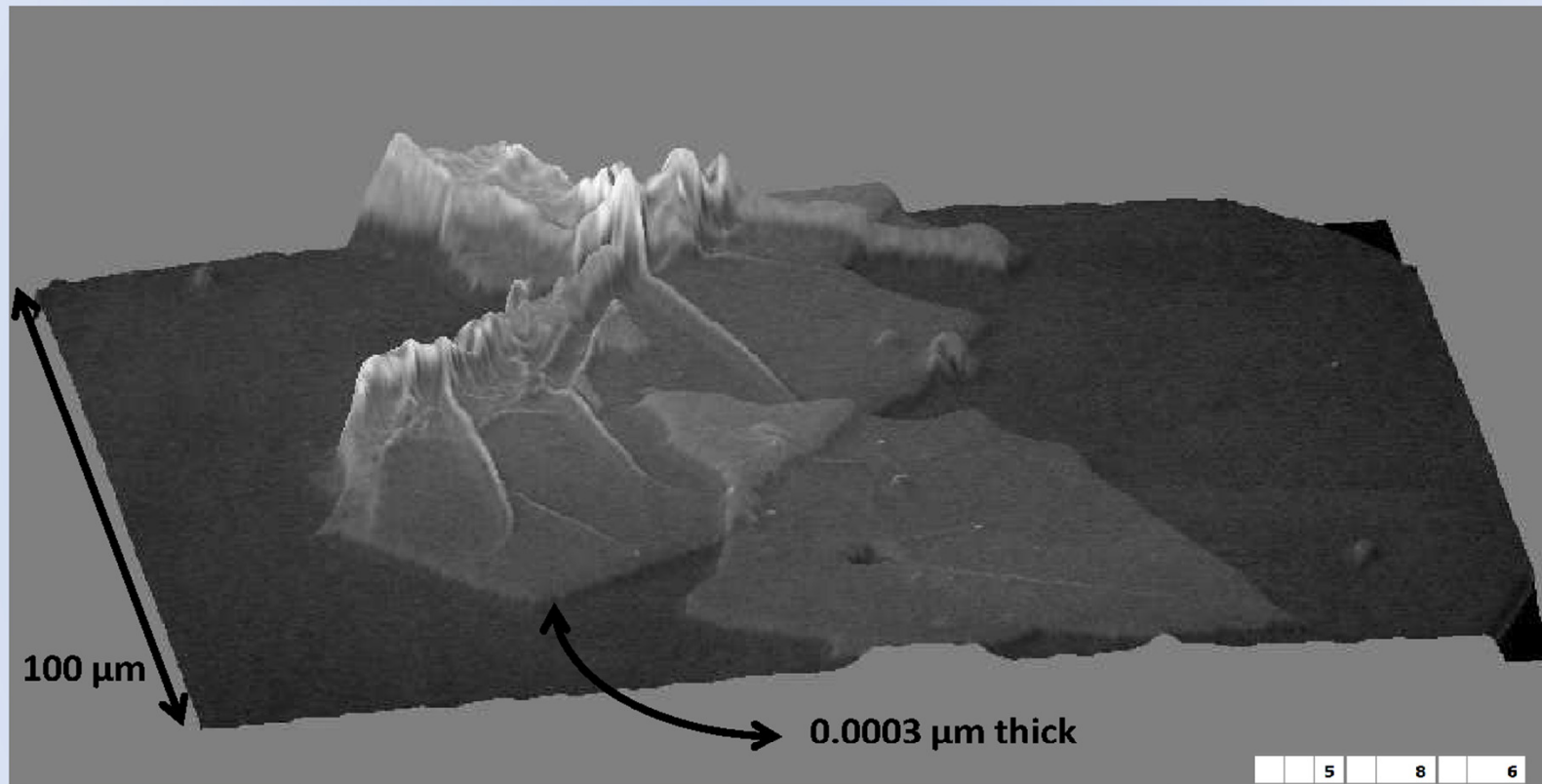


## Content

- 1 Puzzle
- 2 Imaging needs in Graphene science
- 3 Current imaging techniques
- 4 Backside Absorbing Layer Microscopy BALM
- 5 Absorbing Antireflecting Layers
- 6 Puzzle solution
- 7 Images of Graphene monolayers in Air and in Water
- ~~8 Kinetics of Graphene Reduction by  $\text{N}_4\text{H}_2$~~
- ~~9 Interaction with external agent (surfactant)~~
- 10 Water intercalation between graphene monolayers
- ~~12 Other 2D crystals: Example of  $\text{MoS}_2$~~

**Puzzle: which technique was used ?  
(graphene oxide monolayers)**



*(you may also chose sudoku grid)*

		5		8		6
6		7		2		1 9
9	1	2		3 6		
		4				5
8	5	6				9 7 3
2						4
			4 9		1 5 2	
1	2		6		4	8
5			2		6	

## Current needs for graphene imaging

Qualify/monitor graphene production

Control/monitor graphene sheet deposition/organization

Give local number of graphene sheets in graphene layers

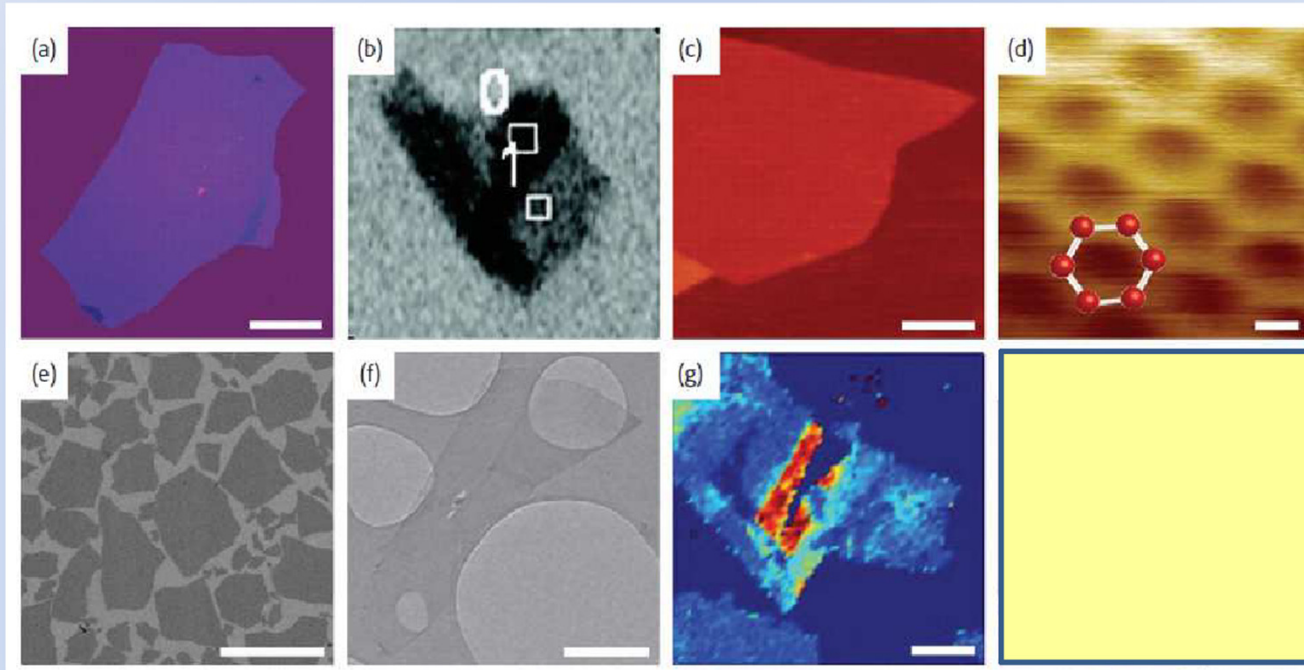
See graphene in water (Preparation and use)

