Catalytic CVD of Graphene on Graphite and Resist-Free Method for Multi-Electrodes Devices

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Outline

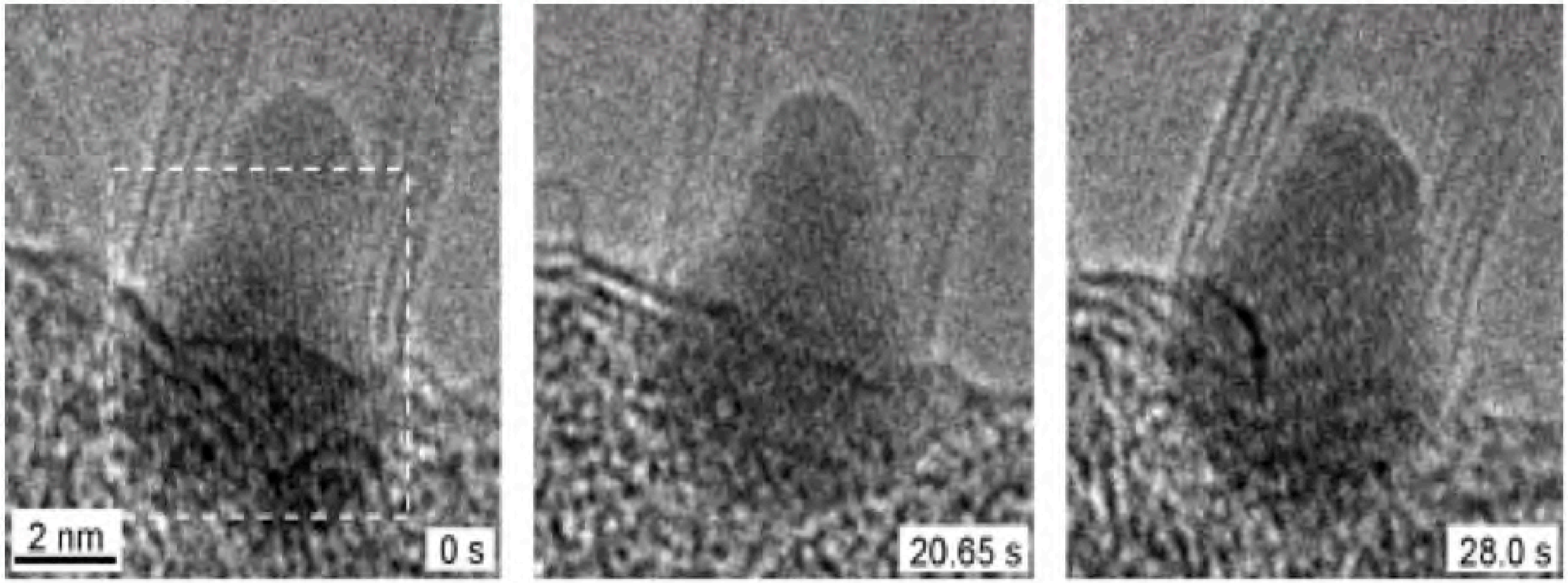
Catalytic Growth of C-Nanostructures

- Metal nanoparticles (NPs) on HOPG
- Chemical Vapor Deposition (CVD) on HOPG
- Scanning Tunneling Microscopy (STM) of C-structures

Graphene Device Fabrication

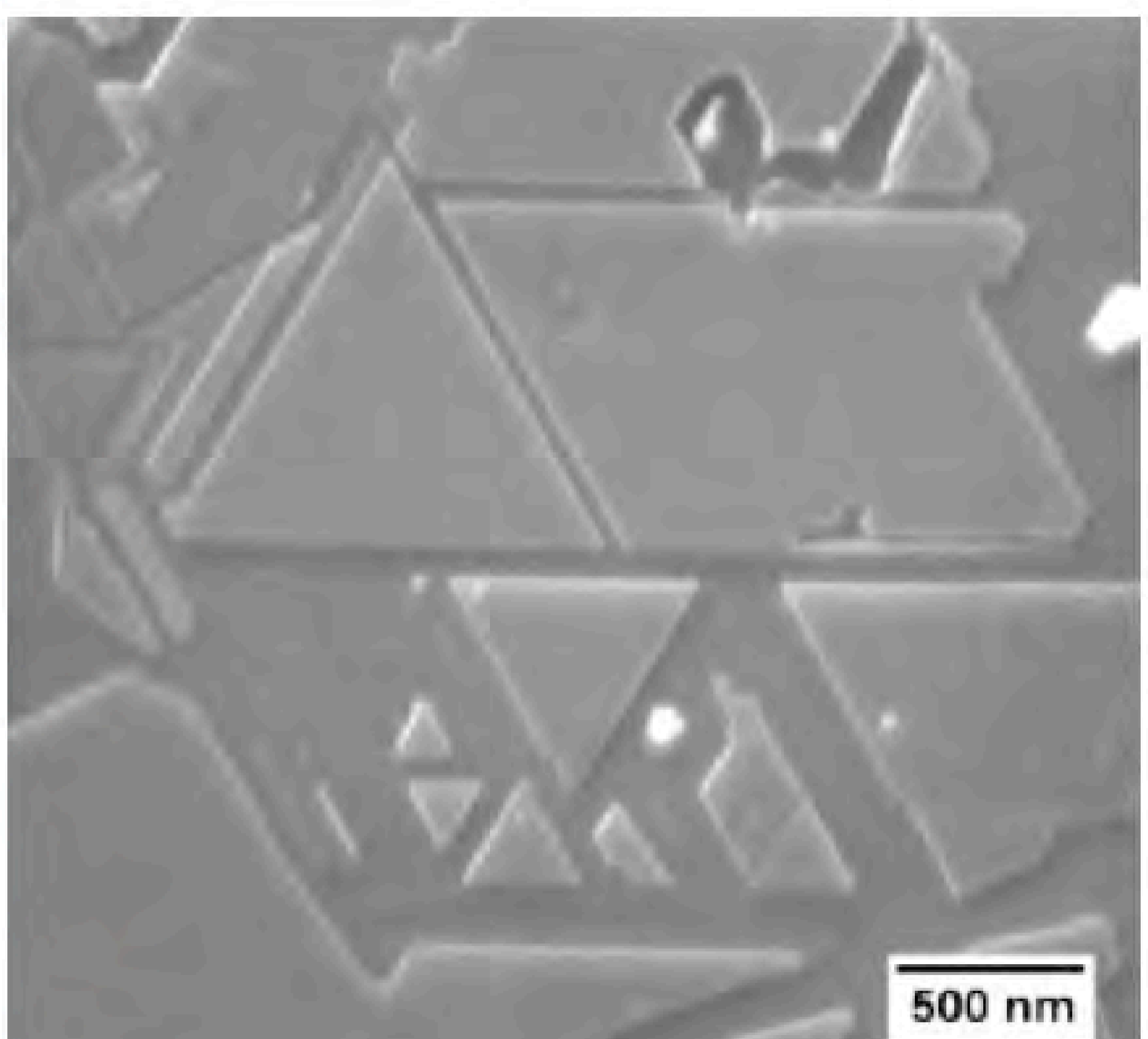
- Direct-masking technique
- Graphene/graphene-like device fabrication
- Characterization of the charge transport

Nanoparticles (NPs) to Structure Graphene

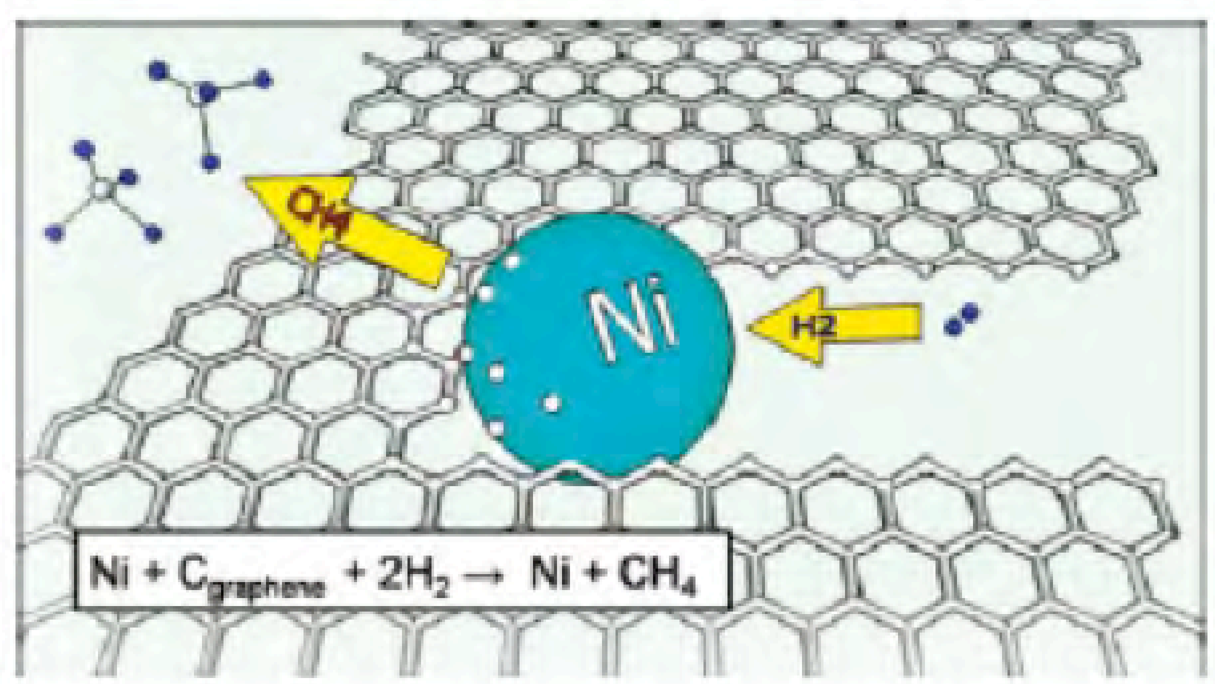


Yoshida *et al.* Nano Lett. **9**, 3810 (2009)

Catalytic Growth of Nanostructures



Ci *et al.*, Adv. Mater. **21**, 1 (2009)

