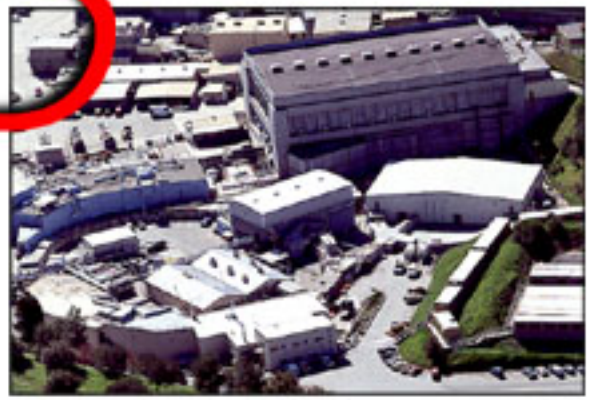
STANFORD SYNCHROTRON RADIATION LABORATORY

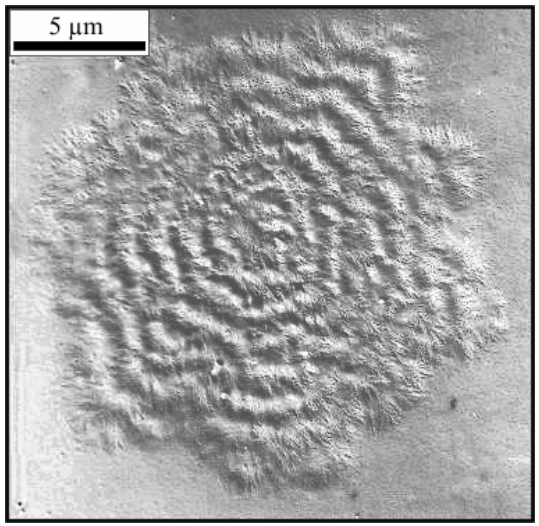
SSRL



Everything You Ever Wanted to Know About SAXS But Were Afraid to Ask

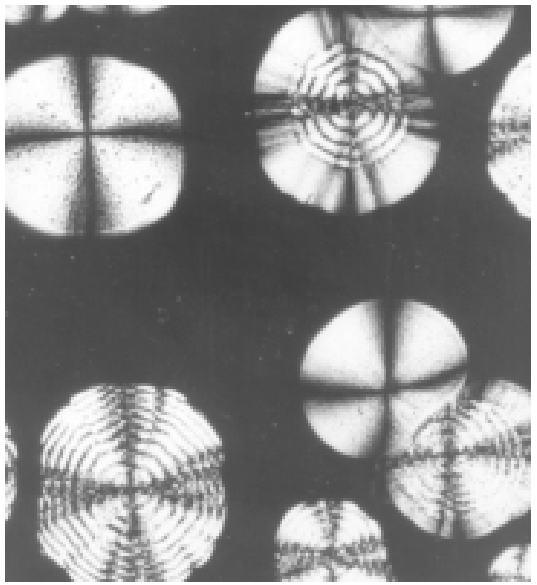
John A Pople
Stanford Synchrotron Radiation Laboratory,
Stanford Linear Accelerator Center, Stanford CA 94309

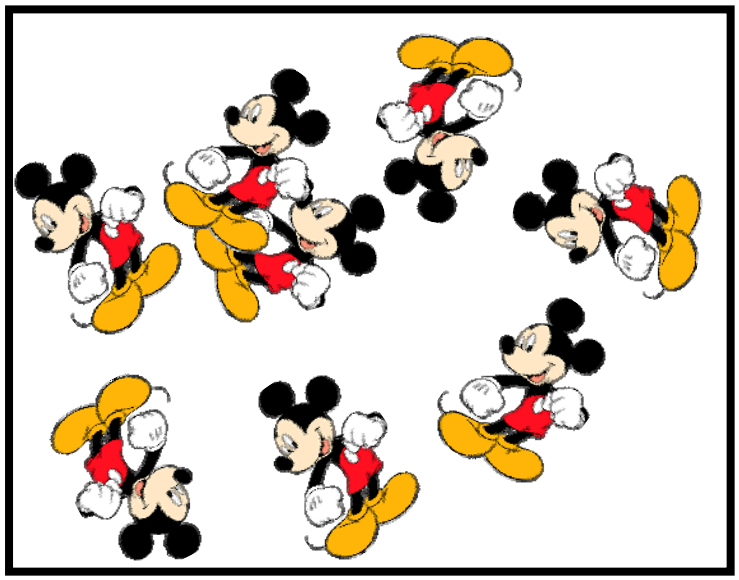
Scattering or Microscopy?



Microscopy good for:

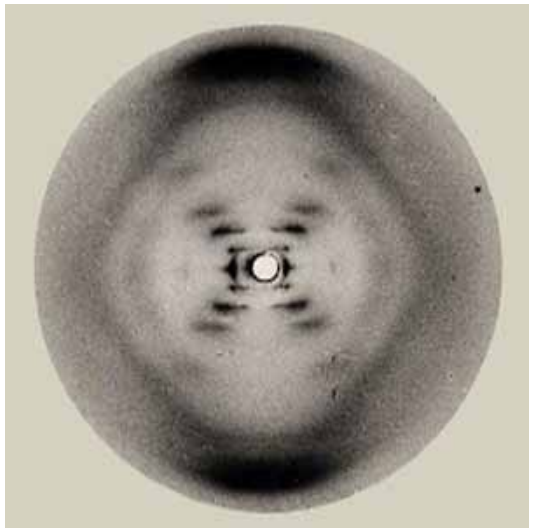
- Local detail
- Surface detail
- Faithfully represents local complexities





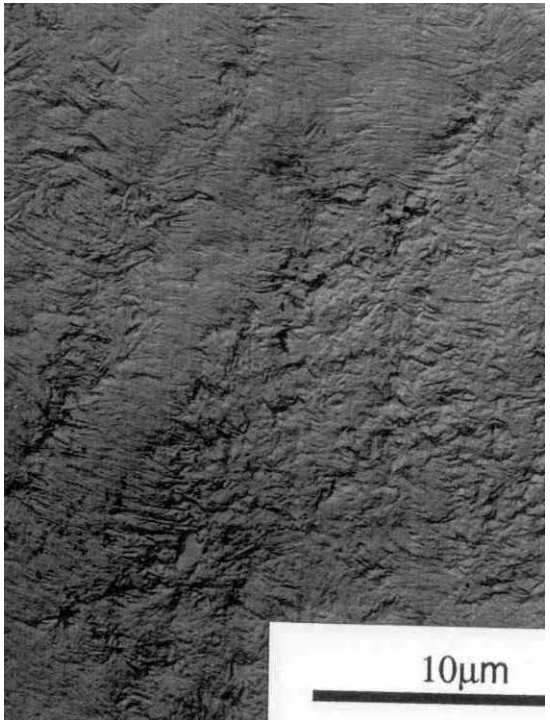
E.g. if objective is to monitor the degree to which Mickey's nose(s) and ears hold to a circular micromorphology… use microscopy not scattering

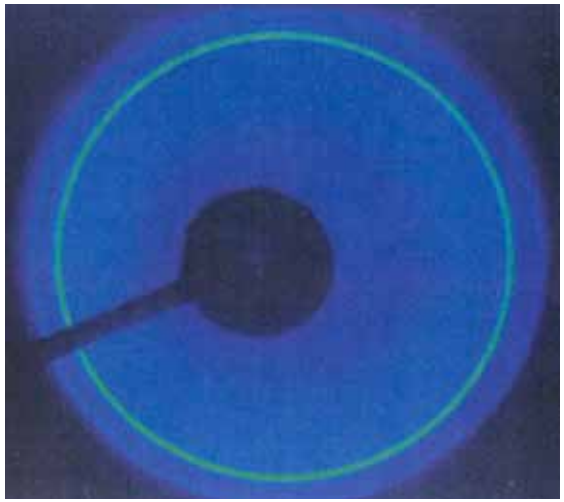
Scattering or Microscopy?



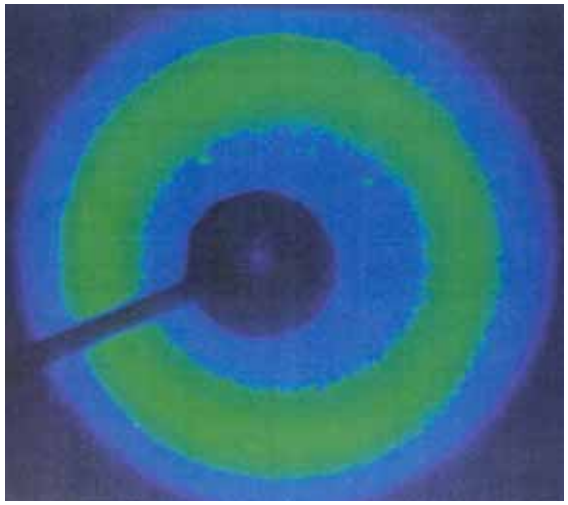
Scattering good for:

- Global parameters, distributions; 1st order
- Different sample states
- Non destructive sample preparation
- In-situ transitional studies

