


3C-SiC nanotubes with an external diameter ~ 300 nm with {200} facetted and dense sidewalls (40-100 nm)

L. Latu-Romain, M. Ollivier et al.
Journal of Physics D: Appl. Phys. 46, Fast Track Comm 092001 (2013) ***Highlights 2013***

SiC nanotubes

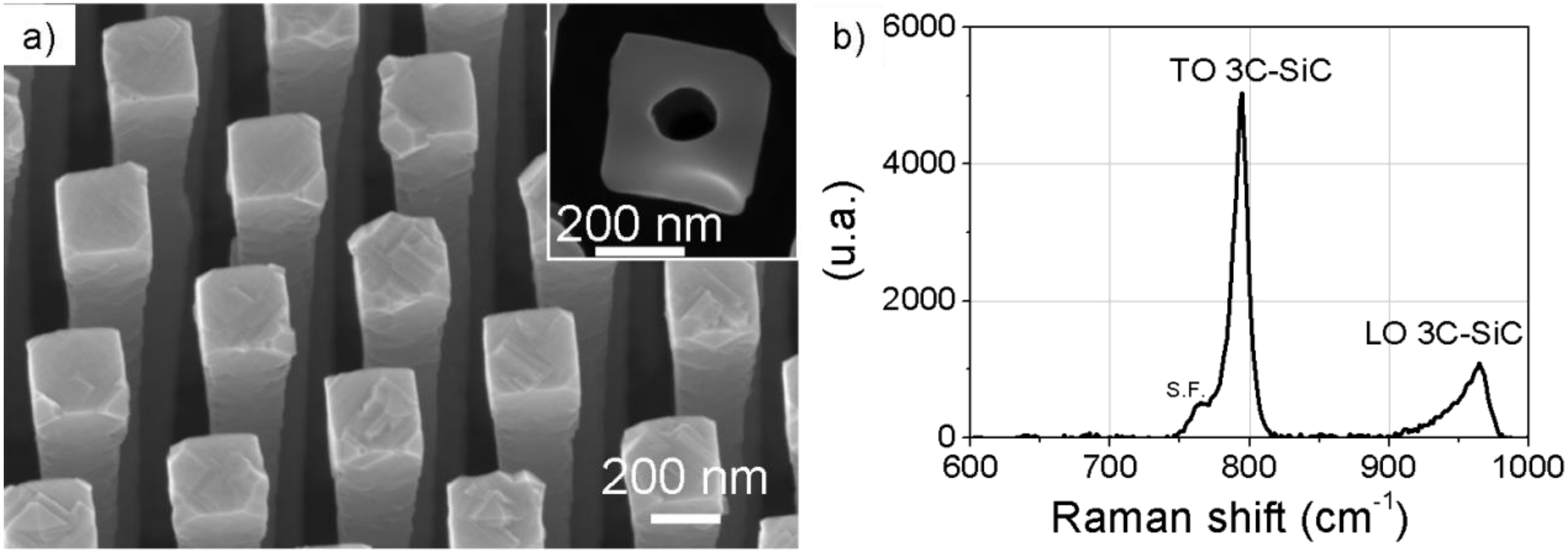




a) Long SiC nanotubes de SiC and in inset SEM image after a FIB cut.
b) Raman Spectrum of 3C-SiC nanotubes (514, 5 nm ; 0,1 mW). Stacking faults can be put in evidence.

- Long SiC nanotubes can also be obtained: {200} dense sidewalls 3C-SiC nanotubes.

SiC nanotubes



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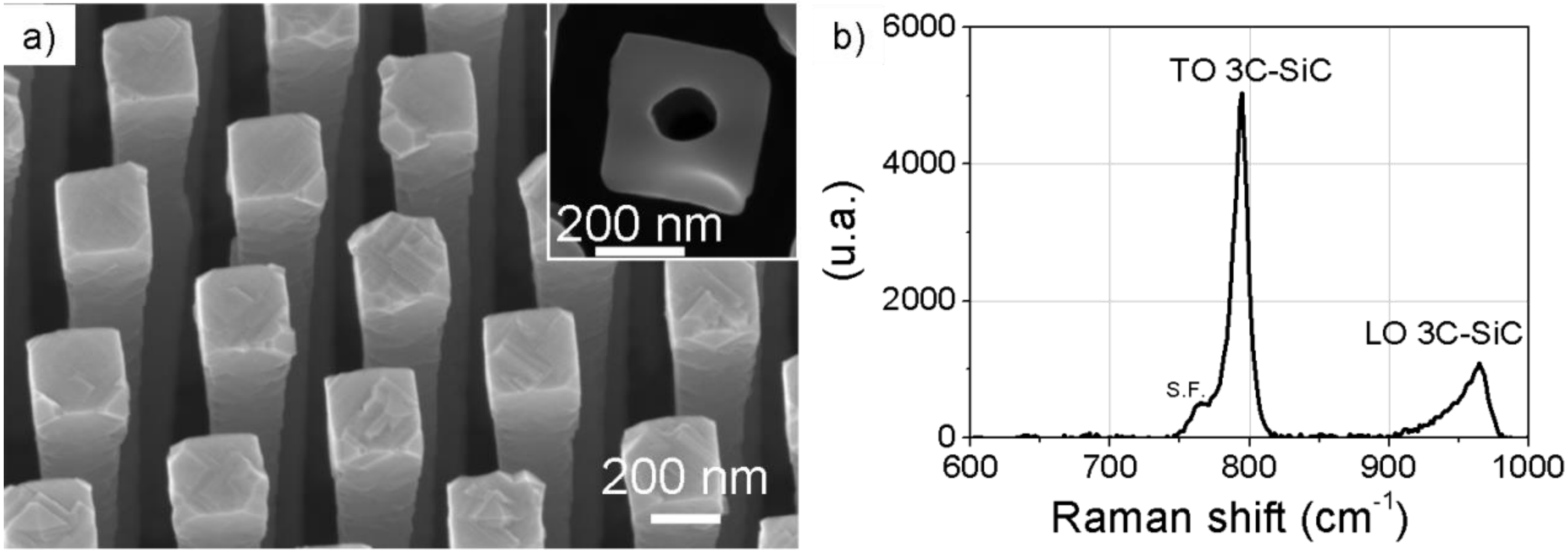
- Si out-diffuses through SiC ! Same limiting step.

SiC nanotubes



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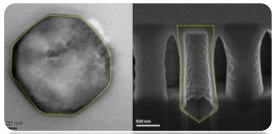
- Long SiC nanotubes can also be obtained:

{200} dense sidewalls 3C-SiC nanotubes.