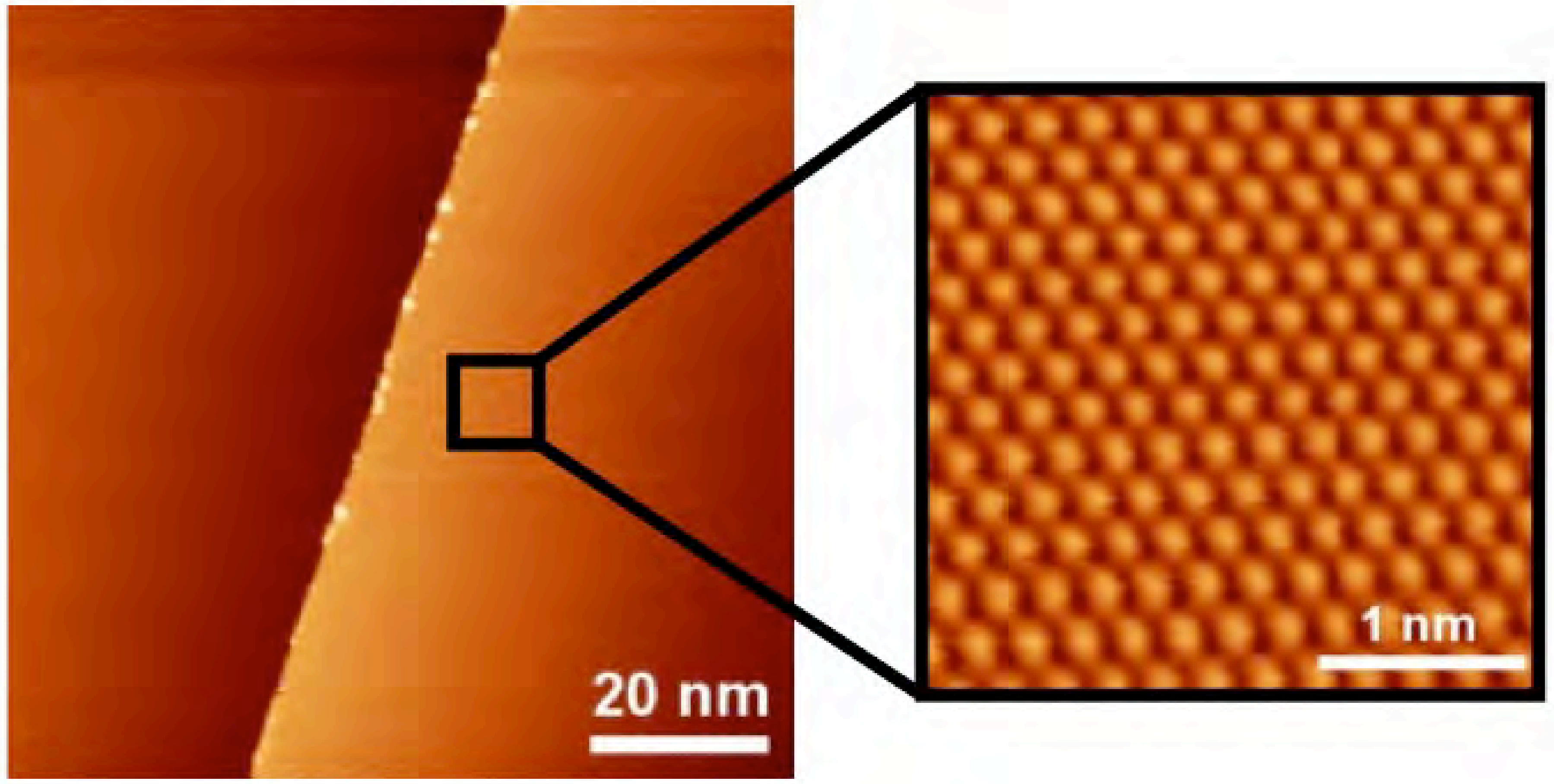


Campos et al. Nano Lett. **9** 2600 (2009)

Etching of Carbon-based Nanostructures

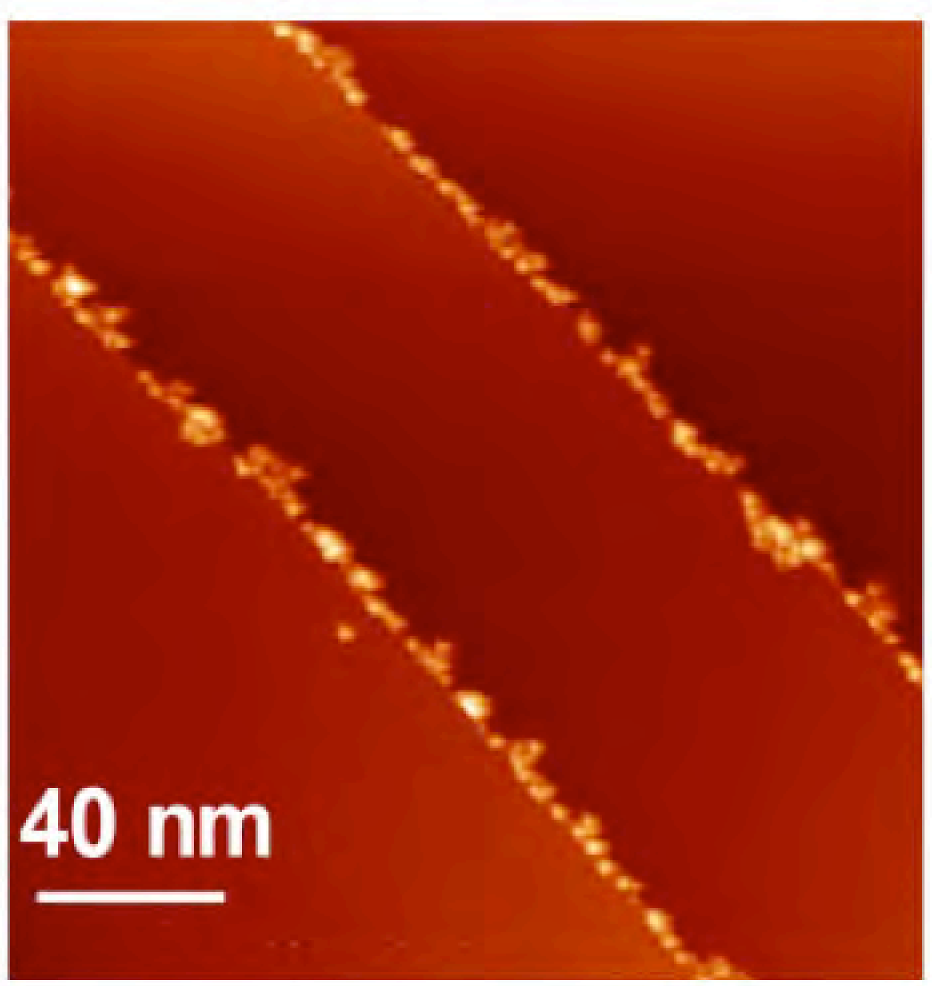
Highly Oriented Pyrolitic Graphite (HOPG)

Cleaved HOPG

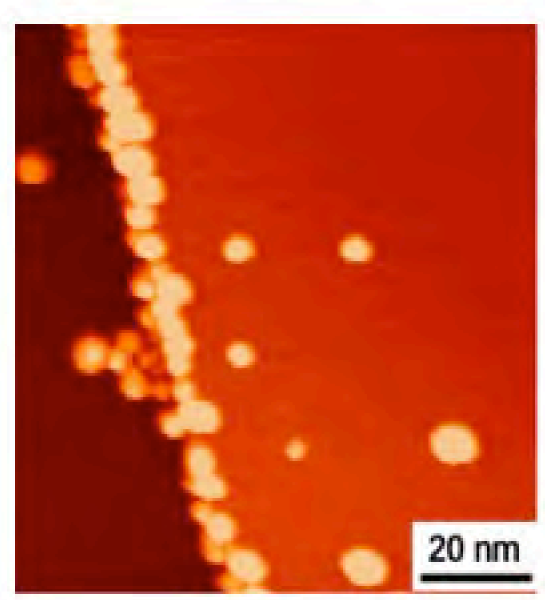


Atomically flat planes with step edges
(degassing @ 900°C after cleavage)

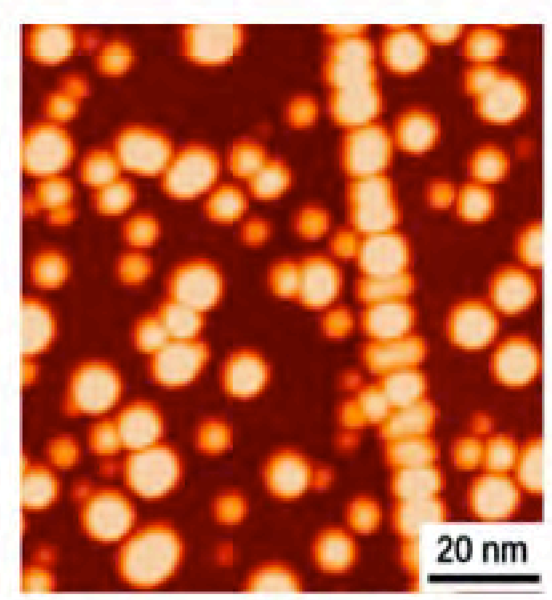
Fe deposition (<1ML)



