Small Angle X-Ray Scattering at NCD beamline, in ALBA synchrotron light source

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Small Angle X-Ray Scattering

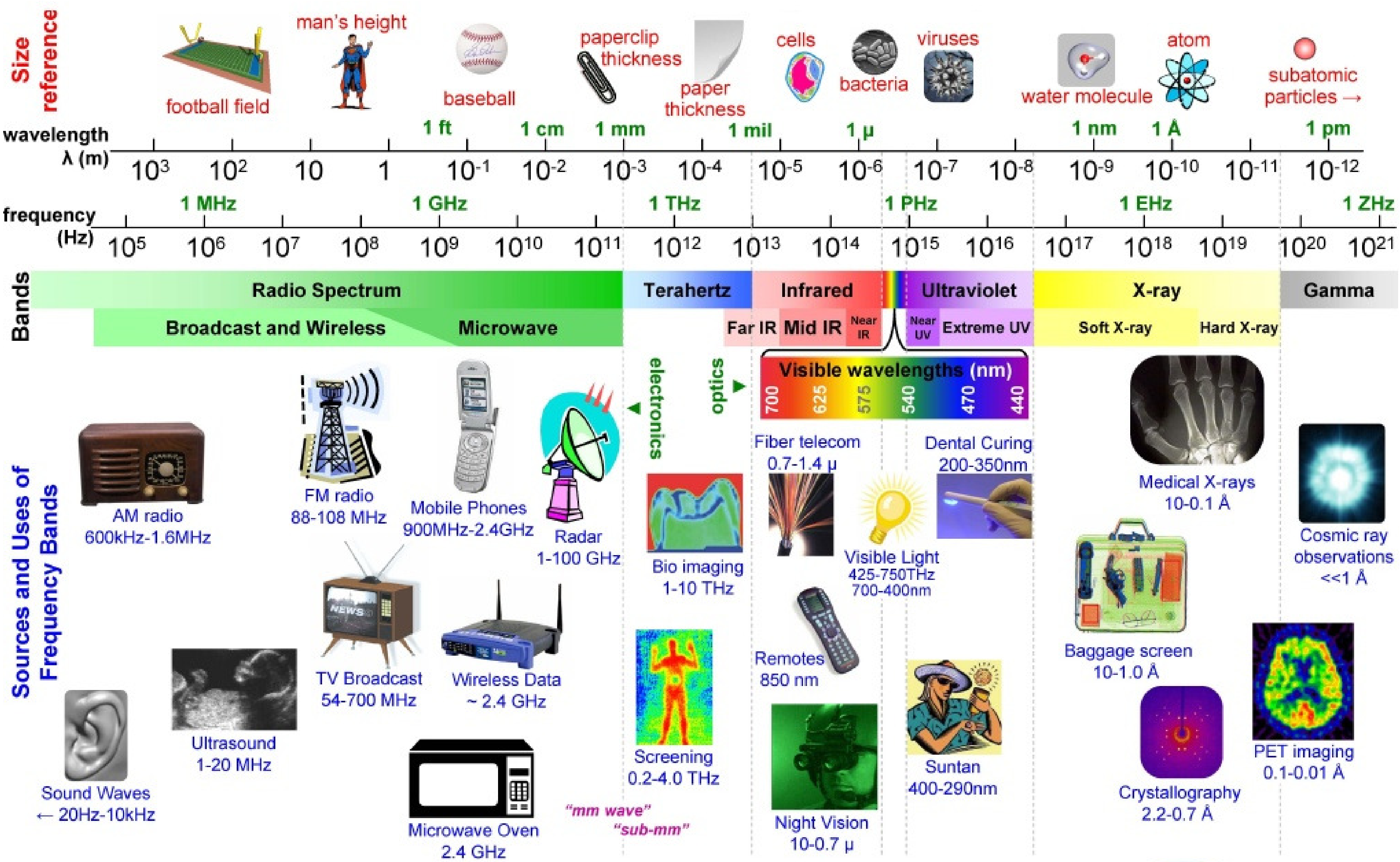
<u>A wide range of fields:</u>

Medicine
Biology
Chemistry
Physics
Archaeology
Environmental and conservation sciences
Materials

<u>A wide range of systems:</u>

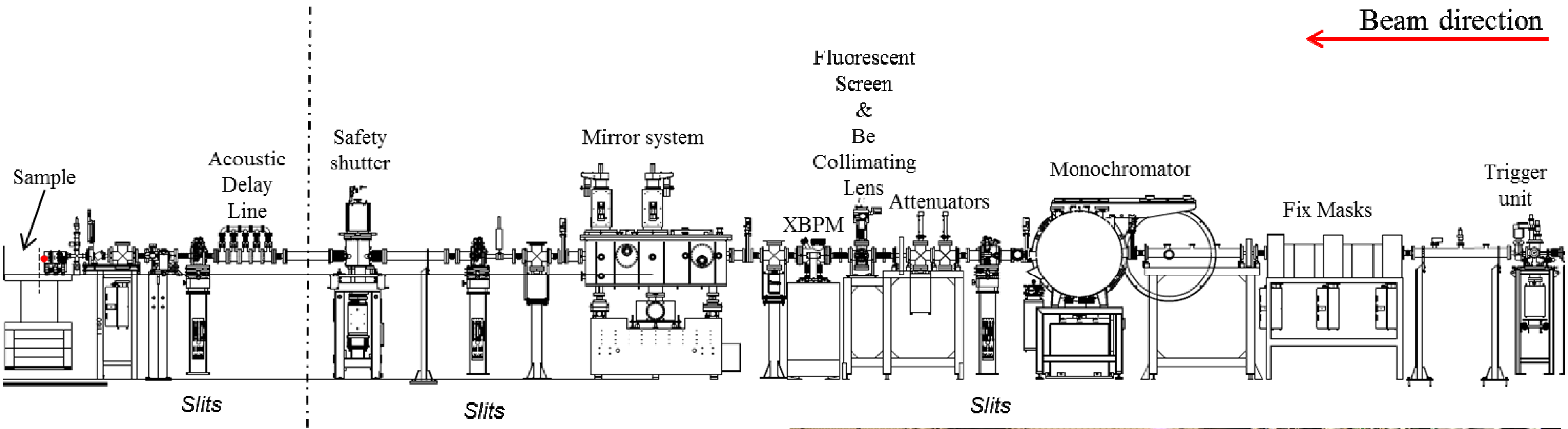
Polymer processing
Self assembly of mesoscopic metal particles
Colloids
Inorganic aggregates
Liquid crystals
The supramolecular organisation in biological systems
The structure and function of muscle filaments
Corneal transparency

