12th European FIB Users Group Meeting (EFUG 2008)
29-09-2008 Maastricht, The Netherlands

Customization of Water Vapour Assisted FIB Milling on Diamond Technology

Simone Magni, Alanna S. Campbell, Eugene Gregoryanz

Centre for Science at Extreme Conditions

School of Physics and Astronomy

University of Edinburgh

Outline

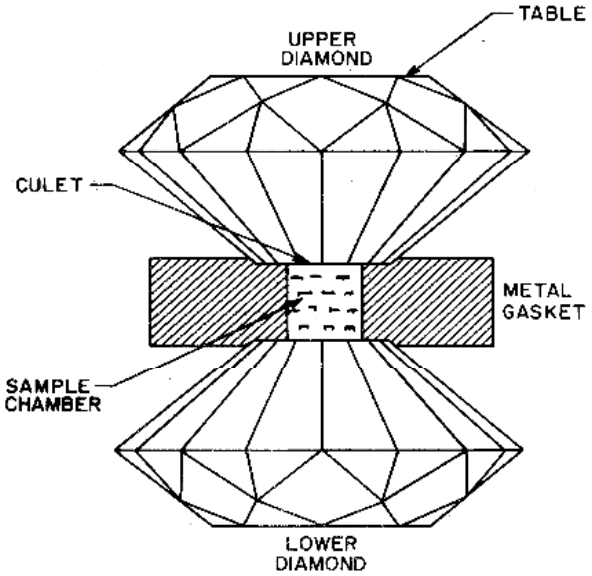
- Diamond Anvil Cells (DACs)
- Motivations
- Customization of Water Assisted Milling

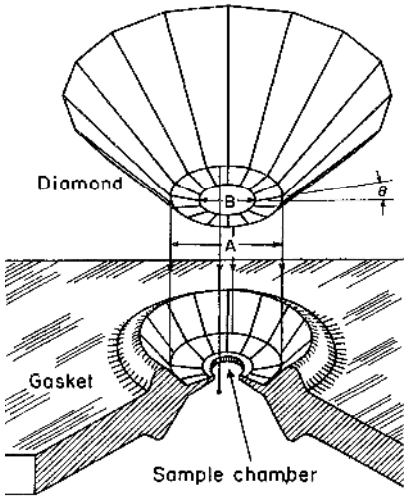
- Open Questions:
  - What is the value of the diamond sputtering rate?
  - Effects related to the H_2O assisted Milling

Diamond Anvil Cells

Diamond Anvil Cells (DACs) are employed to study the properties of matter at high compressions and over a wide temperature range.
Such a system…
•…can achieve pressures up to 300 GPa
• …allows *in situ* access for X-ray diffraction and/or optical spectroscopies (IR, Raman…)

However only a minute sample can be 'squeezed'…

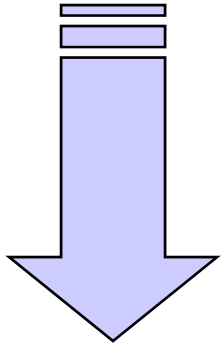


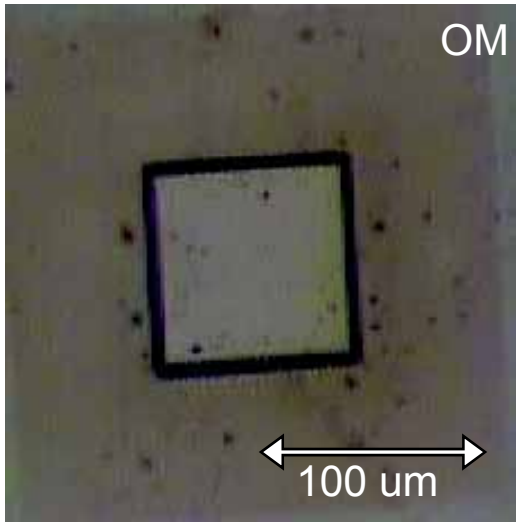


H. Mao & R.J. Hemley Rev. Mod. Phys. 66, 671 1994
A. Jayaraman Rev. Mod. Phys. 55, 65 1983

Motivations

A FIB can carve a niche (~100x100x5 um^3) into the diamond culet allowing a bigger sample volume which means…