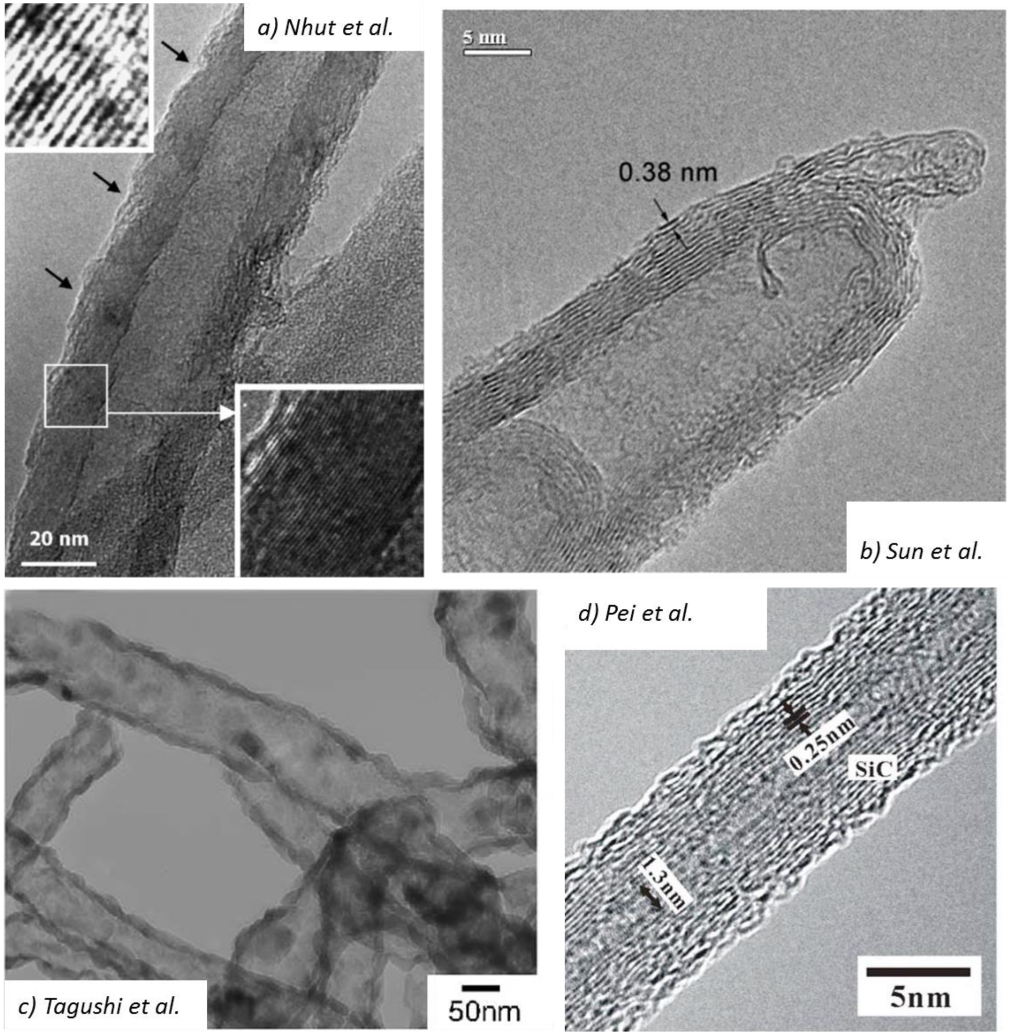
- Si out-diffuses through SiC ! Same limiting step.
- Carburization pressure allows to control the out-diffusion speed (chemical potential gradient).

State of the art -Discussion



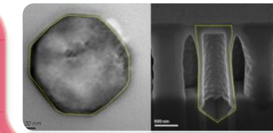
In the litterature, C NT siliciuration leads to SiC NWs and SiC NTs.

Porous sidewalls.

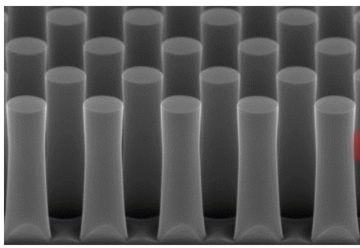


J.M. Nhut et al. Catalysis Today, 76(1), 11–32, (2002)
T. Taguchi et al. Physica E: Low-dimensional systems and nanostructures, 28(4), 431–438, (2005)
X.H. Sun et al. Journal of the American Chemical Society, 124,14464–14471, (2002)
L.Z. Pei et al. Journal of Applied Physics, 99, 114306, (2006)

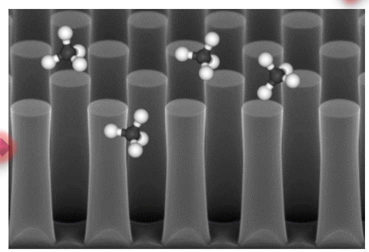
By carburization:
Control of the size with dense sidewalls
Good crystalline quality



Carburization mechanisms

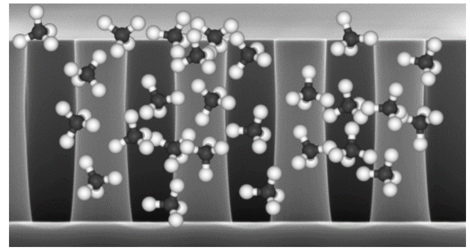


Silicon nanowire

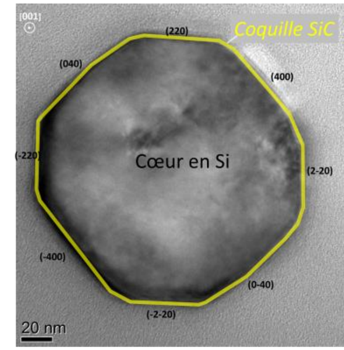


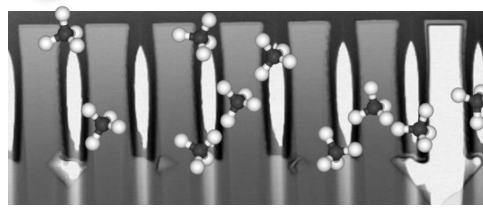
Carburization from 800°C to 1100°C at Patm: growth of very thin SiC layer (2 nm)

Carburization at 1100°C and Patm, limited by Si out-diffusion

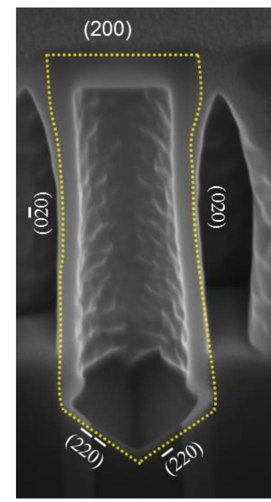


Single crystalline Si-SiC core-shell nanowire
(3 nm thick and continuous shell)



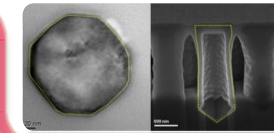


Carburization at 1100°C and and at a low pressure, Si out-diffusion is increased



Cubic SiC nanotube with dense sidewalls and a controlled size.

Existence diagram



To the generalization of growth mechanisms

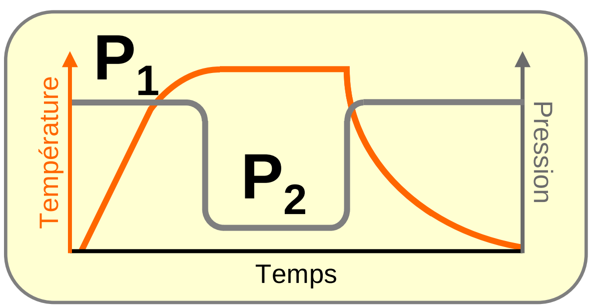
Existence diagram:

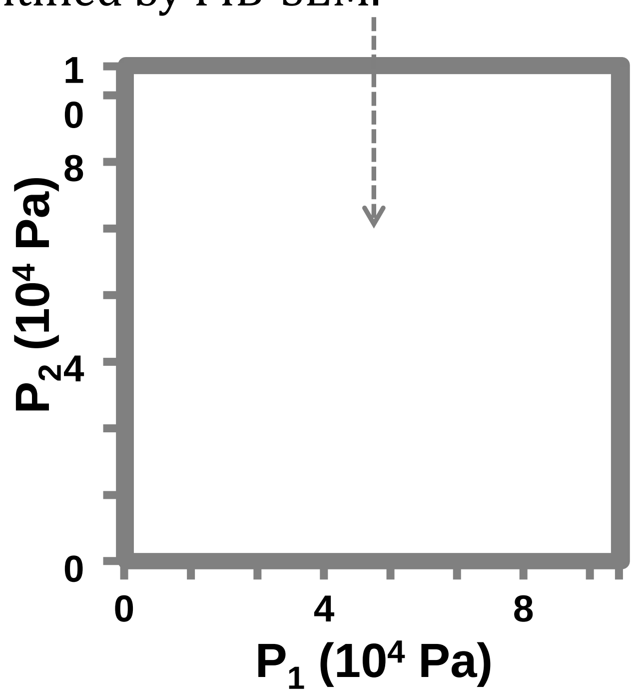
X Axis: pressure during the heating temperature, P_1

Y Axis: pressure during the carburization dwell, P_2

For carburization experiments at 1100°C during 15 minutes with 200 nm diameter Si nanowires.