The electromagnetic spectrum



http://ebookbrowse.com/sura-electromagnetic-spectrum-full-chart-pdf-d14713735

BL11-NCD layout



Source: in-vacuum undulator

Monochromator: Double Si(111) crystals
fixed exit, 5-15keV

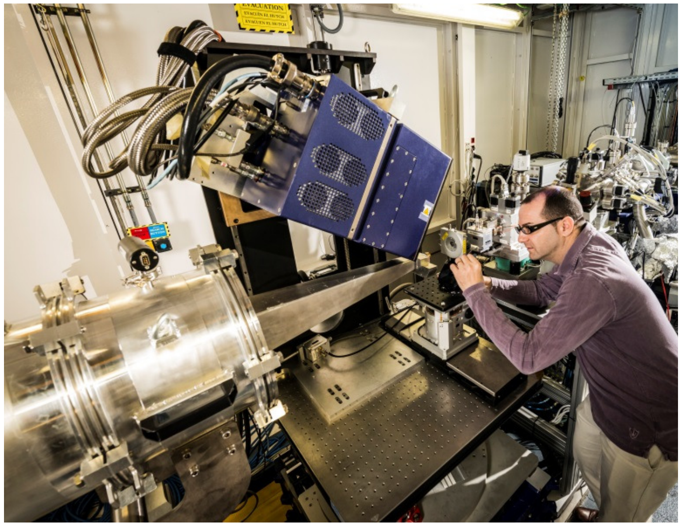
Optics: Plane, Rh coated Si substrate
Sagital radius: flat (>50 km)
Toroidal focusing mirror,
Rh coated Si substrate

Beamline specifications

Wavelength (Energy) range	0.9 - 1.9 Å (6.5 -13 keV)
Flux at sample	>1.25 10^{11} ph/s 1.24 Å for a beam current of 250 mA
Bandpass (ΔE/E)	< 10^{-4}
Beam size at sample	Variable between ~65 - 1200 μm horizontally ~30 - 265 μm vertically
Beam divergence at sample	<0.5x0.1 $mrad^2$
Q range SAXS	0.0066-0.7 $Å^{-1}$
2Θ range WAXS	3.0° - 62°

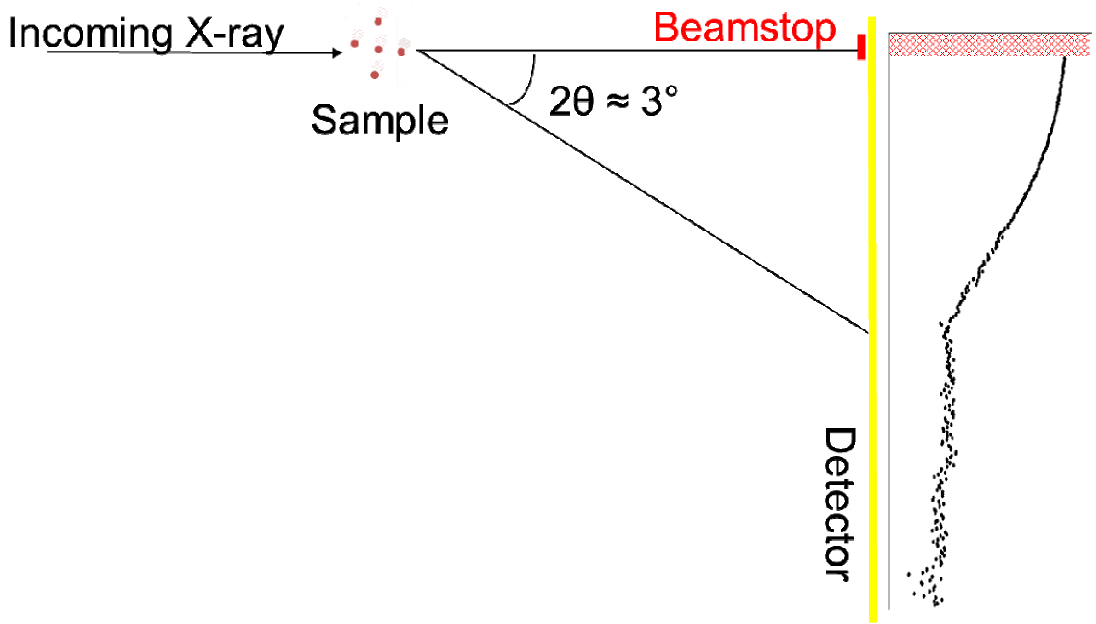
Sample environments

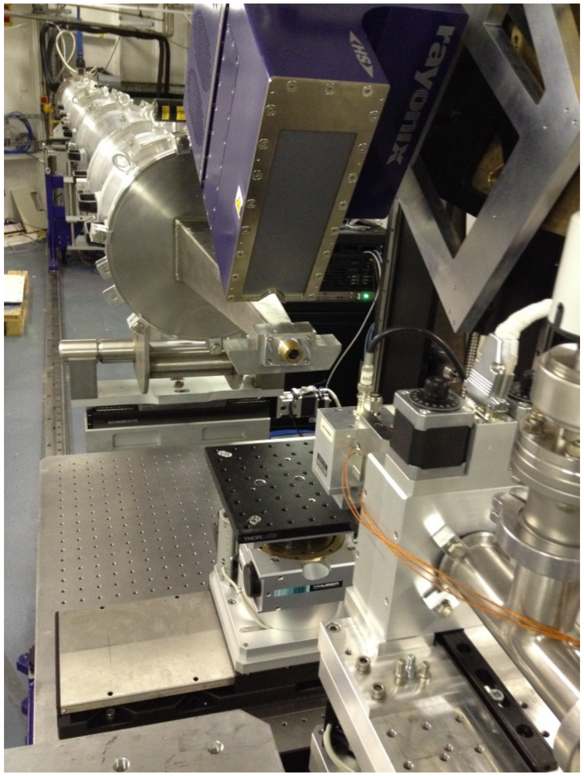
- Thermo-stated liquid cell (Required volume on demand)
- Non thermo-stated liquid cell rack for 24 samples (Volume 30ml)
- Thermo-stated ladder for 6 capillaries (1mm or 2mm diameter)
- Non thermo-stated ladder for 20 capillaries (1mm diameter)
- One film holder for 45 samples
- 1 Linkam stage for 1mm capillaries
- 1 Linkam stage for 22mm diameter films