Probe/monitor graphene chemistry

Visualize intercalation of impurities

Probe local conductivity

## Main current Imaging Techniques



Si/SiO <sub>2</sub> substrate	(a)	Interference - Scale bar	= 20 $\mu\text{m}$
	(b)	Ellipsometry -	?
	(c)	AFM-	1 $\mu\text{m}$
	(d)	STM-	0.0001 $\mu\text{m}$
	(e)	SEM-	20 $\mu\text{m}$
	(f)	TEM-	0.5 $\mu\text{m}$
	Si/SiO <sub>2</sub> substrate	(g)	Raman imaging

[From Kim, Kim and Huang, Materialstoday 13 \(2010\) 28](#)



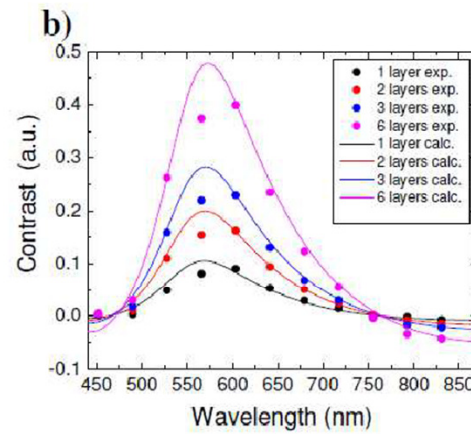
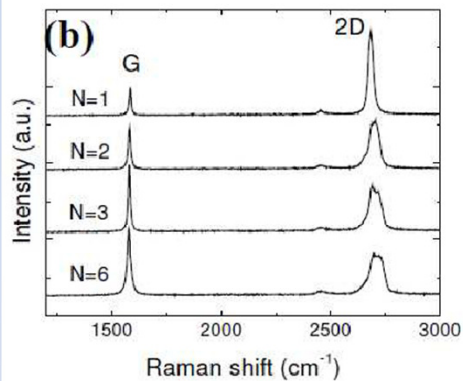
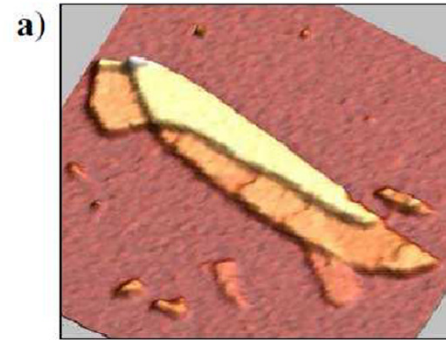
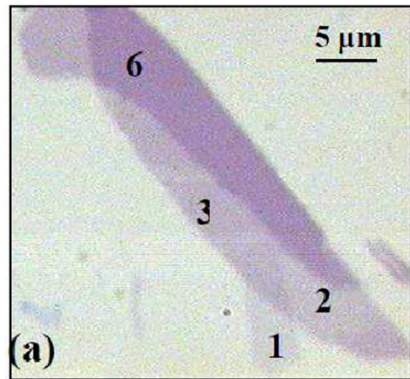
## Most current OPTICAL Imaging Techniques

Si/SiO<sub>2</sub> substrate +

Raman

+

Confocal

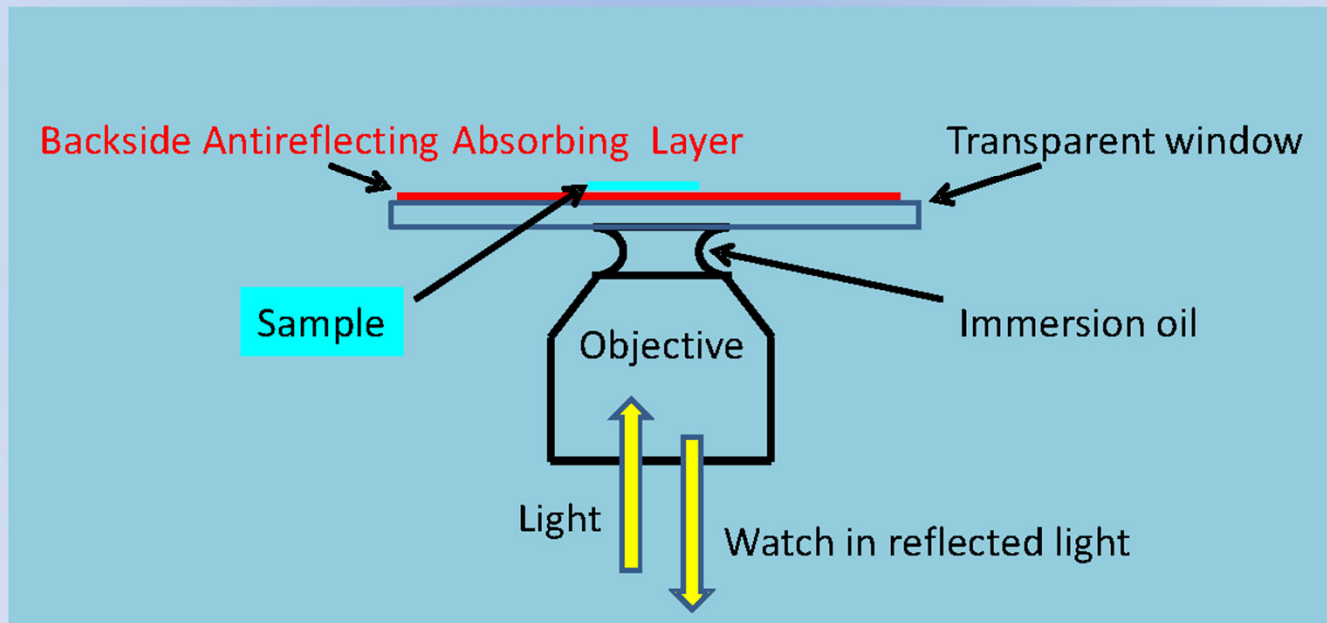


From arXiv:0705.2645v1 [cond-mat.matr-sc], Casighari et al. 2007

## Backside Absorbing Layer Microscopy (BALM)

Lateral resolution 300 nm

Z- Resolution 1  $\mu\text{m}$



## Backside Anti-Reflecting Absorbing Layers

### Why is it so sensitive ?

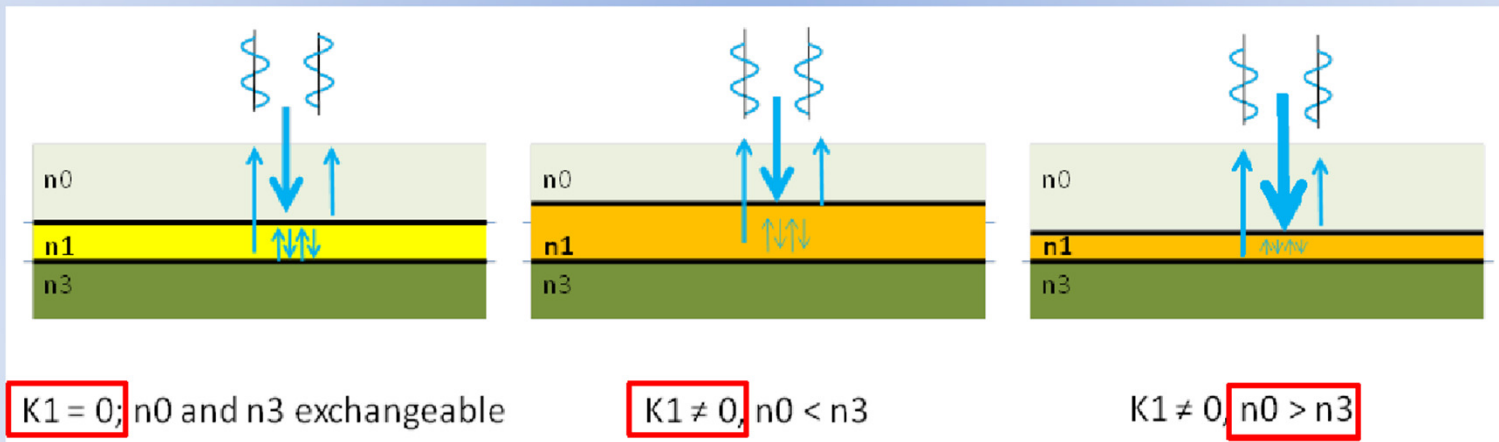
Contrast =  $\frac{I_{obj} - I_{back}}{I_{obj} + I_{back}}$  Max. with background extinction  $I_{back} = 0 \Rightarrow$  AR Surfaces

