Small, low-cost SAXS sample environments to fit your hand luggage

…and your wallet

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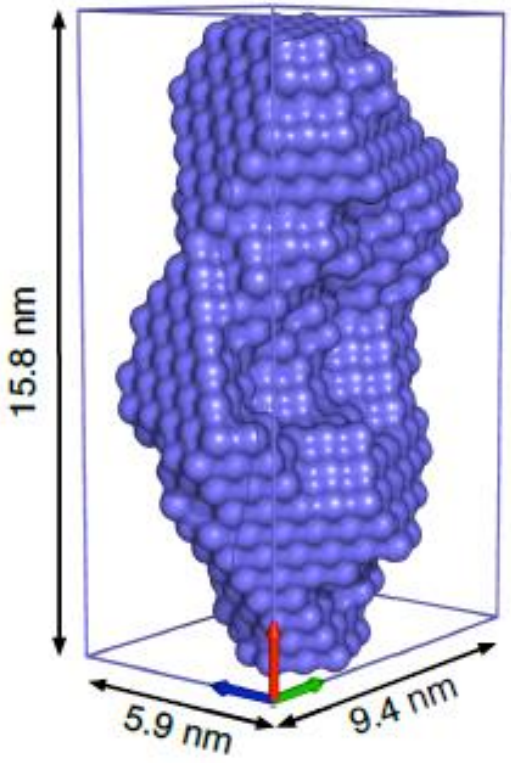
Science Symposium on Advances on Sample Environment and Experimental Control, Lund 2015

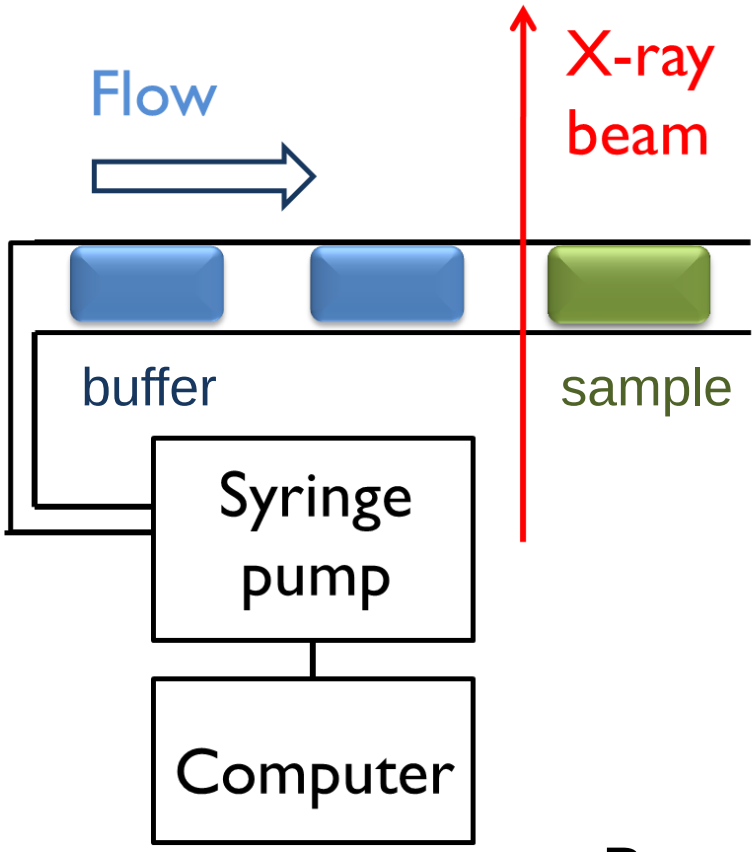
1. Automated protein solution scattering

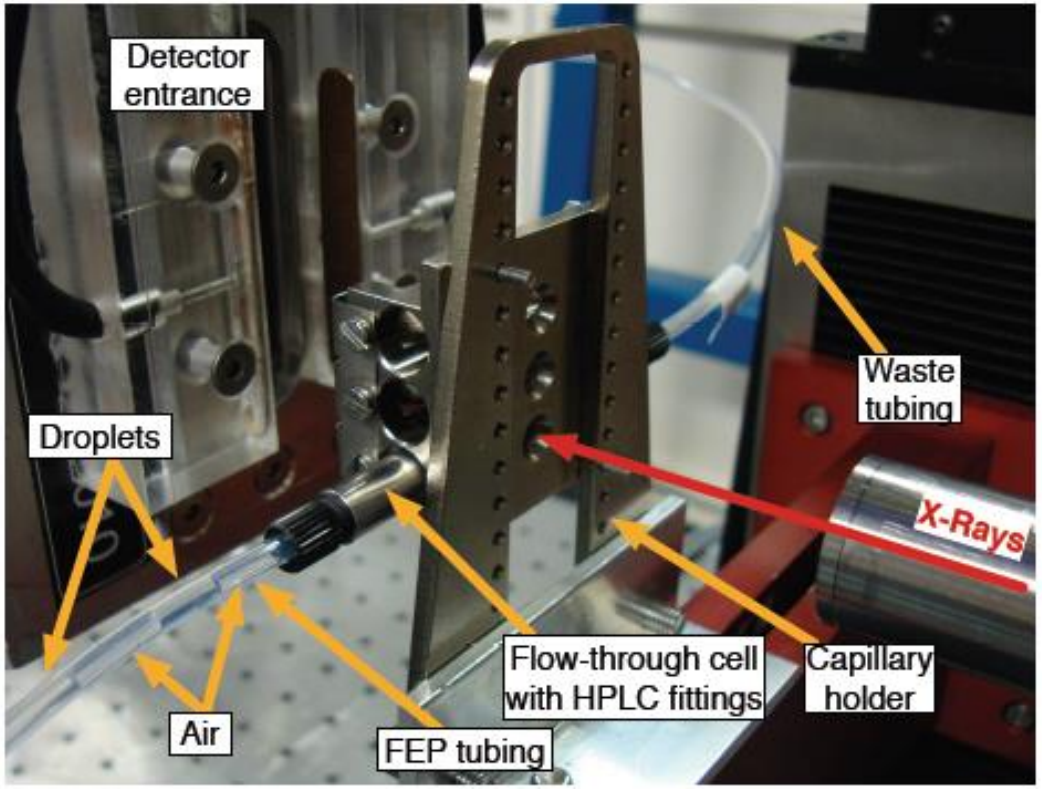
Problem	Solution
Weakly scattering	Bright light
Sensitive to beam damage	Flow sample
Small sample volume	
Low budget	