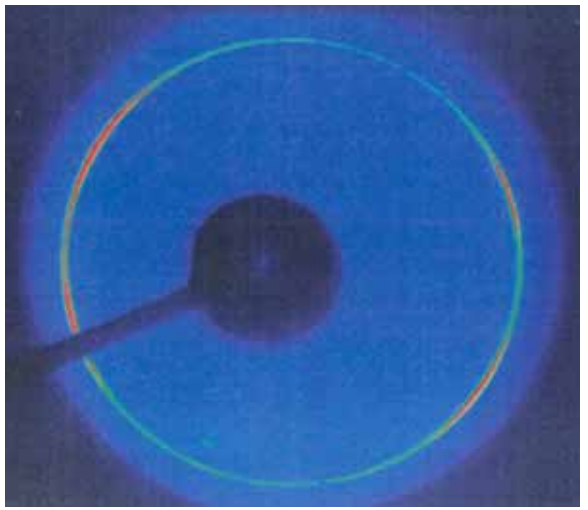




Solid

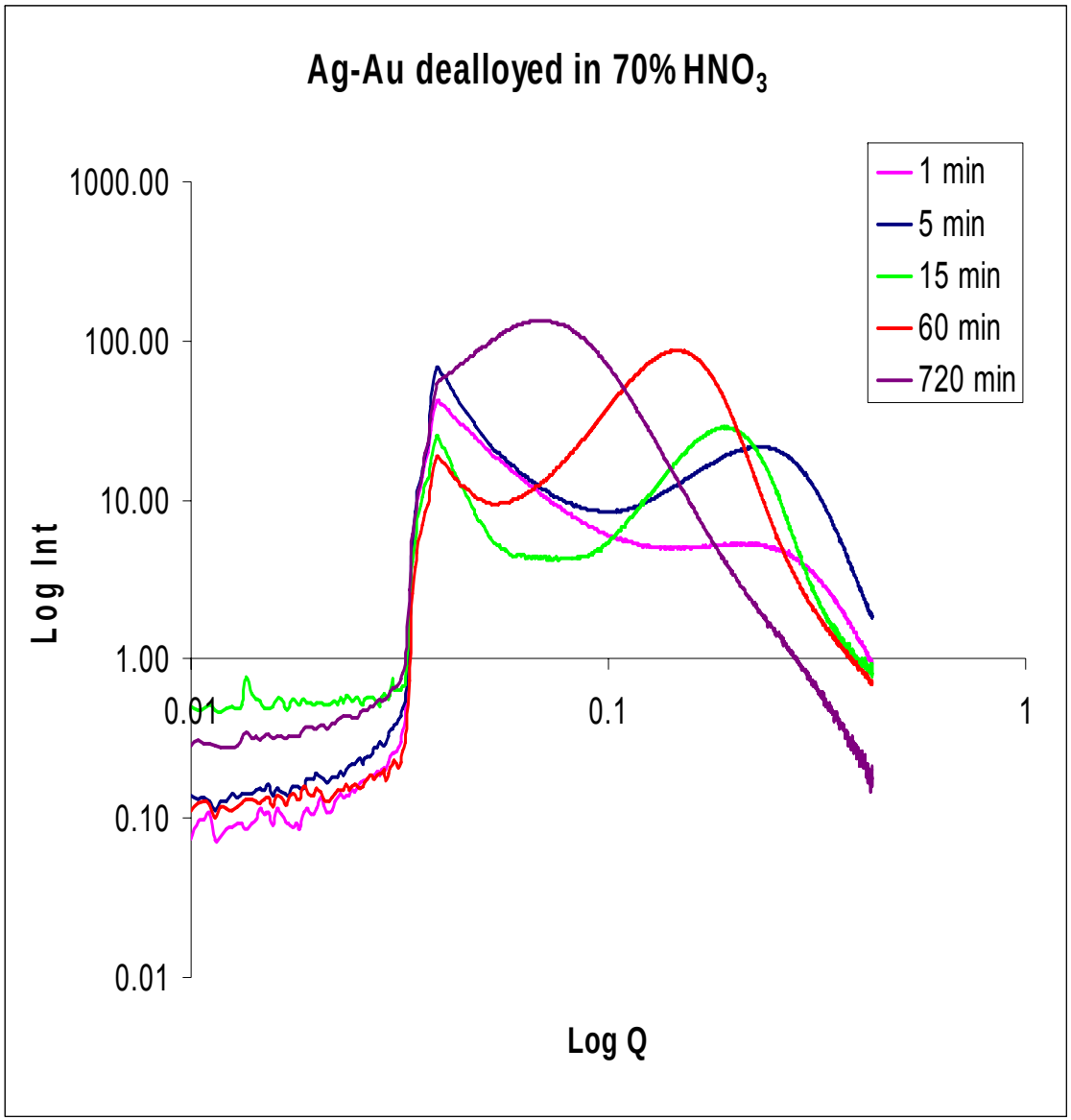


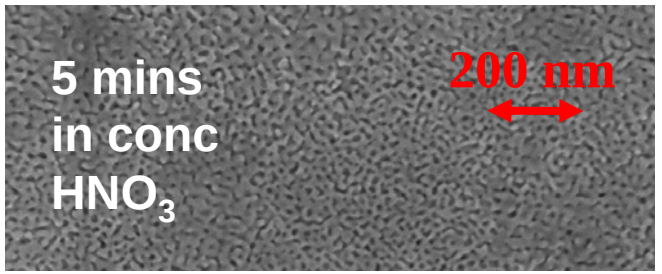
Melted & Sheared

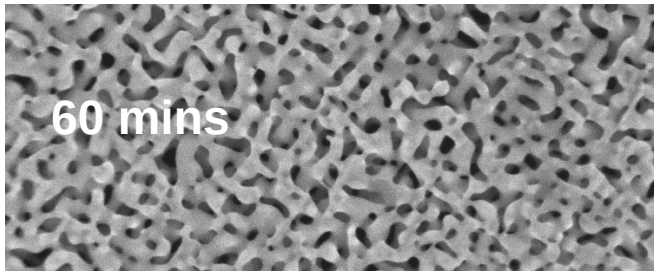


Recrystallized

Complementary Scattering and Microscopy

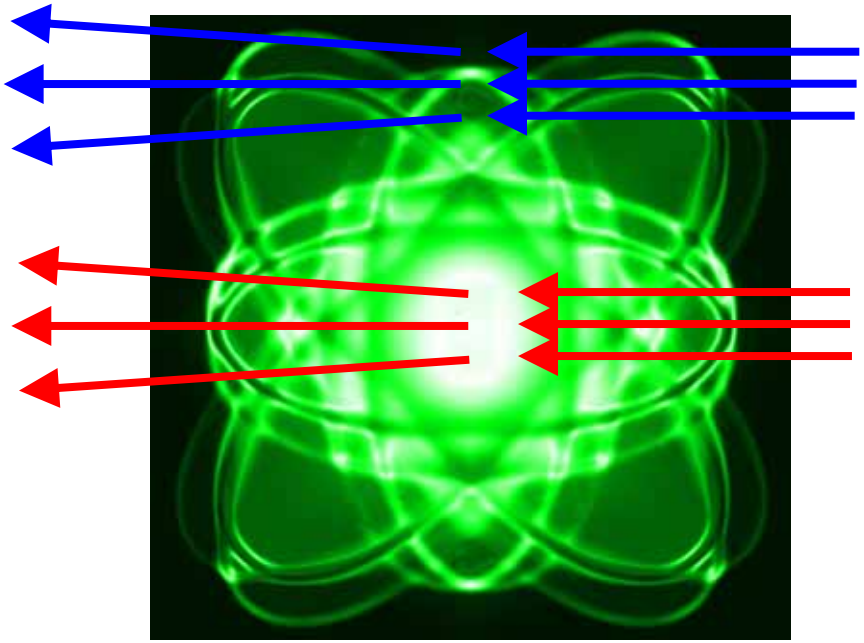






Forming a bi-continuous porous network with ligament width on the nanoscale by removing the less noble element from a binary alloy, in this case Ag-Au (multiple films for trans scattering)

Scattering: Neutrons or Photons?



X-rays

Sensitive to electron density contrast

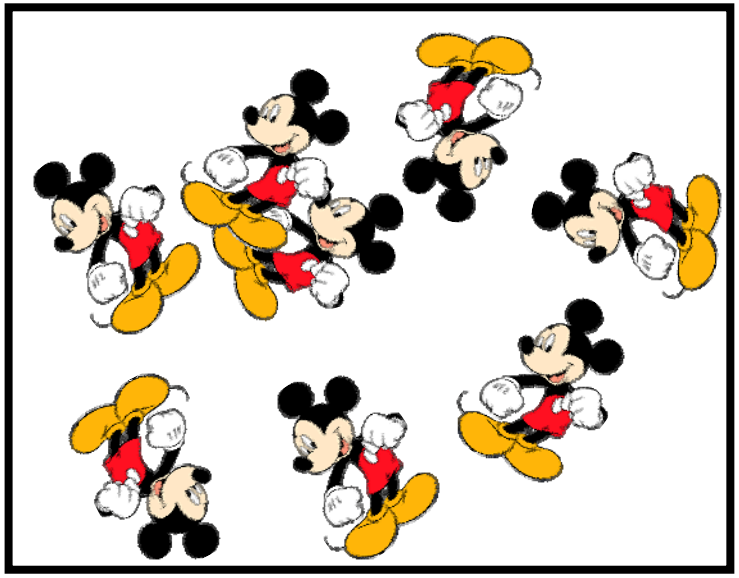
Neutrons

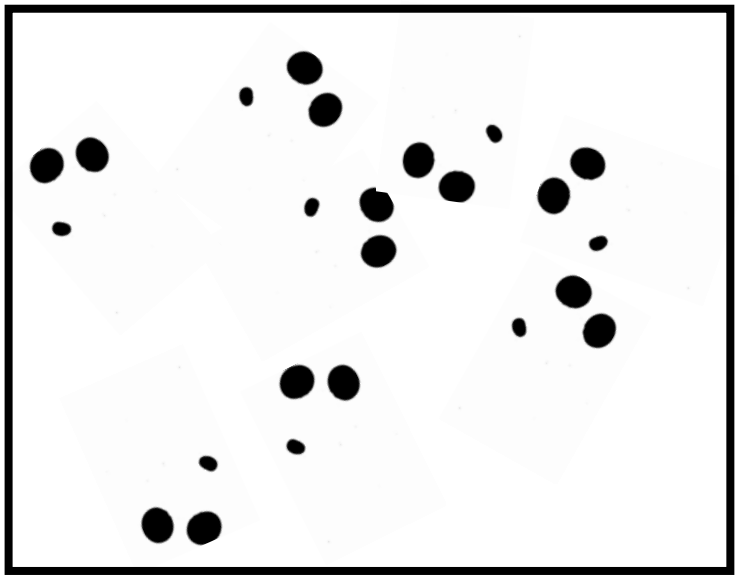
Sensitive to nuclear scattering length contrast

Neutron scattering: Deuteration allows species selection

X-ray scattering:
Relatively small sample quantities required
Relatively fast data acquisition times - allows time resolved effects to be characterized

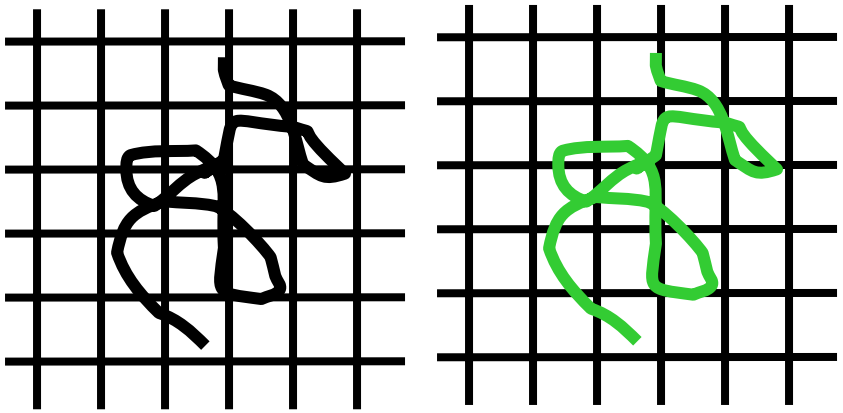
Scattering: Neutrons or Photons?





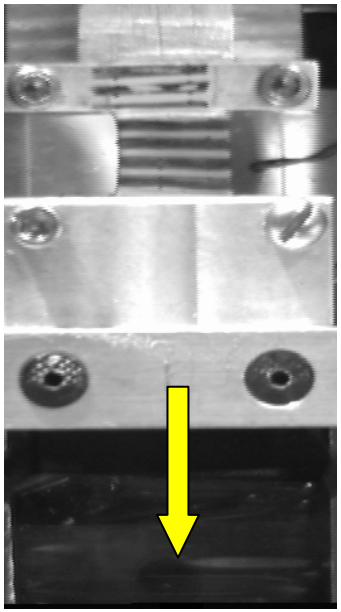
Neutrons: Deuteration allows species selection

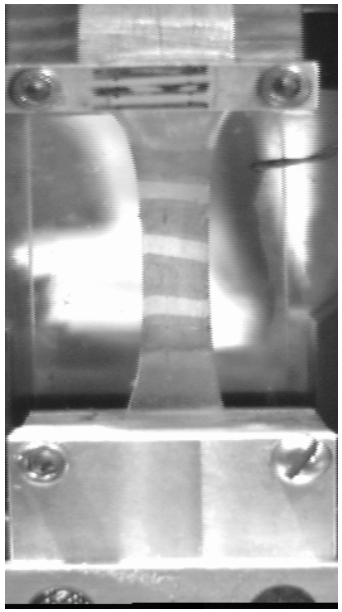
This essentially permits a dramatic alteration to the 'visibility' of the tagged elements in terms of their contribution to the reciprocal space scattering pattern



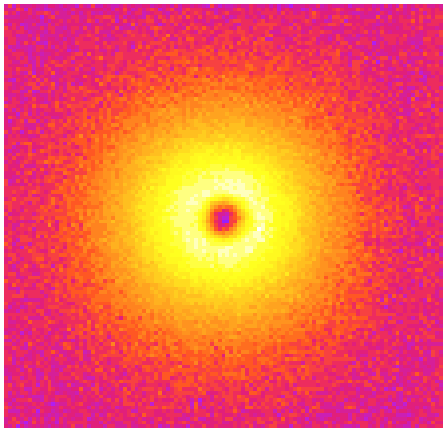
Atom	Scattering length ($\times 10^{12}$ cm^2)	Incoherent scattering ($\times 10^{24}$ cm^2)
^{1}H	-0.374	80
^{2}D	0.667	2

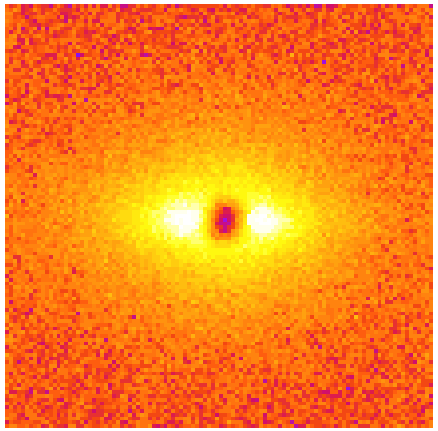
Scattering: Neutrons or Photons?