### What is new?

Incident  $n_0$

$$\tilde{n}_1 = n_1 - jk_1$$

Emergent  $n_3$



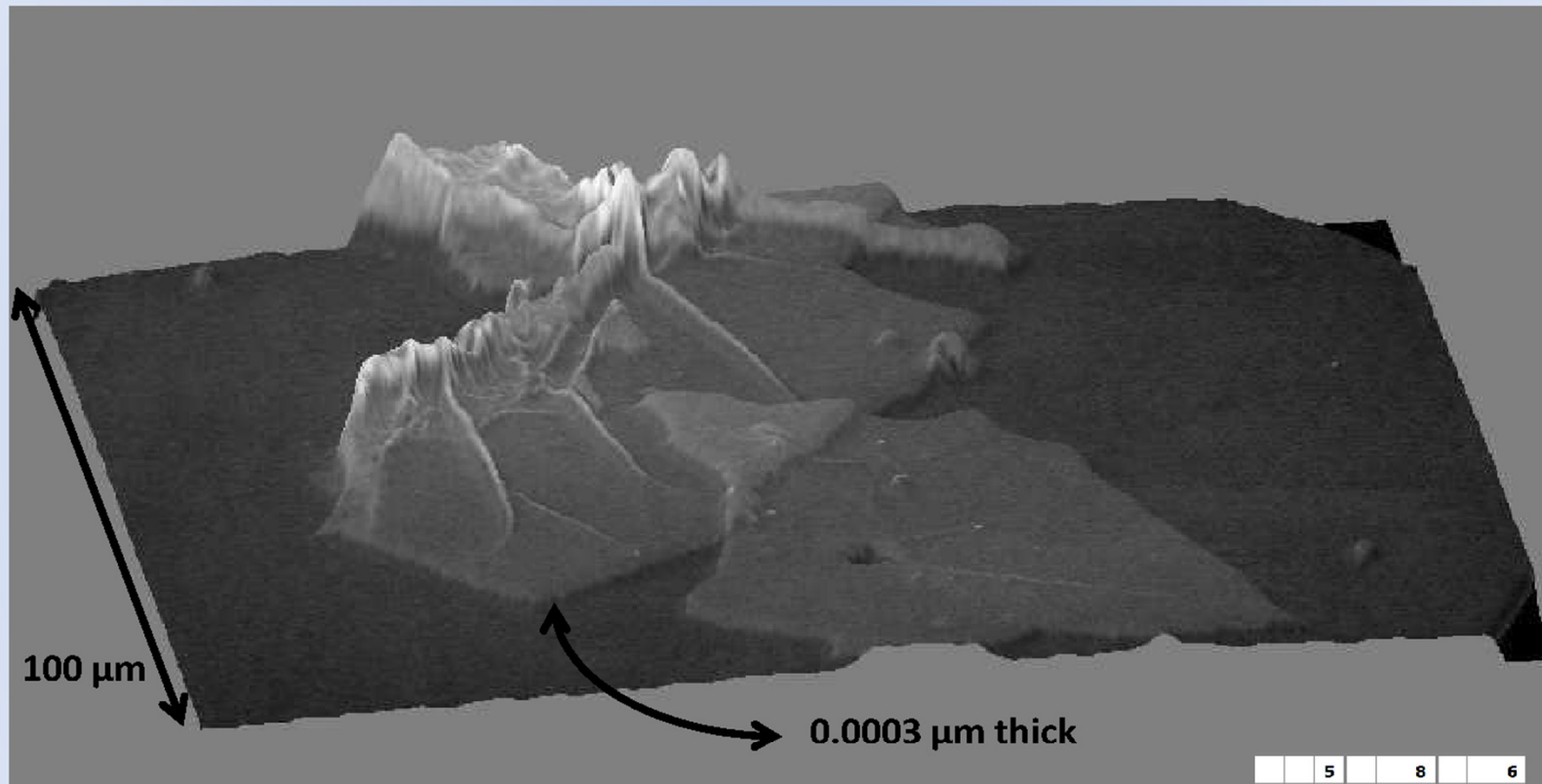
New AR surfaces

transparent and conductive



BALM

**Puzzle: which technique was used ?  
(graphene oxide monolayers)**



*(you may also chose sudoku grid)*

		5		8		6
6		7		2		1 9
9	1	2		3 6		
		4				5
8	5	6				9 7 3
2						4
			4 9		1 5 2	
1	2		6		4	8
5			2		6	