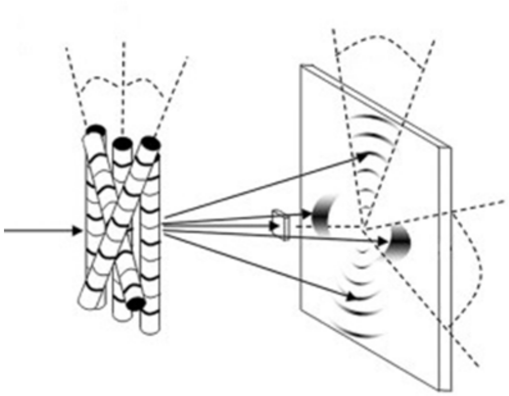


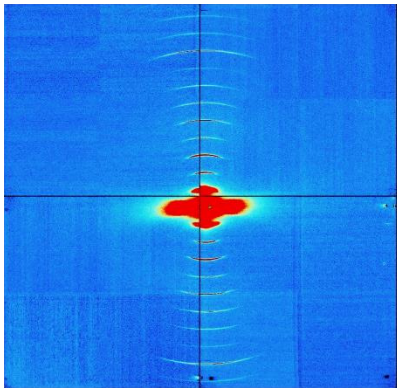
	Controller	Pumping system	Linkam Stage	
Sample type			Capillary	Film
Model	T95	LNP954	HFSX350-CAP	THMS600
Max Temp (ºC)	1500	-196	350	600
Max rate (ºC/min)	200	100	30	150

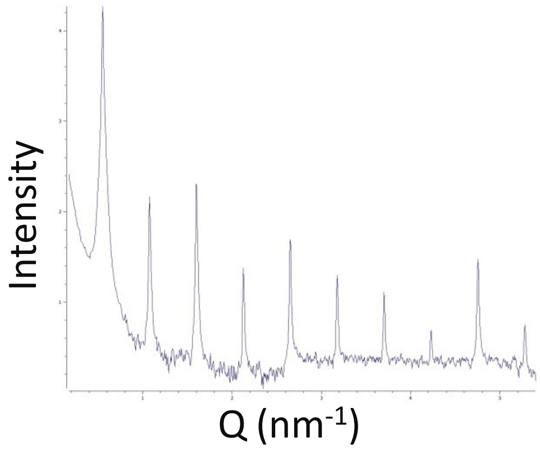
Experimental setup



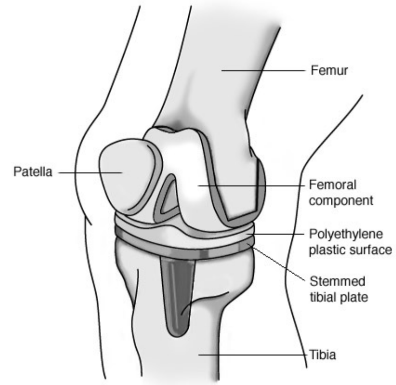


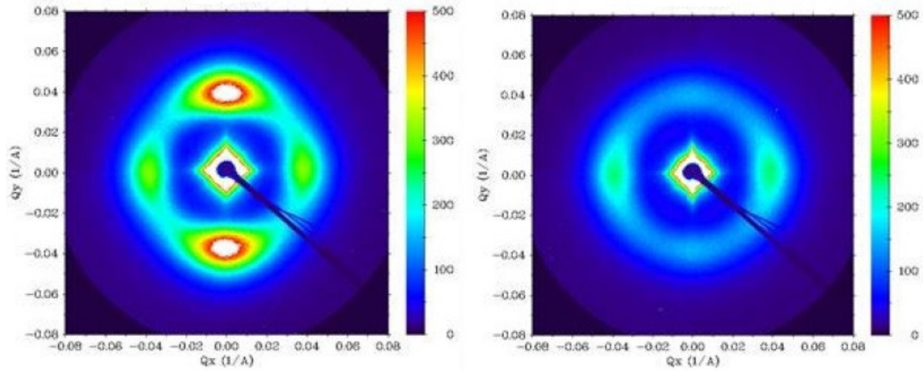


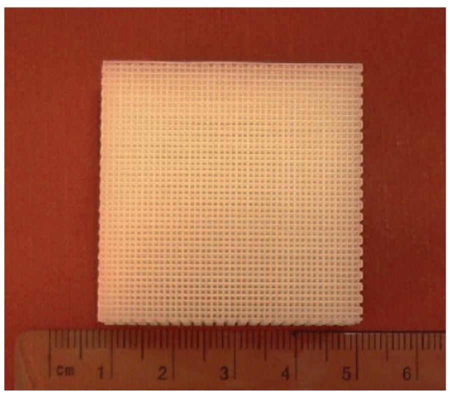


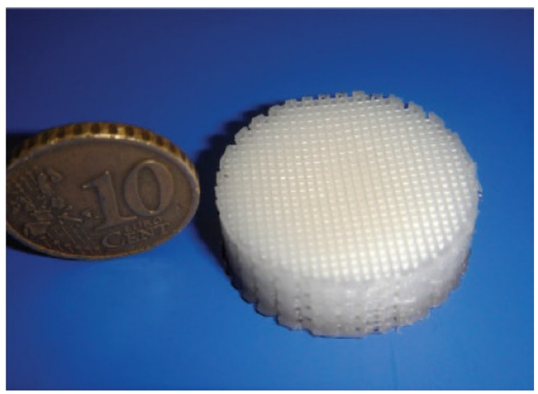


SAXS in regenerative medicine



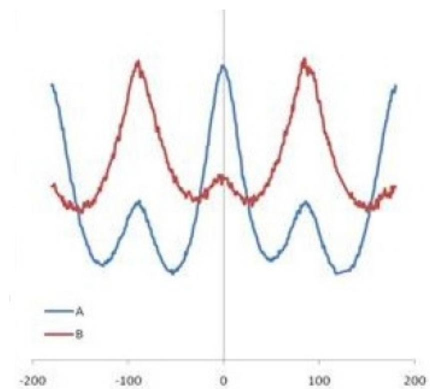






Domingos M., Dinucci D., Cometa S., Alderighi S., Bártolo P., Chiellini F. International Journal of Biomaterials. 2009. 239643