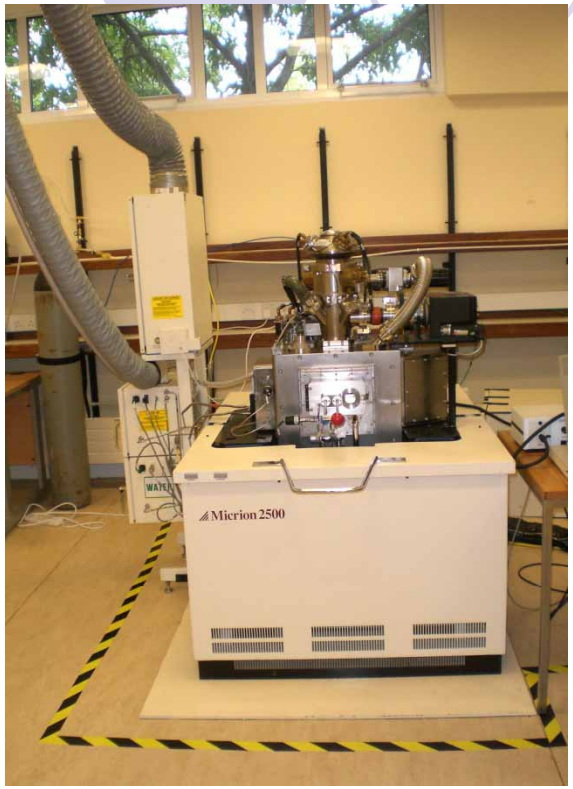


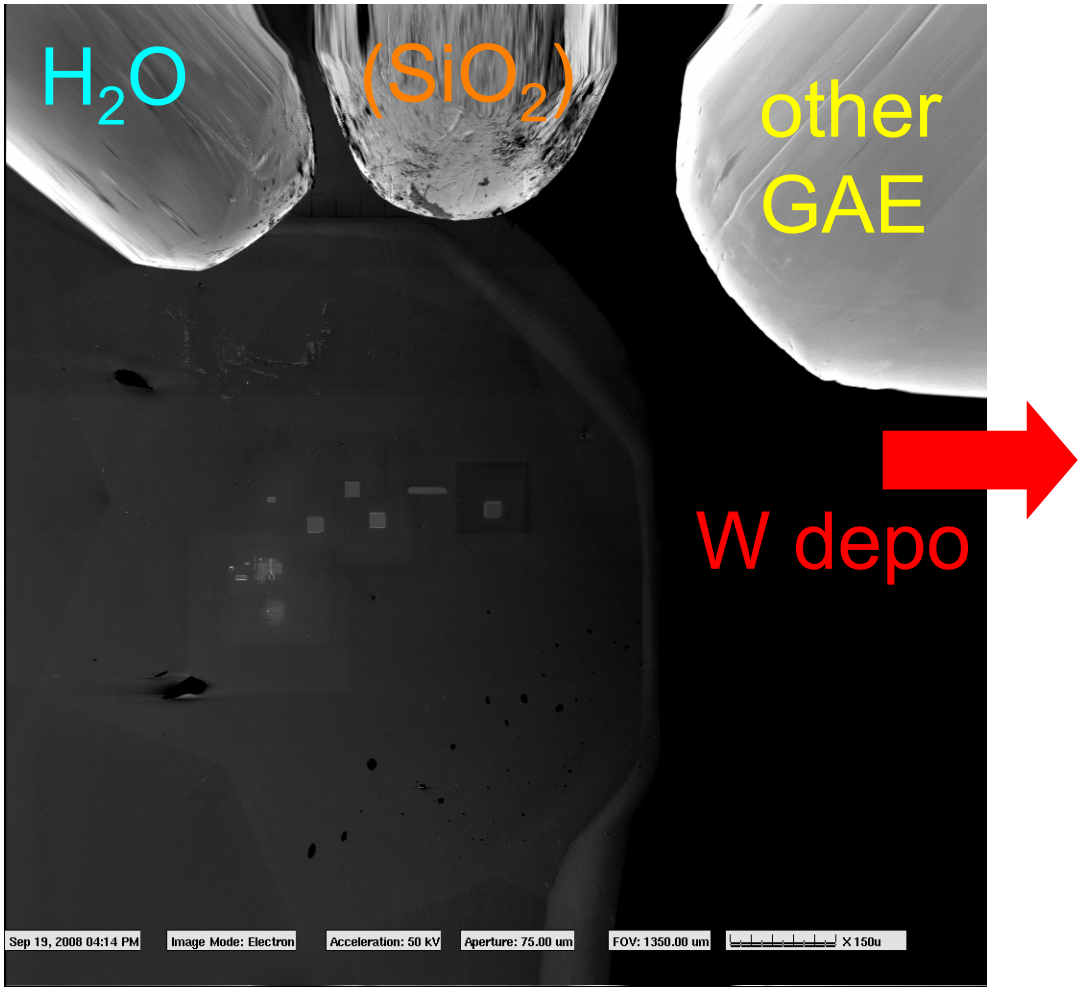


…opening the way to inelastic X-ray scattering, Fermi surface measurements and estimation of transport properties for high pressure physics!