## Answer

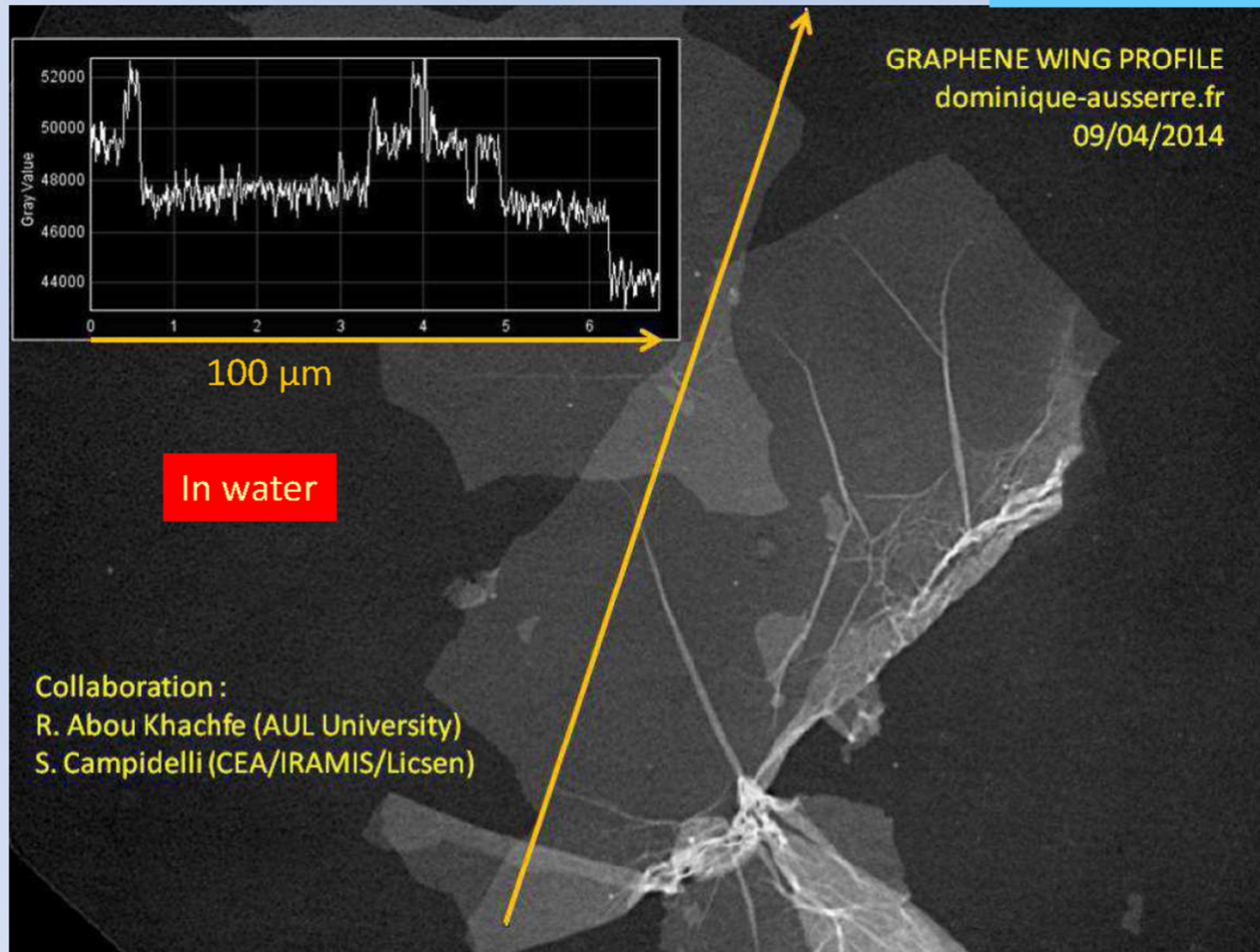
<b>4</b>	<b>3</b>	<b>5</b>	<b>9</b>	<b>1</b>	<b>8</b>	<b>7</b>	<b>2</b>	<b>6</b>
<b>6</b>	<b>8</b>	<b>7</b>	<b>5</b>	<b>4</b>	<b>2</b>	<b>3</b>	<b>1</b>	<b>9</b>
<b>9</b>	<b>1</b>	<b>2</b>	<b>7</b>	<b>3</b>	<b>6</b>	<b>5</b>	<b>8</b>	<b>4</b>
<b>3</b>	<b>4</b>	<b>1</b>	<b>8</b>	<b>7</b>	<b>9</b>	<b>2</b>	<b>6</b>	<b>5</b>
<b>8</b>	<b>5</b>	<b>6</b>	<b>1</b>	<b>2</b>	<b>4</b>	<b>9</b>	<b>7</b>	<b>3</b>
<b>2</b>	<b>7</b>	<b>9</b>	<b>3</b>	<b>6</b>	<b>5</b>	<b>8</b>	<b>4</b>	<b>1</b>
<b>7</b>	<b>6</b>	<b>8</b>	<b>4</b>	<b>9</b>	<b>3</b>	<b>1</b>	<b>5</b>	<b>2</b>
<b>1</b>	<b>2</b>	<b>3</b>	<b>6</b>	<b>5</b>	<b>7</b>	<b>4</b>	<b>9</b>	<b>8</b>
<b>5</b>	<b>9</b>	<b>4</b>	<b>2</b>	<b>8</b>	<b>1</b>	<b>6</b>	<b>3</b>	<b>7</b>