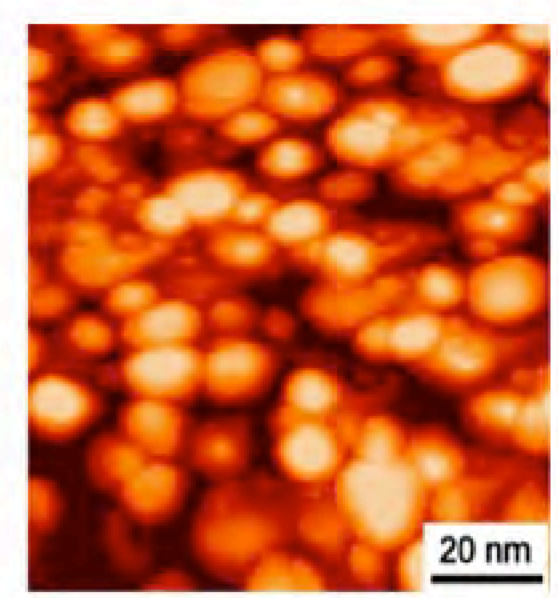
e-beam sublimation at room temperature



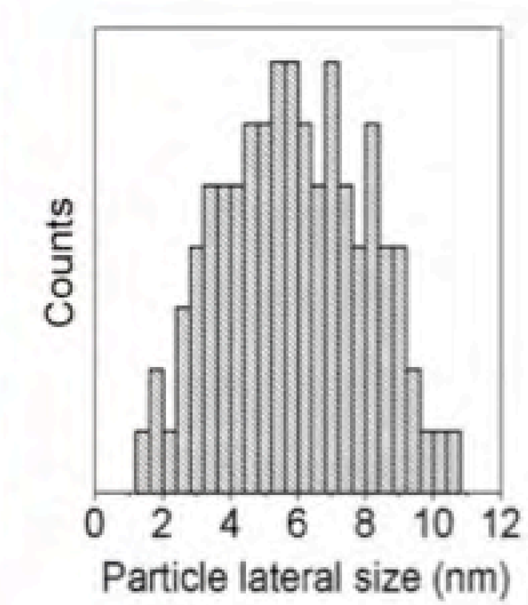
0.3 ML



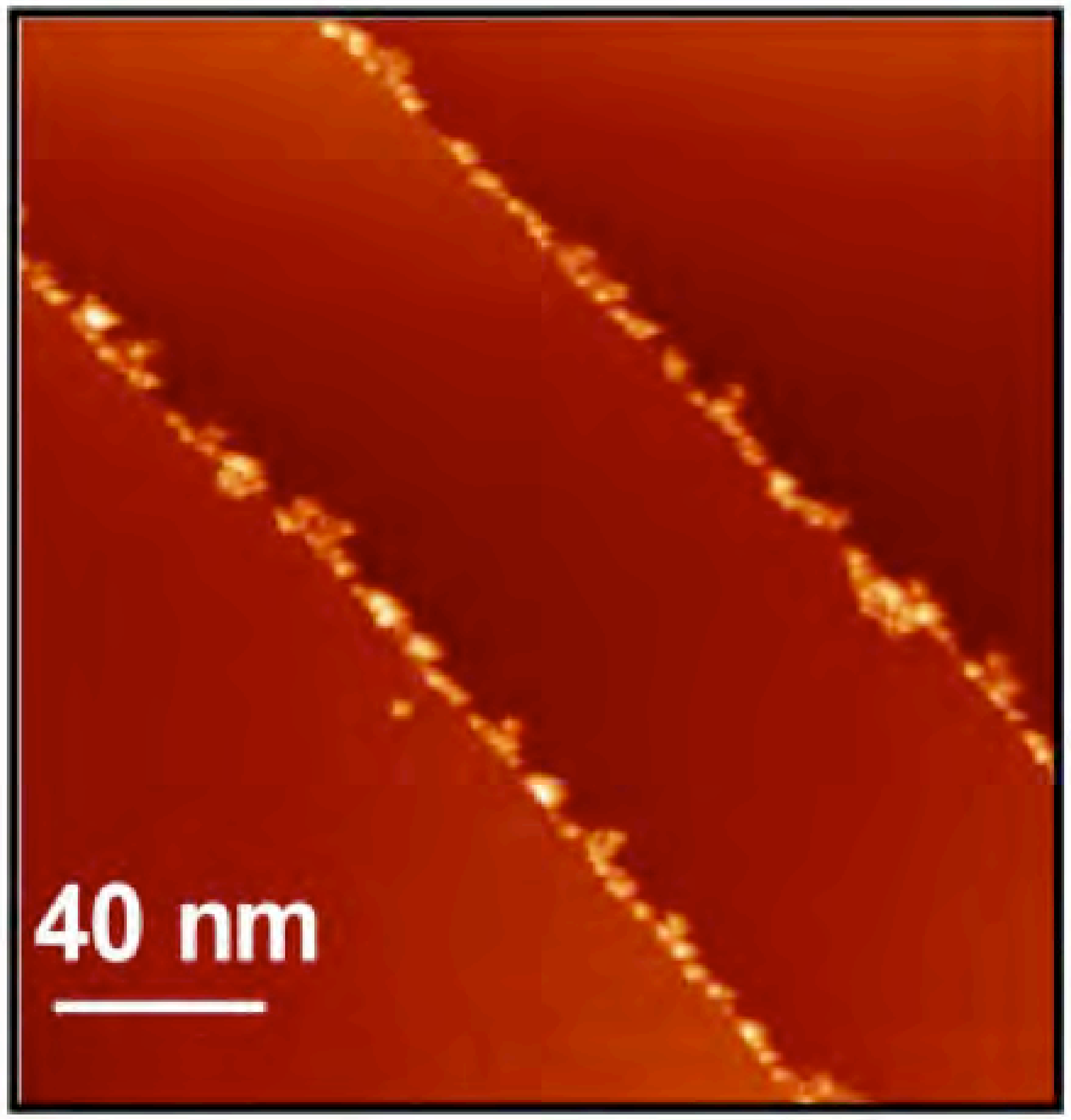
2 ML*



6 ML

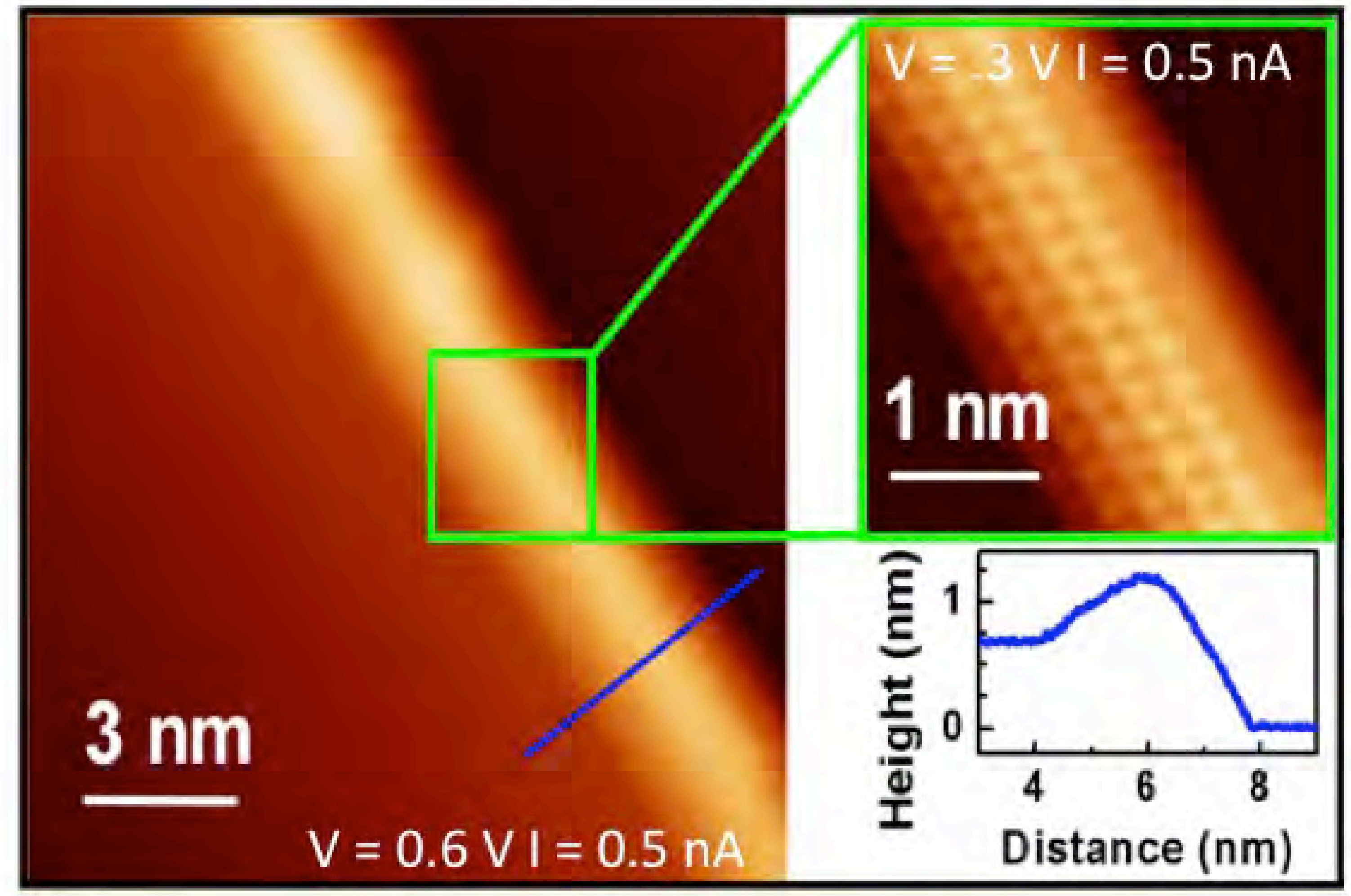


Growth of Curved Graphene Sheets (CGS) – Fe <1ML



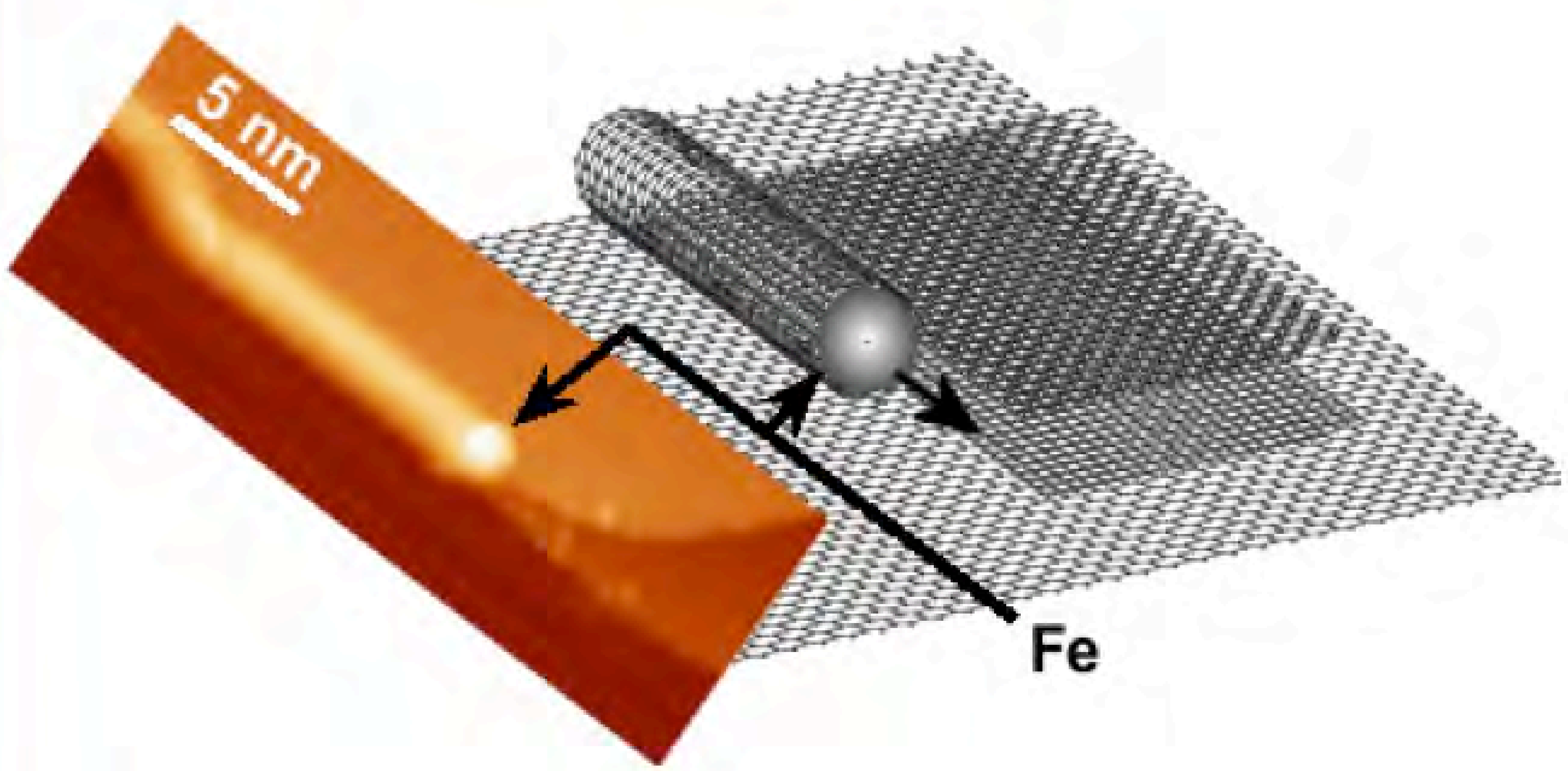
CVD

CH_4 @ 900°C
p=$2x10^{-4}$mbar



Kholmanov *et al.* PRB **79**, 233403 (2009)