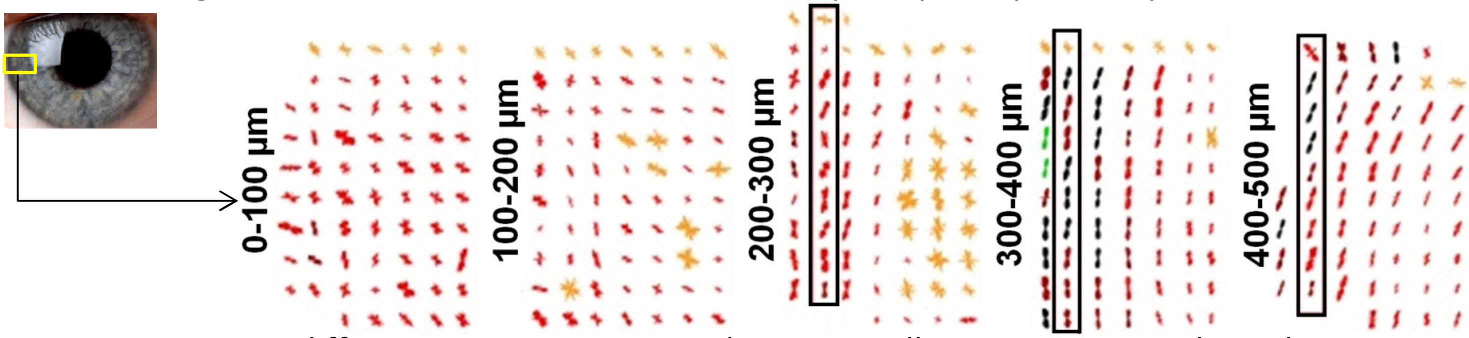
Square and disc shape scaffolds



Mitchell G.R., Domingos M., Bartolo P., Advanced Materials Research. 2012. 506: 11-14

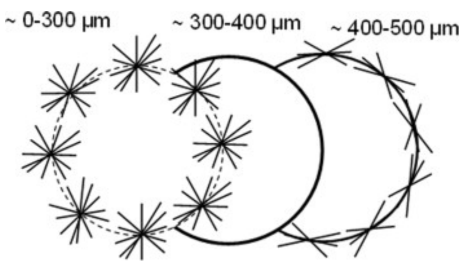
Improving the success of corneal transplantation procedures

- Corneal blindness or visual impairment is the fourth cause of sight loss globally ~5.1% (WHO, 2014)
- Corneal graft failure rate is ~35% within the first 5 years post operatively (http://emedicine.medscape.com/)



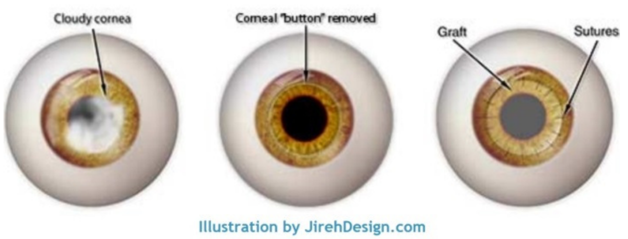
X-ray diffraction representing predominant collagen orientation throughout consecutive tissue sections (~100 μm each) from peripheral cornea

Severe corneal blindness in human
(http://www.cehjournal.org/article/corneal-blindness-prevention-treatment-and-rehabilitation/)

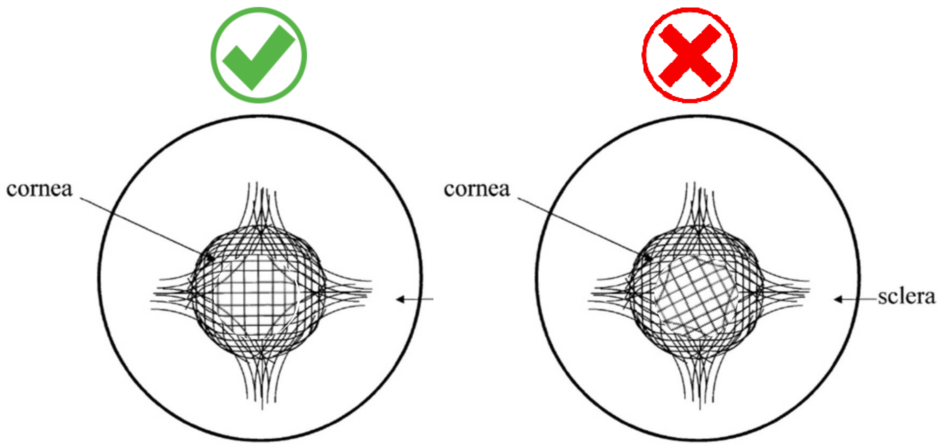


Kamma-Lorger C.S., et al., Collagen and mature elastic fibre organisation as a function of depth in the human cornea and limbus. J Struct Biol.2010; 169:424-30.

Schematic overview of Penetrating Keratoplasty (PKR)