## Answer to other Puzzle: The technique was BALM

Backside Absorbing Layer Microscopy

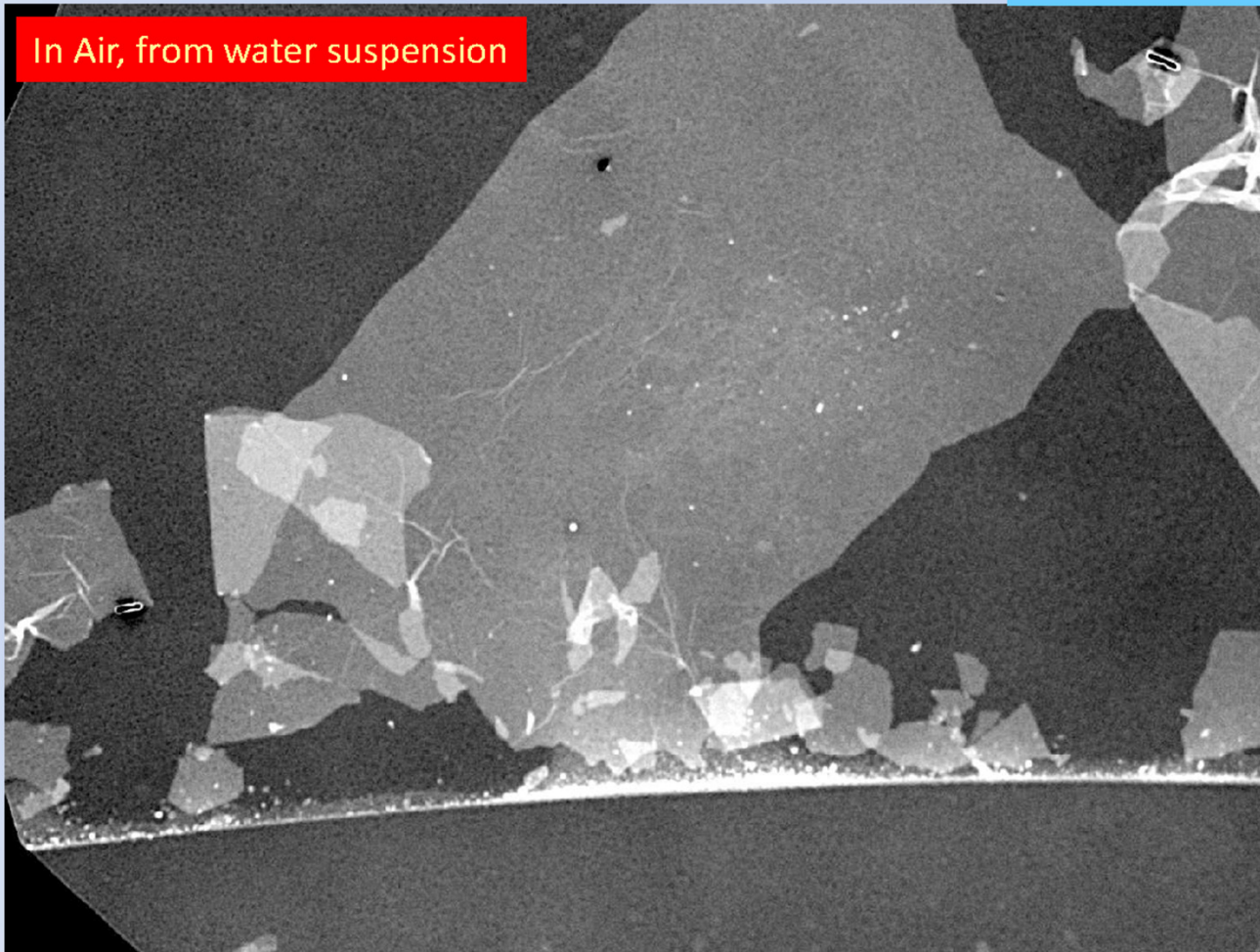
*Looks like AFM*



# Graphene (GO) sheets

Collaboration S. Campidelli, CEA Saclay

Looks like SEM

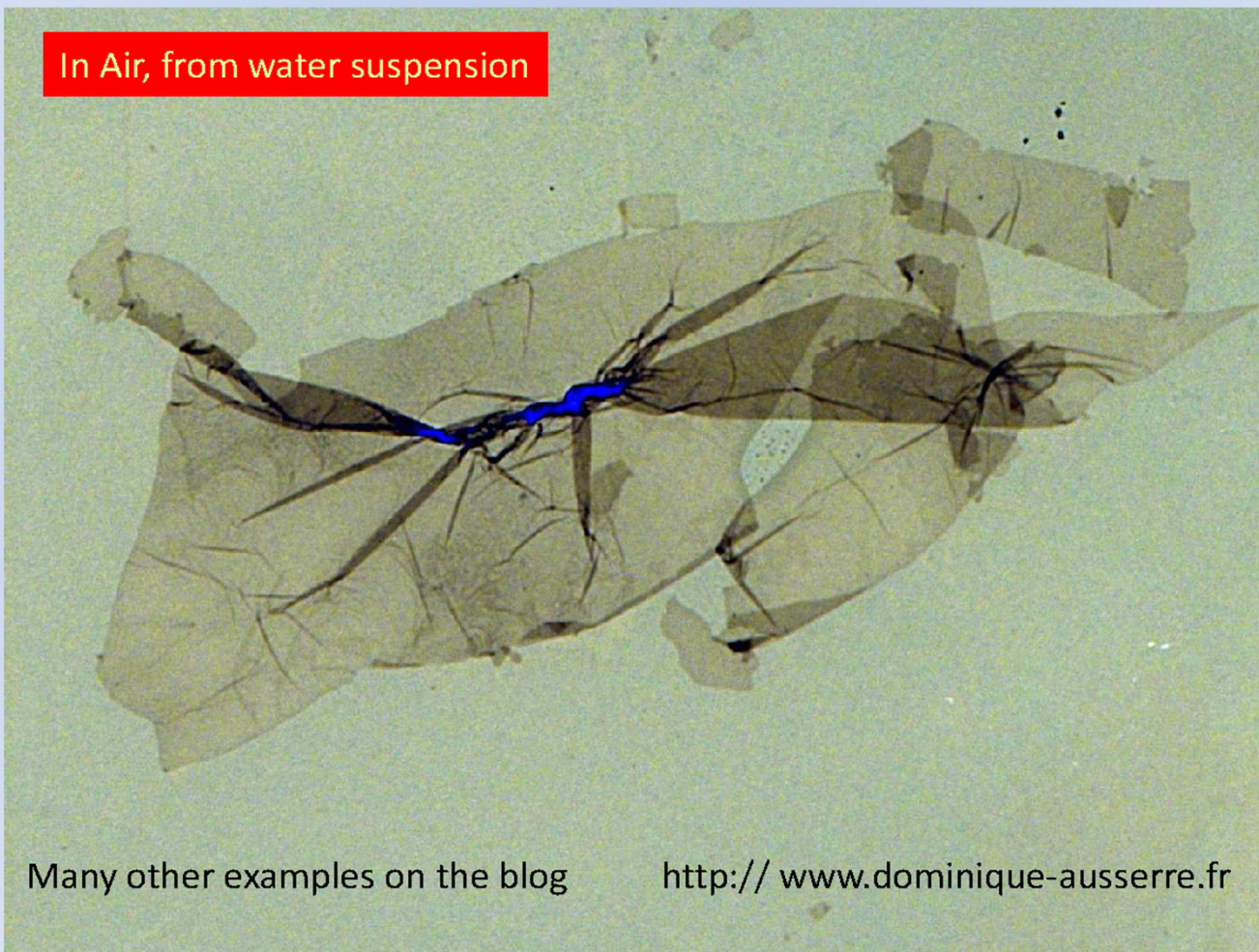


Dominique Ausserré – Nanotek 2014 - San Francisco 141203



## Graphene (GO) sheets observed in white light

In Air, from water suspension



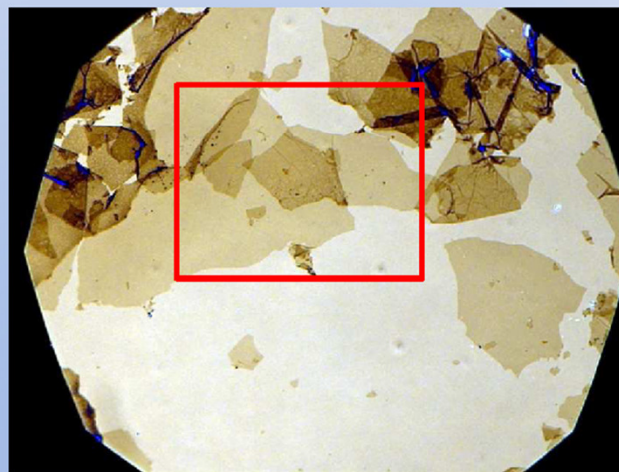
Many other examples on the blog

[http:// www.dominique-ausserre.fr](http://www.dominique-ausserre.fr)

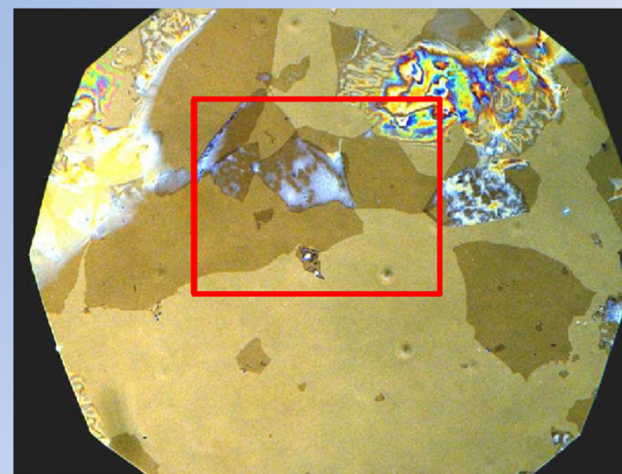
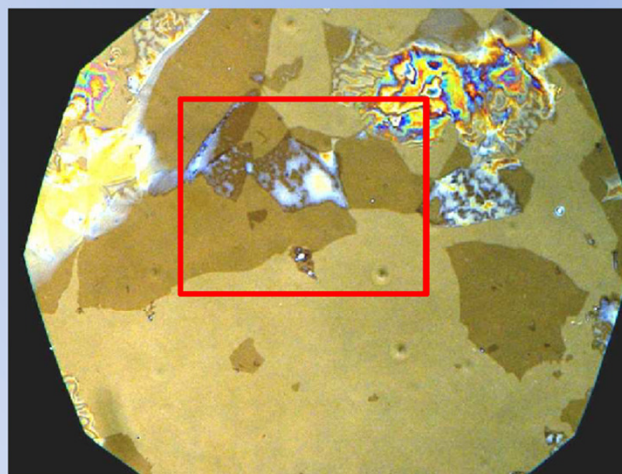
# Same graphene sheets in air and in water