Photos of deformation





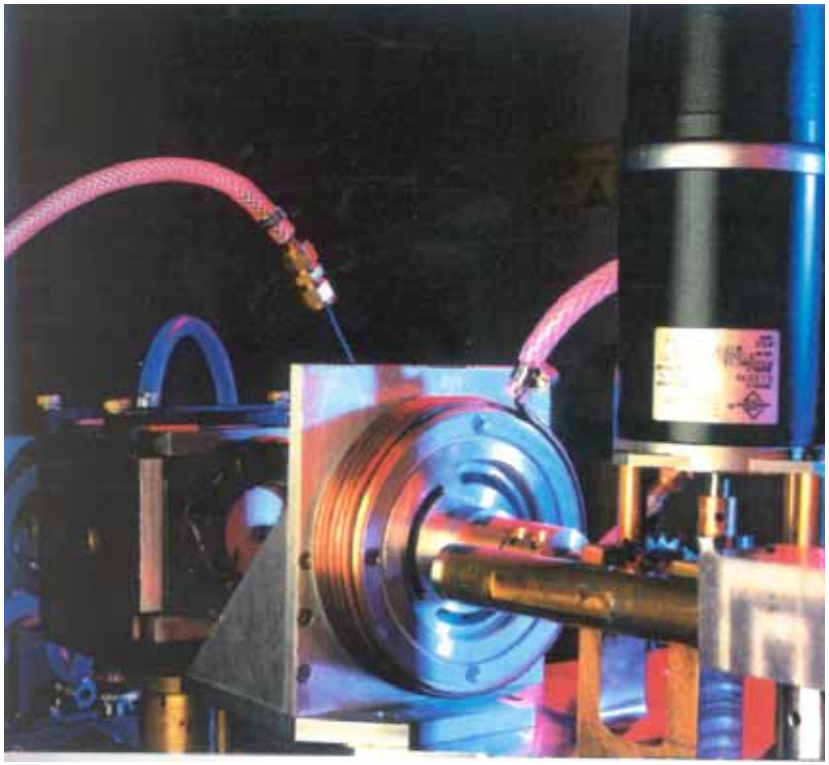
SANS patterns

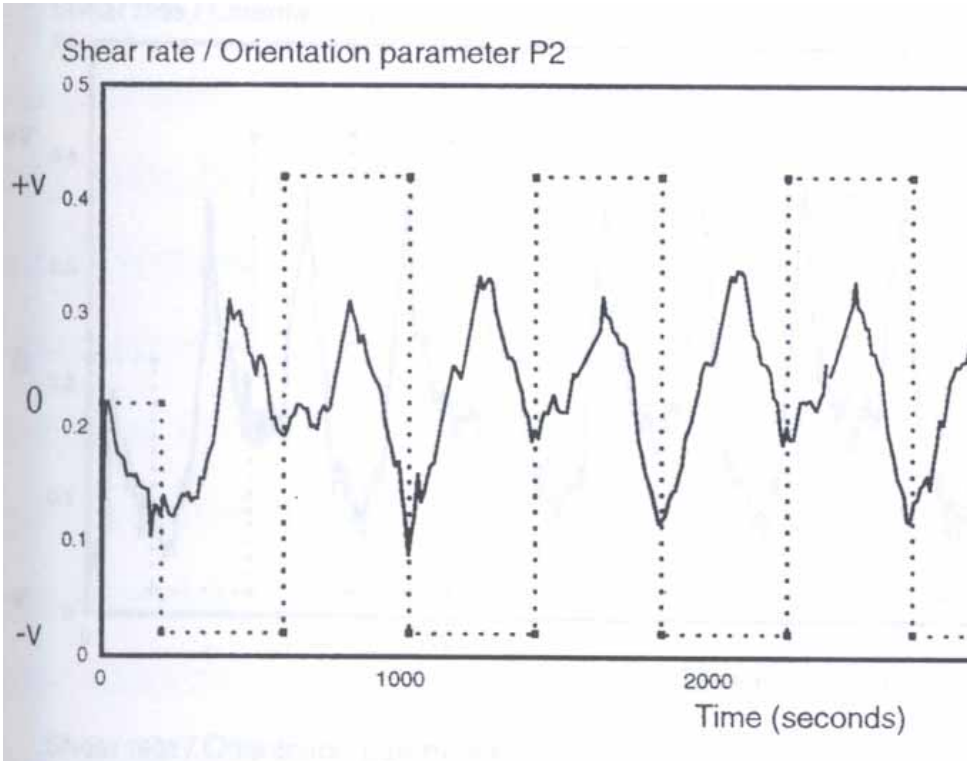
λ = 0%

λ = 300%

Scattering: Neutrons or Photons?

X-rays: Order of magnitude better spatial resolution
Fast data acquisition times for time resolved data





Oscillatory Shearing of lyotropic HPC – a liquid crystal polymer

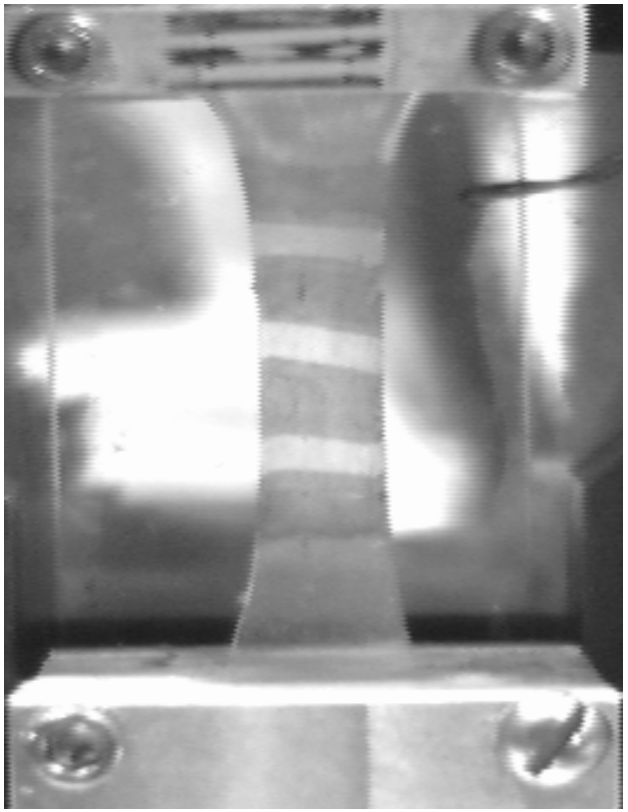
X-ray Scattering: Transmission or Reflection?

Need to be conscious of:

Constituent elements, i.e. absorption cutoffs

Multiple scattering

Area of interest: surface effect or bulk effect



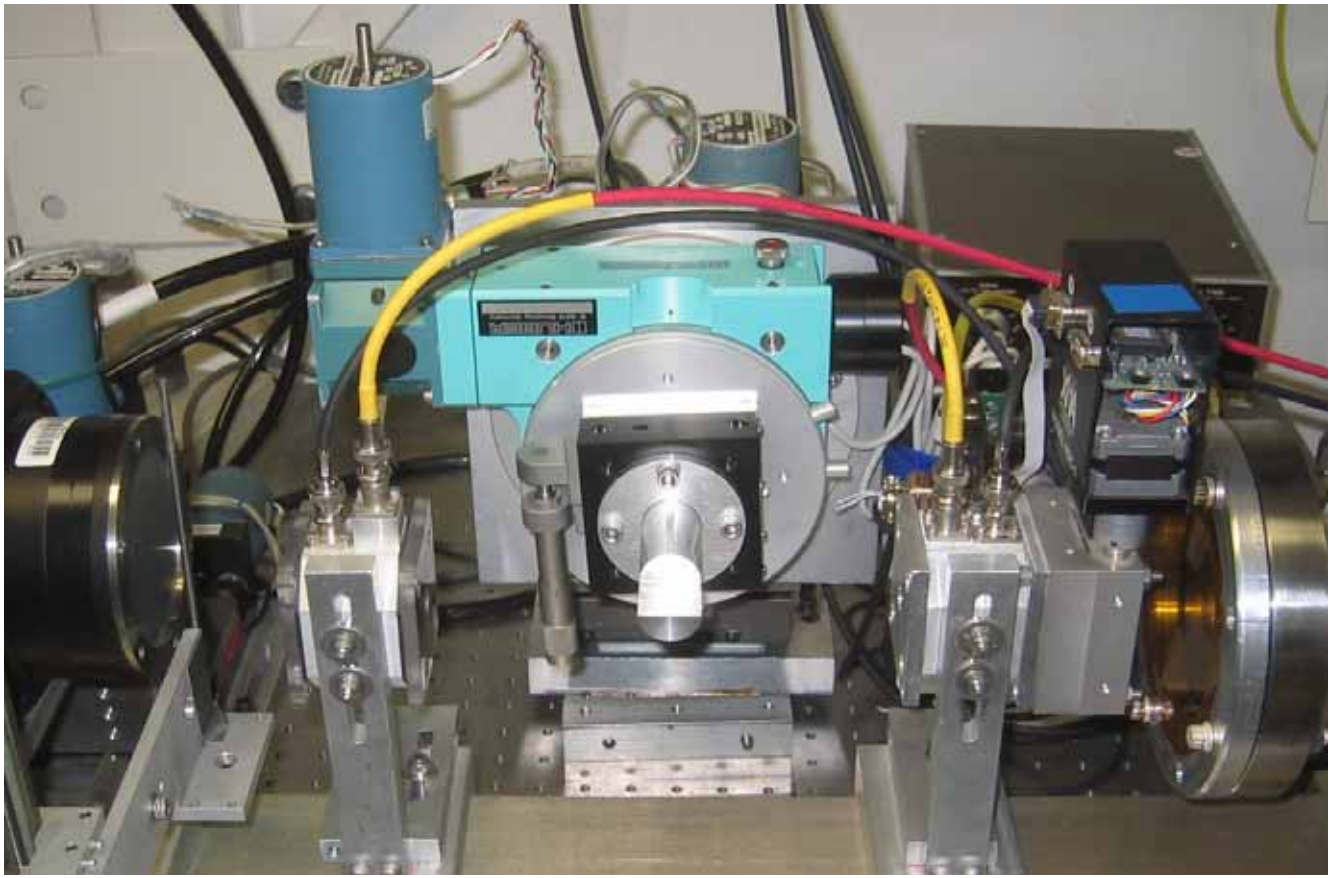
Transmission geometry appropriate for:

- Extracting bulk parameters, especially in deformation
- Weakly scattering samples: can vary path length

X-ray Scattering: Transmission or Reflection?