The kind of nanostructure is identified by FIB-SEM.





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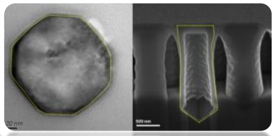
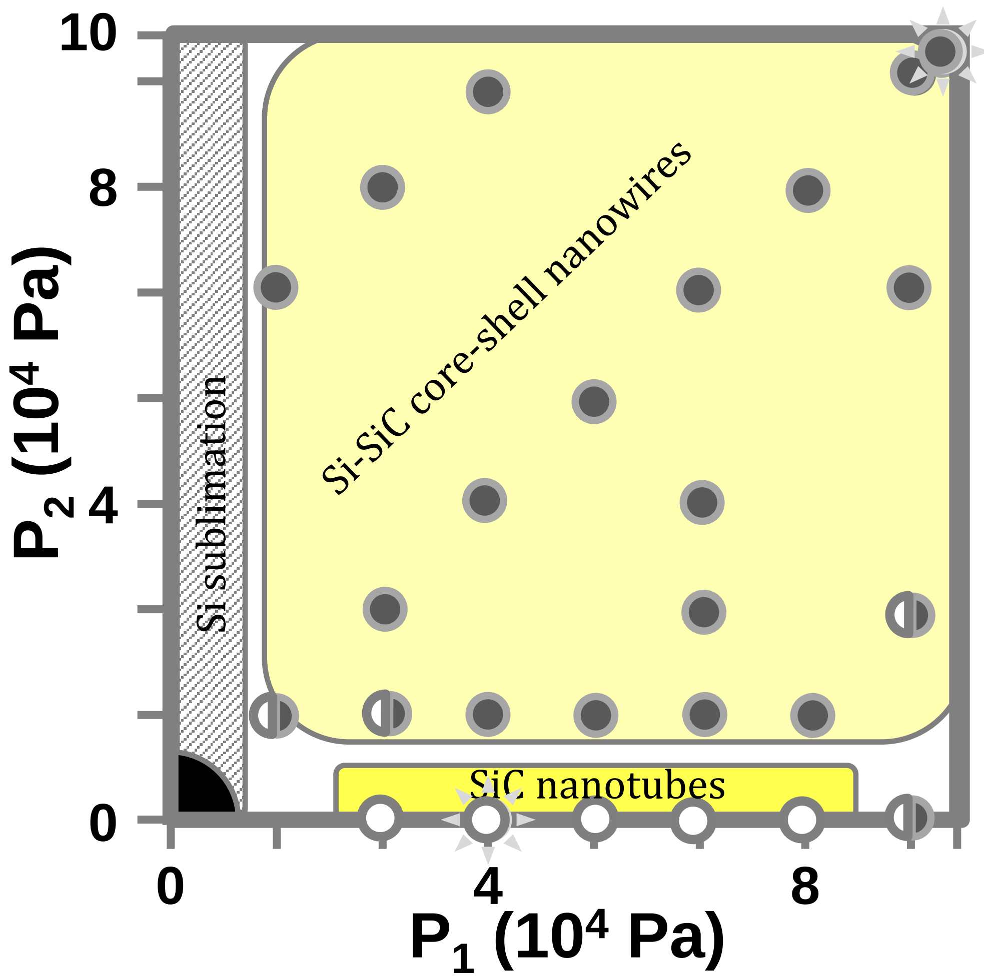
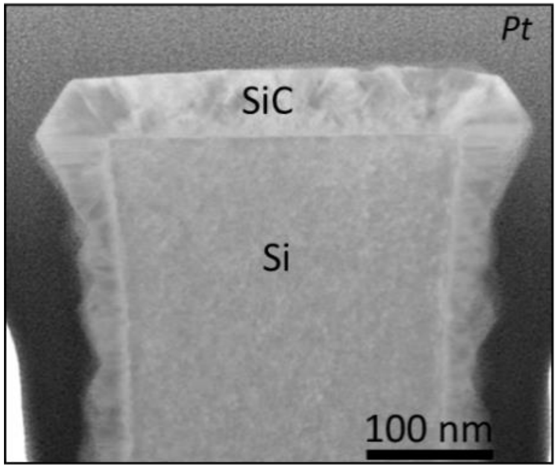






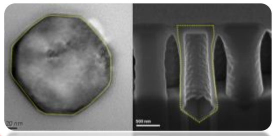
Transversal STEM image of a Si-SiC core-shell NW (P_1,P_2)

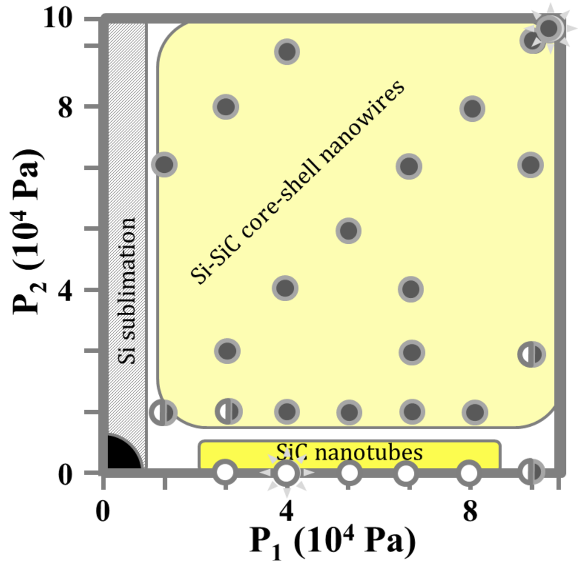
i) If P_1 and P_2 are high: Si-SiC core-shell nanowires

ii) If P_1 high and P_2 low: SiC nanotubes

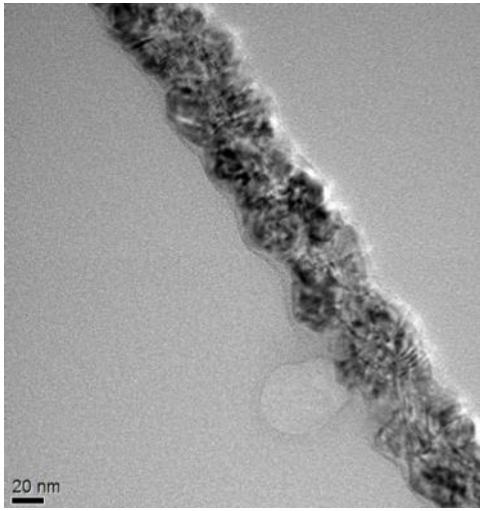
L. Latu-Romain, M. Ollivier, Journal of Physics D: Appl. Phys, topical review 47, 203001 (18pp) (2014)

SiC nanowires





The existence domain of SiC nanowires is necessarily very narrow



TEM image of a SiC nanowire with 50 nm diameter:
Complete carburization of a Si nanowire.

Growth direction:<111>
The SiC NW contains a high density of structural defects.

The way to obtain single crystalline SiC NW remains a challenge…
An alternative has to be proposed.