CSEC FIB Laboratory

School of Physics & Astronomy - UoE

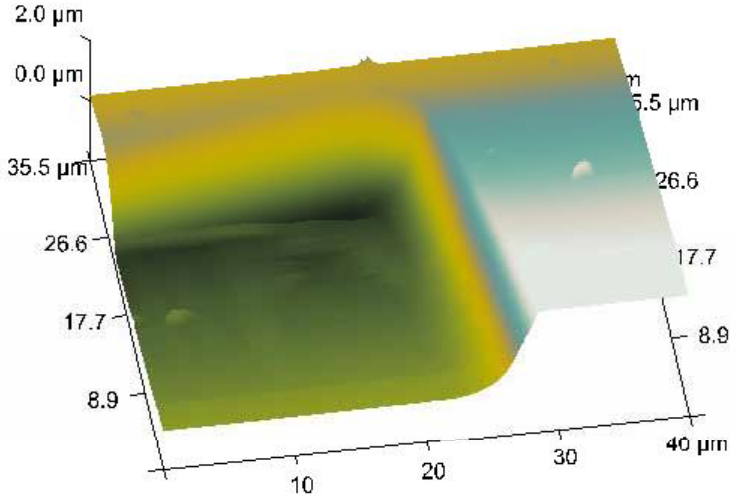


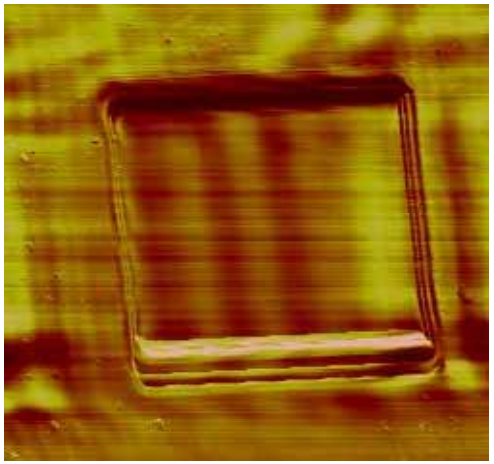


- **FEI - Micrion 2500**
- MFG: 1998
- Acceleration Energy: 10-50 kV
- Beam Currents: 5 pA - 11nA

Customization of Water-Vapour Milling (1)

First attempts: H_2O injection without changing dwell time and refresh time…

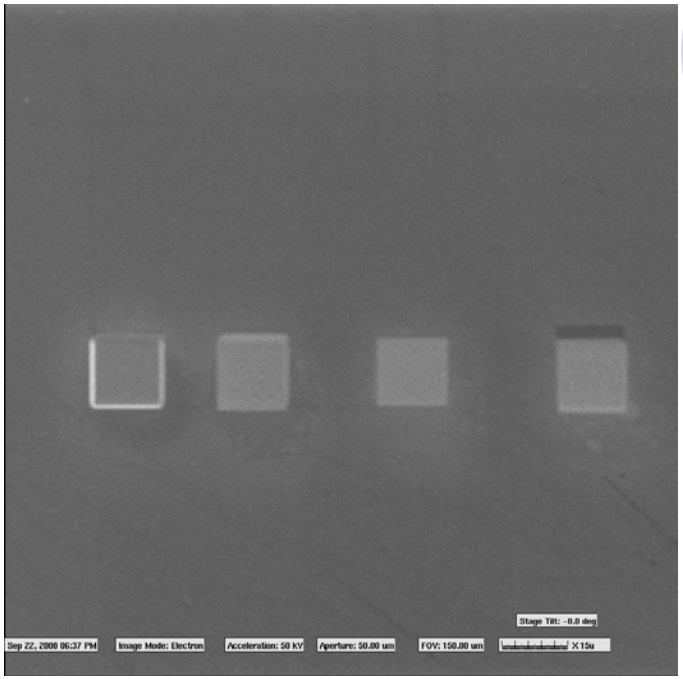




	Standard milling (3.0us) w/o e.f.g.	Standard milling (3.0us) w/t e.f.g.	Standard milling (3.0us) + H2O inject.
Sputtering rate $\left[\mu m^3/nC\right]$	0.078	0.083	0.218
Enhancing fact.	1.0	1.1	2.8

Customization of Water-Vapour Milling (2)

Devised an *ad hoc* milling recipe for H_2O (dwell 0.5 us, refresh 10.0ms, c.f. standard milling 3.0 us and 10.0 us)