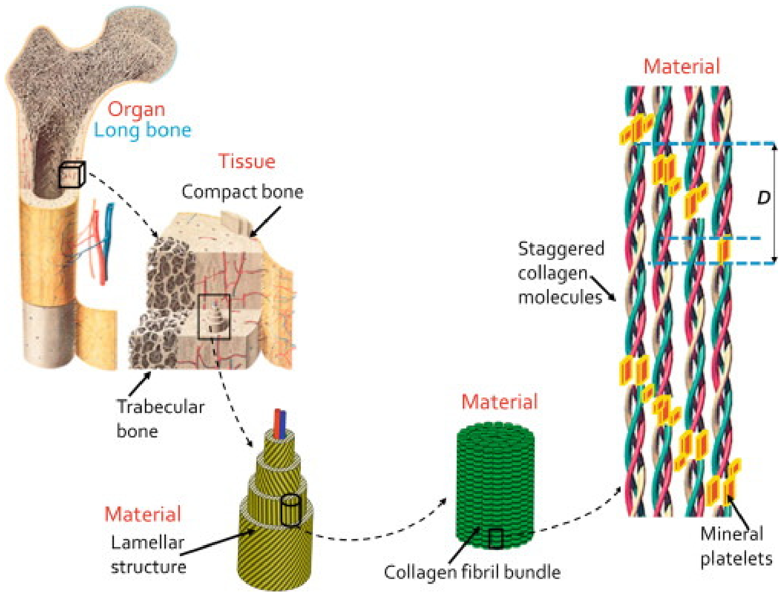


Graft positioning during PKR

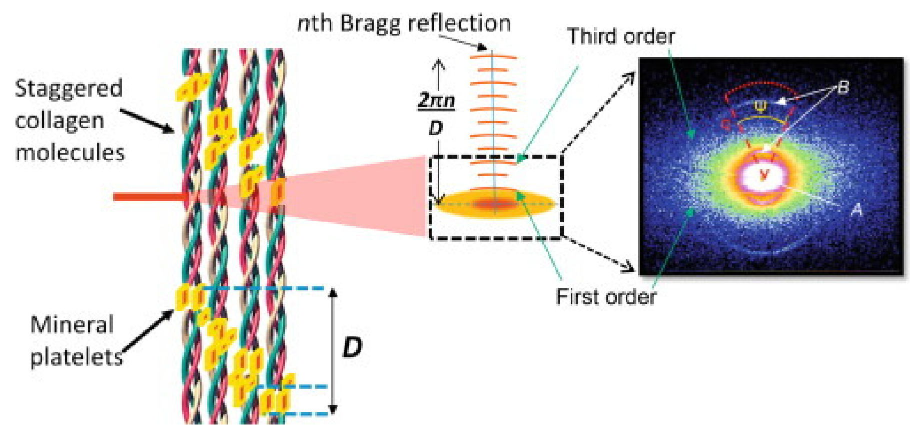


Match collagen orientation between donor and recipient tissue
- ✓ Improved biomechanical stability post-operatively
- ✓ Increased chances of success

SAXS in bone matrix nanomechanics



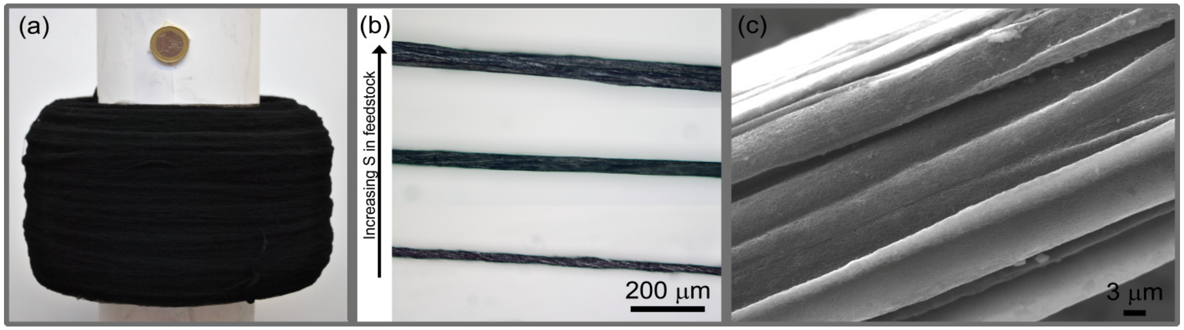
Strain, stress, and other mechanical parameters determination at small scales (< 100 nm) in nanostructured biomineralized composites.

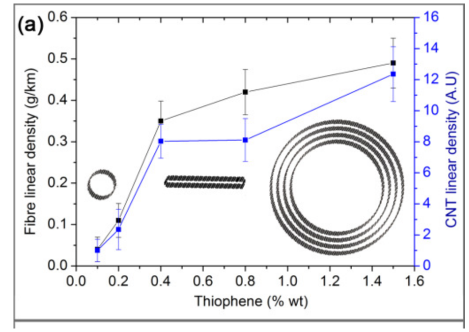


Karunaratne *et al.*, Methods in Enzymology, **532**, 2013, Pages 415–473

Characterisation of yarn-like carbon nanotube (CNT) fibres (1)

The research group has a process to produce kilometres of continous macroscopic fibres of CNTs

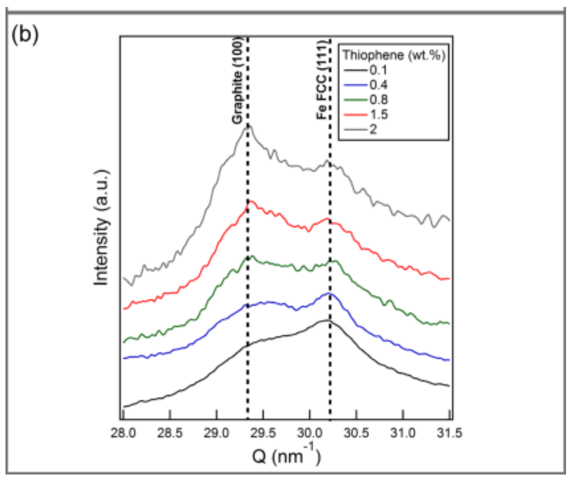




Synchrotron XRD confirms the increase of graphitic layers at turbostratic separation

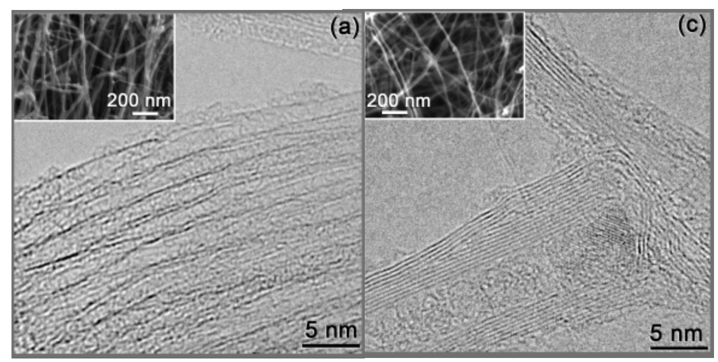
S ↑ in feedstock → Intensity ratio [(100)Graphite / (111)Fe]

They can tailor thee type of nanotubes through the addition of sulphur precursor (thipohene)