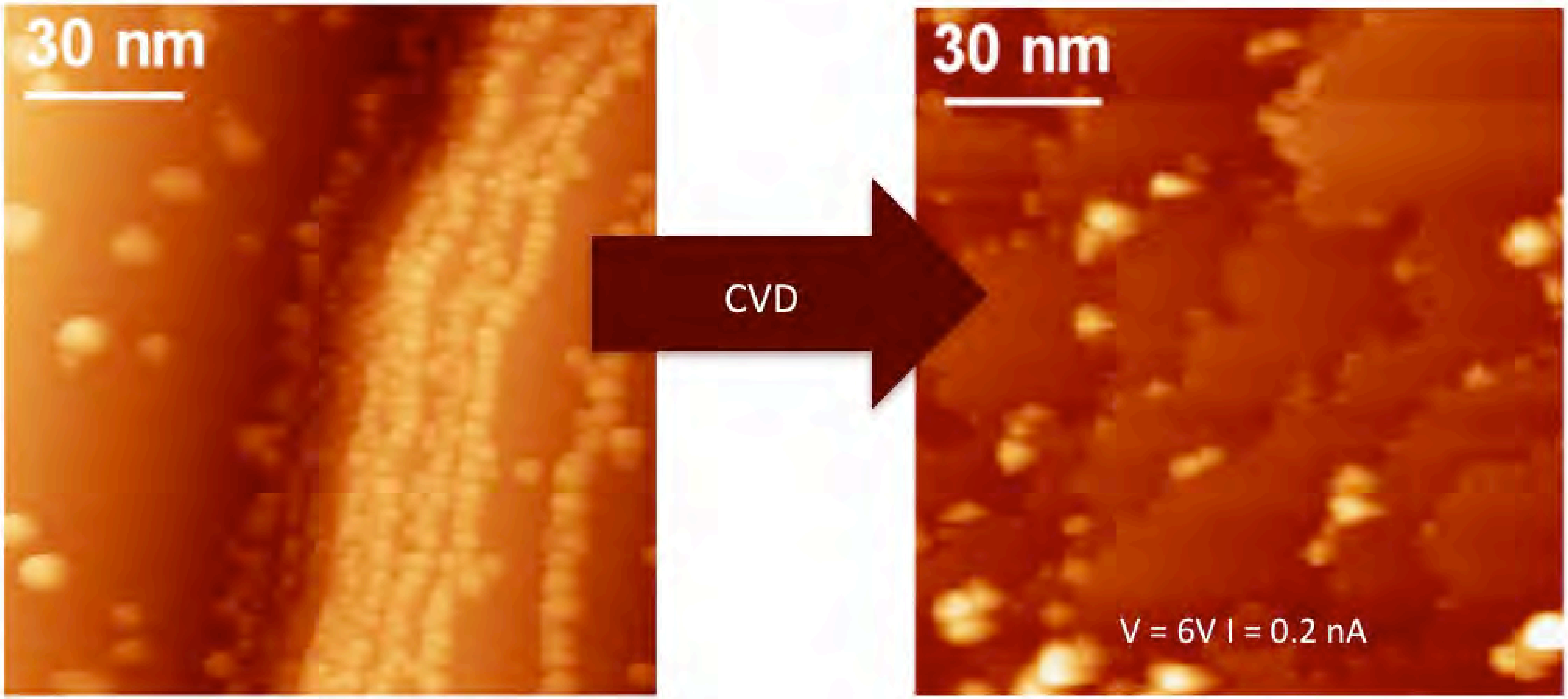
1. abrupt edges → continuously smooth curved graphene sheet (CGS)
2. structures similar to CNT (no Fe)



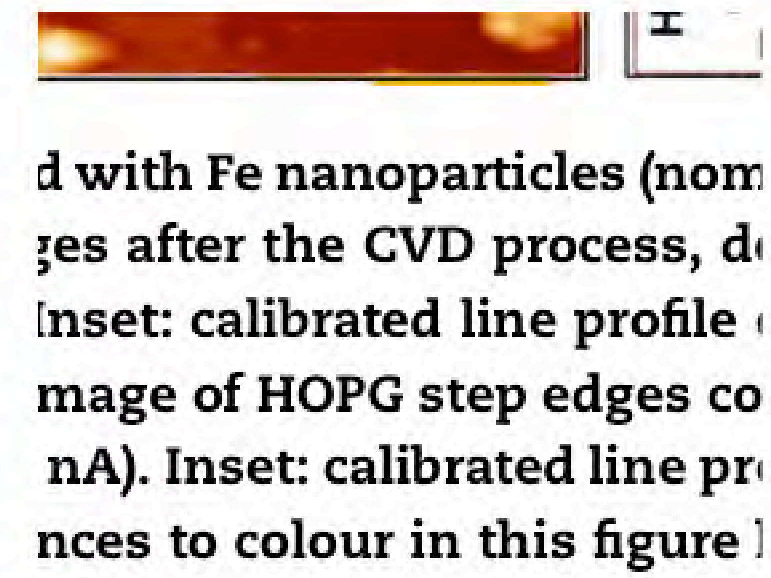
CH_4 decomposition at the edge C-atoms and creation of chemical bonds

C-atoms can meet both edges leading to the growth of CGS (*zipper-like* mechanism).

Growth Along the Edges – Fe <1ML

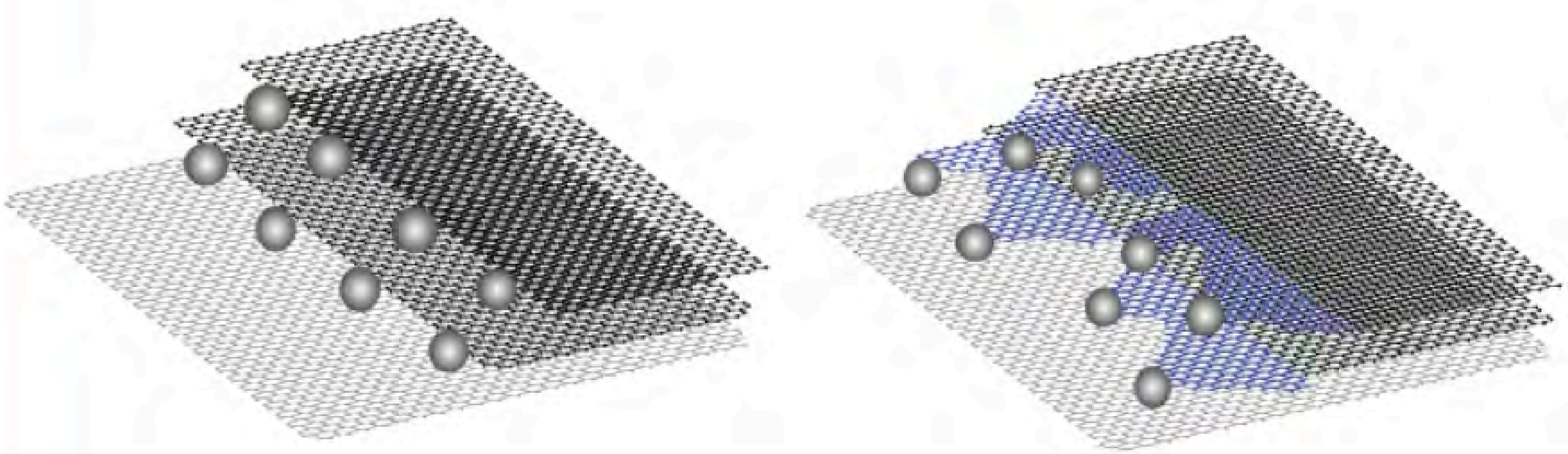


irregular edges, large terraces


d with Fe nanoparticles (nom
ges after the CVD process, d
Inset: calibrated line profile
mage of HOPG step edges co
nA). Inset: calibrated line pr
nces to colour in this figure

Reference (no Fe)

Kholmanov *et al.* Carbon **48**,1619 (2010)



grown graphene is not distinguishable from original terrace (no crystallographic difference)

role of defect for the shape

Graphene Island Growth – Fe > 15ML

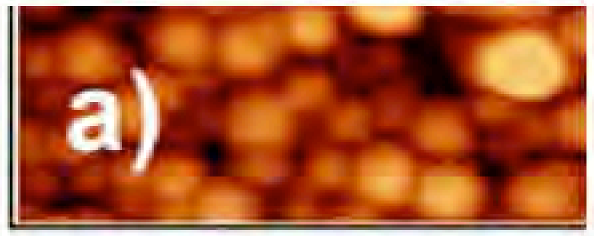


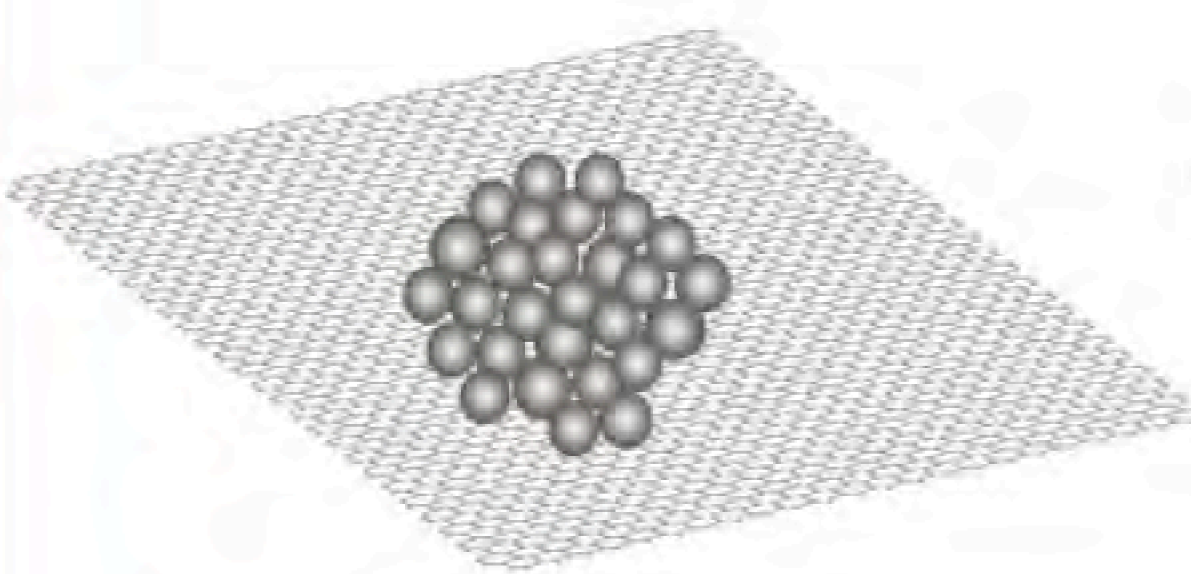
Fig. 3 – (a) STM image of 15 N
to CVD process (*V* = −2.5 V *I*
support and surrounded
graphene layer grown or
atomically resolved STM ima
in the bottom right of the fi
the web version of this artic

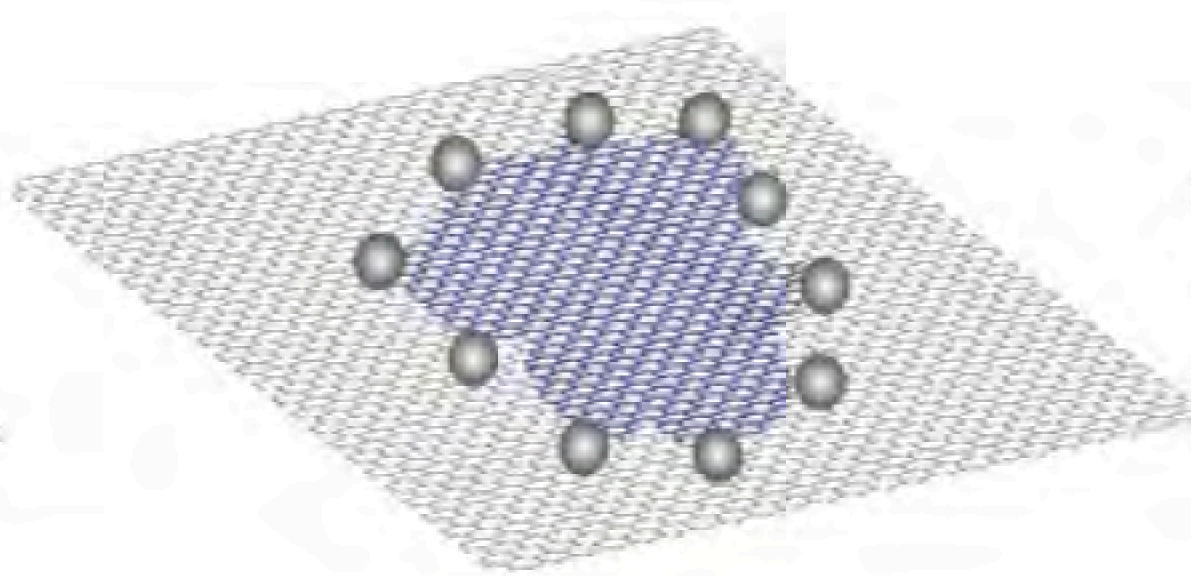
CVD

ML Fe film forming 3D nanoparticles and homogenously covering th
= 0.2 nA). (b) STM images of PGI grown on the terraces of the (0 0 0
metal particles after the CVD reaction (*V* = −2.5 V *I* = 0.6 nA). The circ
GI. The white arrow indicates the grown second graphene layer. Th
age of PGI (*V* = −0.08 V *I* = 0.1 nA). The blue line indicates a calibrated
gure. (For interpretation of the references to colour in this figure leg
le.)