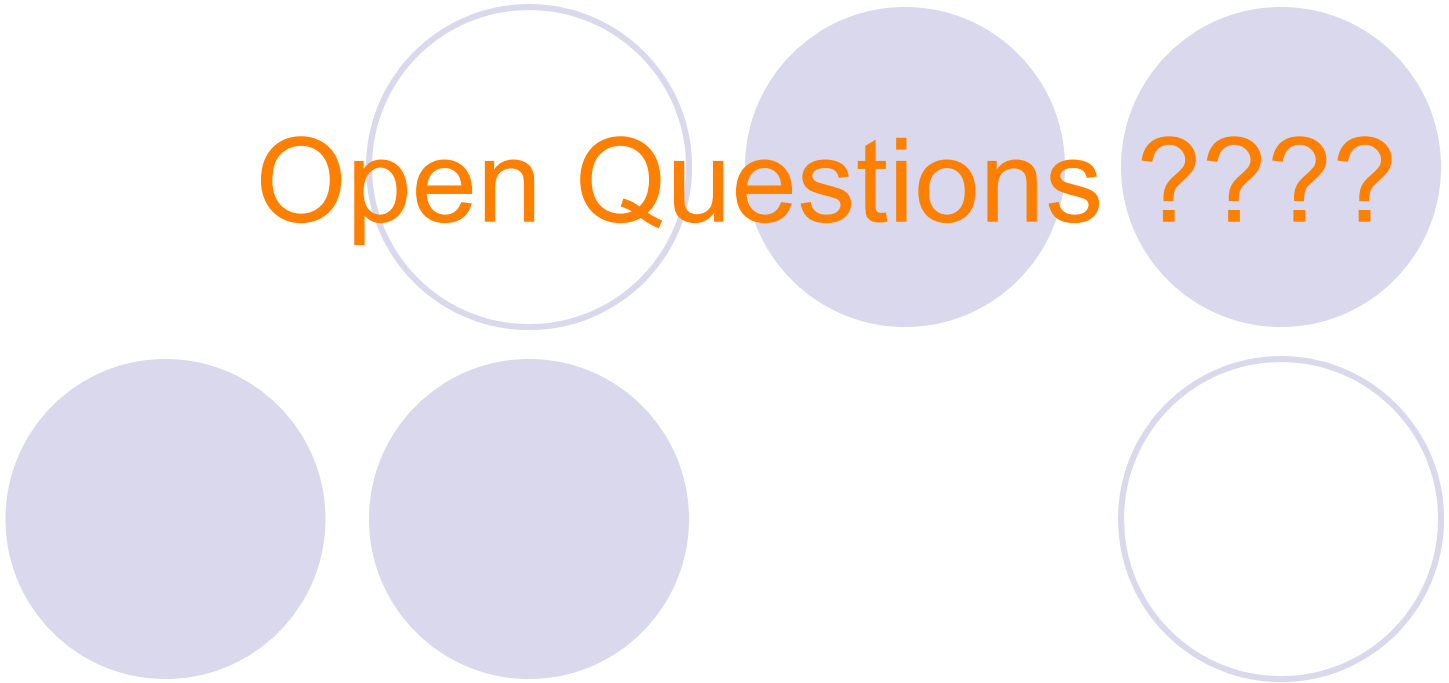


Recipe	Sputtering rate $\left[\mu m^3 / nC\right]$	Enhancing factor
Standard milling (3.0us)	0.126	1.0
Devised Recipe (0.5us) only!	0.133	1.1
Standard milling (3.0us) and H_2O injection (3.0 torr)	0.346	2.8
Devised Recipe (0.5us) and H_2O injection (3.0 torr)	0.724	5.8

T.J. Stark et al. J. Vac. Sci. Technol. B 13, 2565 (1995)

P.E. Russel et al. J. Vac. Sci. Technol. B 16, 2494 (1998)

Open Questions ????



Experimental Sputtering rate of Diamond?

- E.g. in Orloff's book [1] two values:

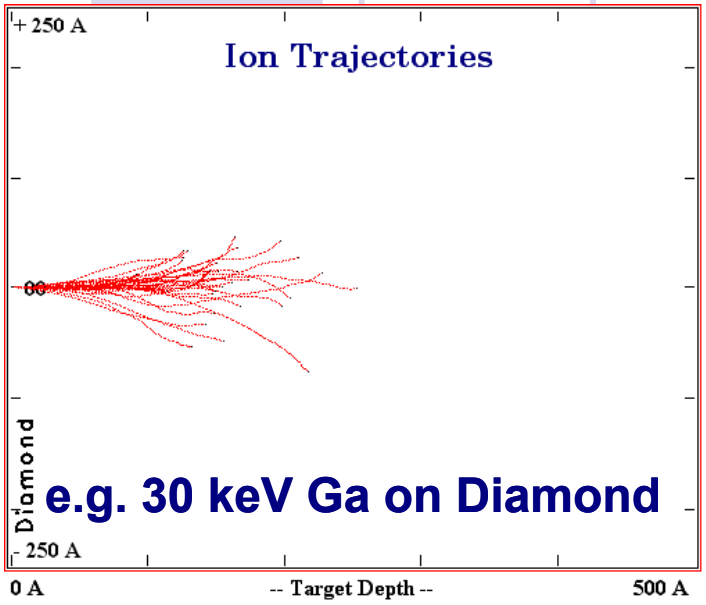
$$Y_t = 0.15,\ 0.18 \left[\mu\mathrm{m}^3 / \mathrm{nC} \right]$$

- In literature [2,3] (independent from the acceleration!):

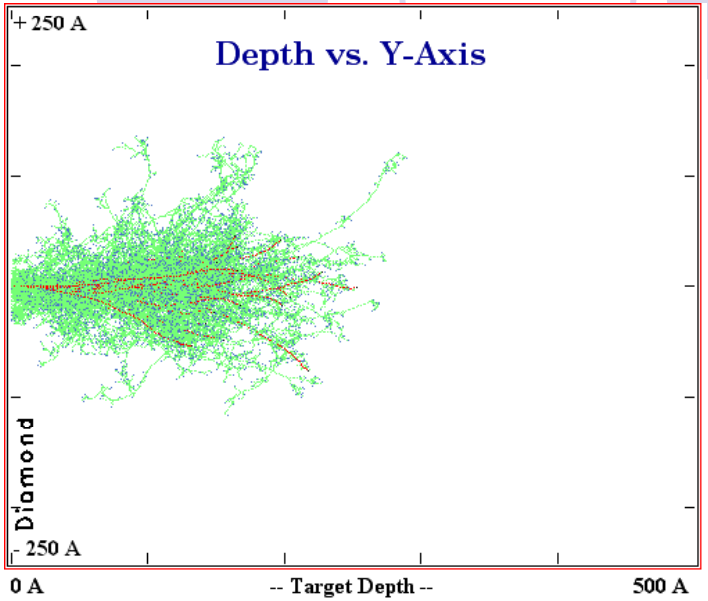
$$Y_t = 0.09 \left[\mu\mathrm{m}^3 / \mathrm{nC} \right]$$