Low S → SWCNTs

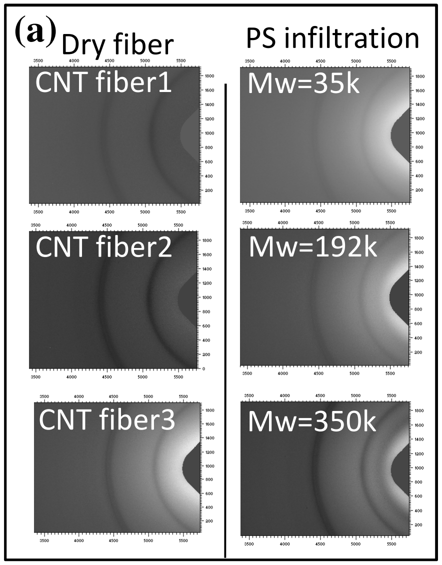
High S → MWCNTs



Reguero *et al.*, Controlling carbon nanotube type in macroscopic fibers synthesized by the direct spinning process , 2014, Chemistry of Materials, **26 (11)**, 3550-3557 DOI: 10.1021/cm501187x

Characterisation of yarn-like carbon nanotube (CNT) fibres by synchrotron X-ray diffraction (2)

High-performance composites through polymer infiltration in mesoporous CNT fibres



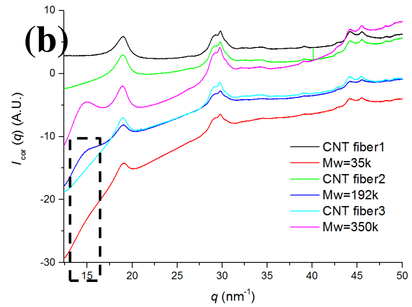
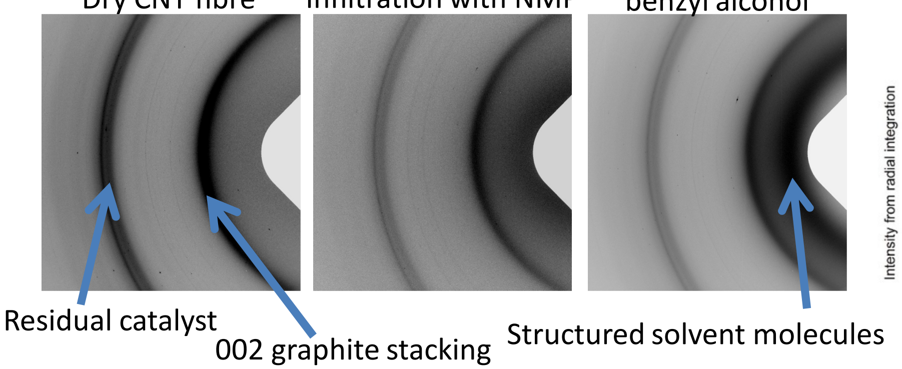
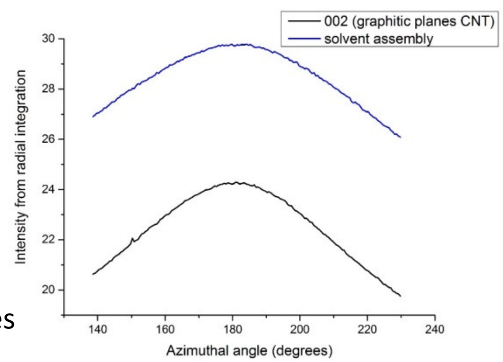


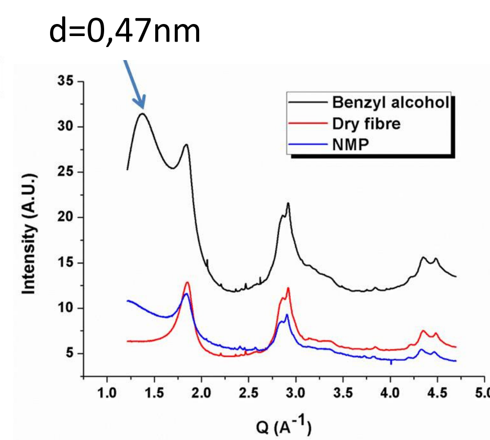
Figure: 1D WAXS patterns (a) of CNT fibres and that infiltrated with polystyrene (PS) of different molecular weight, together with integrated scattering patterns (b) after azimuthal integration.

Further information at NCD science highlights:
http://www.cells.es/en/media/news/multiscale-structural-characterisation-of-yarn-like-cnt-fibres-by-synchrotron-x-ray-diffraction

Liquid interaction with mesoporous CNT fibres and evidence of π–π stacking

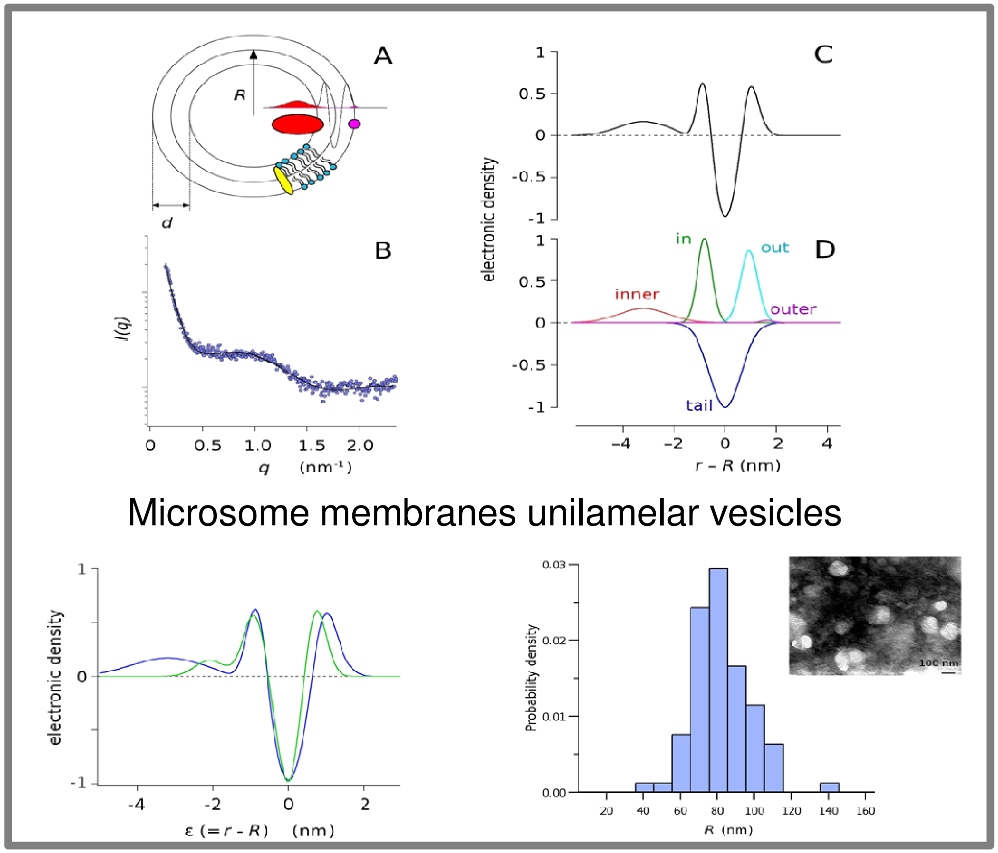






SAXS for the study of the use of membrane vesicles for cystic fibrosis research (1)

From scattering theory, analytic expressions are derived for the bilayer form factor over a spherical geometry, assuming the lipid bilayer electron density to be composed of a series of Gaussian shells.