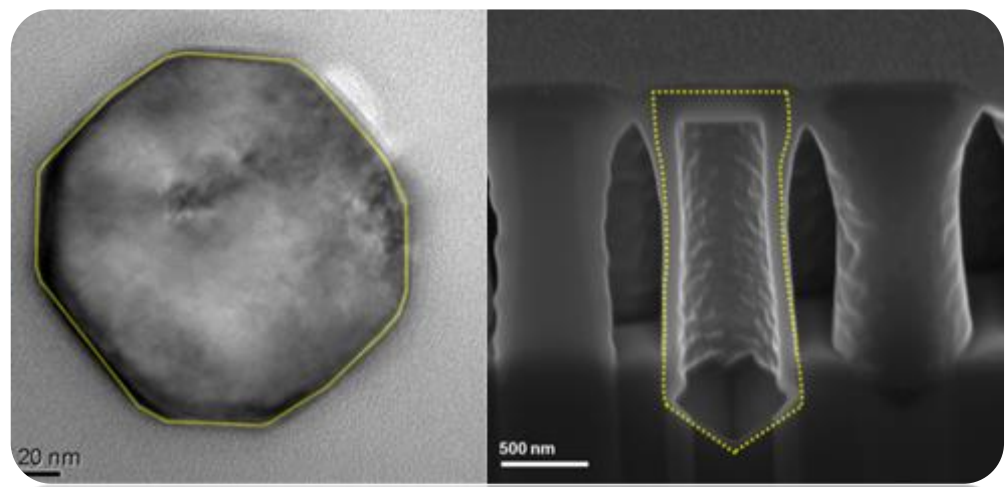
Si nanowires carburization

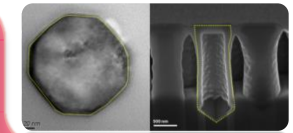
Two original nanostructures:

-single crystalline Si-SiC core-shell nanowires, with a doping control of the Si core,
-SiC nanotubes with a high crystalline quality and dense sidewalls

With controlled sizes (external diameter, lenght, spacing of the network).

Carburization pressure modifies the chemical species concentration gradient.
This is the key parameter to tune the Si out-diffusion through SiC.

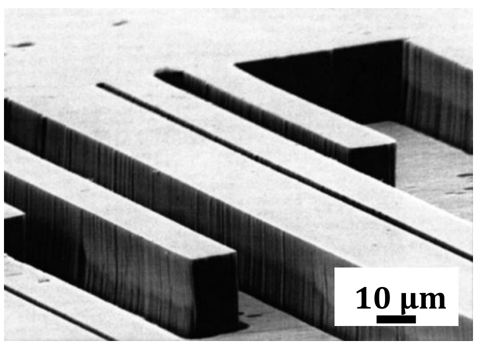




Alternative Approach

Bulk and epitaxial growth of silicon carbide has made a lot of progress, single crystalline 4H- and 6H-SiC wafers can be grown with a controlled doping level.

Top-down approach by plasma etching (« top-down approach »):
inductively coupled plasma etching in SF_6/O_2 plasma, already studied for micro-trenches fabrication (MEMS applications)



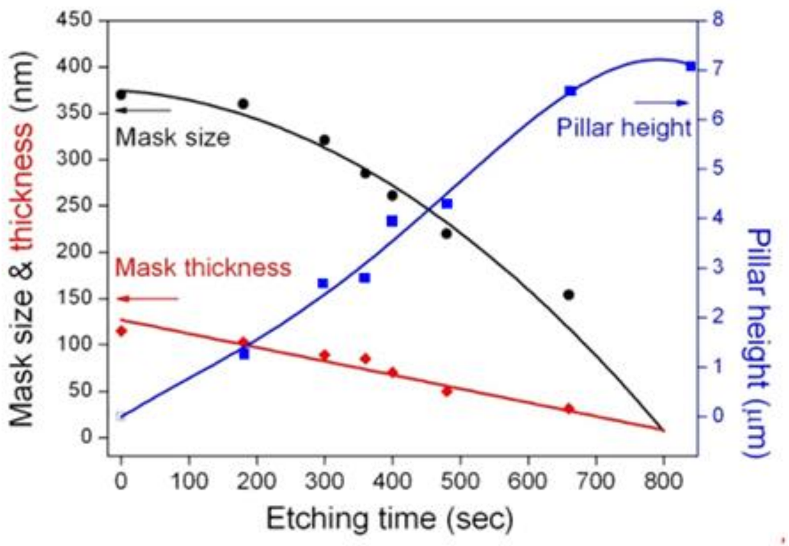
Kim *et al.* Thin Solid Films 447–448 (2004) 100

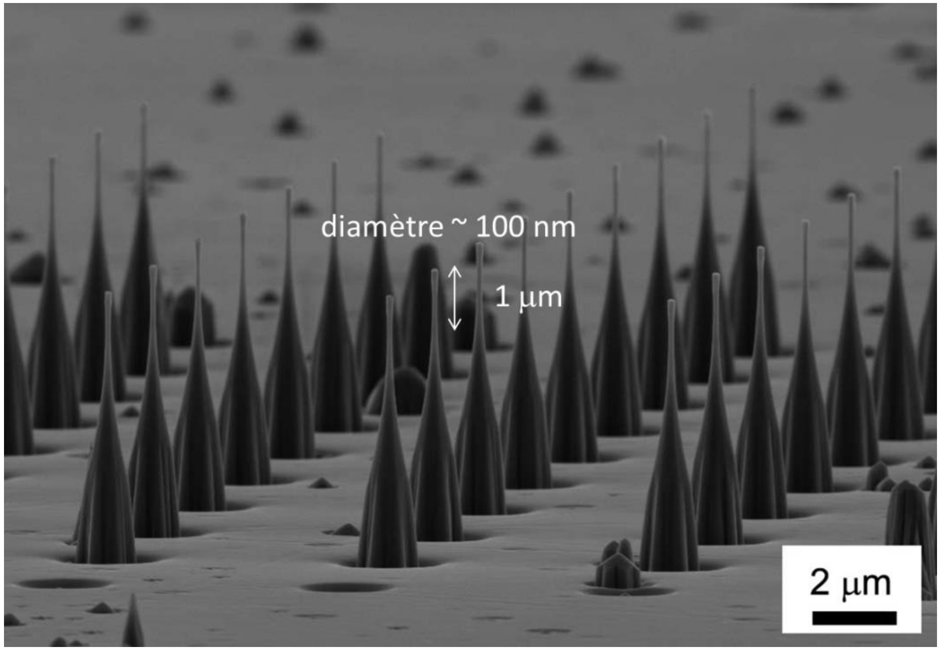
$$
\begin{aligned}
[SF_6] + e^- &\longrightarrow [SF_5^+] + [F] + 2e^- \\
\langle SiC \rangle + 8[F] &\longrightarrow [SiF_4] + [CF_4] \\
\text{ou} \quad \langle SiC \rangle + [SF_x^+] &\longrightarrow [SiF_x] + [CF_y] + [SF_z^+]
\end{aligned}
$$

Challenge? Etch the material in order to get a high aspect ratio with a small diameter …to approach the nanowire

Alternative Approach

Process parameters study (chemical nature of the mask–Cu, **Ni**, Al-, polarization, gas dilution…)



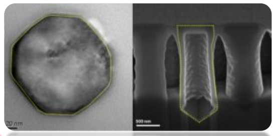


SiC nanopillars with an aspect ratio of about 7 (and a total length of 7 µm) and a diameter of about 100 nm on the latest 1 µm

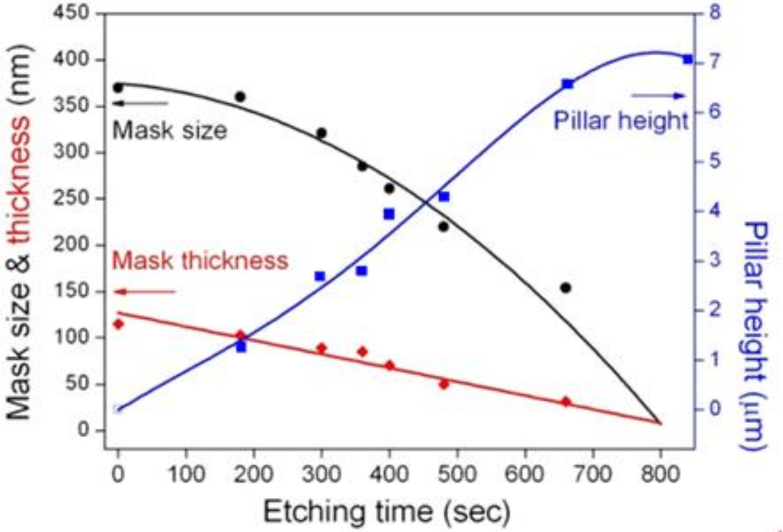
Object that can be integrated into a transitor…