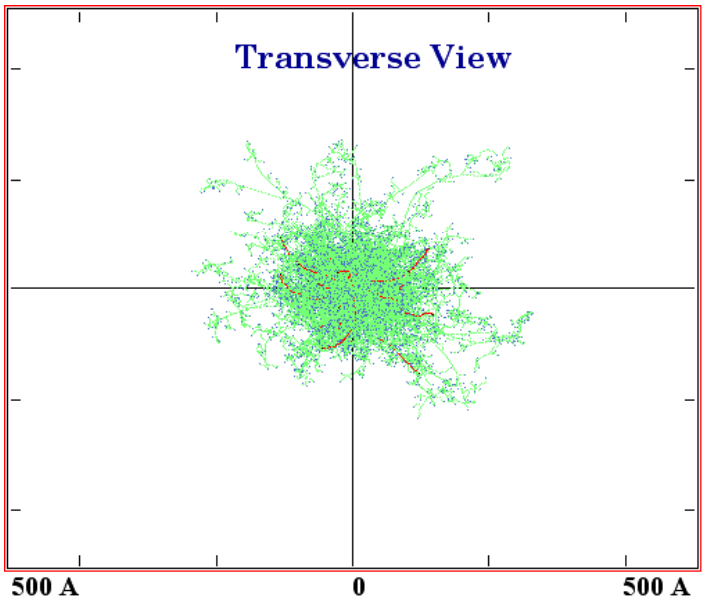
[1] Cap.6 in J. Orloff et al. "High Resolution Focused Ion Beams: FIB and Applications" Kluver Academic (2003)
[2] Adams et al. J. Vac. Sci. Technol. B 21, 2334 (2003) ⇒ 25 keV
[3] A. Stanishevsky Thin Solid Films 398, 560 (2001) ⇒ 50 keV

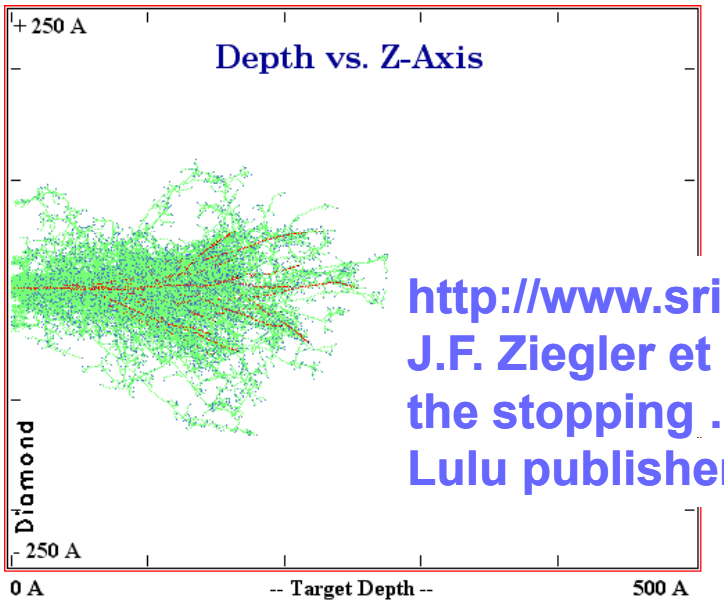
‘Theoretical’ Sputtering rate for Diamond