Reflection geometry appropriate for:

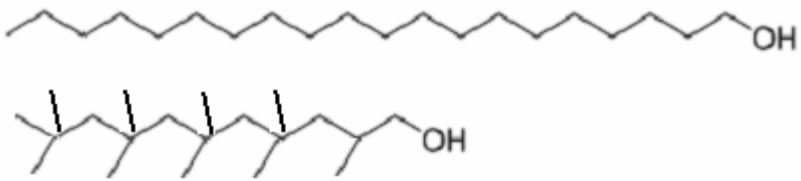
- Films on a substrate (whether opaque or not)
- Probing surface interactions

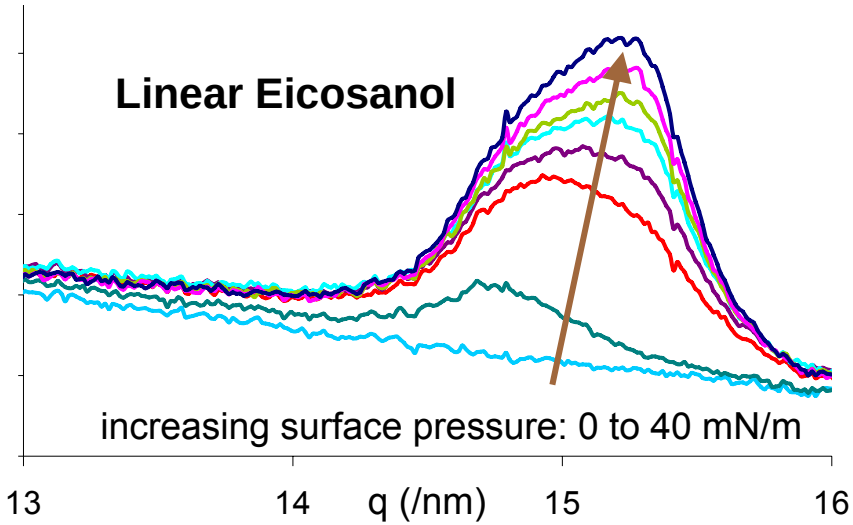


Rheology of Straight and Branched Fatty Alcohols

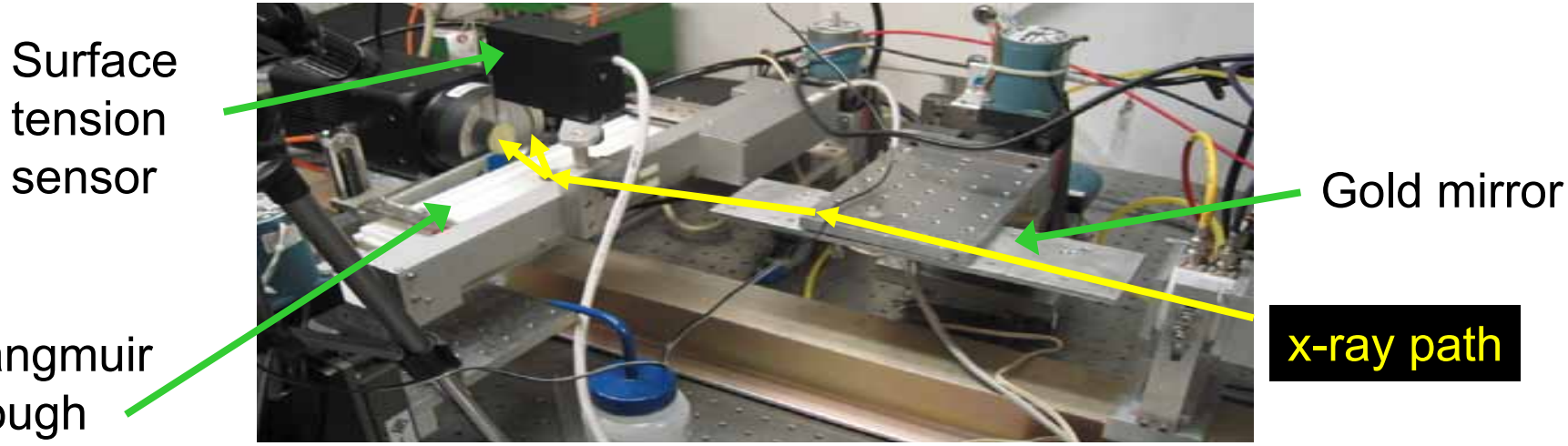
Study phase transitions of Langmuir monolayers of mixed fatty alcohols in terms of molecular branching and surface tension

linear eicosanol ($C_{20}H_{42}O$, MW = 298)



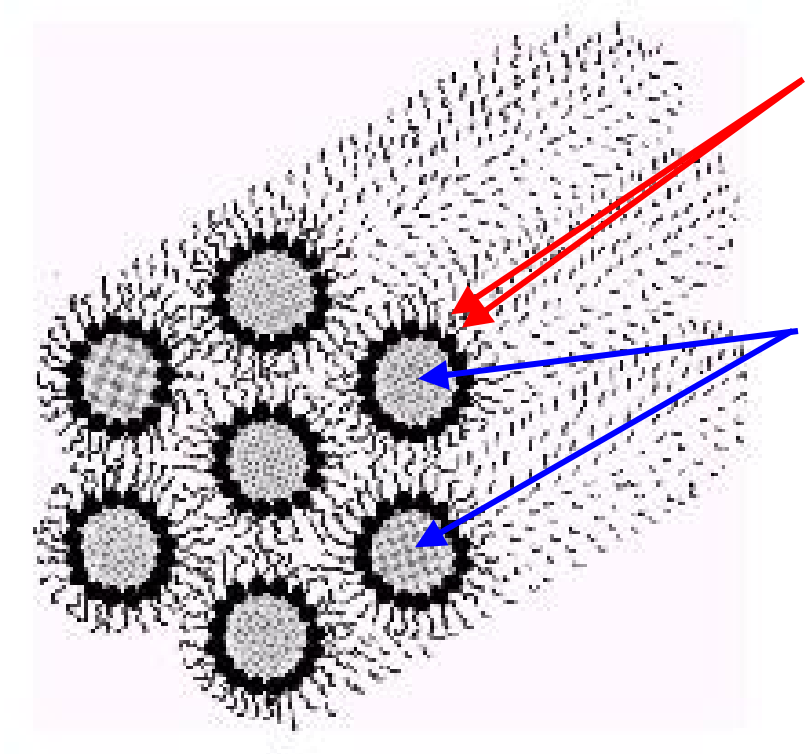


branched eicosanol ($C_{20}H_{42}O$, MW = 298)



X-ray Scattering: SAXS or WAXS?

No fundamental difference in physics: a consequence of chemistry



WAXS patterns contain data concerning correlations on an intra-molecular, inter-atomic level

SAXS patterns contain data concerning correlations on an inter-molecular level: necessarily samples where there is macromolecular or aggregate order

As synthesis design/control improves, SAXS becomes more relevant than ever before

X-ray Scattering: SAXS or WAXS?

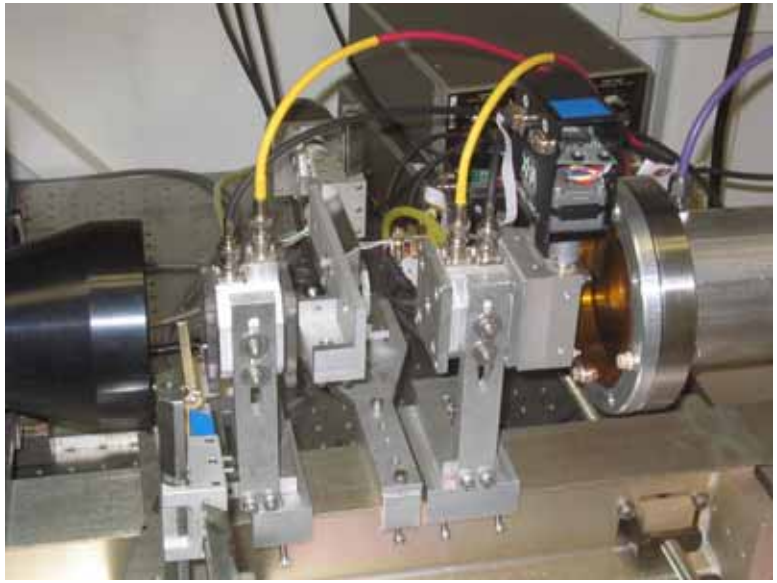
Experimental consequences

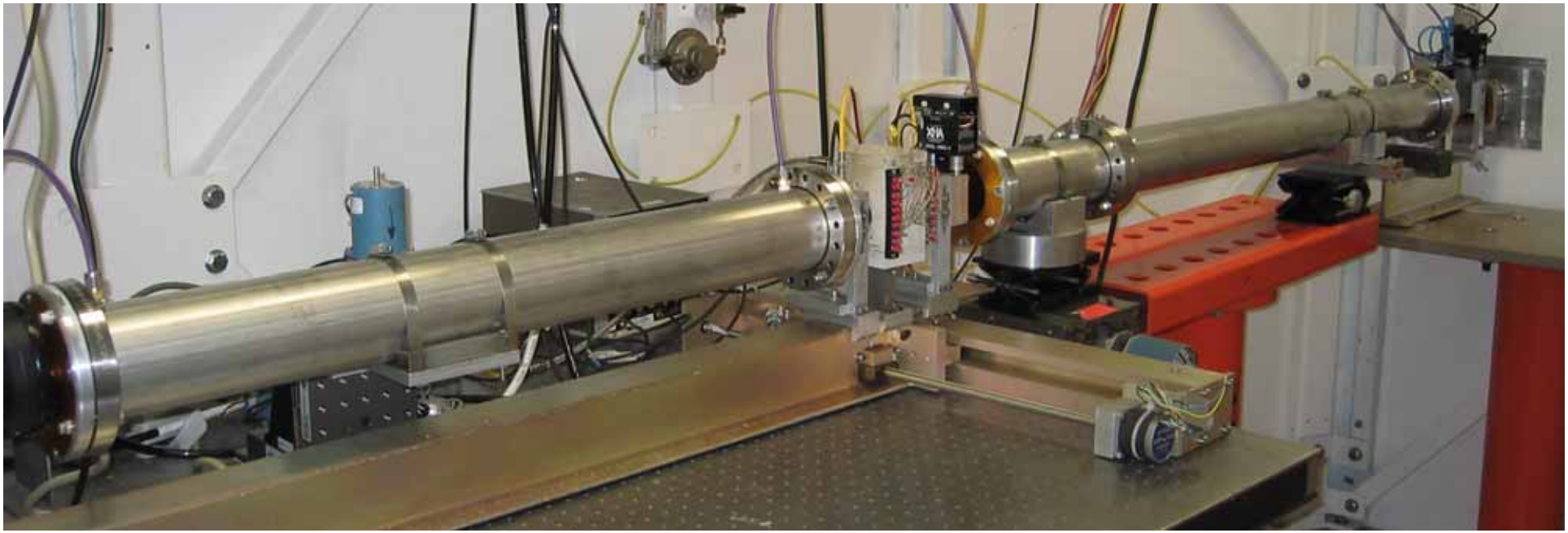
WAXS: Detector close to sample, consider:

- Distortion of reciprocal space mapping
- Thermal effects when heating sample
- No ion chamber for absorption

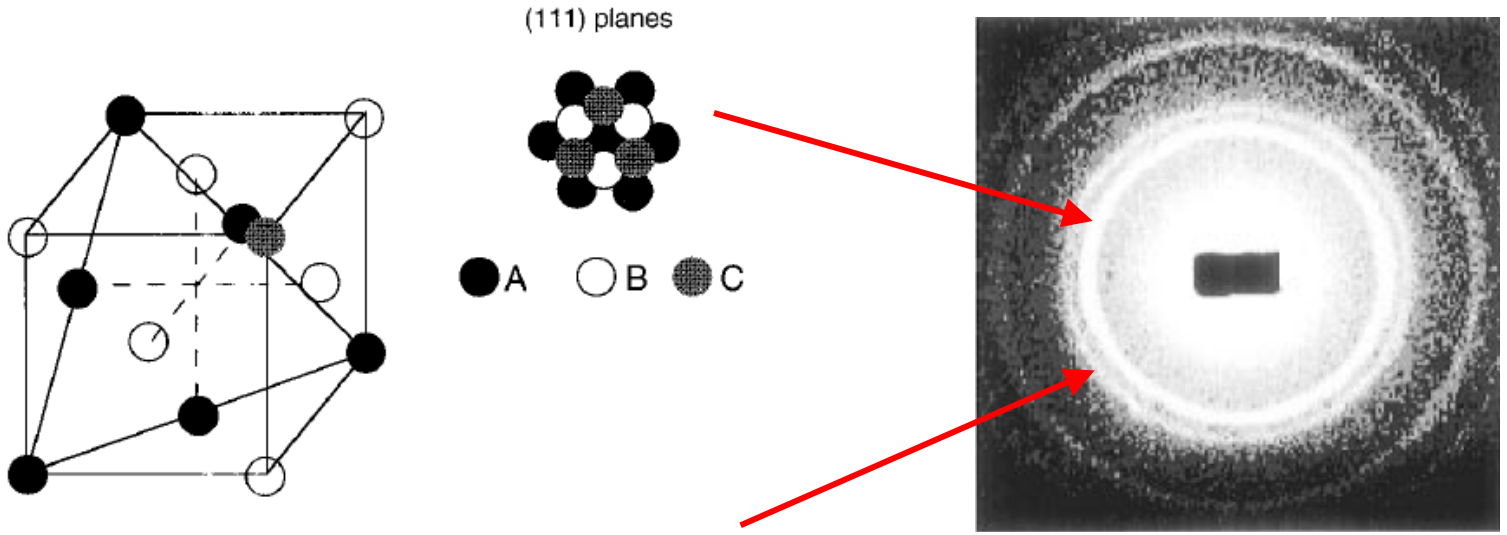
SAXS: Detector far from sample, consider:

- Absorption from intermediate space
- Interception of appropriate q range





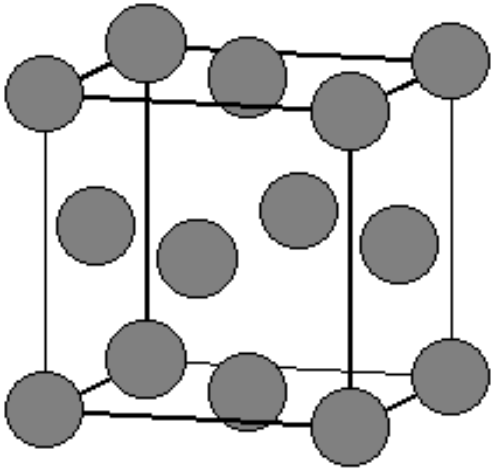
Recognizing Reciprocal Space Patterns: Indexing



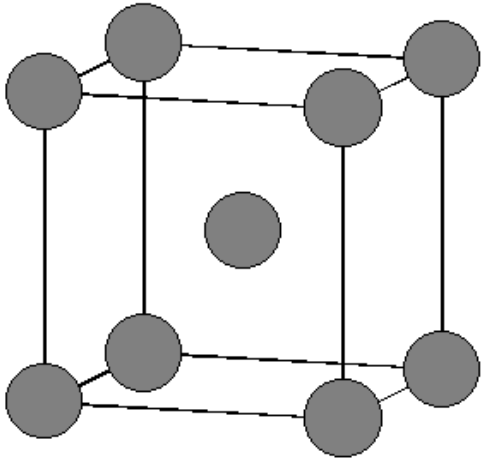
Face centered cubic pattern from diblock copolymer gel

Recognizing Reciprocal Space Patterns: Indexing

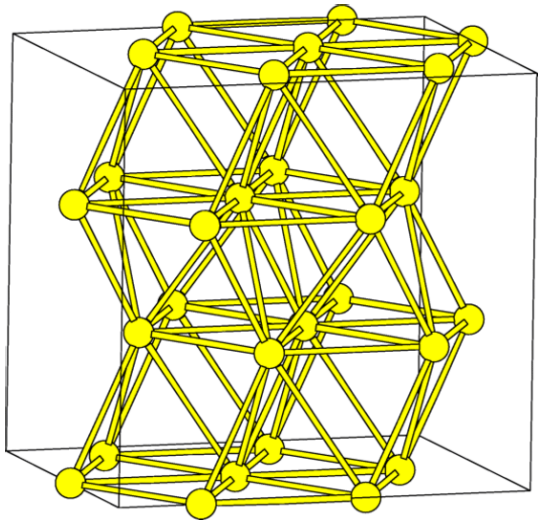
Real space packing



Face centered cubic



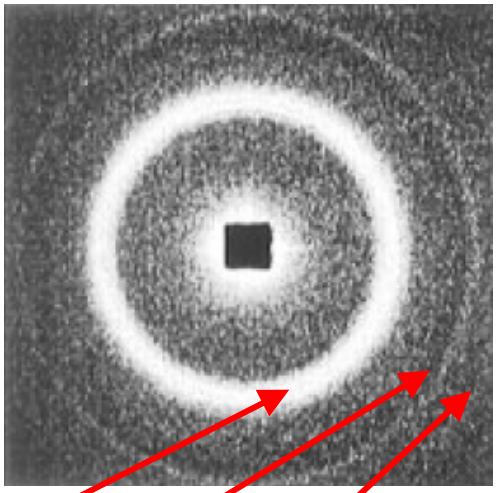
Body centered cubic



Hexagonal

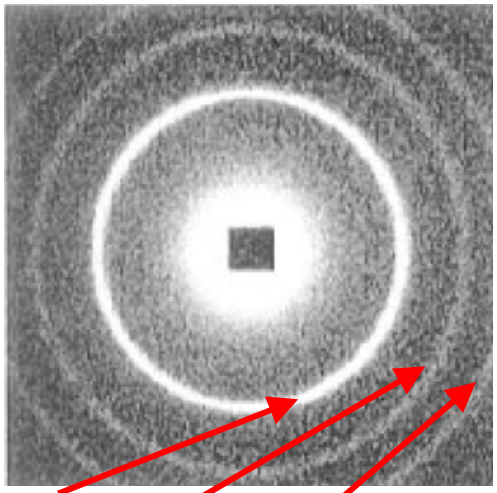
Reciprocal space image

(unoriented domains)