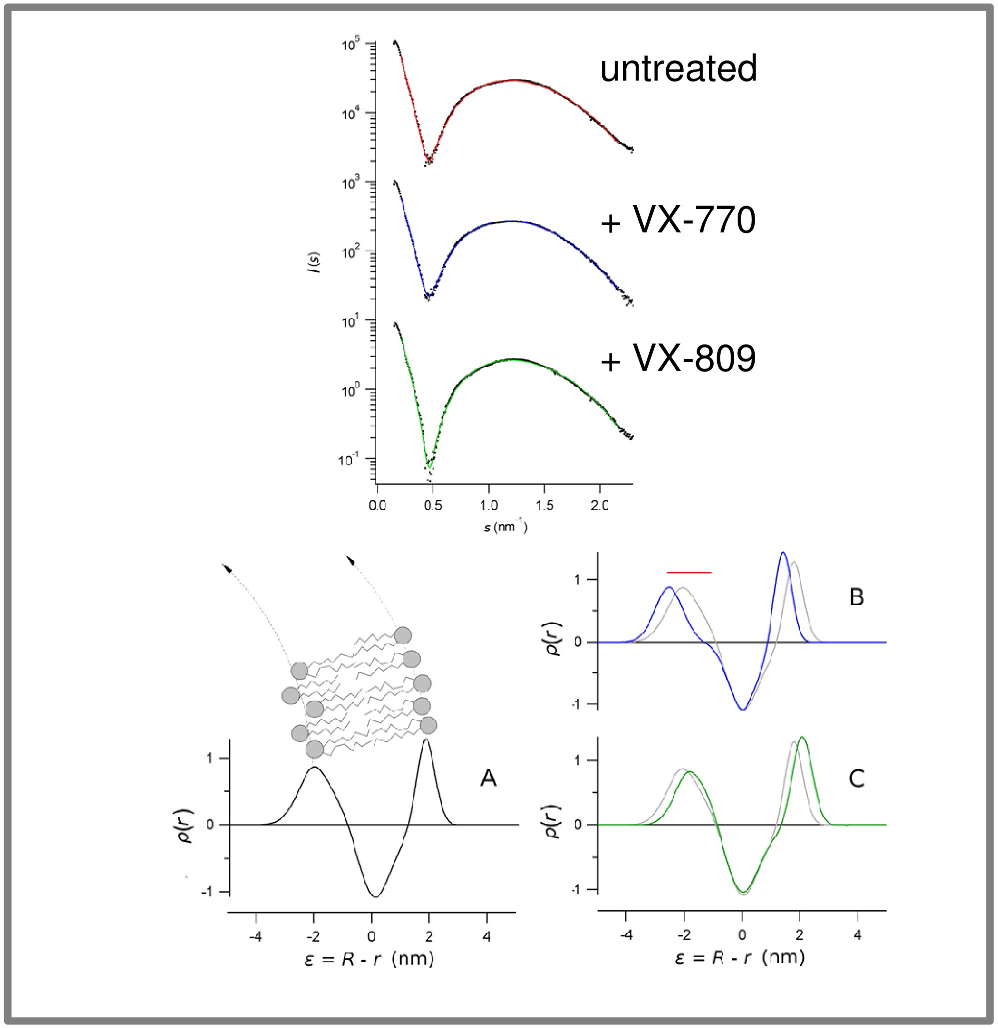


A) Sketch of the model of a vesicle consistent with the measured SAXS data.
B) SAXS spectra of the WT-CFTR membranes
C) The electronic density profile was calculated from the five Gaussian model
D) The decomposition of each singular Gaussian used to model the electronic density

Electron density profiles of from microsome vesicles wall of native cells (green) and cells overexpressing CFTR (blue; protein involved in cystic fibrosis). Micrographs of vesicles are shown.

Baroni et al., Direct interaction of a CFTR potentiator and a CFTR corrector with phospholipid bilayers, European Biophysics Journal, July 2014, Volume 43, Issue 6-7, pp 341-346

SAXS for the study of the use of membrane vesicles for cystic fibrosis research (2)