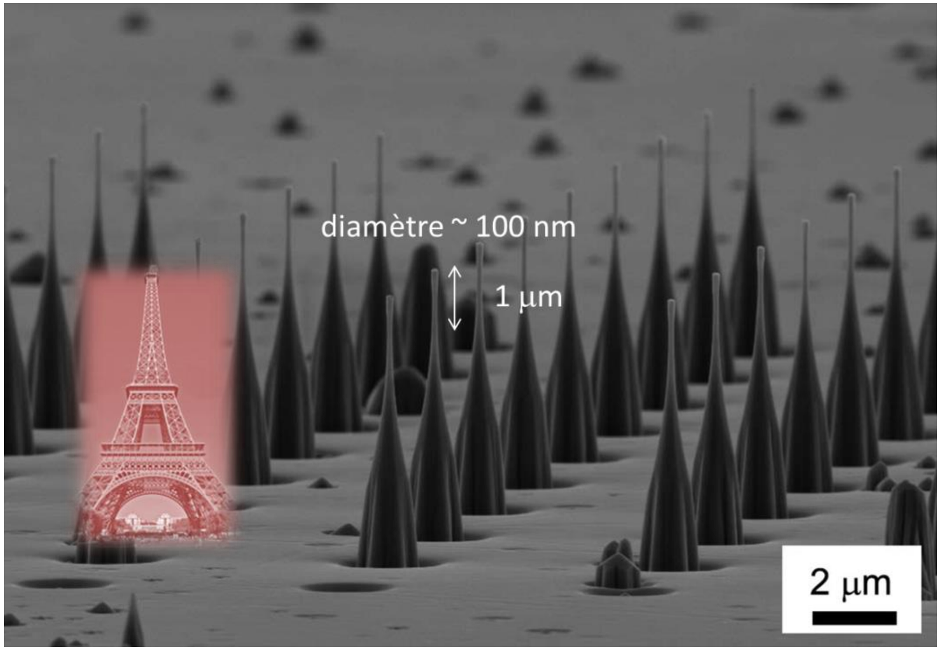
J.H. Choi, L. Latu-Romain, E. Bano et al. J. Phys. D: Appl. Phys., 45, 235204, (2012)

Alternative Approach



Process parameters study (chemical nature of the mask–Cu, **Ni**, Al-, polarization, gas dilution…)





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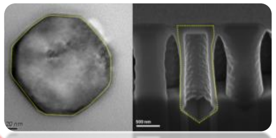
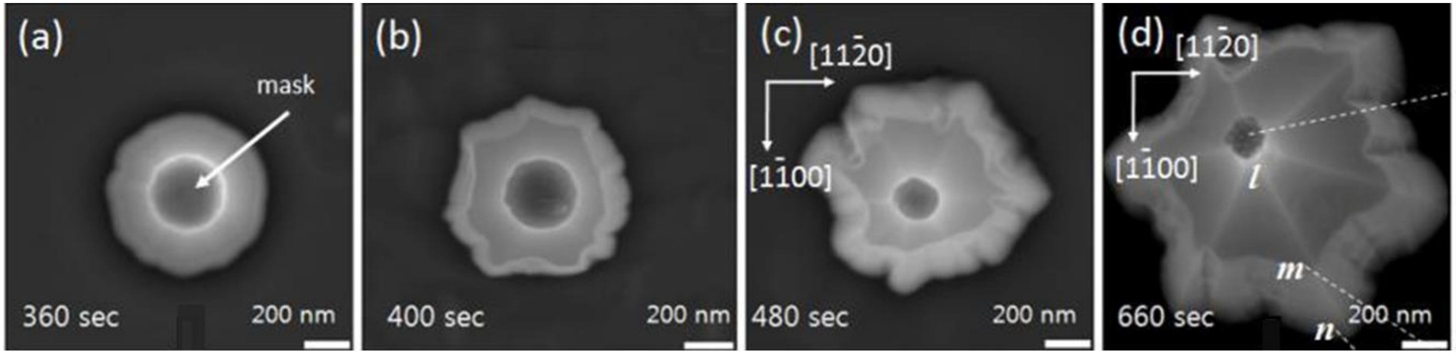
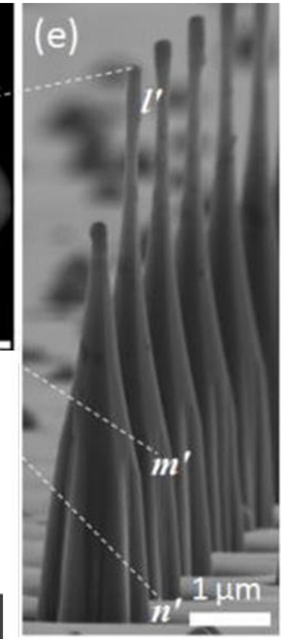
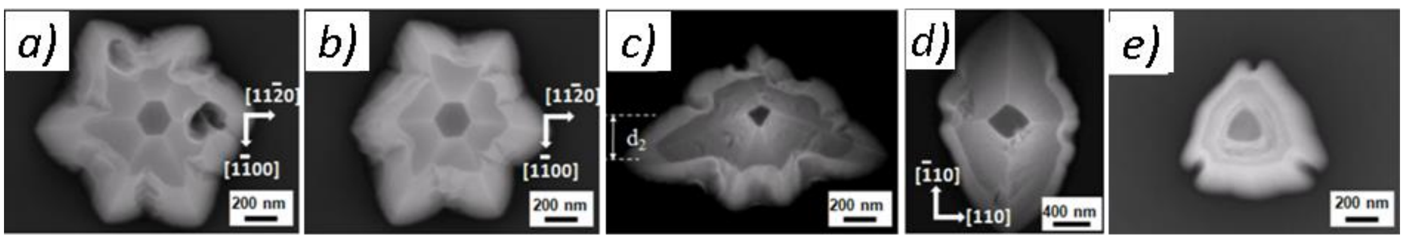


Alternative Approach





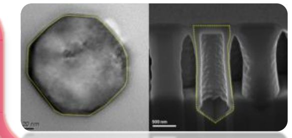
6H-SiC etching vs etching time (Ni mask).



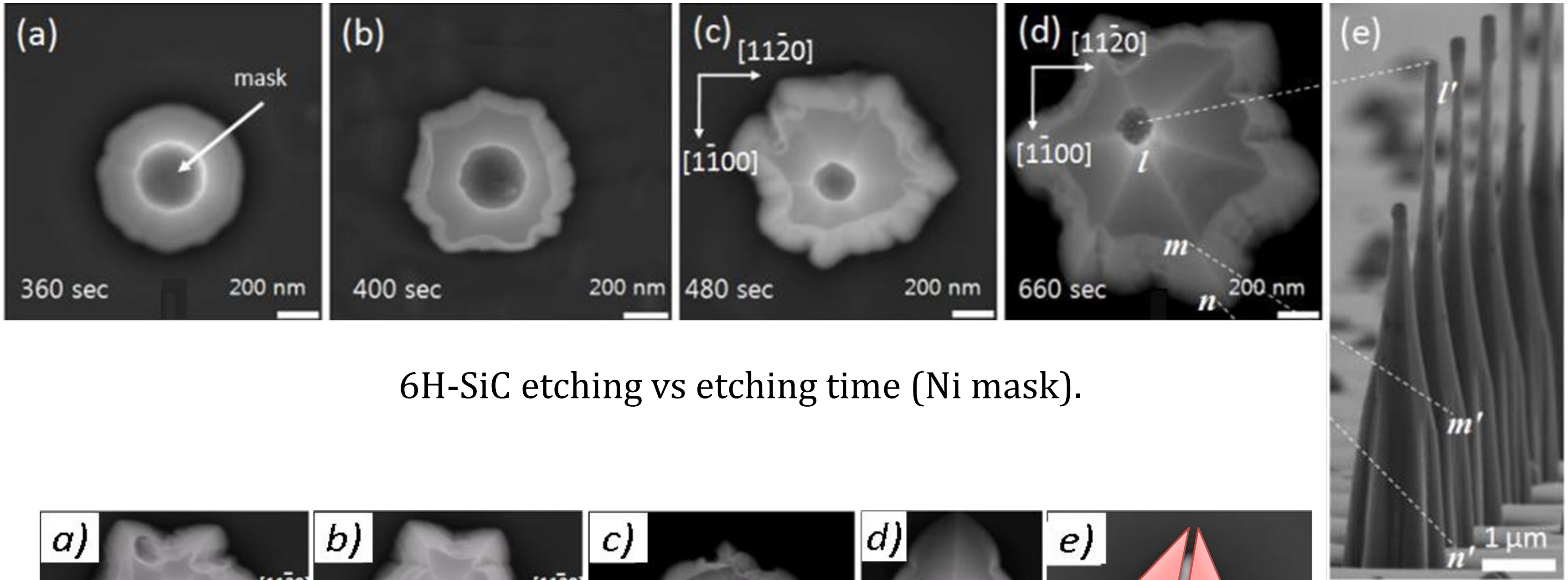
4H-SiC (0001)	6H-SiC (0001)	6H-SiC (11$\bar{2}$0)	3C-SiC (001)	3C-SiC (111)