e.g. 30 keV Ga on Diamond

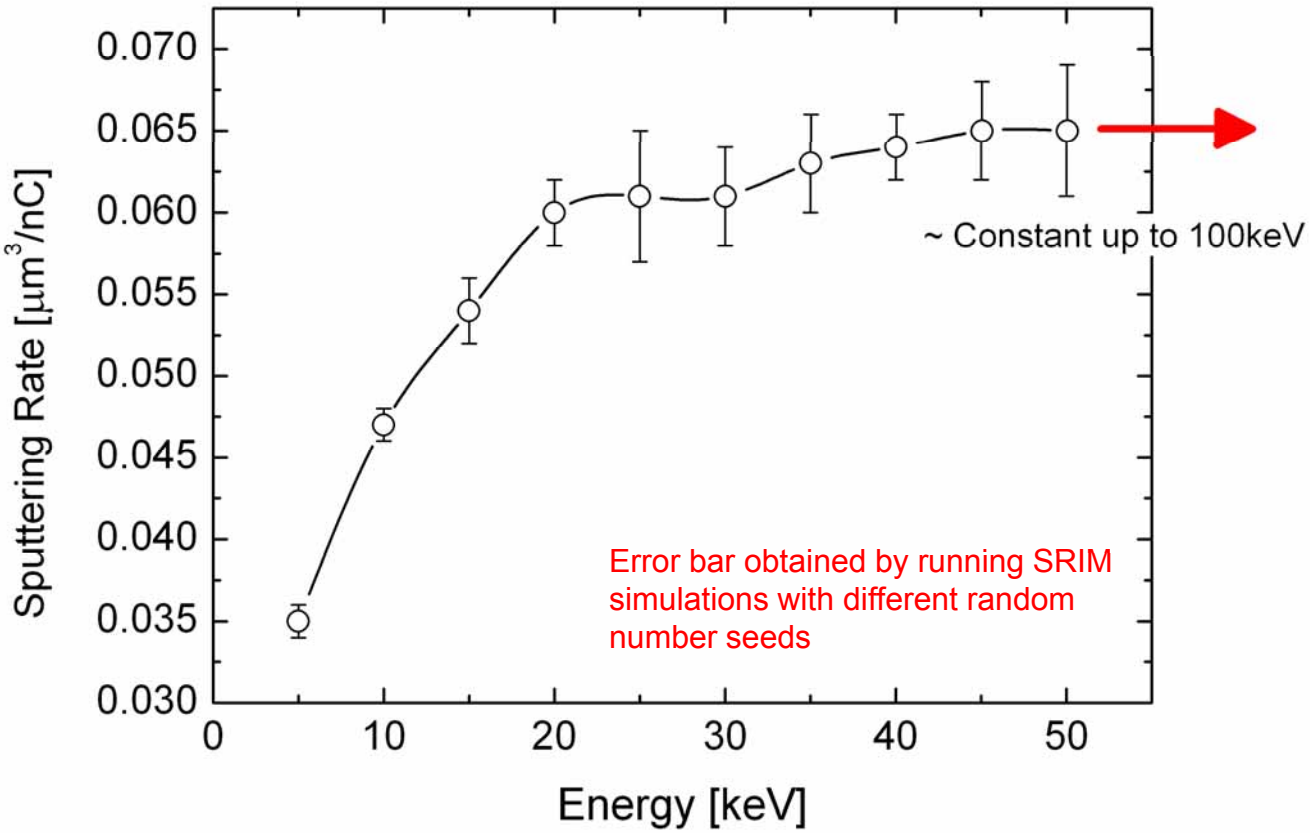






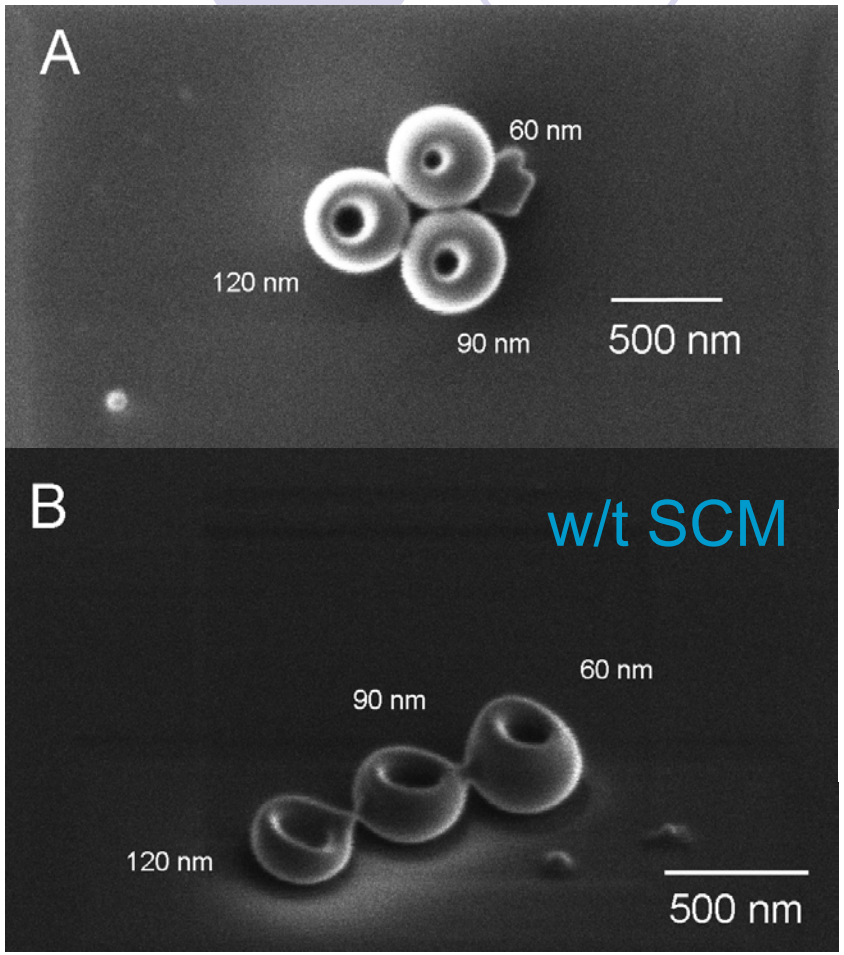
http://www.srim.org;
J.F. Ziegler et al. “SRIM: the stopping …”, Lulu publisher (2008)

Sputtering rate vs. Energy (0°)

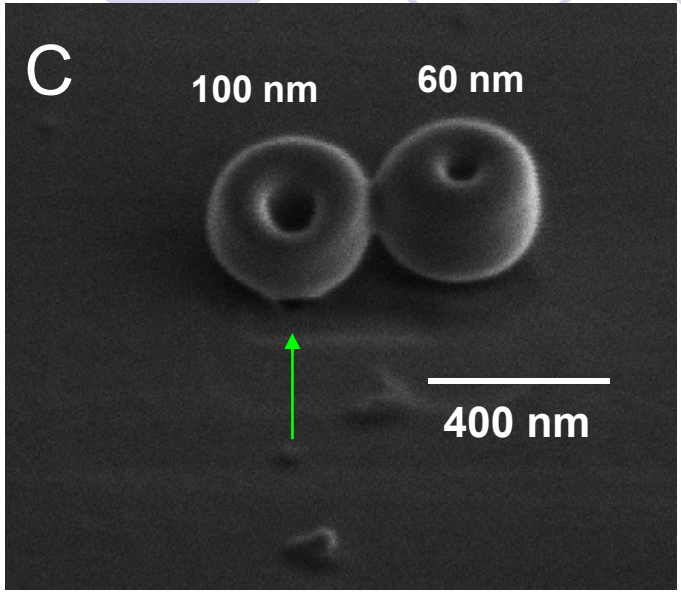


- SRIM simulation matches the experimental sputtering yield within a factor of two (underestimating contribution of low energy atoms) **Datta et al. Phys. Rev. B 63, 25407 (2001)**
- Whatever the experimental value is, it is constant for E > 20 keV