Kholmanov *et al.* Carbon **48**, 1619 (2010)

large number of isolated graphene islands
(tens of nms)



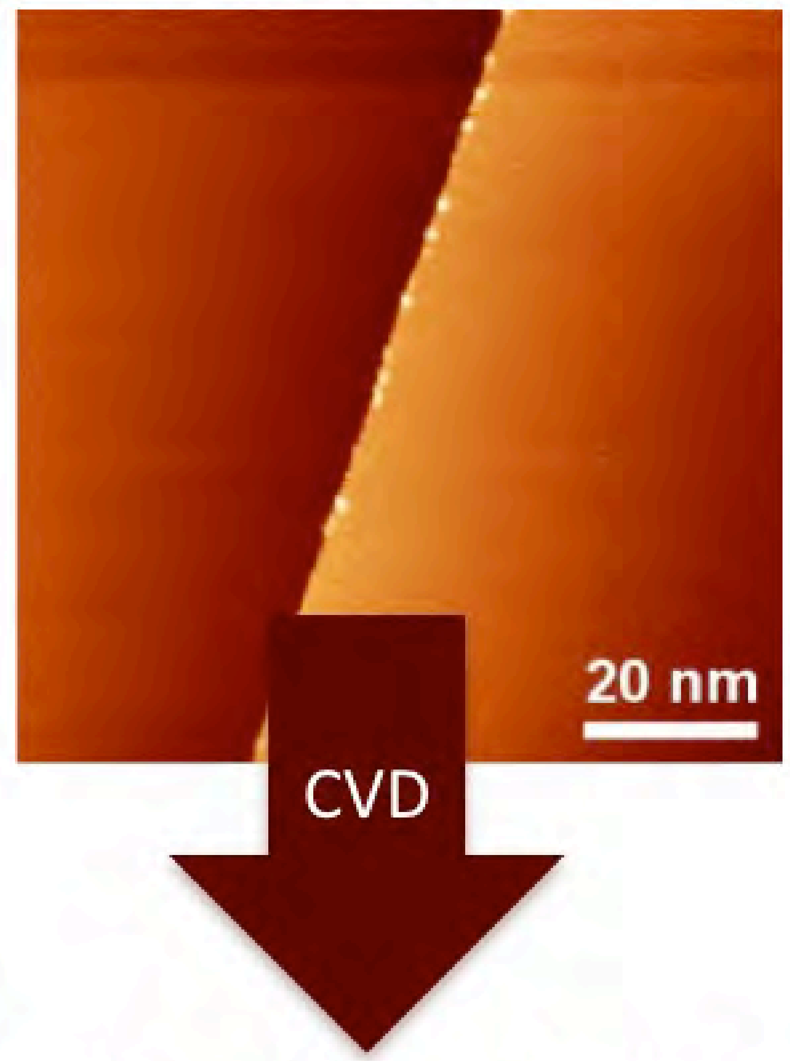


Fe evaporation *vs.* CH_4 decomposition

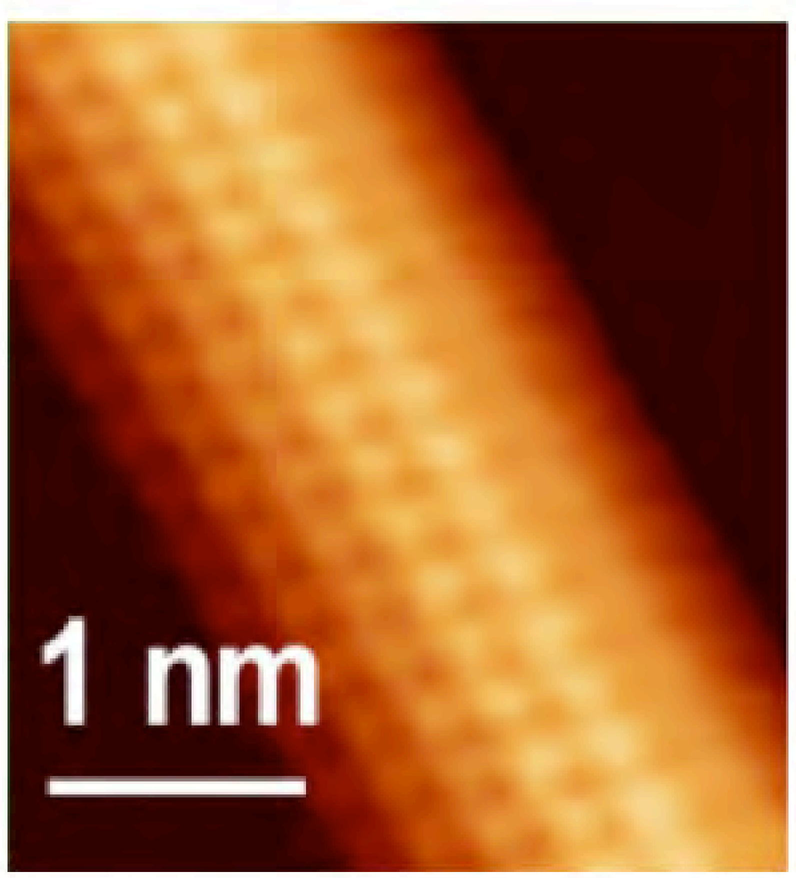
Possible role of defect for the shape

I. Conclusion

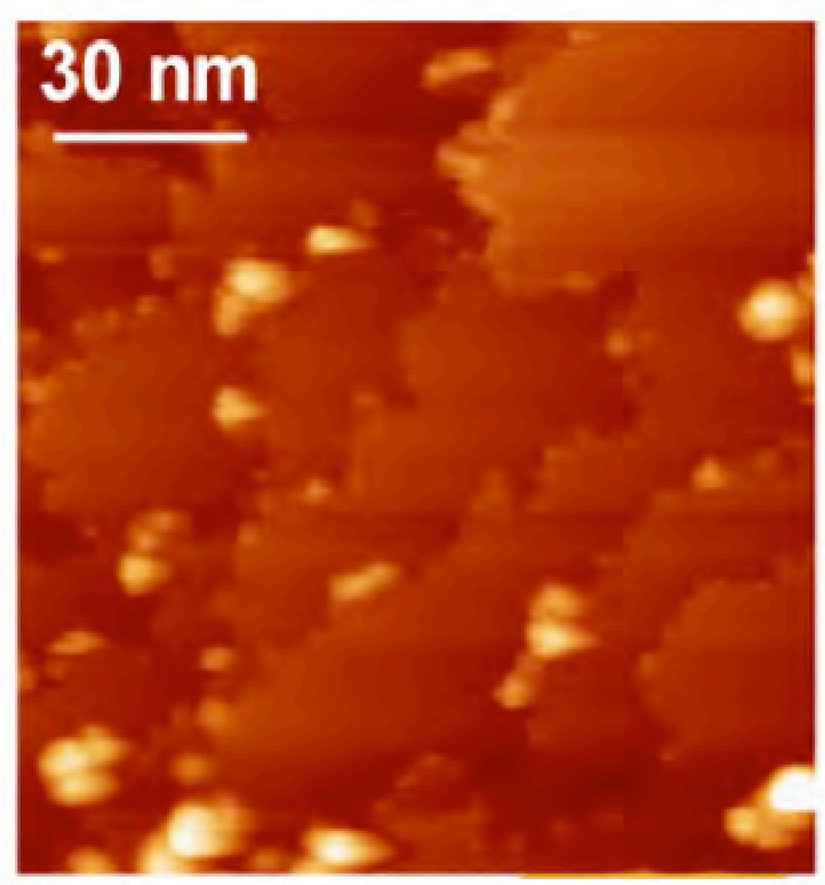
Cleaved HOPG



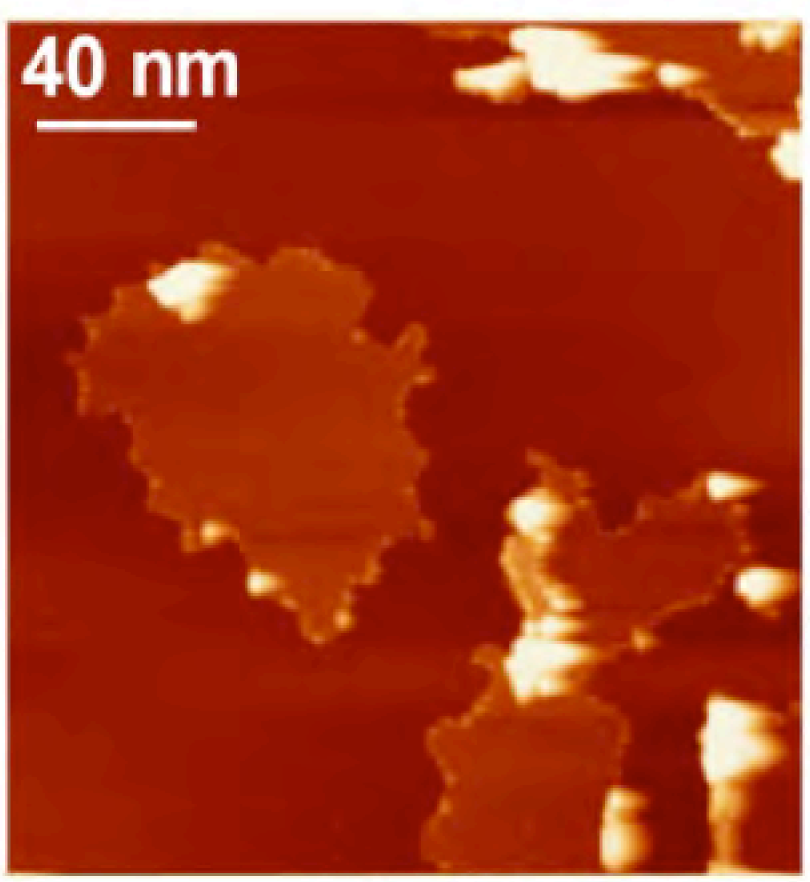
Carbon Nanostrutures



Tubular (CNT-like)



Terraces



Islands

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Graphene Device Fabrication

- Direct-masking technique
- Graphene/graphene-like device fabrication
- Characterization of the charge transport