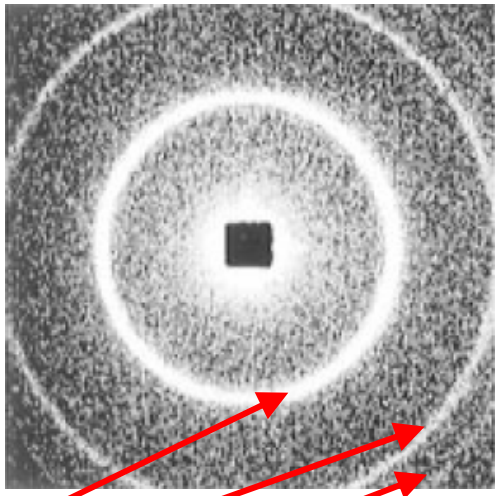


Normalized peak positions

≡1; =√4/3; =√8/3



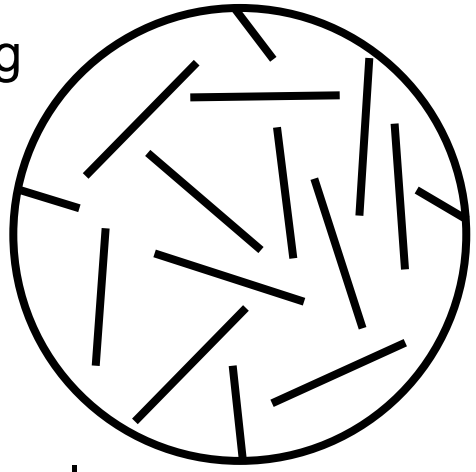
≡1; =√2; =√3

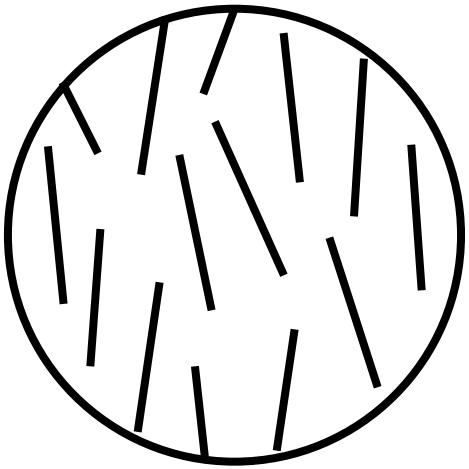


≡1; =√3; =√4

Recognizing Reciprocal Space Patterns: Preferential Orientation

Real space packing



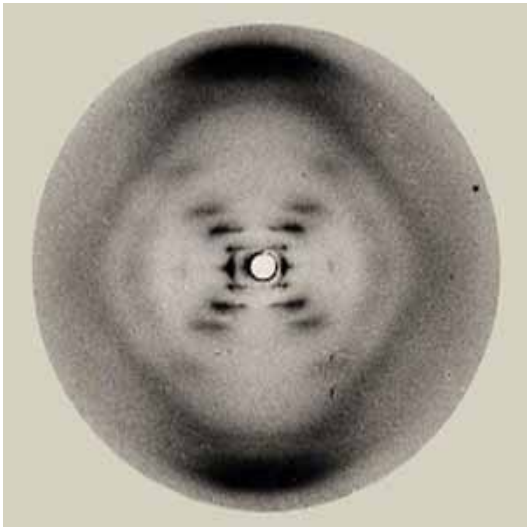


Hydrated DNA

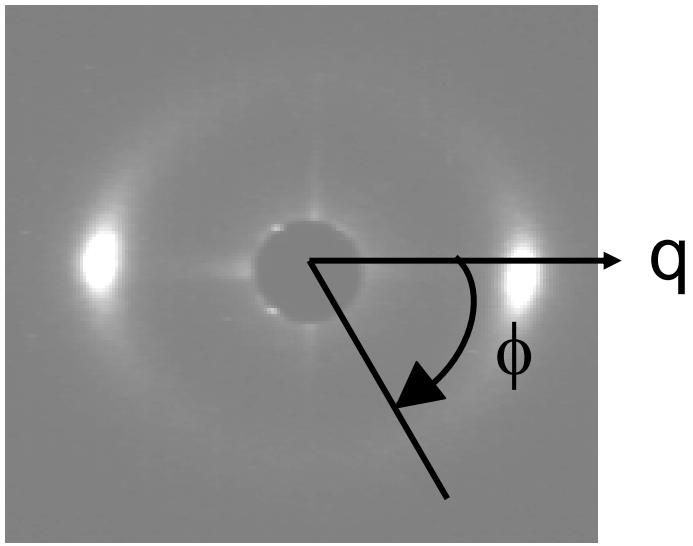
Reciprocal space image

Randomly aligned rods

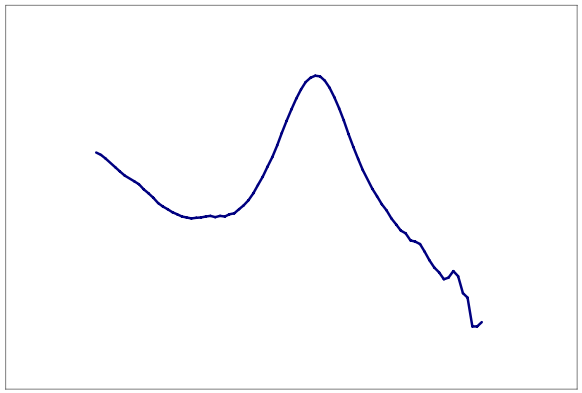
Preferentially aligned rods



Extracting Physical Parameters from X-ray data

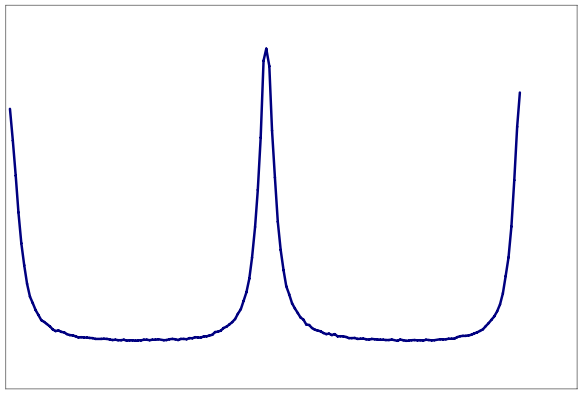


I(q)



q

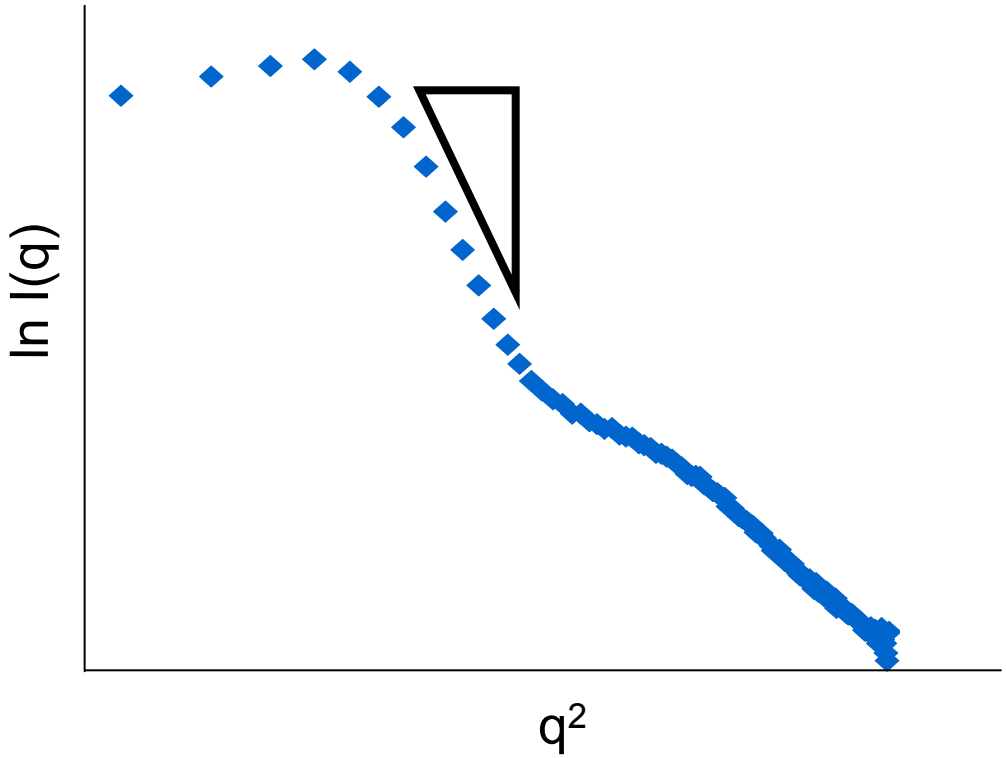
I(ϕ)



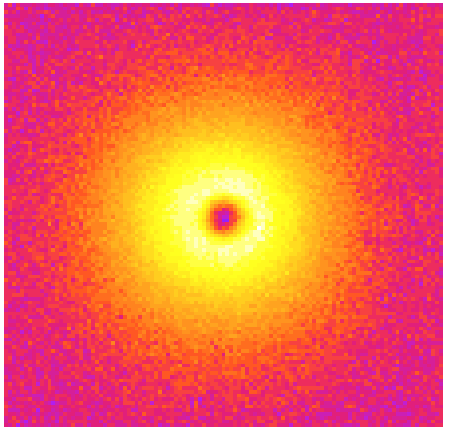
ϕ

Extracting Physical Parameters from X-ray data

Molecular size: Radius of gyration (R_g)



Guinier plot



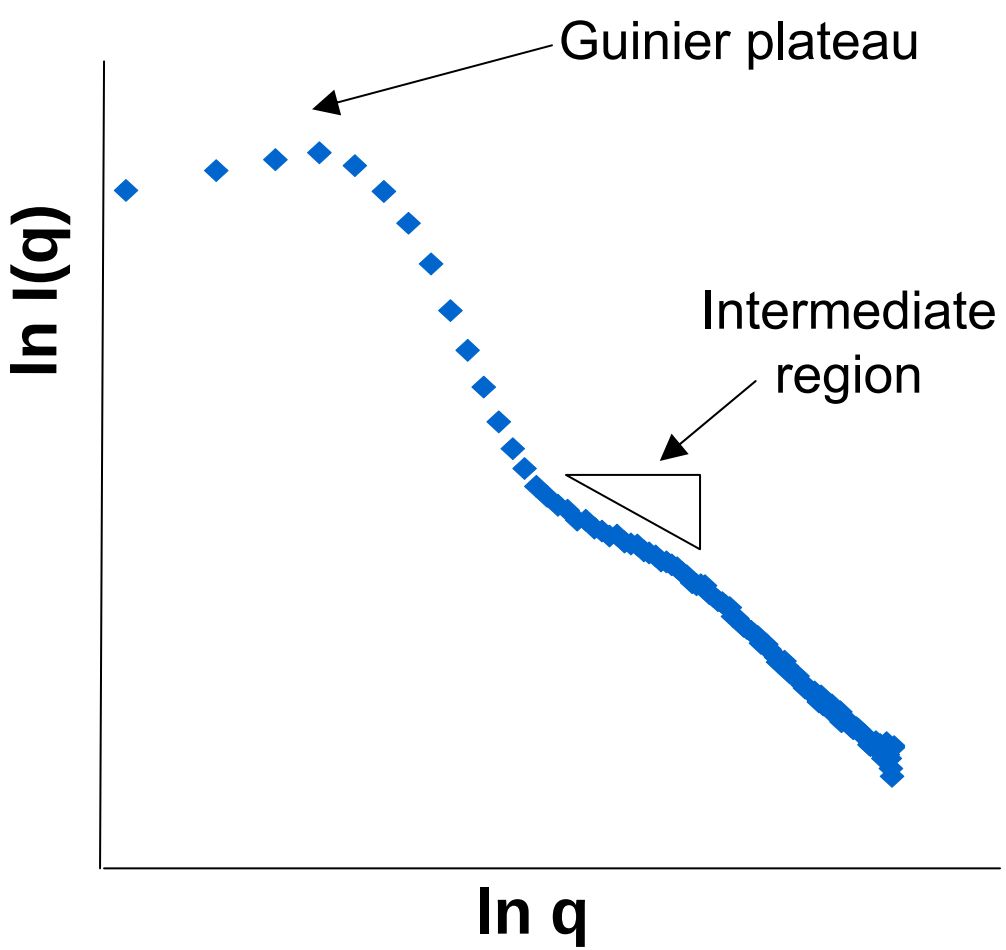
$$I(q) = I(0) \exp [-q^2 R_g^2 / 3]$$

$$R_g^2 \alpha \ln I(q) / q^2$$

Guinier region: $q < 1 / R_g$

Extracting Physical Parameters from X-ray data

Molecular conformation: Scaling exponent



Gradient of profile in intermediate region implies fractal dimension of scattering unit