Top view of the nanopillars after long etching time for various polytypes and crystallographic orientations.

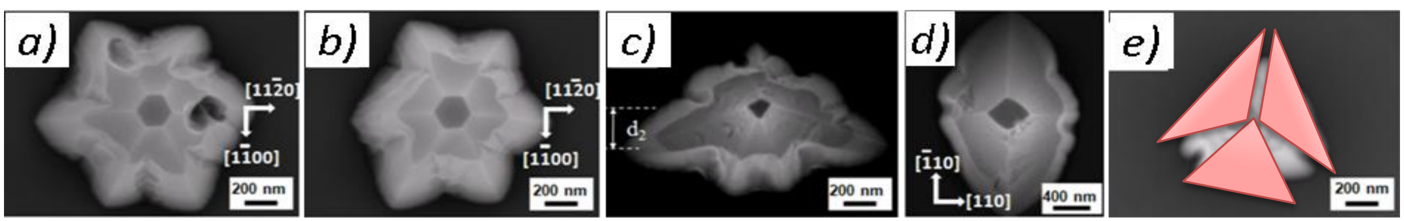
J.H. Choi, L. Latu-Romain, E. Bano et al., Materials Letters, 87, 9-12, (2012)



Alternative Approach



6H-SiC etching vs etching time (Ni mask).

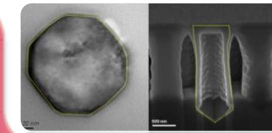


4H-SiC (0001)	6H-SiC (0001)	6H-SiC (11$\bar{2}$0)	3C-SiC (001)	3C-SiC (111)

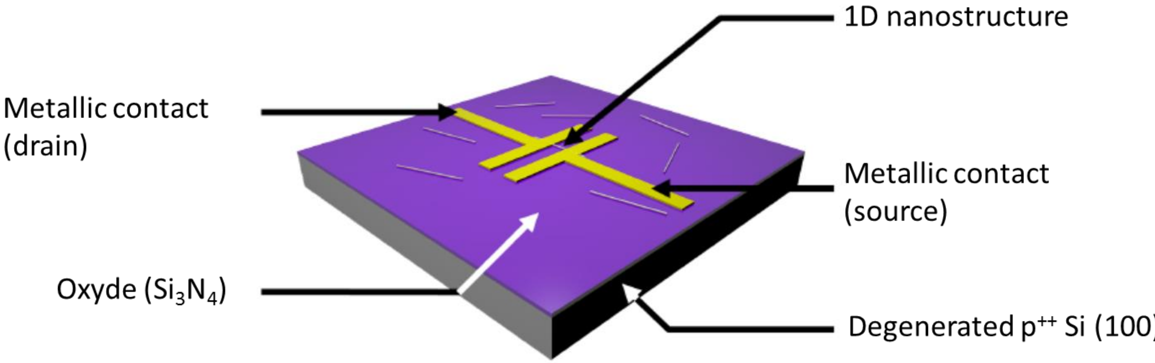
{111} équivalent planes

Top view of the nanopillars after long etching time for various polytypes and crystallographic orientations.

For long etching time, nanopillars are facetted, making appears planes of lowest surface energy, **Wulff theorem**

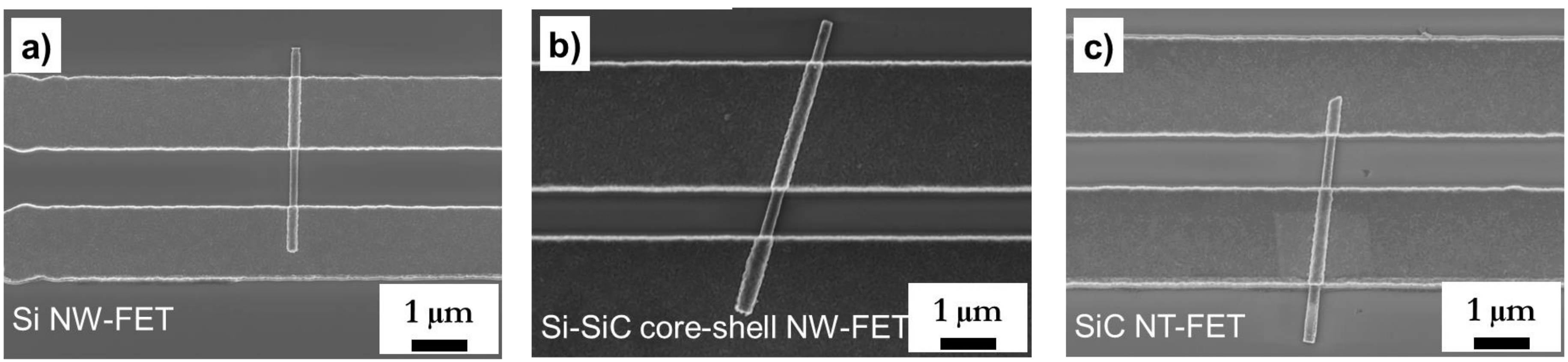


SiC-1D nanostructures into nano-Field Effect Transistors



LTM-IMEP-LAHC Collaborations

B. Salem and E. Bano



SEM images of:

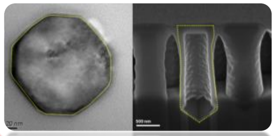
a) a Si NW-FET;

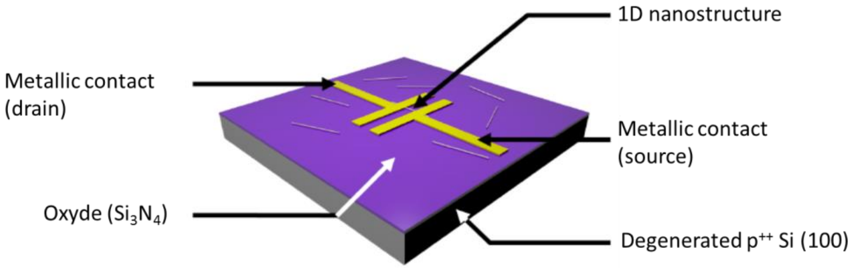
b) a Si-SiC core-shell NW-FET;