Gudrun Lotze (PhD student), Prof Nick Terrill (Diamond), Dr Katsuaki Inouye (Diamond), [Ian Hamley (Reading)]

Low-cost flow cell design

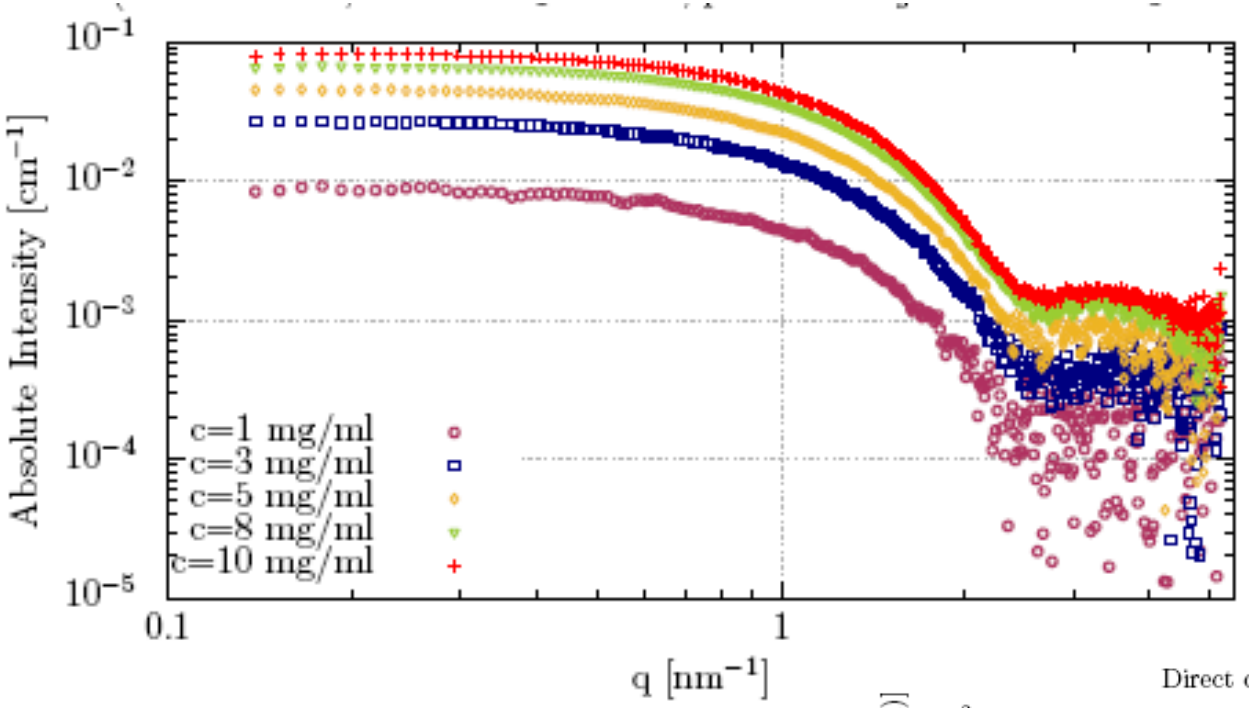




Beamline I22, Diamond

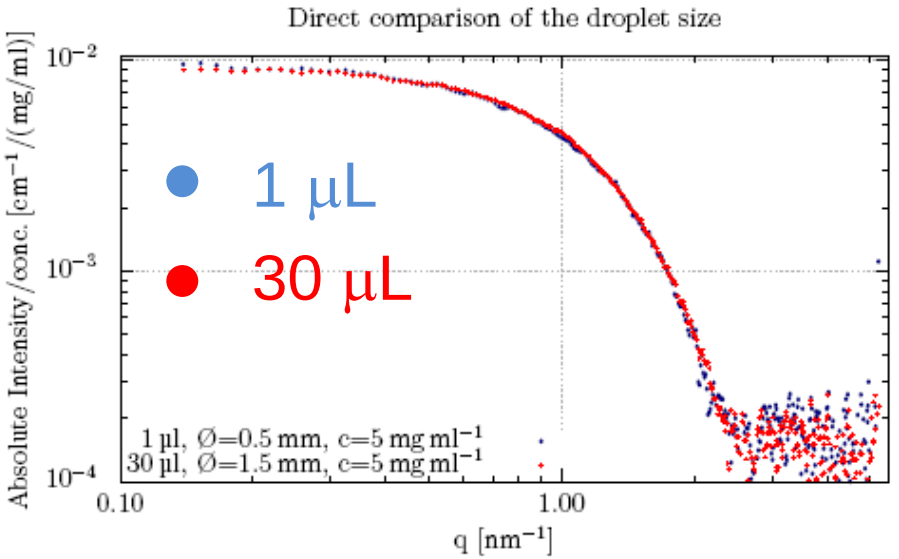
Also: MAXlab I911-4; Petra III; ESRF ID02