100  $\mu\text{m}$

In air



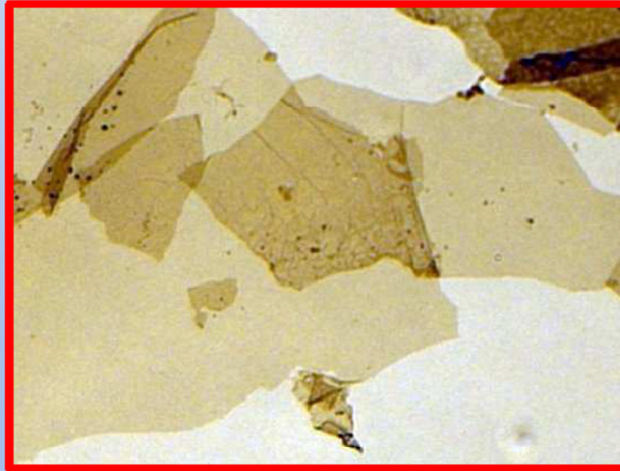
in water



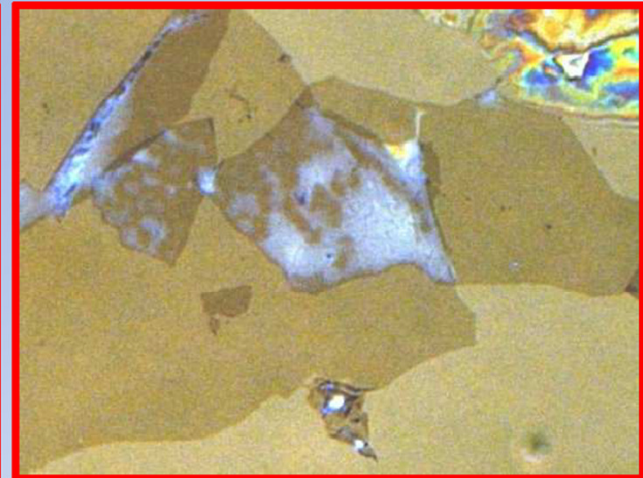
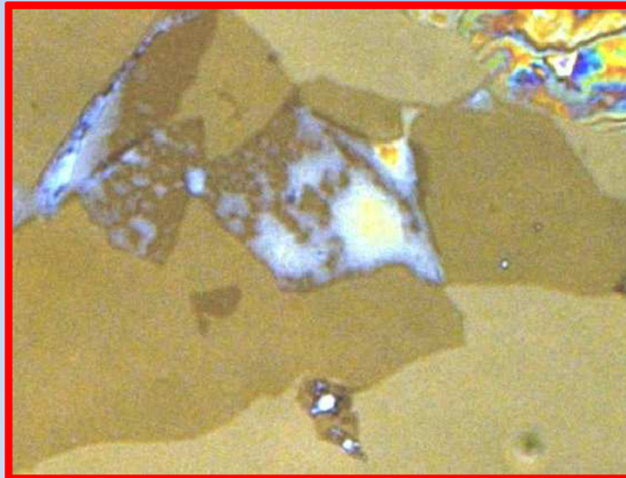


Same graphene sheets in air and in water: zoom x 2.5

In air



in water

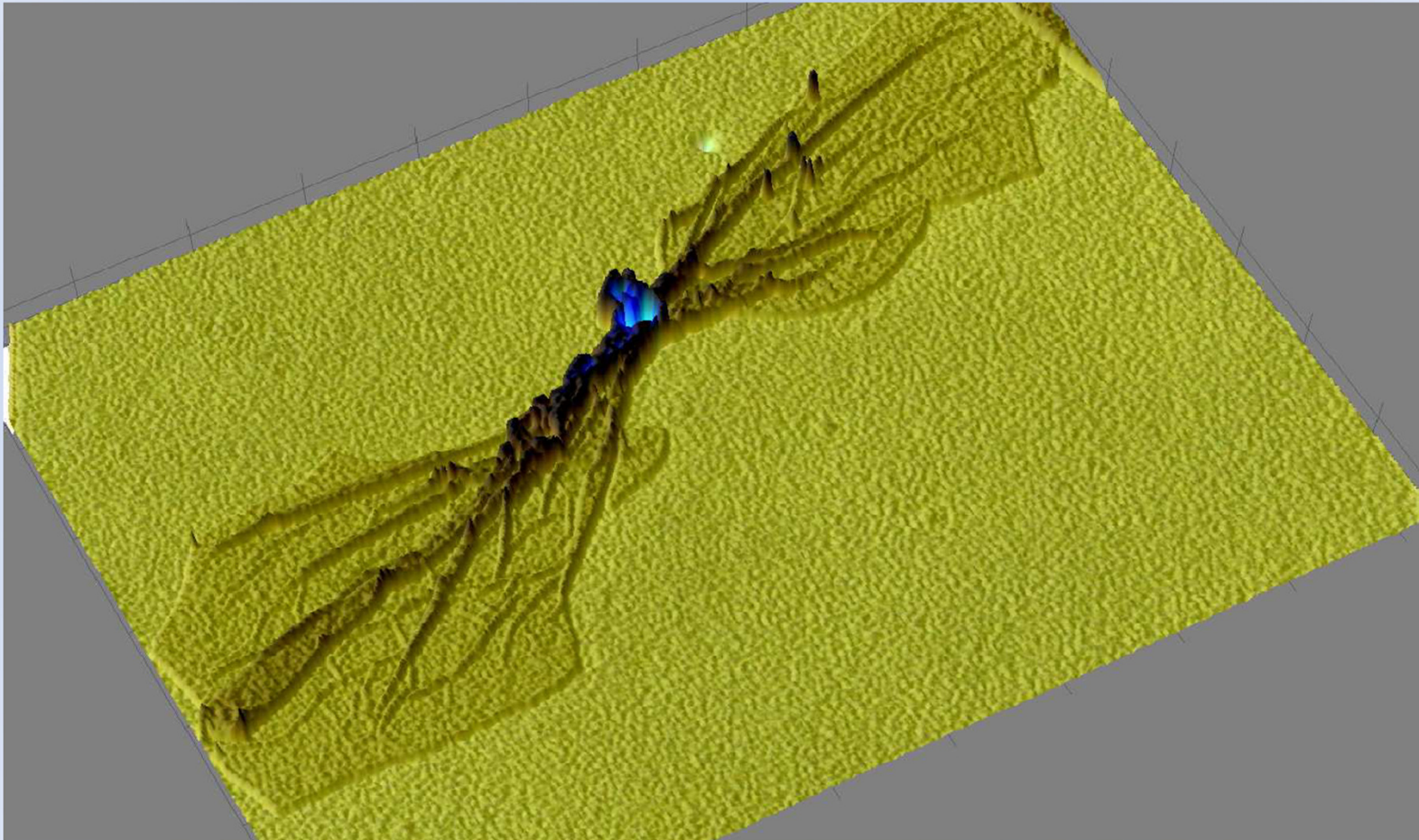




## BALM answer to current needs in graphene imaging

- Qualify/monitor in situ graphene production
- Control/monitor graphene sheets deposition/organization - Switch from air to water
- Count local number of superimposed graphene sheets
- Probe/monitor in situ graphene chemistry
- Visualize impurity intercalation
- Probe local conductivity
- Do all these things at the same time on a same sample without destroying it
- Do all these things fast and at a low cost

Thanks for your attention !



*Dominique Ausserré – Nanotek 2014 - San Francisco 141203*

**Dominique Ausserré**, Institut des Molécules et Matériaux du Mans, CNRS UMR 6382

[ausserre@univ-lemans.fr](mailto:ausserre@univ-lemans.fr)

**blog** [http:// www.dominique-ausserre.fr](http://www.dominique-ausserre.fr)

**Collaboration with:**

*Refahi Abou Khachfe, AUL University, Beyrouth, Liban*

*numerical calculations*

*Stephane Campidelli, CEA/DSM/IRAMIS, Saclay, France*

*graphene*

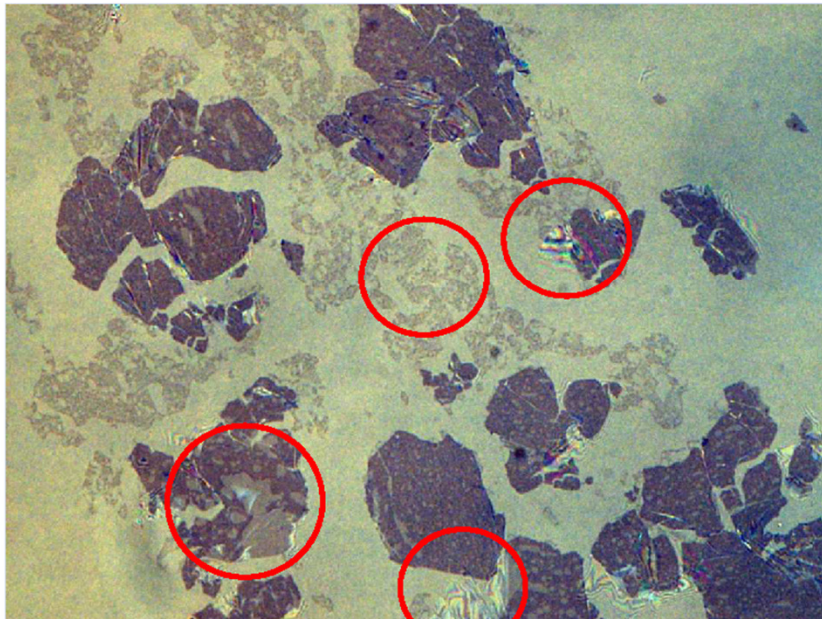
**Granted by ANR:** PNANO-07-050

**BALM commercialized by the Startup:** Watch Live S.A.S. (created today)

**Informations**

[ausserre@gmail.com](mailto:ausserre@gmail.com)