Device Fabrication : Going *Ion*- and *Resist*-free

<u>Device fabrication:</u>

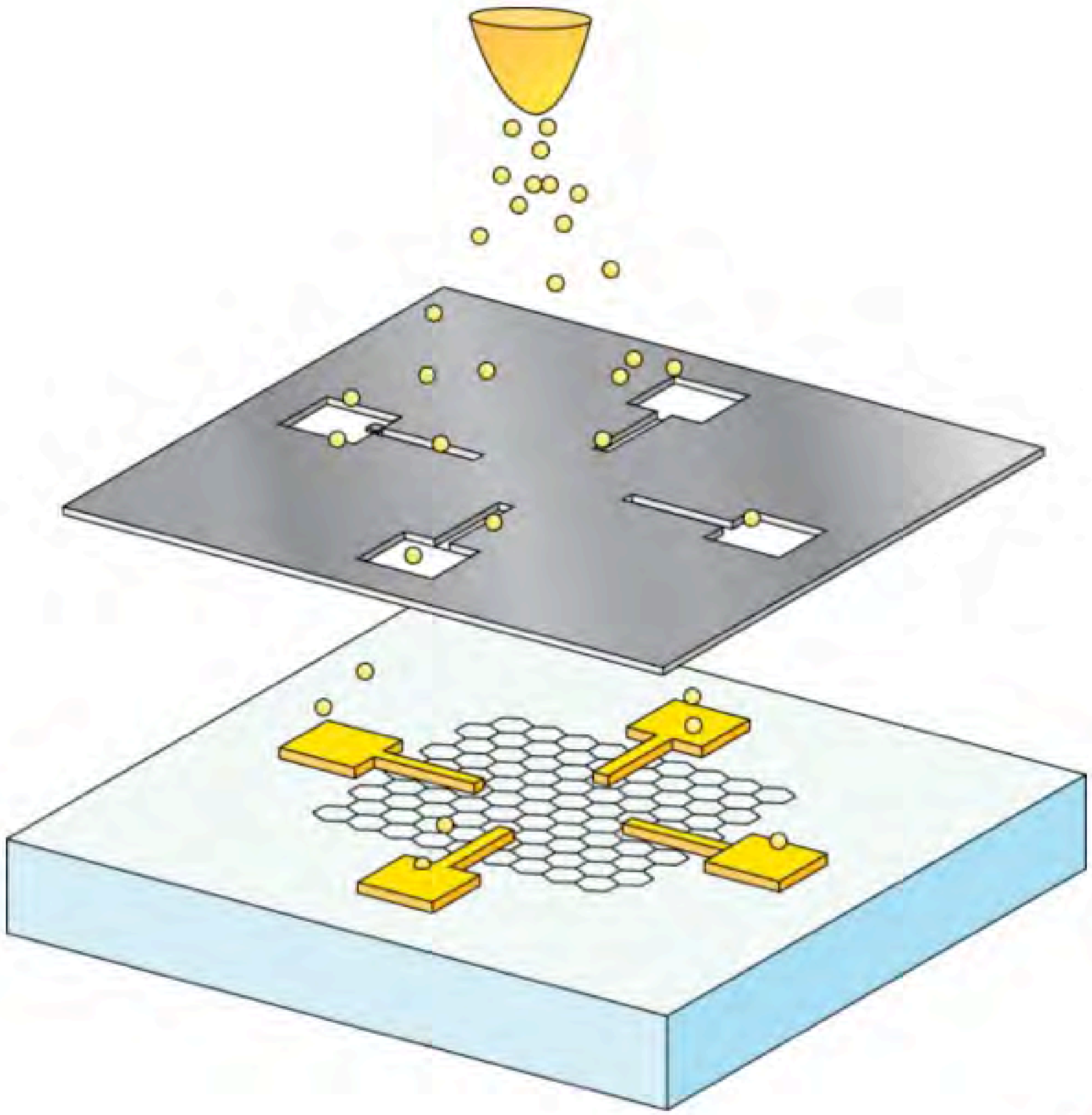
Conventional techniques to fabricate graphene-based-devices:

e-beam lithography (photo-resist residues, lift-off)
FIB lithography (ion implantation, amorphization)
others…

post-fabrication treatments to recover intrinsic properties

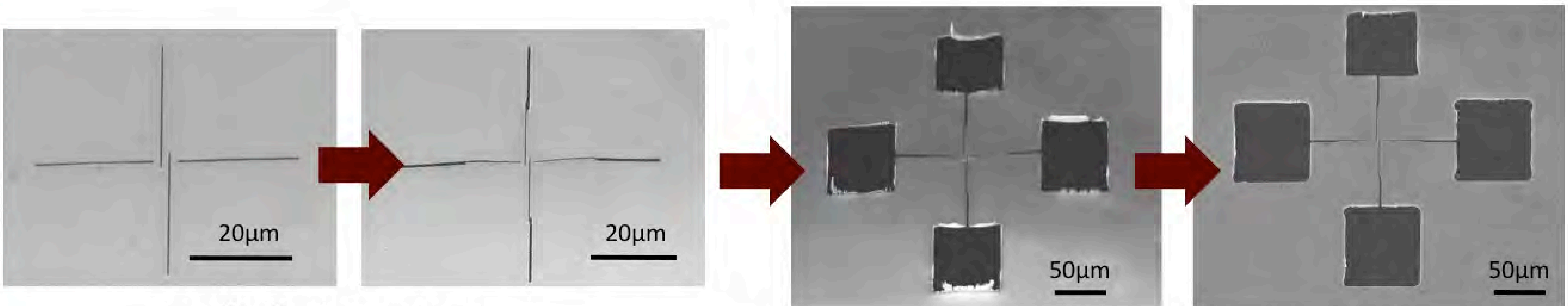
Direct masking on graphene/graphene-like material

- the technique;
- membrane fabrication and alignment;
- study of the charge transport;



Membrane Fabrication

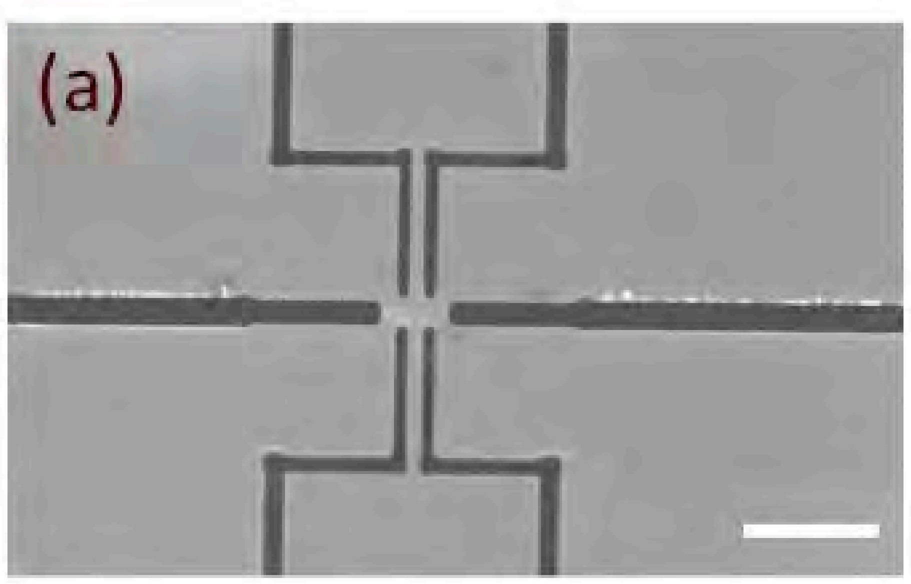
Patterns are fabricated on Si_3N_4 membranes (~250nm thickness) by XeF_2-assisted ion-beam etching (FIB) lithography. FIB offers great versatility in the pattern shape.



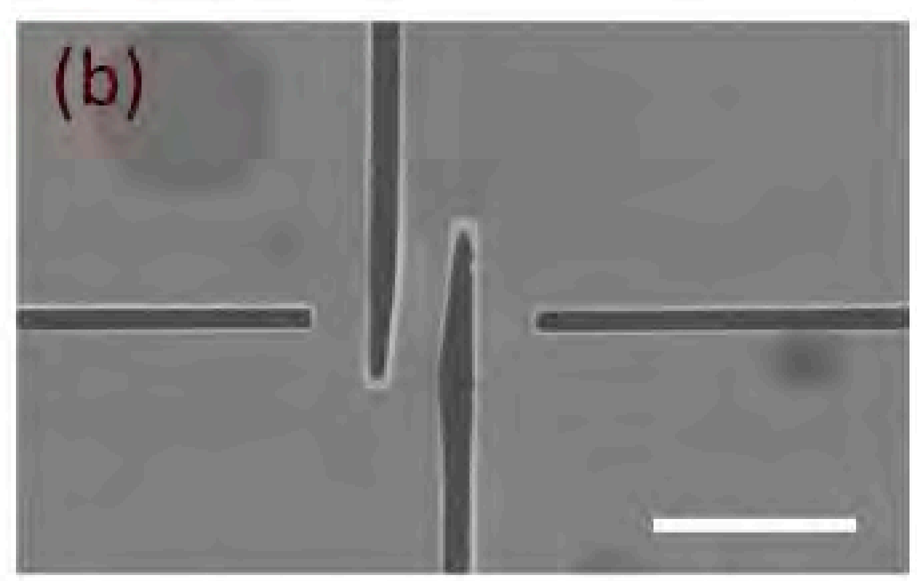
smaller leads (10x0.1µm)
→ small FIB currents (~10s pA)

larger electrodes (50x50µm)
→ large FIB currents (~nA)

fine polishing



Hall bar



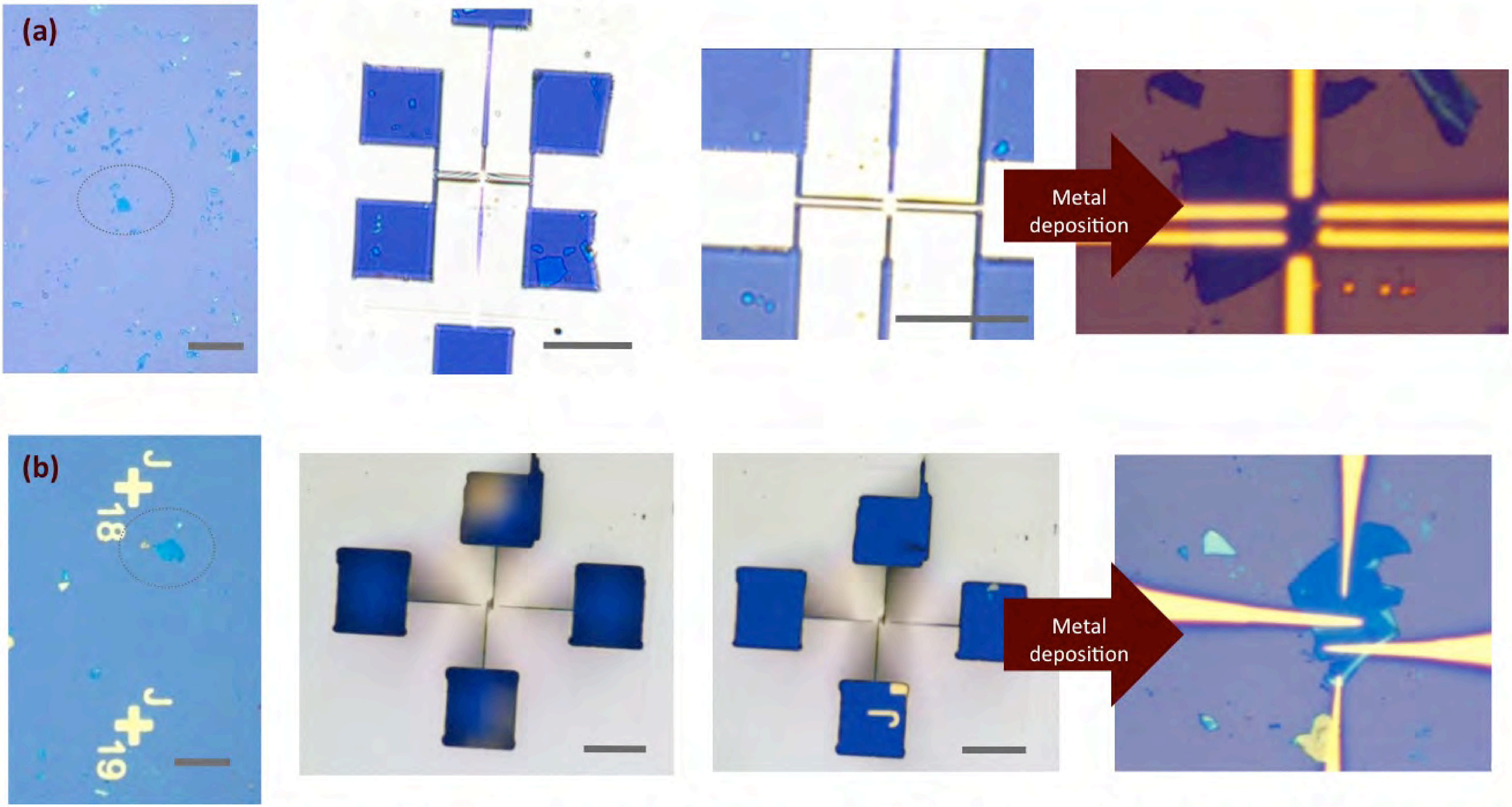
4-pr interdigitated

Van der Pauw

(Scale bar is 10µm)