Proof of concept: lysozyme



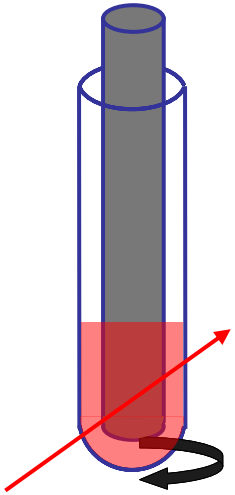
Left: automated concentration series
1-10 mg/ml
1.5mm diameter
30 µL

30x smaller?
0.5mm diameter
1 µL droplets



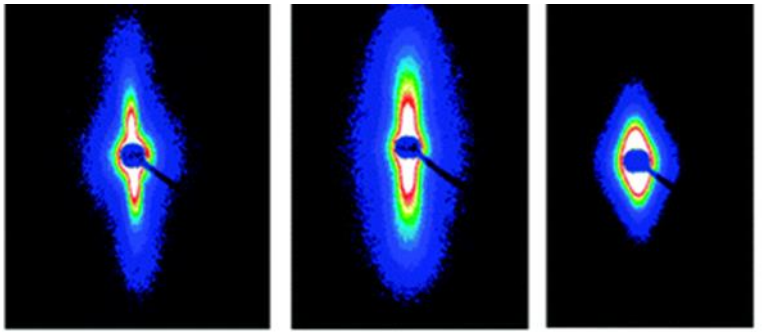
R_G = 1.55 ± 0.01nm
Literature range: 1.43-1.60 nm

2. Couette cell



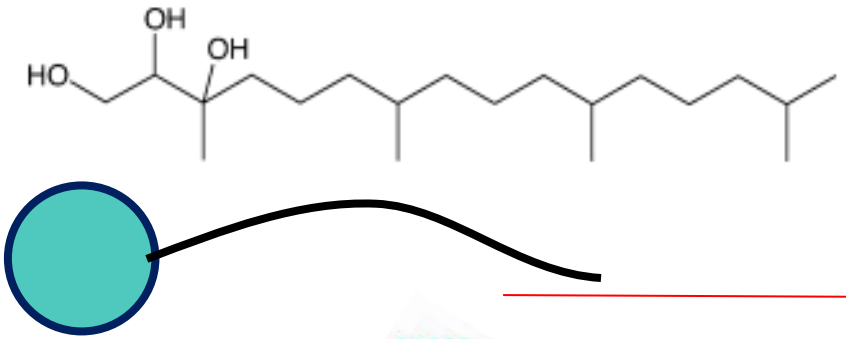
Inter-changeable cylinders eg
Polycarbonate (SAXS)
Quartz NMR tubes
(Neutrons; Polarised Raman)
Sample volume 70 µL

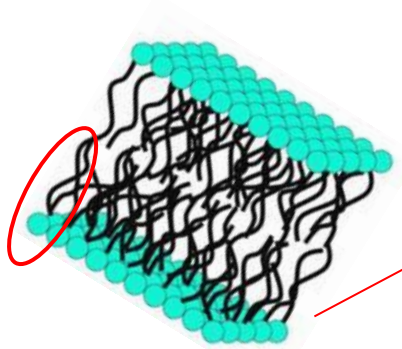
Small-angle x-ray scattering of fibrils from naphthalene-diphenylalanines

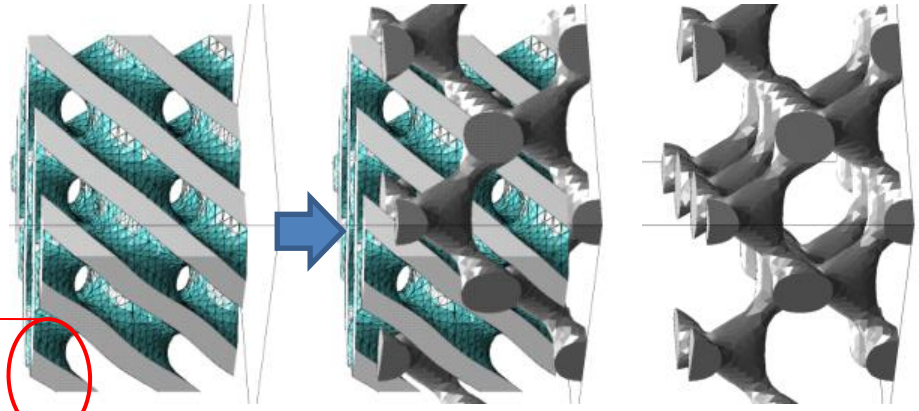


L. Chen, G. Pont, K. Morris, G. Lotze, A. Squires, L. Serpell and D. J. Adams, Chem. Commun., 2011, 47, 12071-12073
Data from MAX-lab I7-11

Periodic nanomaterials







Electrodeposition

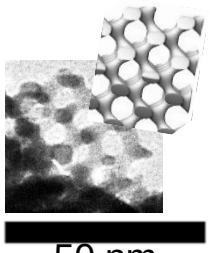
Lipid cubic phase
"Double diamond" Pn3m
a=6.5nm

Mesoporous metal (platinum)

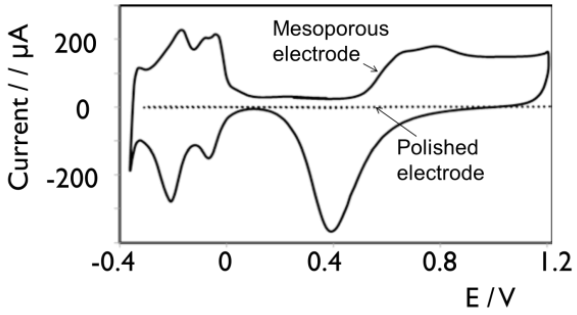
"Single diamond" Fd3m
a=13nm

Metal and lipid template have different symmetries

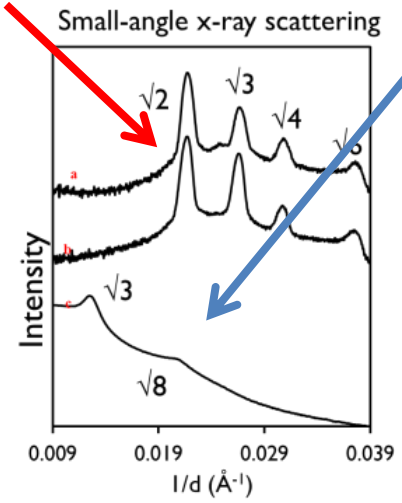
TEM:



Electrochemistry:

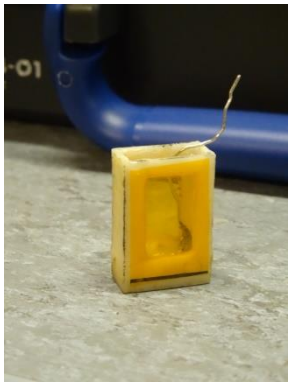


1000x surface area

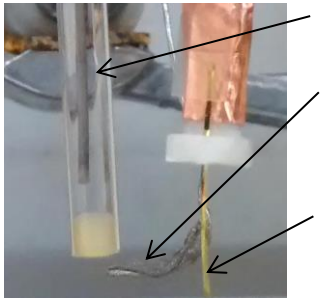


S. Akbar, J. Elliott*, M. Rittman & A. Squires*, *Advanced Materials 2013, 25, 1160–1164*

3. In-situ electrodeposition



3-D printed
ABS (“Lego”)
Kapton windows
Path length 7mm

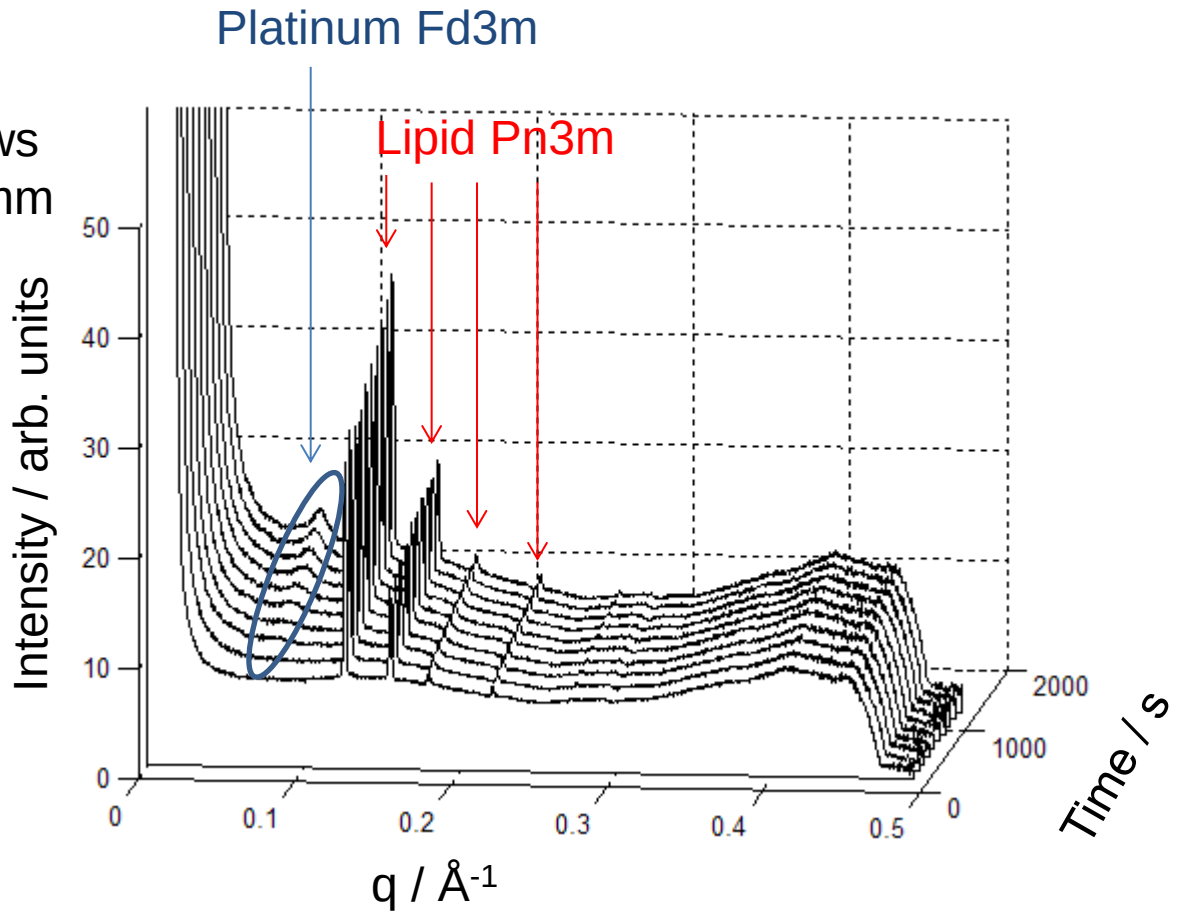


Reference

Counter

Gold foil working electrode

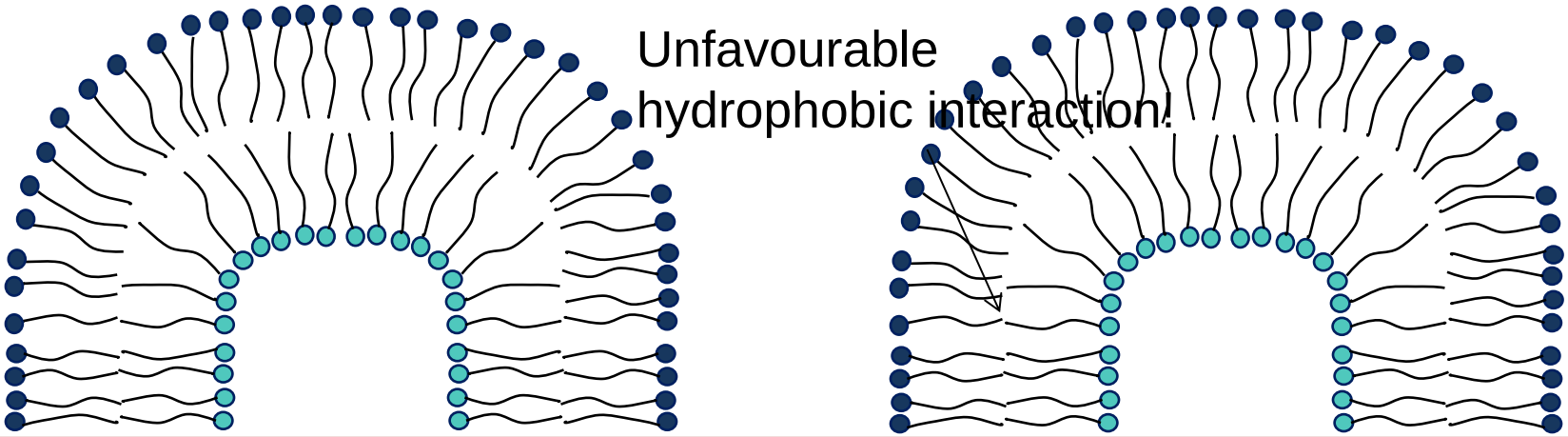
Diamond I07



Metal is *deposited* asymmetrically

Why asymmetric deposition? Consider the interface…

External Water



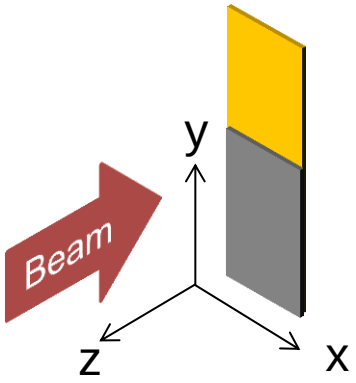
Bicontinuous cubic phase

Larsson,
Curr. Op. Coll. Int. Sci
2000

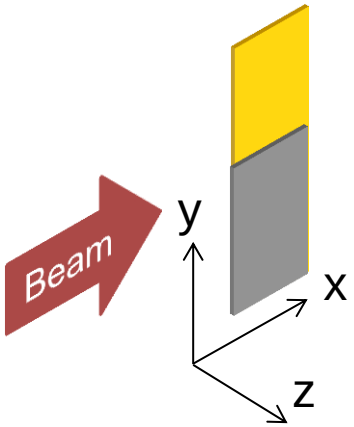
is shown in Fig. 3. In these calculations of the bilayer mid-surface, the bilayer always becomes closed. This feature is consistent with the particle surfaces observed in cryo-TEM images. This means that the particle contains two kinds of water spaces; one is open towards the outside water medium and the other is a closed water compartment.