FIB Drilling of Nanocavities in PS spheres



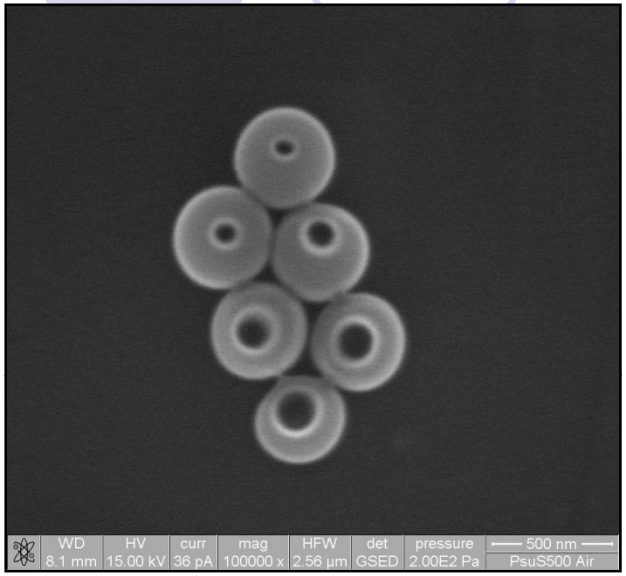
Ion images (30kV, 1pA, ETD)



Drilling of PS microspheres (∅ 460nm) with a circular pattern at 1pA:
A) and C) without SCM
B) With SCM
C) The 100-nm hole is passing-through but what about the 60-nm hole?

S. Magni et al. EMAG proc. in Journal of Physics: Conference Series (2008)

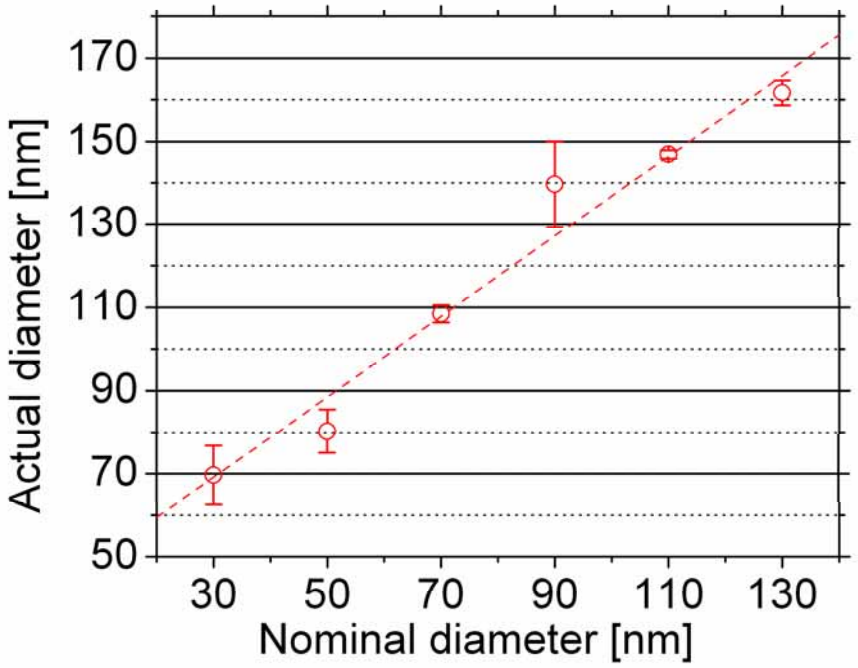
Effect of the Water assisted Milling



∅	Time
30 nm	3”
50 nm	5”
70 nm	9”
90 nm	15”
110 nm	17”
130 nm	25”

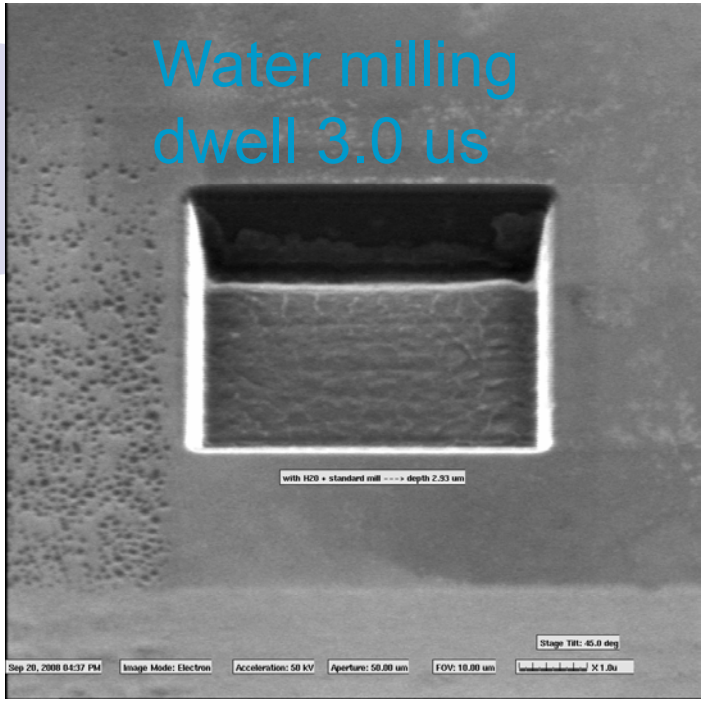
Holes FIB drilled with SCM with increasing diameters of the circular pattern (cf. table). Same dose (nominal depth).