Rod q^{-1}

Coil in good solvent $q^{-5/3}$

Sphere q^{-4}

Molecular Conformation in Dentin

John H Kinney
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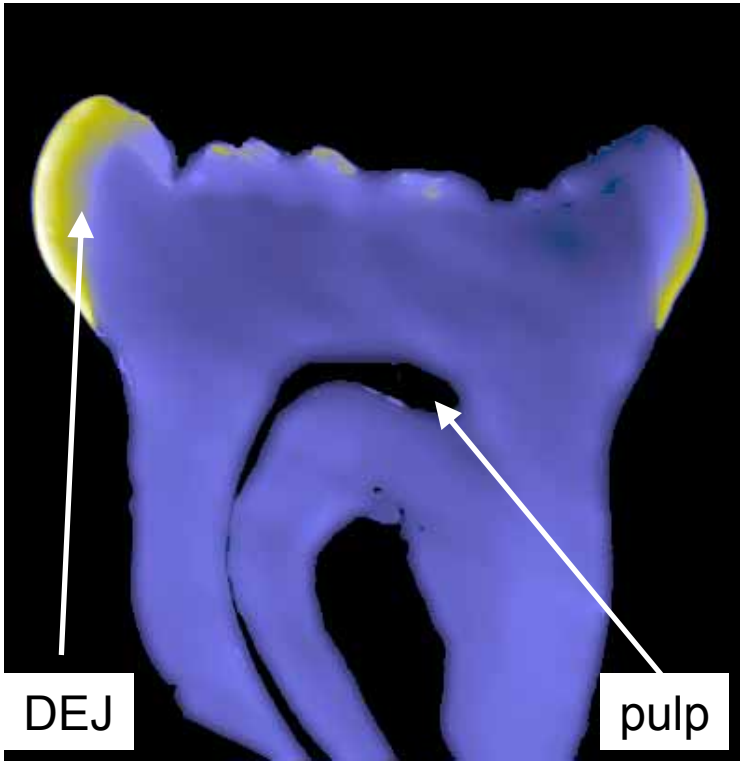
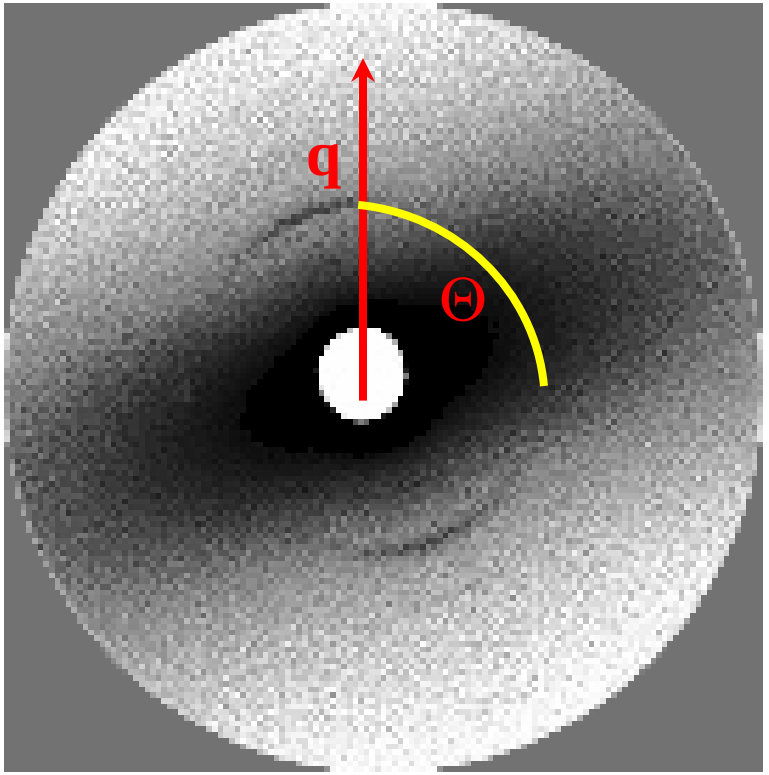
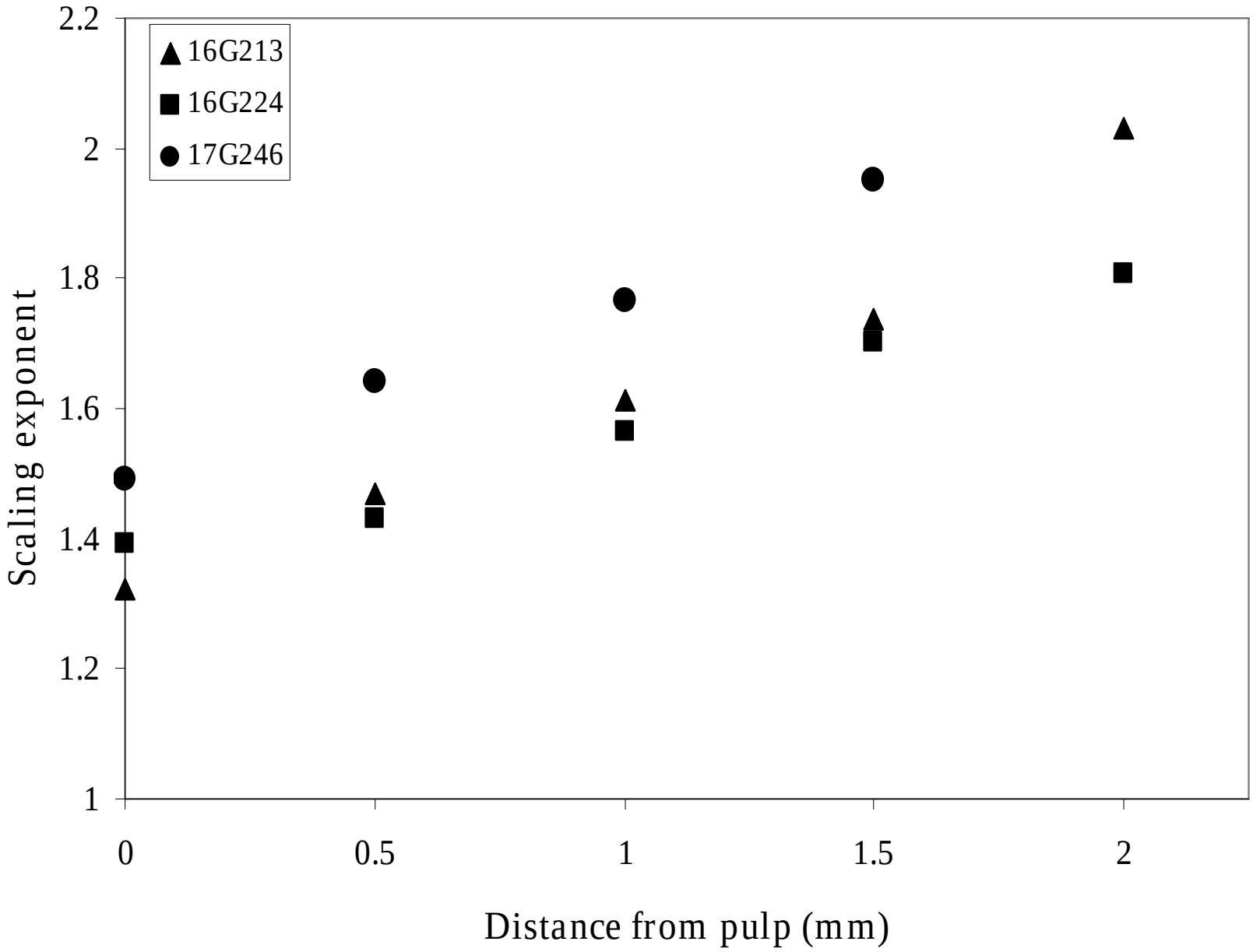
University of California, San Francisco, CA 94143





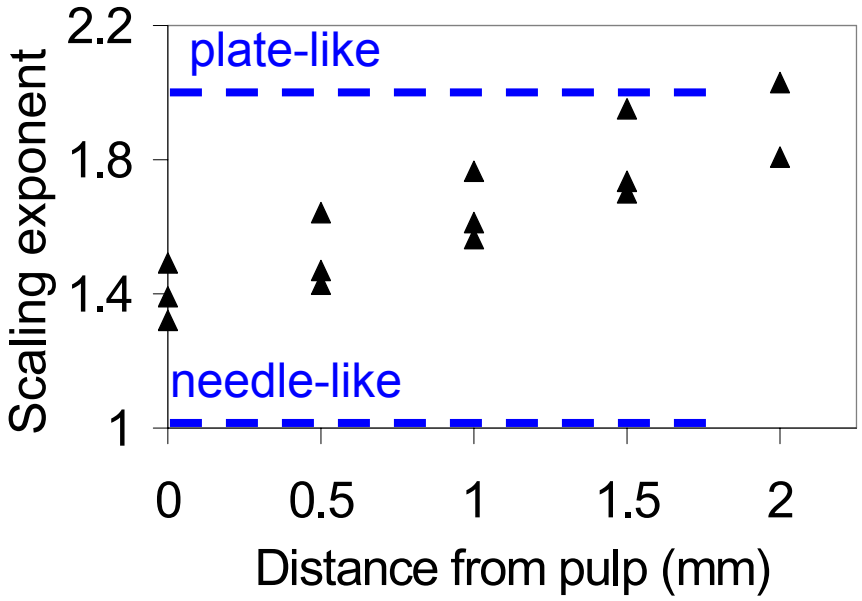
SAXS pattern

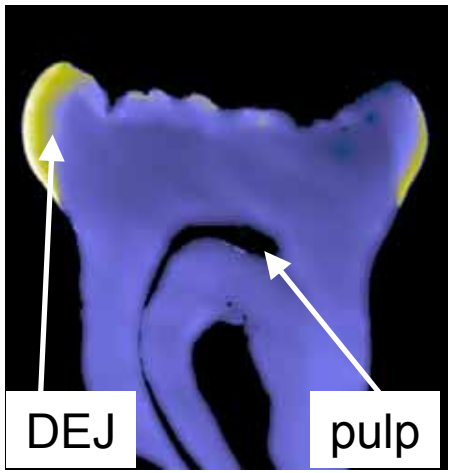
Molecular Conformation in Dentin



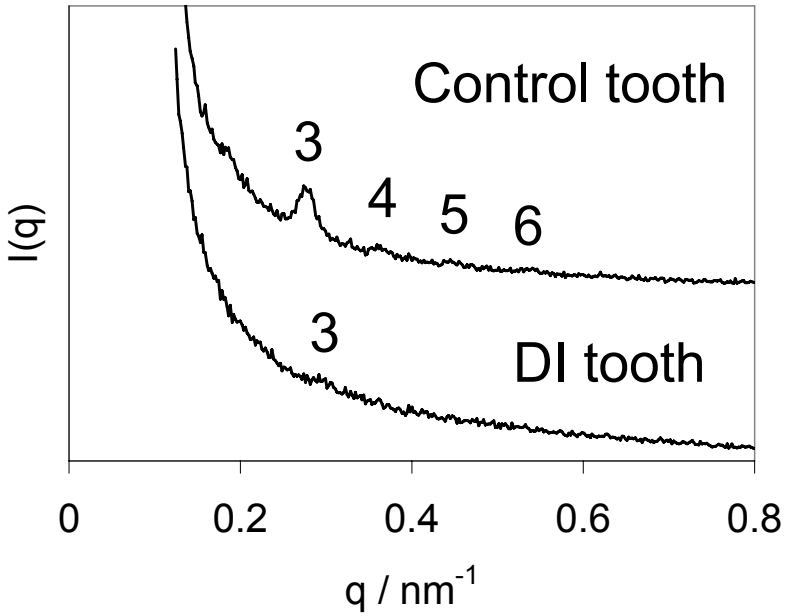
Molecular Conformation in Dentin

Shape change of mineral crystallites from needle-like to plate-like from pulp to dentin-enamel junction (DEJ).



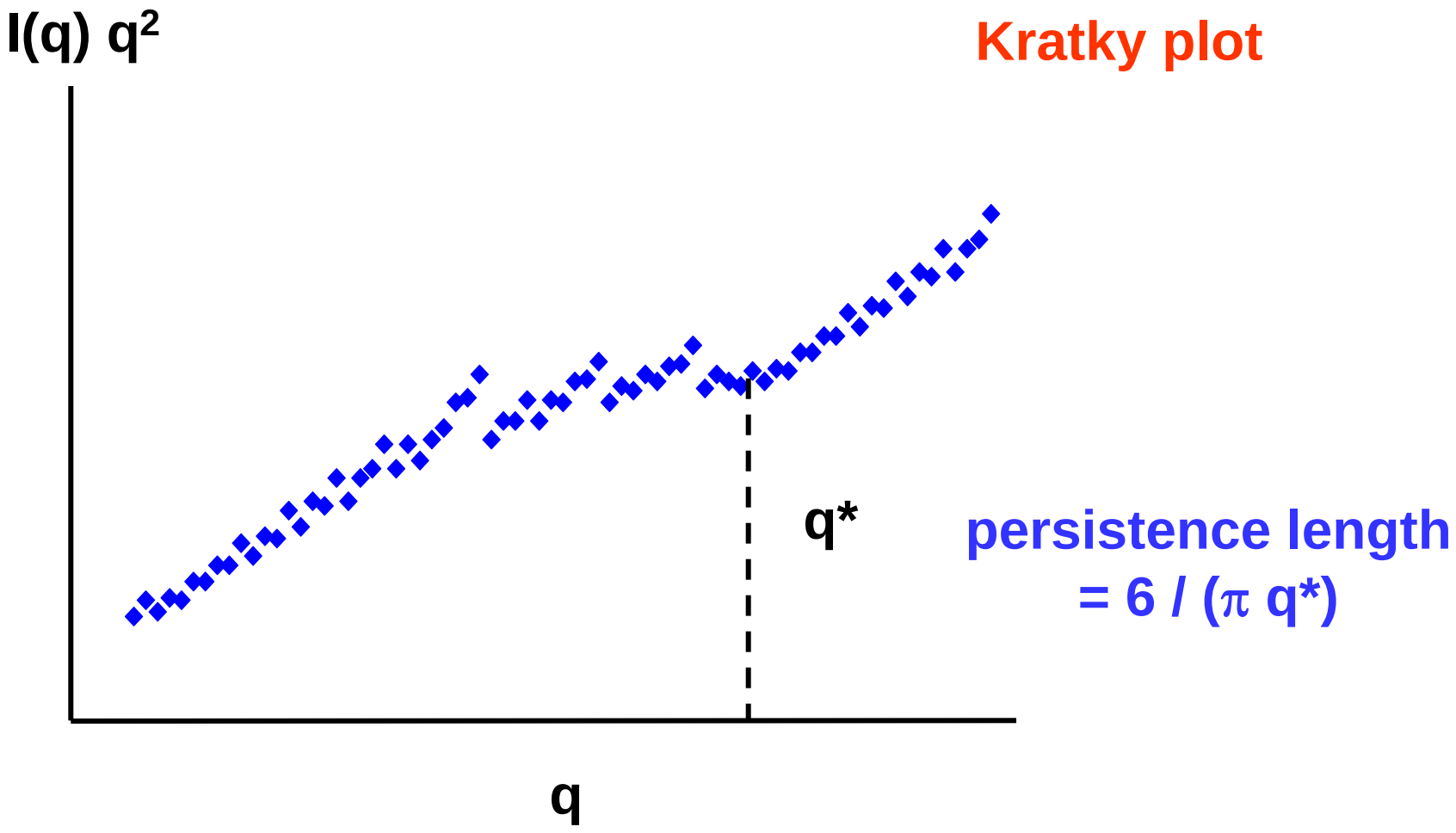


Dentinogenesis imperfecta (DI) teeth shown to exhibit impaired development of intrafibrillar mineral: characteristic scattering peaks are absent from the diseased tooth.