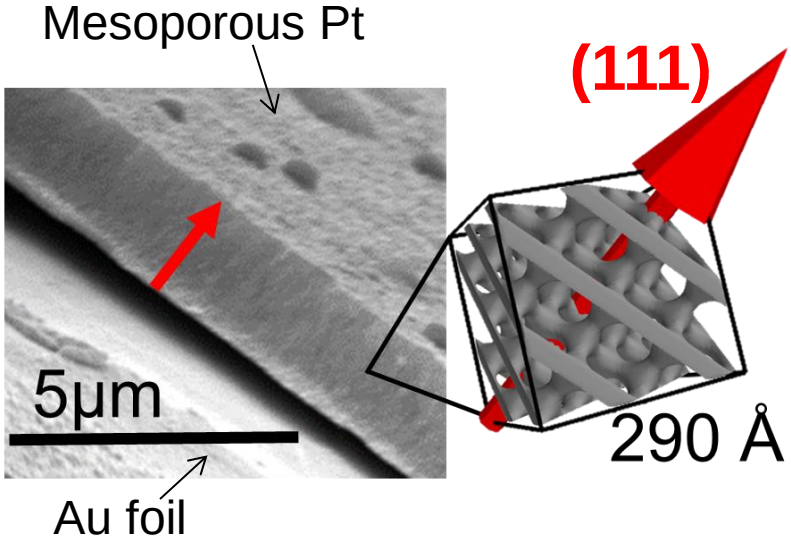
Orientation in mesoporous platinum

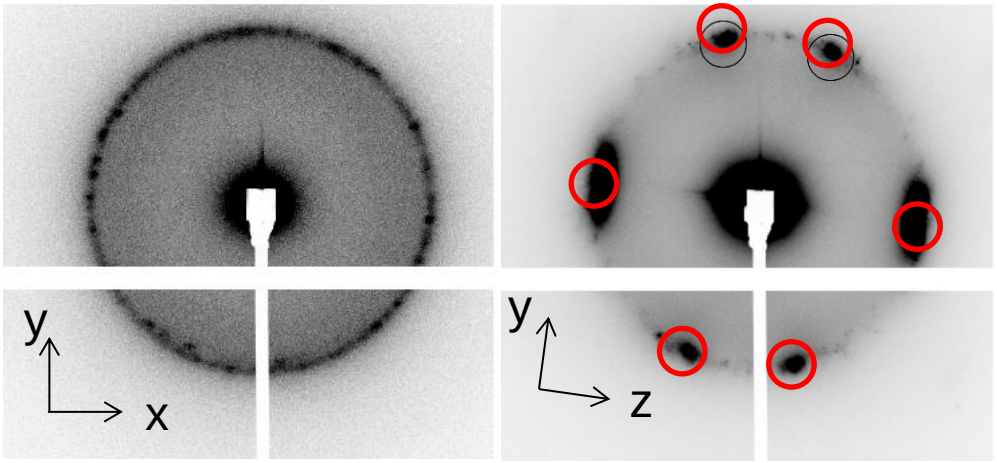
Transmission



Quasi-
grazing incidence

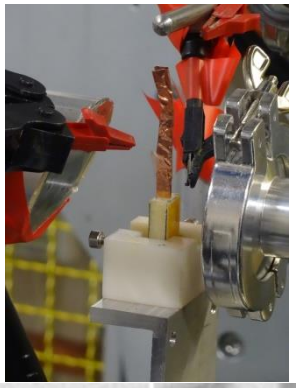




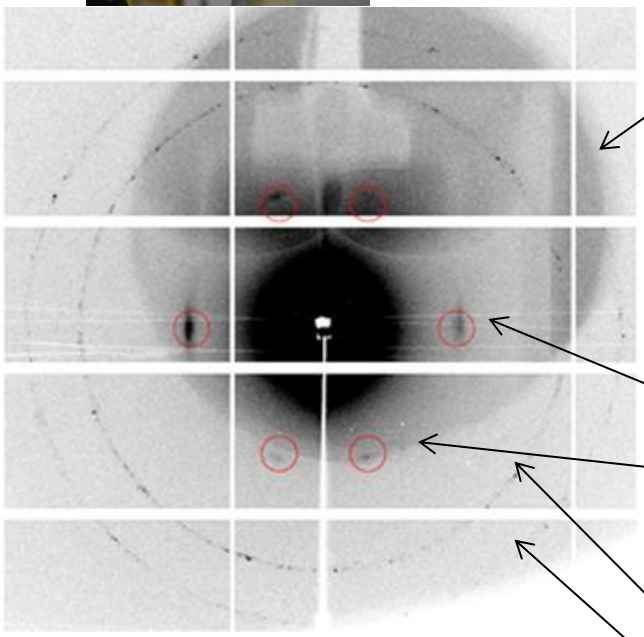


I911-4 MAX-lab

Is the lipid template oriented?