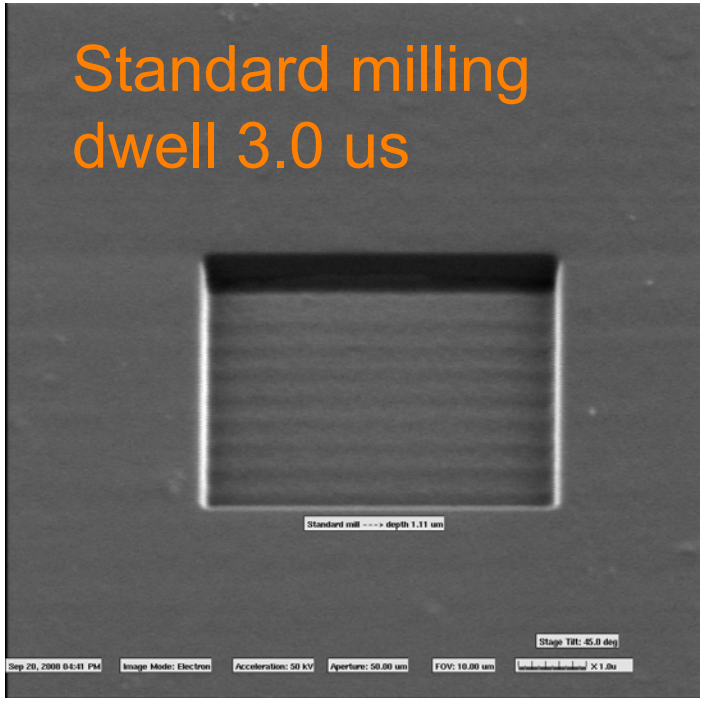
Actual diameter vs. nominal diameter is reported in the plot.

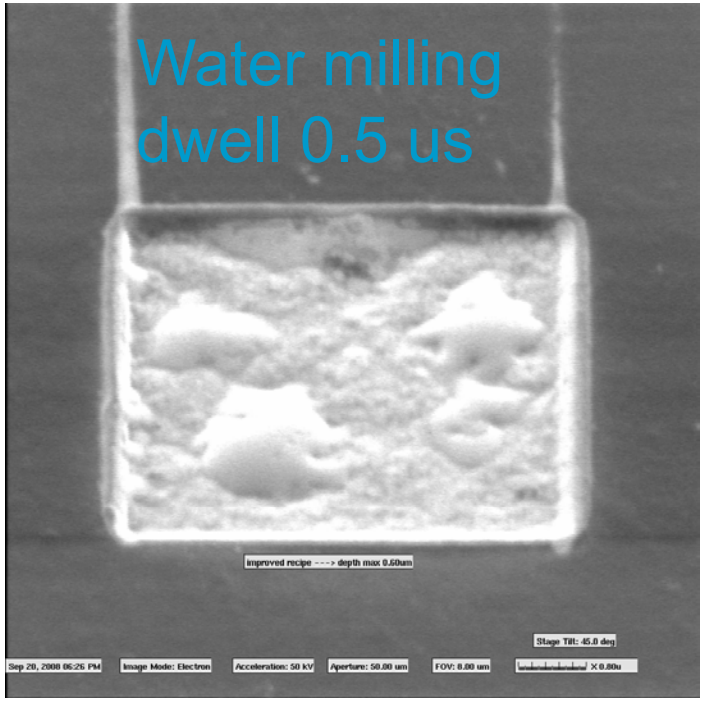


Effects on Silicon (??)

By changing the dwell time we switch from a >2x enhancement to a 5x retarding etching of Si!







Further developments

- Determination of the experimental sputtering rate for diamond (with different parameters / techniques)
- Study damages on the diamond surface (Sputter damage, partial graphitization, ion implantation…)
- Try lower acceleration voltages to minimize the damage
- feedback from high pressure experiments…

Acknowledgements

- People at CSEC for stimulating discussions
- Nhan Phan (School of Physics, University of Edinburgh) for taking AFM measurements
- C.R. Thomson (Building Superintendent School of Physics, University of Edinburgh) assistance in setting up the laboratory
- Technical Staff of the School of Physics (UoE) for helping during FIB installation

Appendix: Sputtering Yield/Rate

Sputtering Yield:

$$Y_t \equiv \frac{\text{number of sputtered particles}}{\text{number of entering ions}} \left[\text{atoms}/\text{ions}\right]$$

Sputtering Rate (Volume Yield):

$$Y_r = \frac{dV}{dQ} \quad [\mu\text{m}^3/\text{nC}]$$

Conversion factors:

$$Y_t = 96.4 \frac{\rho Y_r}{m}$$

for Ga, m in amu and ρ is in [g/cm^3]