Extracting Physical Parameters from X-ray data

Molecular conformation: Persistence length of coiled chain



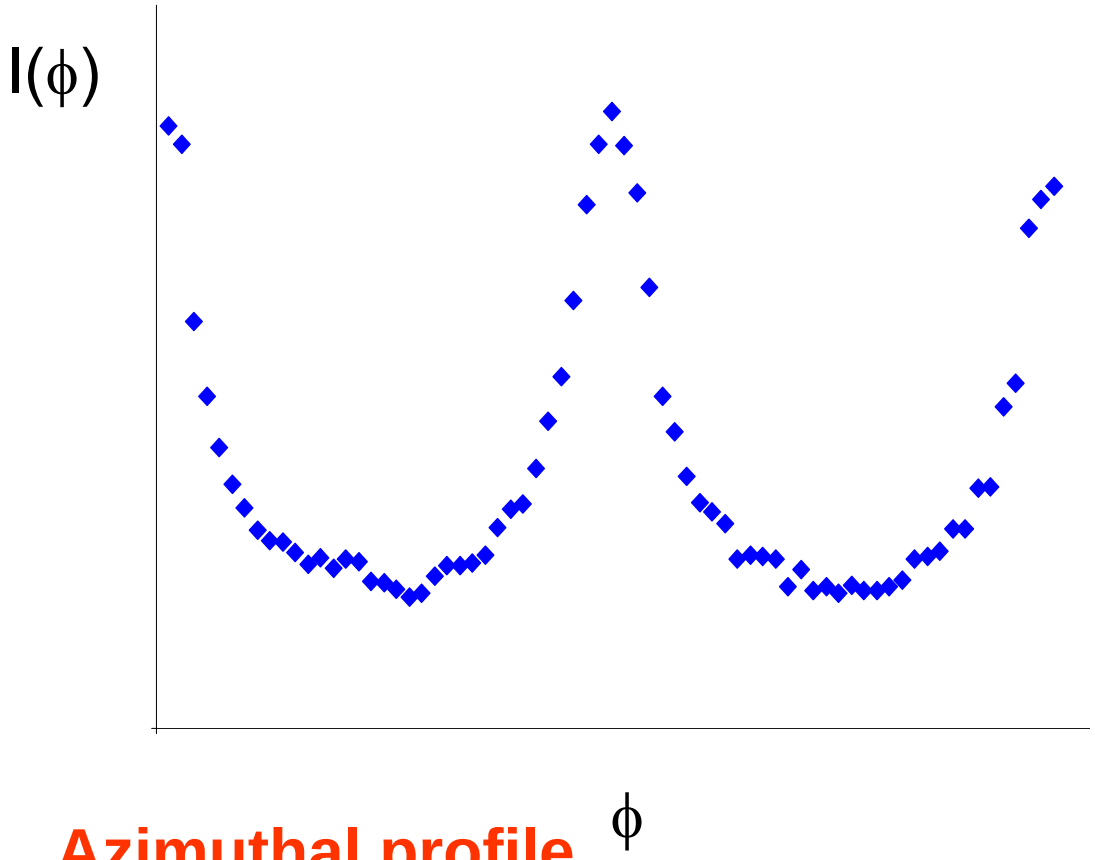
Extracting Physical Parameters from X-ray data

Molecular orientation: Orientation parameter P_2

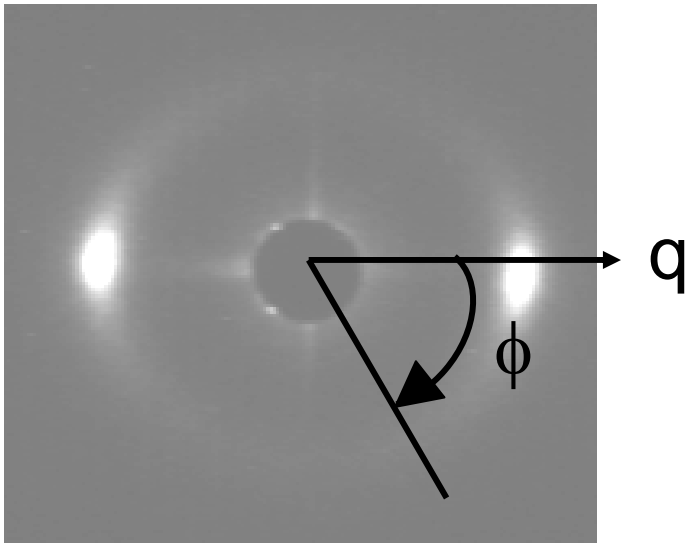
$$\langle P_{2n}(\cos\phi)\rangle = \frac{\int I(s,\phi)\, P_{2n}(\cos\phi)\sin\phi\, d\phi}{\int I(s,\phi)\sin\phi\, d\phi}$$

Normalized:

$-0.5 < P_2 < 1$



Azimuthal profile



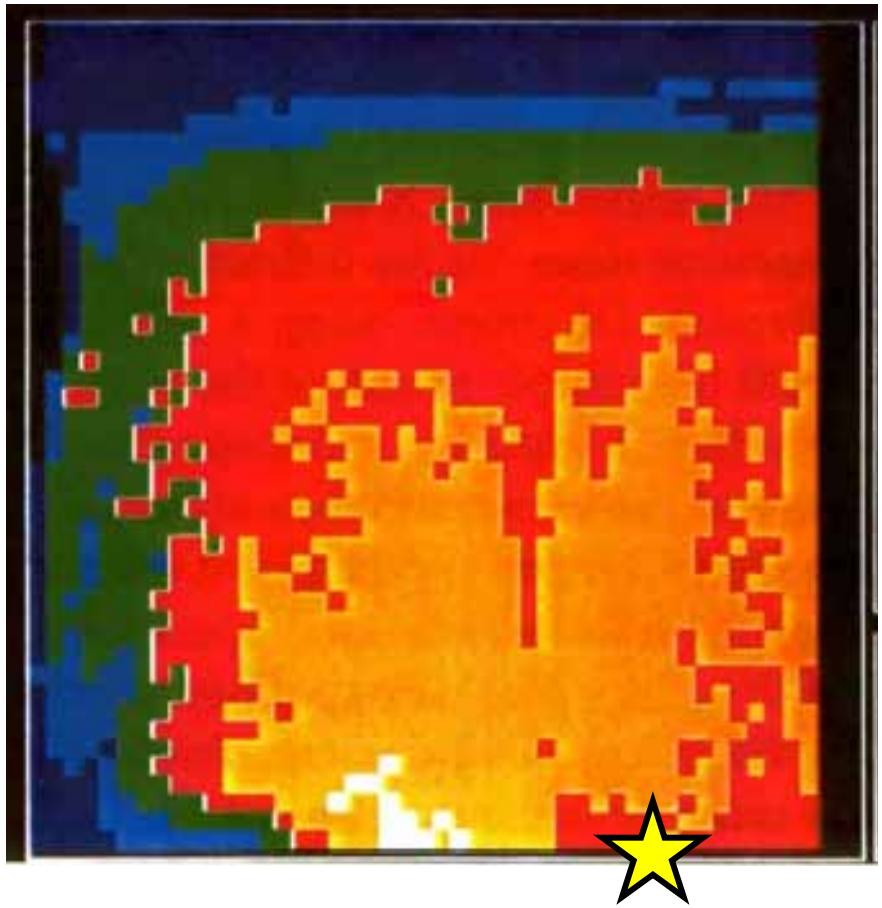
Molecular Orientation in Injection Moldings

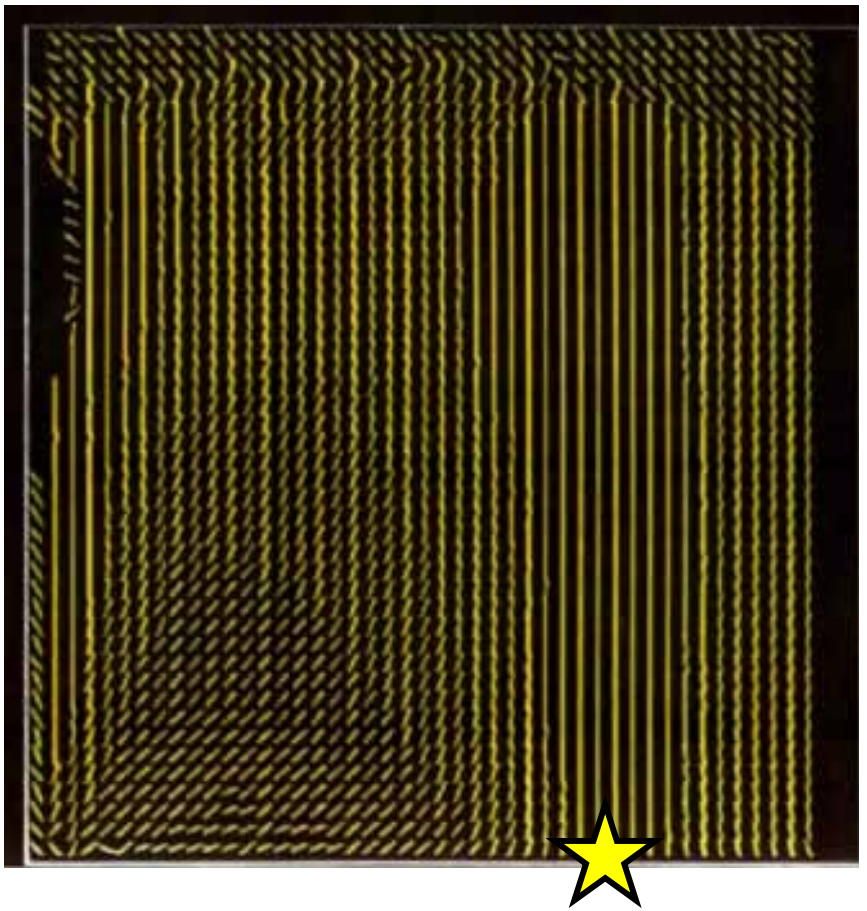
Measuring the degree and inclination of preferential molecular orientation in a piece of injection molded plastic (e.g. hip replacement joints). ~ 1500 WAXS patterns

Marks the injection point

Orientation parameters: $0 < P_2 < 0.3$

Axis of orientation





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