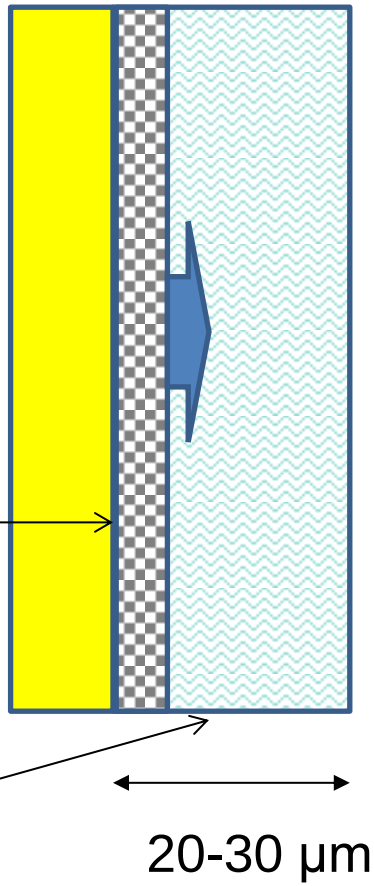
Quasi-
grazing incidence



Ignore this

Oriented
mesoporous
Pt

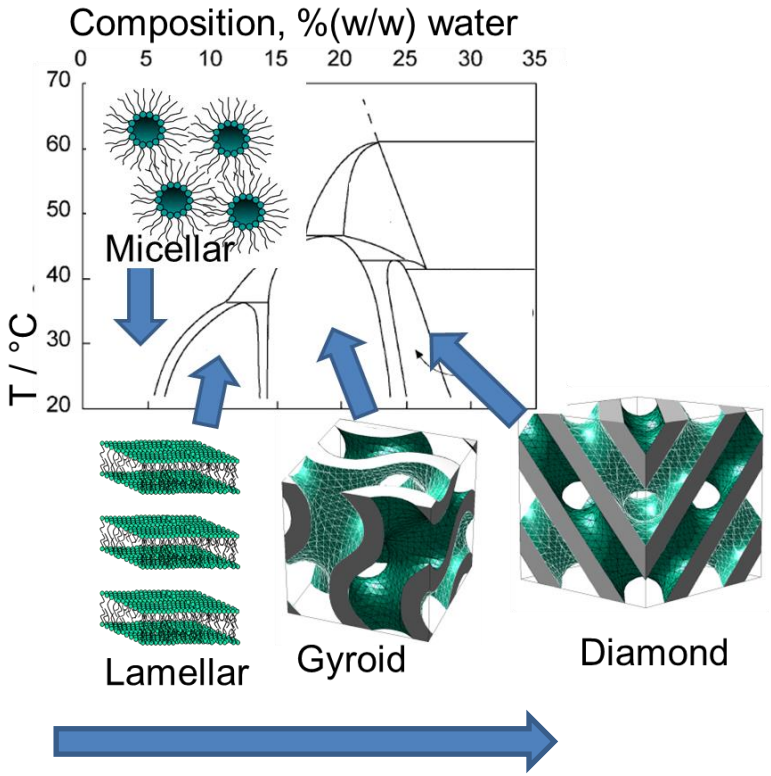
Un-oriented
lipid template

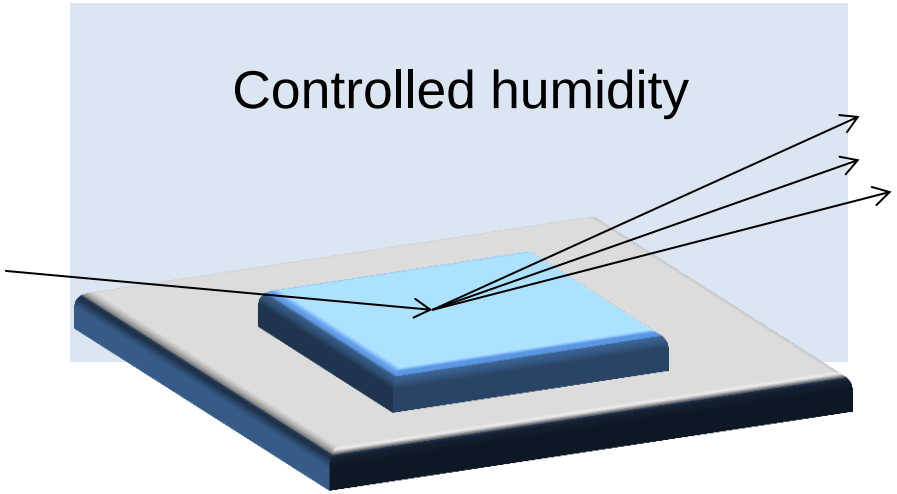


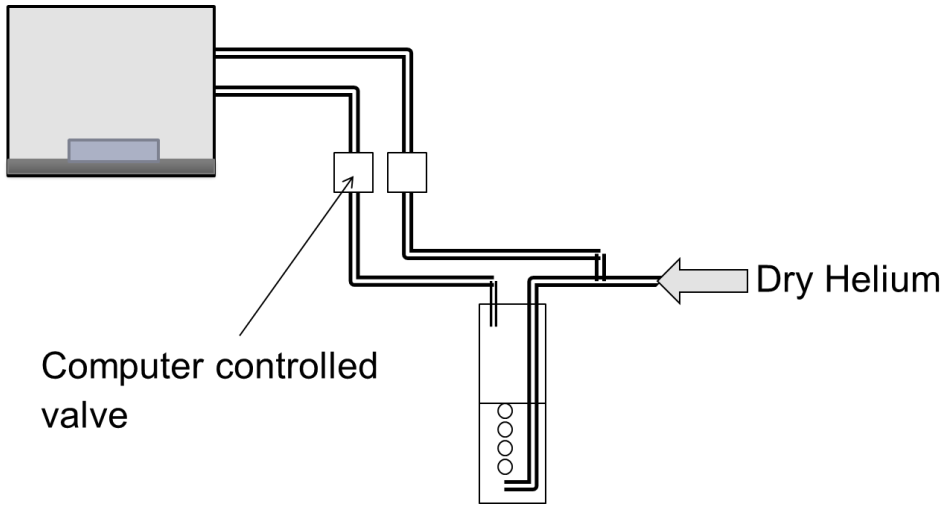
Diamond I07

4. Thin lipid films: GI-SAXS

The idea:

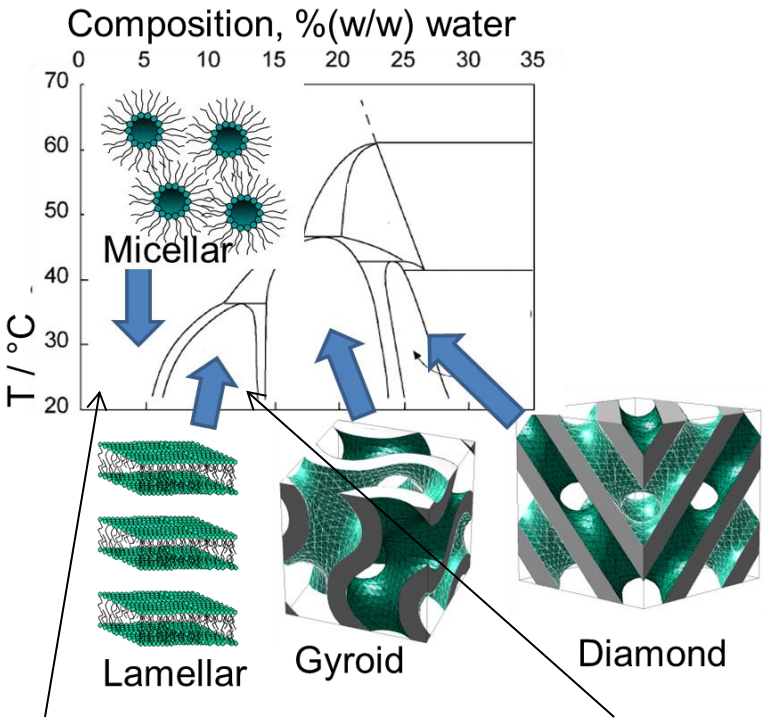