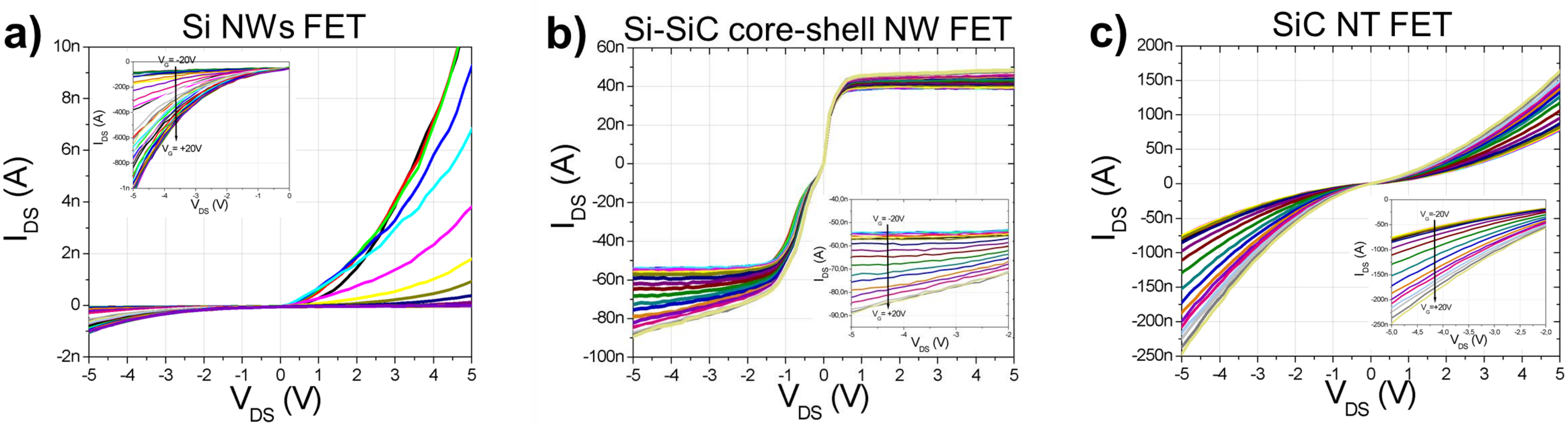
c) c) a SiC NT-FET

with drain and source contacts spaced of 1 μm

SiC-1D nanostructures into nano-Field Effect Transistors





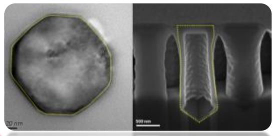


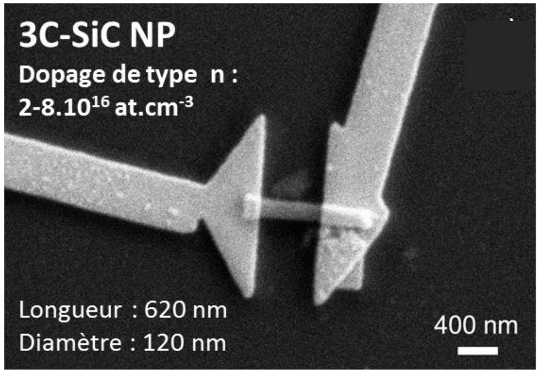
Transistor effect

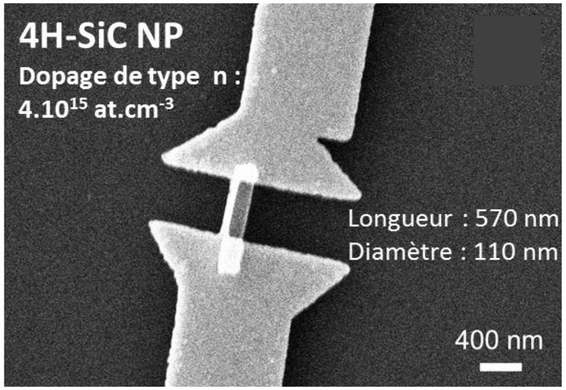
Extracted apparent mobility for these three different nano-FETs is very low (absence of contact annealing)

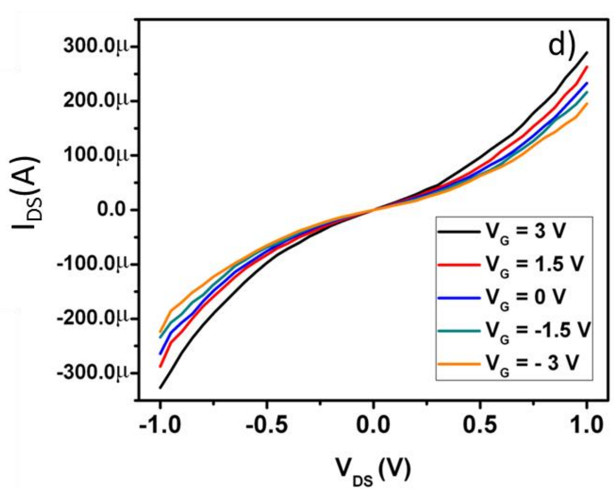
M. Ollivier, L. Latu-Romain et al. Materials Science in Semiconductor Processing, http://dx.doi.org/10.1016/j.mssp.2014.03.020i, (2014)

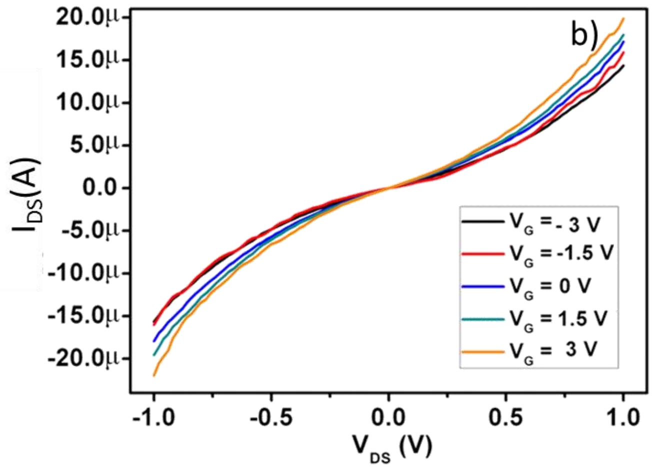
SiC-1D NP into nano-Transistors







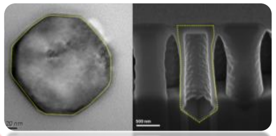




Contact silicidation during the annealing. Transistor effect.
Apparent carrier mobilities (record*): **40 cm^2.V^{-1}.s^{-1} (4H-) and 140 cm^2.V^{-1}.s^{-1} (3C-)**.
Thanks to a high crystalline quality associated to a low doping level.
Cubic polytype mobility > hexagonal polytype mobility.

*W.M. Zhou et al. IEEE Electron Device Letters, 27(6), 463-465, (2006) : **16 cm^2.V^{-1}.s^{-1}***

M. Ollivier, L. Latu-Romain et al. Materials Science in Semiconductor Processing, (2014)

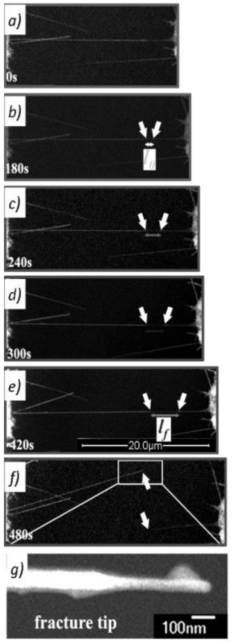


Towards applications

Mechanics applications: new composites

Y. Zhang et al. Advanced Functional Materials, 17:3435–3440, (2007)

Superplasticity of a SiC NW (200%)



For energy:

New anode materials (Li-ion batteries…Si NW…Si-SiC NW)

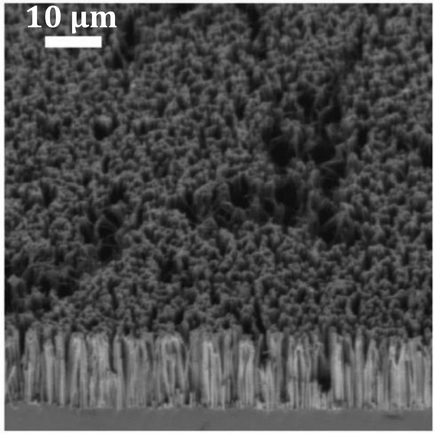
T. Sri Devi Kumari et al. Nano silicon carbide : a new lithium-insertion anode material on the horizon. RSC Advances, 3 :15028–15034, (2013)

Supercapacity:

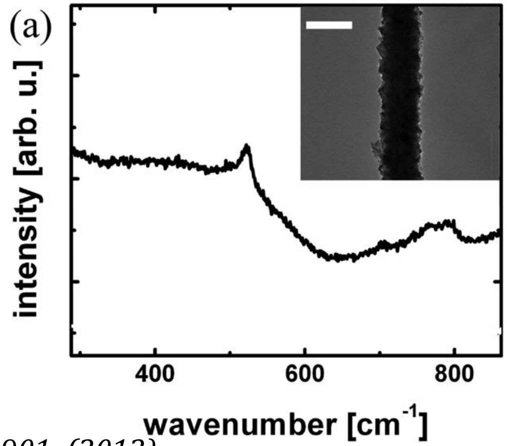
Alper et al.