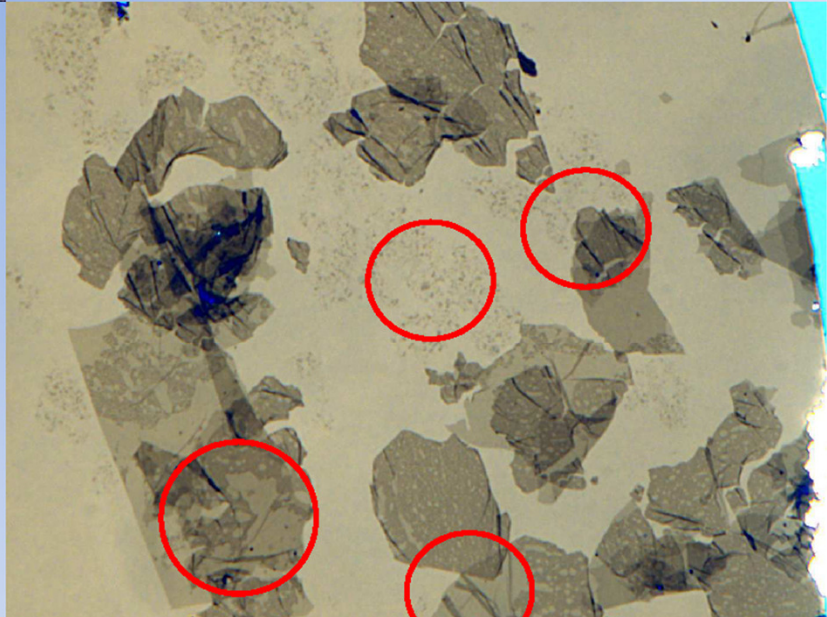




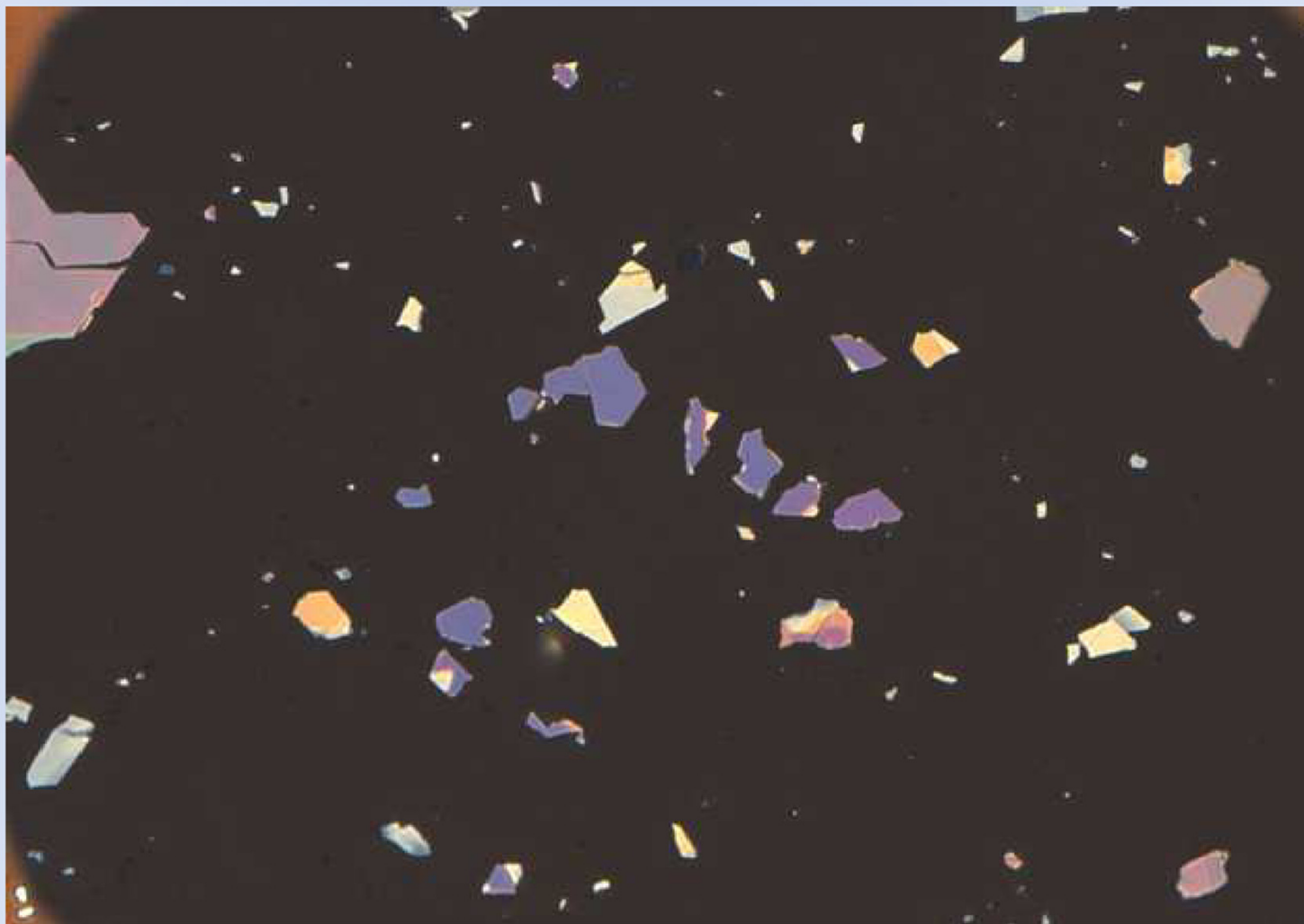
Water

Surface Chemistry

Air



## Graphene analogs: example of MoS<sub>2</sub>



*Dominique Ausserré – Nanotek 2014 - San Francisco 141203*



## Advantages

Simple

Low cost

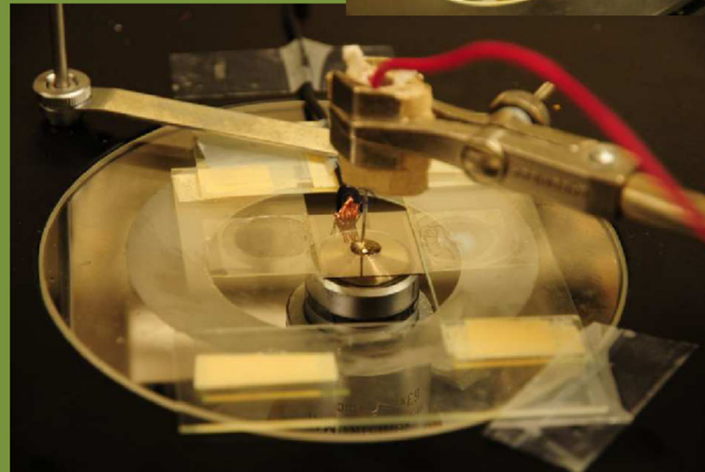
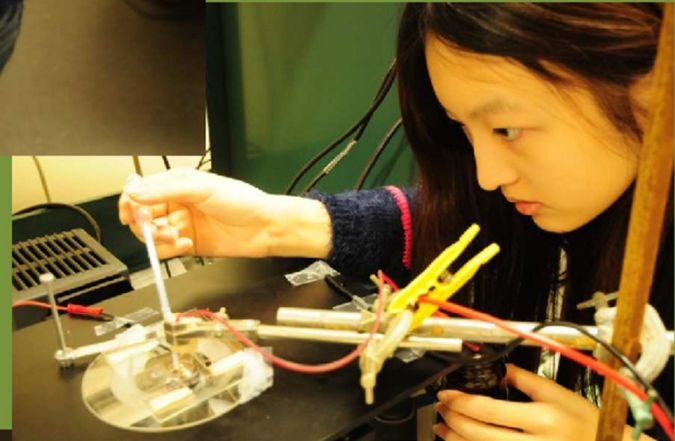
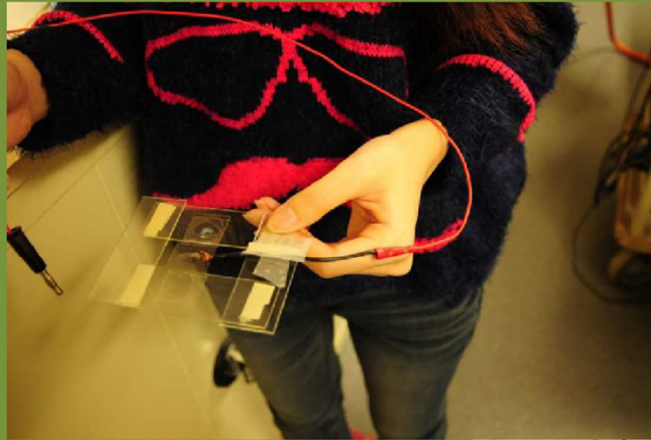
Non destructive

In situ

Real time

Extreme sensitivity

Implementation



Xia Yin

**D. AUSSERRE, R. ABOU KHACHFE, C. AMRA, and M. ZERRAD**

Absorbing Anti-Reflecting Layers

[arXiv:1405.7672v1](https://arxiv.org/abs/1405.7672v1) [physics.optics] 2014

**D. AUSSERRE, R. ABOU KHACHFE, L. ROUSSILLE, G. BROTONS, L. VONNA, F. LEMARCHAND, M. ZERRAD, and C. AMRA**

*J Nanomed Nanotechnol*, **5**:4 (2014); doi: [10.4172/2157-7439.1000214](https://doi.org/10.4172/2157-7439.1000214)

Contact

[ausserre@univ-lemans.fr](mailto:ausserre@univ-lemans.fr)

(+33) 6 08 28 48 00

blog: [www.dominique-ausserre.fr](http://www.dominique-ausserre.fr)

## Première Observation Visuelle de la Double Couche Electrochimique par une Technique d'Imagerie

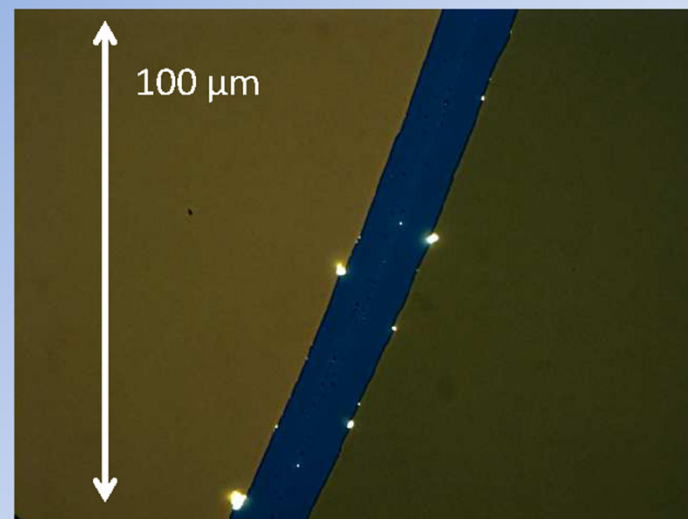
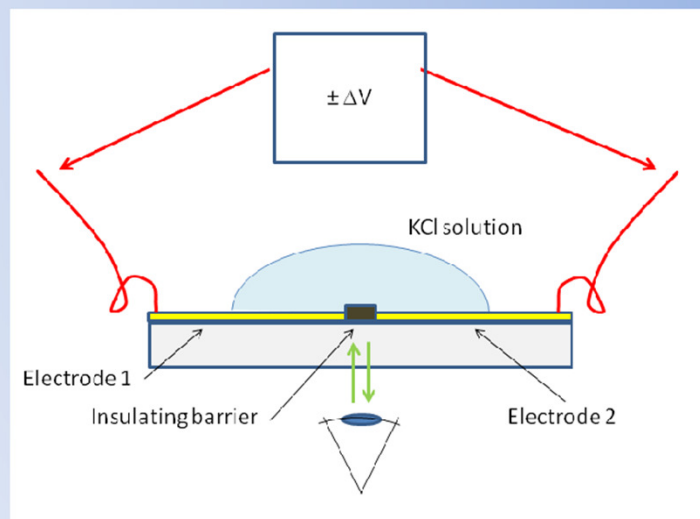
Pre-conference workshop



*Dominique Ausserré, Institut des Molécules et Matériaux du Mans, CNRS UMR 6282.*

*Mathieu Lazerges, UTCBS- ENSCP CNRS UMR 8258 - INSERM U1022*

*Refahi Abou Khachfe, AUL University, Beyrouth, Liban*