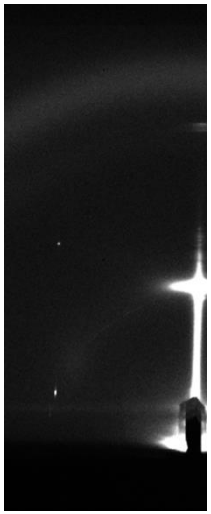
4. Thin lipid films: GI-SAXS

The reality:



90% relative humidity: micellar

95% relative humidity: lamellar



Damp pipe cleaners?

Mist maker?

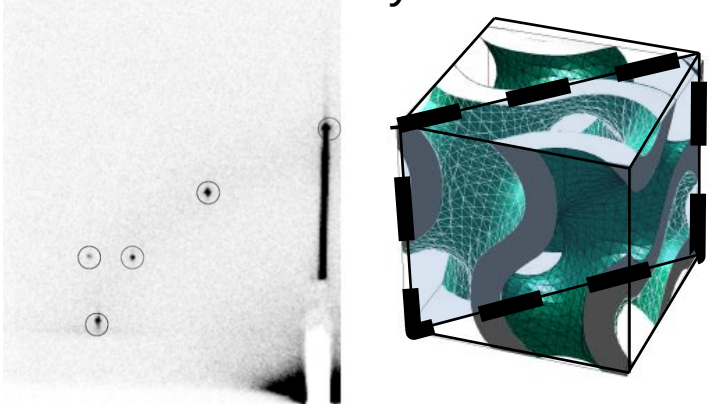
Elettra Austrian SAXS; Diamond I07; ESRF BM28 (XMaS)

4. Thin lipid films: GI-SAXS

The solution: add glycerol.