Thanks to the SiC coating, the supercapacitor remains stable over 10^3 charge/discharge cycles compared to 1 charge/discharge cycle without coating.

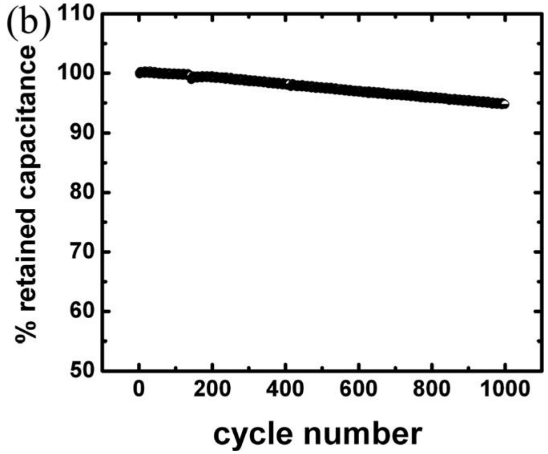
SEM image Si-SiC NWs



Raman spectrum

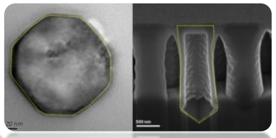


Capacity *vs* cycles number



J.P. Alper, et al. Applied Physics Letters, 100(16):163901, (2012)

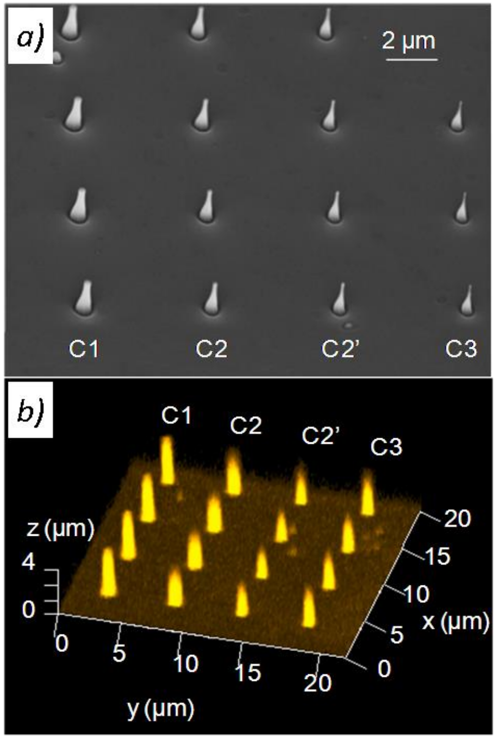
Towards applications



Electronics:
The possible use of one-dimensional SiC nanostructures in nanoelectronics is exclusively restricted for the moment to the study of SiC-NWs Field Effect Transistors

Nano-transitors functioning in extreme conditions
Chemical, gas dectectors (SiC-NT, *ab-initio* studies)

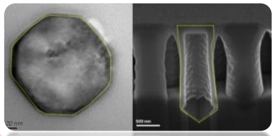
Biology:
Biocompatibility & high developped surfaces



a) SEM view of SiC-NPs obtained by plasma etching
b) 3D reconstructed view of fluorescent DNA on NPs obtained by confocal scanning laser fluorescence microscopy

LMGP collaboration
V. Stambouli, L. Fradetal

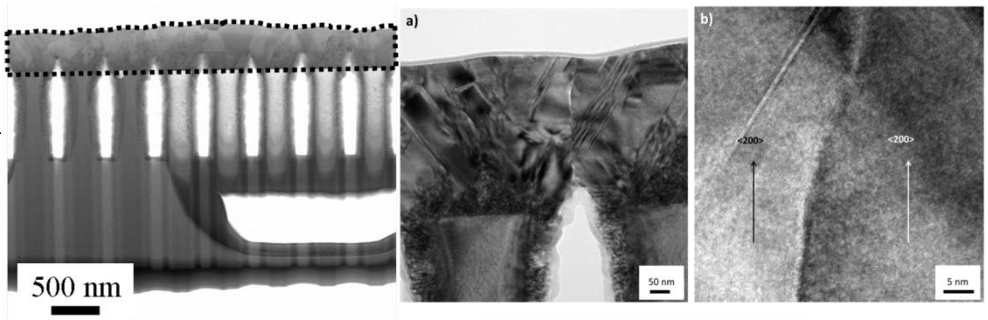
L. Fradetal et al. Journal of Nanoscience and Nanotechnology 14, 3391-3397, (2014)

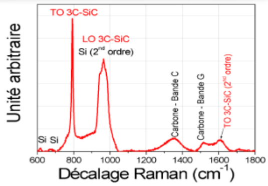


Perspectives

Material:

3C-SiC growth by carburization
3D towards 2D



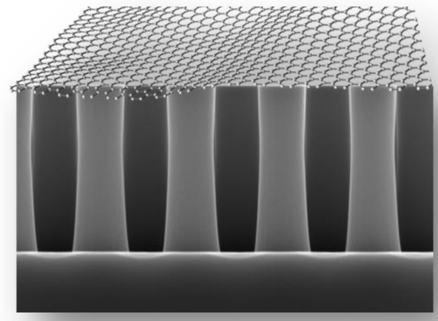


Largeur du mode TO 3C-SiC :11 cm^{-1}

SiC doping

Cubic III-V epitaxy (GaN)

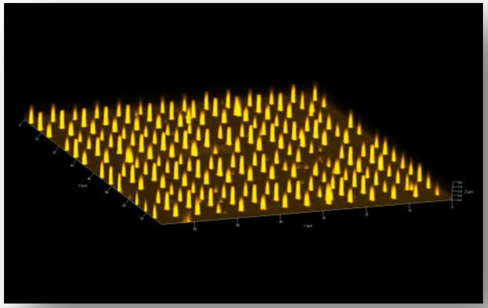
Synthesis of suspended graphene on our arrays



To evaluate the new applications:

testing new anode materials

integrating nanostructures into Bio-nano-sensor (DNA: model molecule…specific molecules)



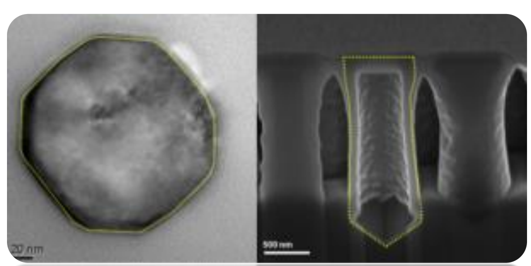
drug delivery

L. Latu-Romain, M. Ollivier Journal of Physics D: Appl. Phys, topical review, 47, 203001 (18 pp), (2014)

Acknowledgments

My colleagues & students: M. Ollivier, J. Choi, M. Martin, V. Stambouli, E. Bano, B. Salem, G. Ferro

Many thanks for your attention



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