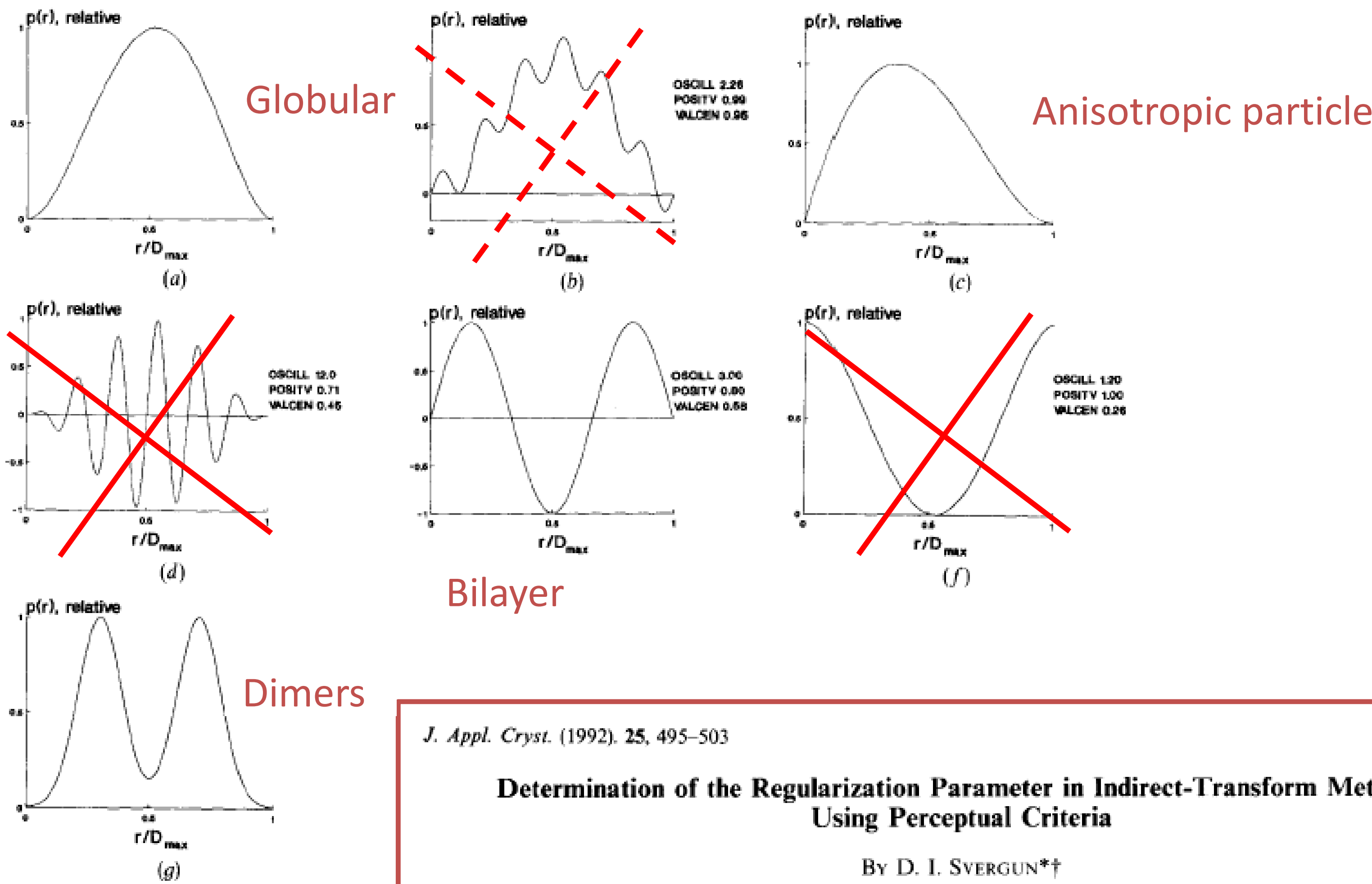


Large unilamellar liposomes (LUV) obtained by extrusion of phospholipids, treated with drugs for the cystic fibrosis therapy.

Electron density of the LUVs wall showing the destabilization induced by drugs.

Partially supported by the Italian Cystic Fibriosis Foundation

The study of particles in solution, some principles



J. Appl. Cryst. (1992). **25**, 495–503

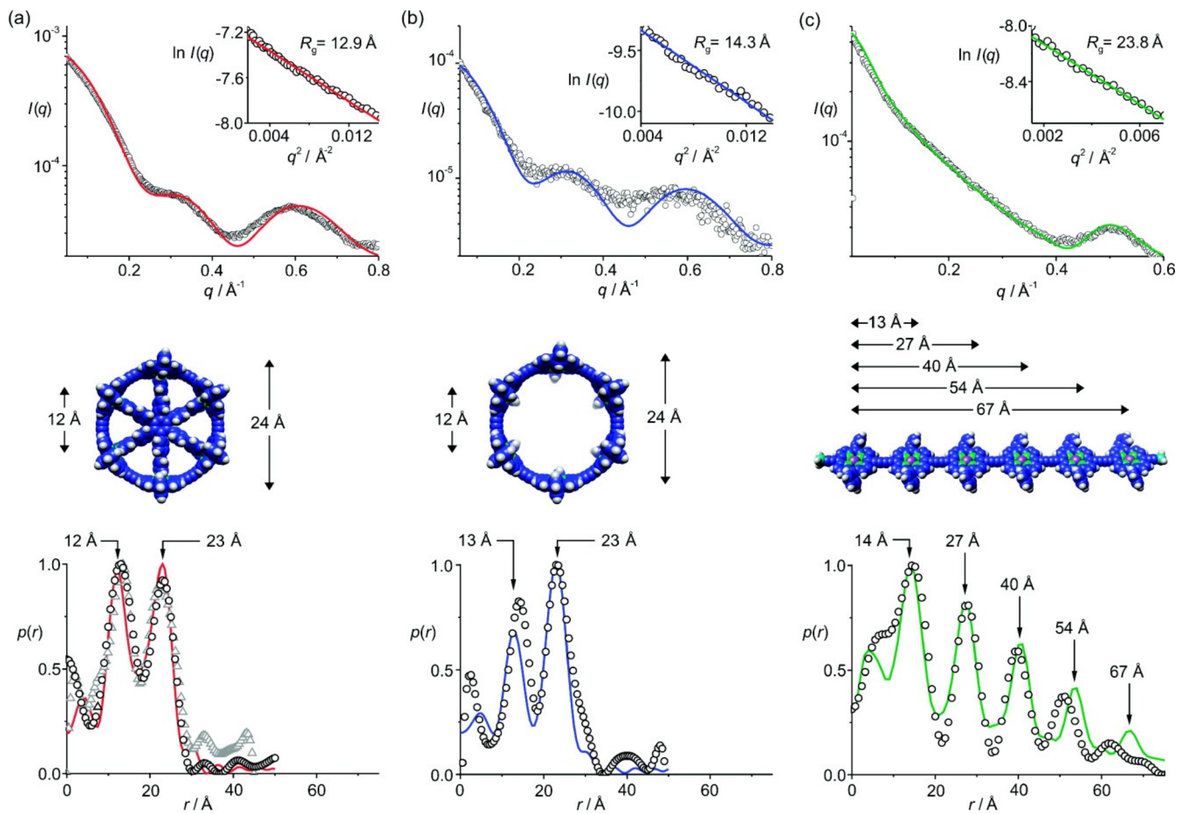
Determination of the Regularization Parameter in Indirect-Transform Methods Using Perceptual Criteria

By D. I. Svergun*†

GKSS Research Center, GKSS-WS, 2054 Geesthacht, Germany

SAXS in Vernier complexes of porphyrin nano-rings

Peak position = Zinc position



O'Sullivan *et al.*, Vernier templating and synthesis of a 12-porphyrin nano-ring, Nature, (2011) 469, 72–75

Beamline staff

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- Juan-Carlos Martínez

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Technician:
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