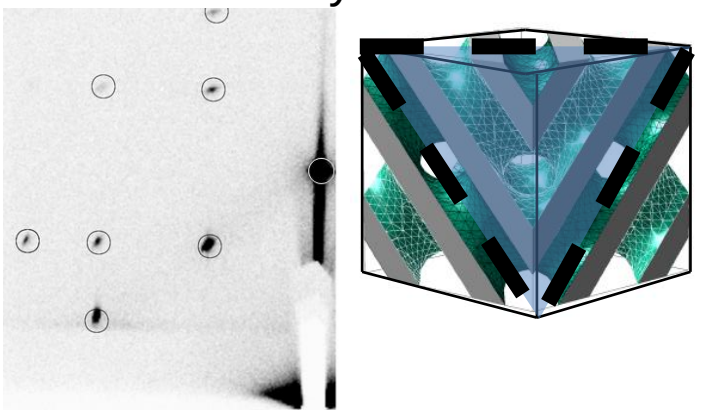
With 20% glycerol, film thickness <1.5 μm :

30% Relative Humidity



Gyroid
[110] parallel to substrate

90% Relative Humidity



Diamond
[111] parallel to substrate

S. Richardson…. A. Squires* & W. Góźdź*, *Langmuir* **2014**, 30, 13510−13515
S. Richardson… & A Squires*, *Chem Commun.* **2015**, 51, 11386

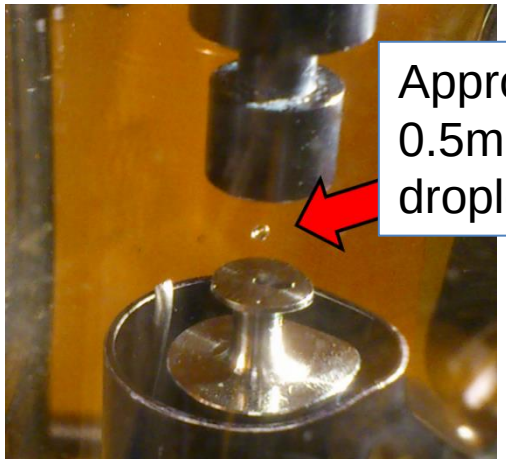
Summary (nanomaterials)

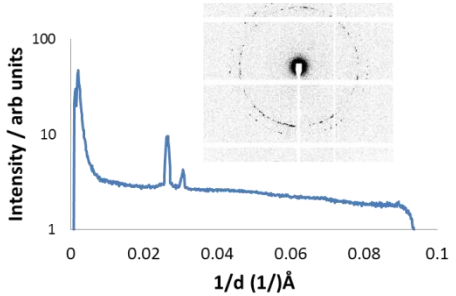
- Templated electrodeposition gives metal with new nanostructure through asymmetric deposition
- Surface-induced orientation of lipid template produces oriented metal nanostructure

SAXS on levitated droplets

(1) Droplets trapped using ultrasound

Beamline: MAXlab I911-4 (Sweden)



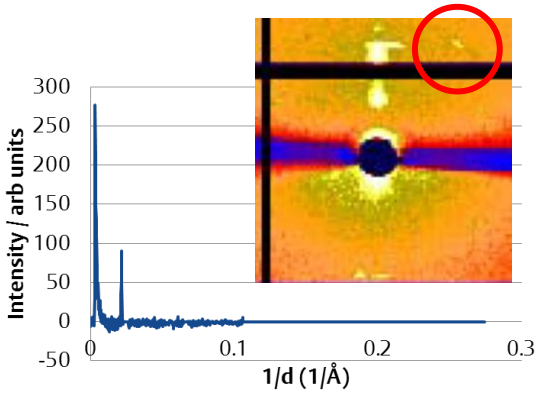


Lipid / glycerol: Cubic phase

(2) Droplets trapped using light

Diamond I22 / Andy Ward (Research Complex at Harwell)

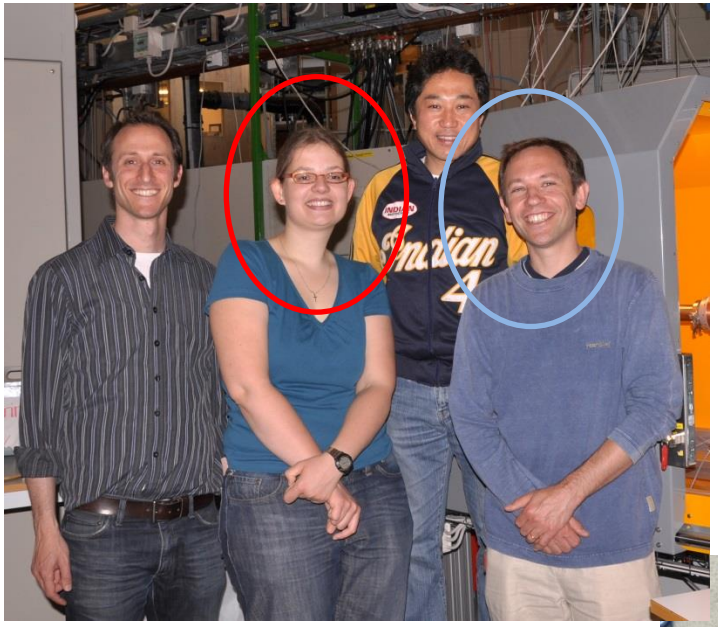
Approx 8 micron droplet



Lipid / glycerol: cubic phase?

Thanks!

Gudrun Lotze



Martyn Rittman

Samina Akbar

Sam Richardson

EPSRC
Pioneering research and skills

diamond