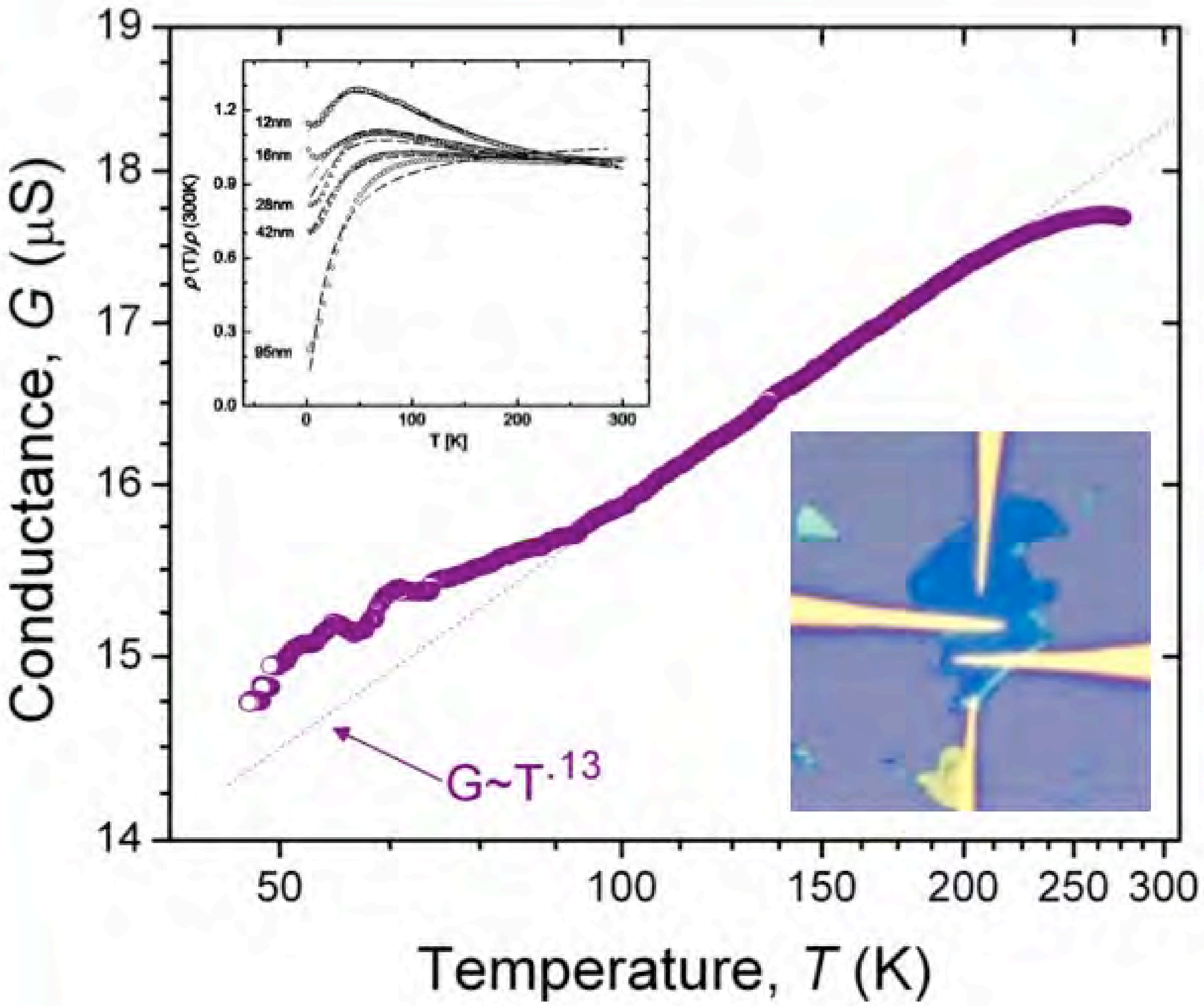
Membrane Alignment

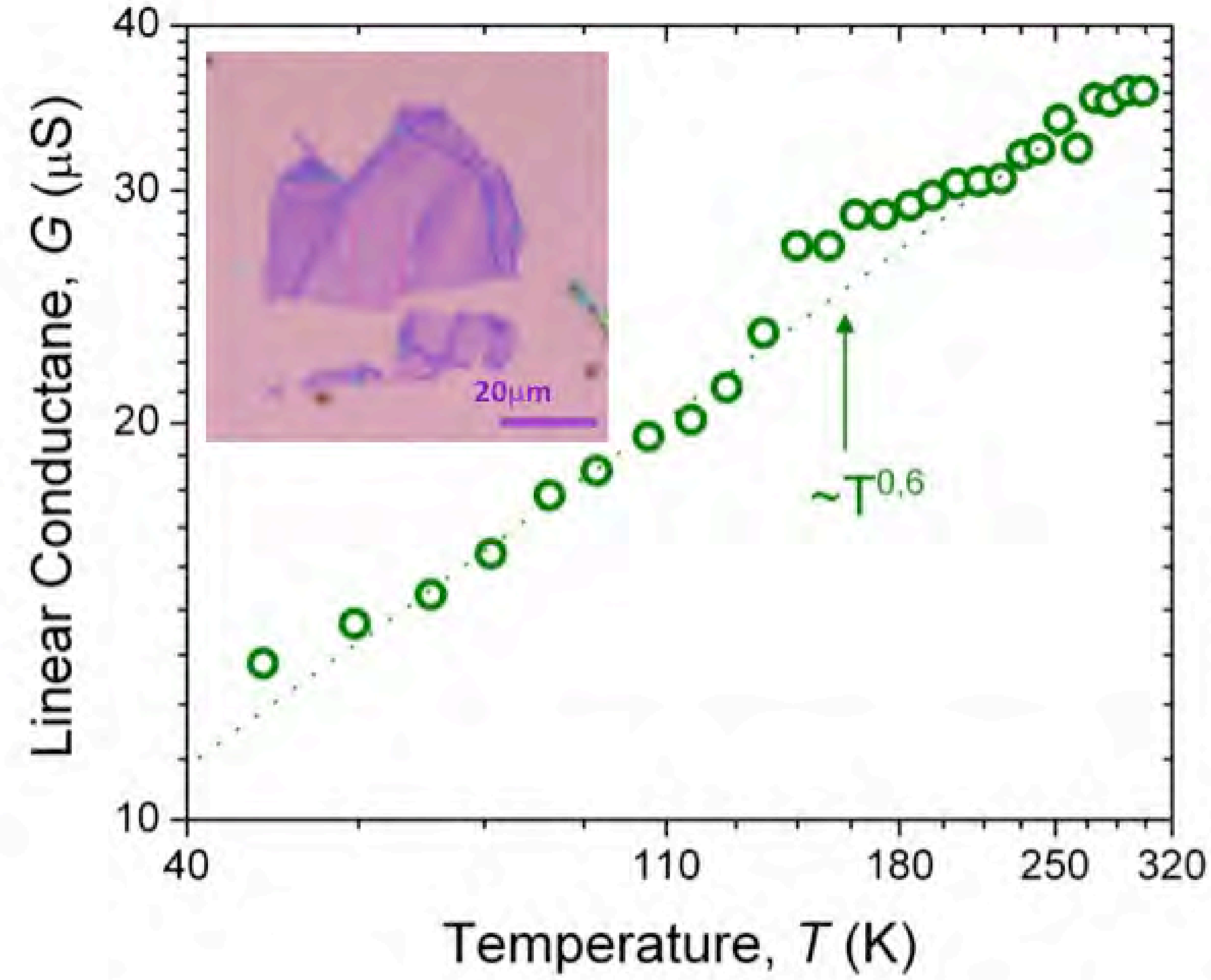
In collaboration with Dr. Grisolia, INSA, Toulouse



Reference marks facilitate sample location and alignment process. Scale bar is 50µm.