*Fig. 2 Common microscopy techniques for imaging GBS. Optical microscopy images of graphene based on (a) interference (scale bar = 20  $\mu\text{m}$ ) (adapted from<sup>1</sup>, with permission, American Association for the Advancement of Science) and (b) ellipsometry (adapted from<sup>34</sup>, with permission, ACS Publication), respectively. Both methods rely on dielectric coated silicon with proper thickness and optimal illuminating wavelength. (c) Atomic force microscopy (AFM, scale bar = 1  $\mu\text{m}$ ) imaging of graphene sheet on  $\text{SiO}_2$  surface (adapted from<sup>1</sup>, with permission, American Association for the Advancement of Science). AFM can give accurate height measurement on smooth surfaces but it is rather low-throughput. (d) Scanning tunneling microscopy (STM, scale bar = 0.1 nm, adapted from<sup>35</sup>, with permission, National Academy of Sciences, U.S.A.) can produce high-resolution, atomic-scale images. But it is too low-throughput for routine imaging. Electron microscopy, such as (e) scanning electron microscopy (SEM, scale bar = 20  $\mu\text{m}$ ) and (f) transmission electron microscopy (TEM, scale bar = 500 nm, adapted from<sup>28</sup>, with permission, Nature Publishing Group) also needs to use special substrates for imaging GBS. (g) Raman imaging (scale bar = 3  $\mu\text{m}$ , adapted from<sup>38</sup>, with permission, Elsevier) is particularly useful for identifying the number of layers for graphene samples. But substrates that have low intrinsic fluorescence and can efficiently dissipate laser heating are required. (h) No techniques are available yet for high-throughput imaging of GBS on plastic surfaces or even in solution (inset).*