Multielectrodes Device I: Conductance

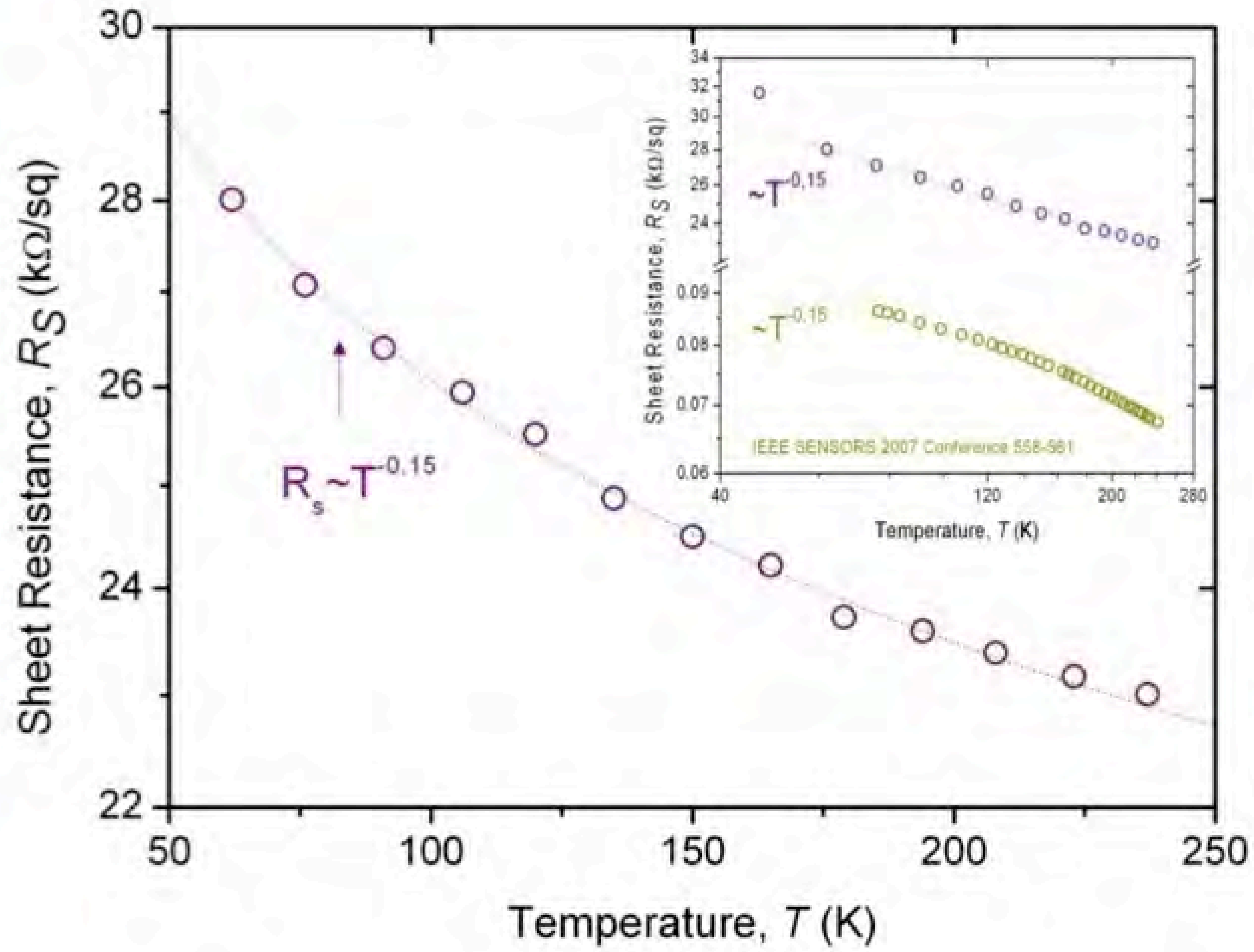


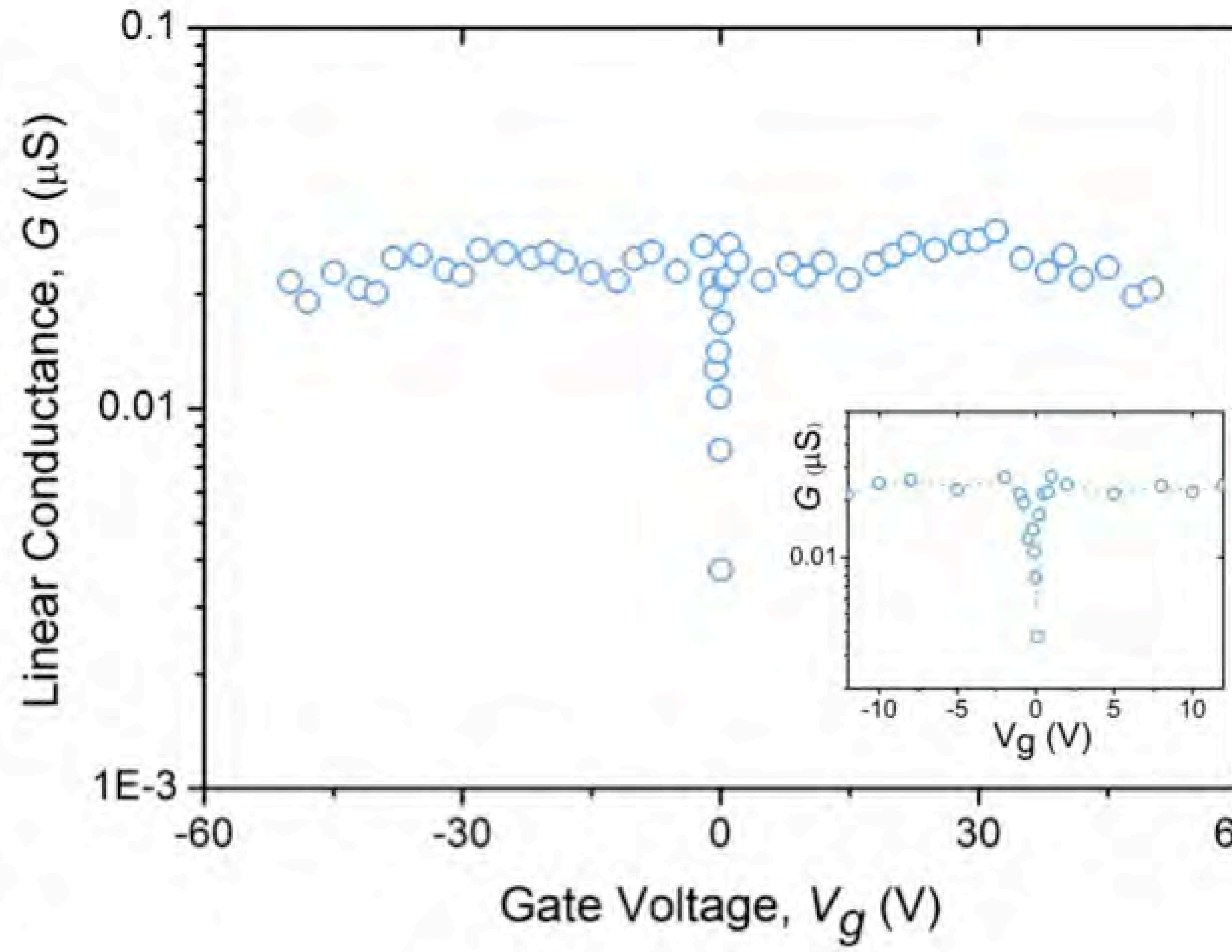


(Left) Temperature dependence of the conductance, G *vs.* T of an FLG (9nm) device. (Inset) Normalized resistivity as function of temperature for various thicknesses graphitic devices [APL **86**, 073104 (2005)].

(Right) Temperature dependence of the conductance for a bi-layer device.

Multielectrodes Devices II: Sheet Resistance and Gate Voltage





(Left) Temperature dependence of the sheet resistance of bi-layer sample. Experimental data are consistent with the literature [IEEE SENSORS 2007 Conference 558].

(Right) Low-temperature gate-modulated charge transport (~42K). The small dip suggests the presence of the Dirac point in the vicinity of V_g=0V.

II . Conclusions

Successful development of a method to electrically contact graphene (or any flat material) which has reduced contamination due to device fabrication process.

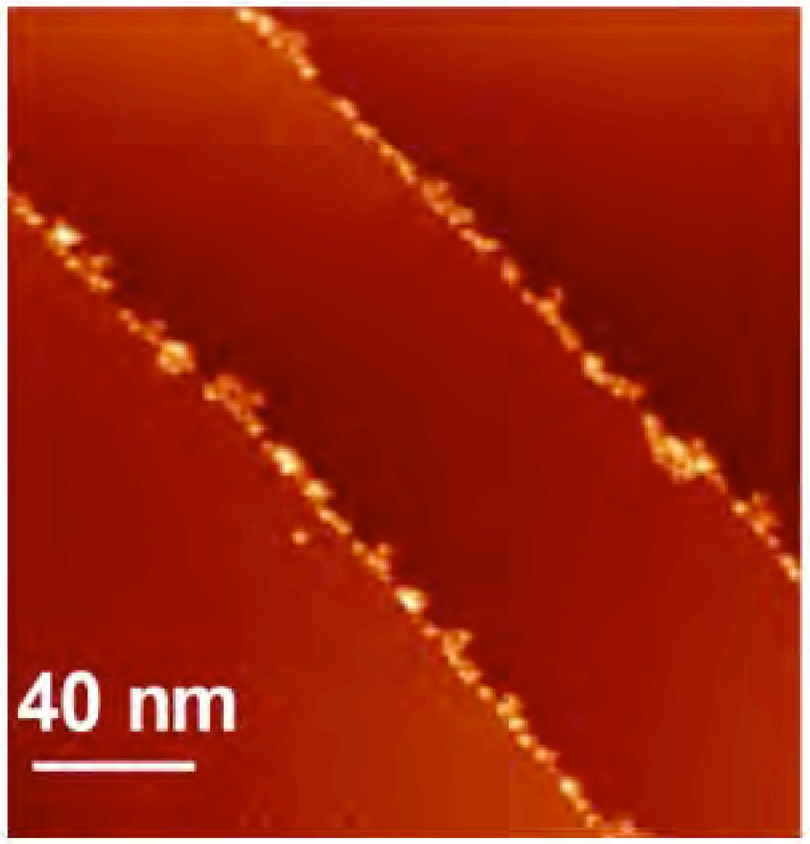
- Membrane's alignment resolution ~1µm ☑
- Membrane's fabrication (FIB → easy and versatile) ☑
- Marked substrate facilitates alignment ☑

- Long FIB times for large patterns → increases the membrane's fragility;
- Membrane planarity respect to the substrate;
- Large size graphene samples.

Compatibility with UHV technology

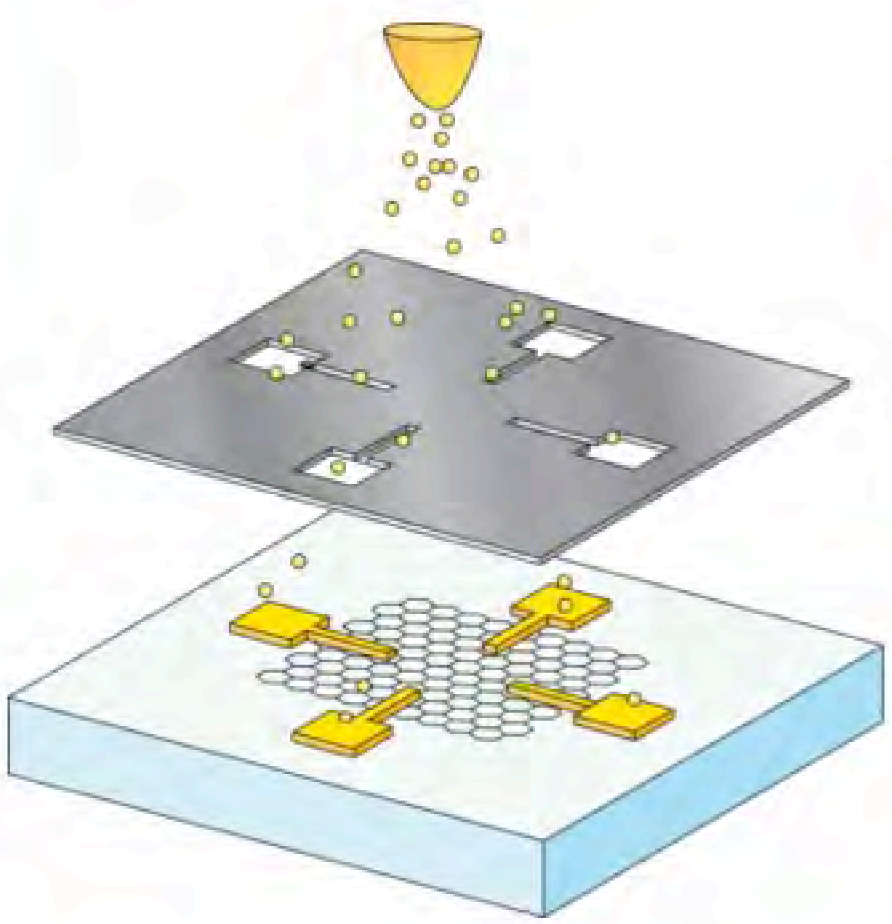
→ *all-in-vacuum* device fabrication and characterization

Acknowledgement



CVD of Graphene on Graphite @ SENSOR
Università di Brescia

E. Cavaliere (Università Cattolica, Brescia)
L. Gavioli (Università Cattolica, Brescia & TASC Laboratories, Trieste)



Stencil on graphene @ CEMES

E. Dujardin (CEMES, Toulouse)
V. Savu and J. Brugger (EPFL, Lausanne)
J. Grisolia (INSA, Toulouse)