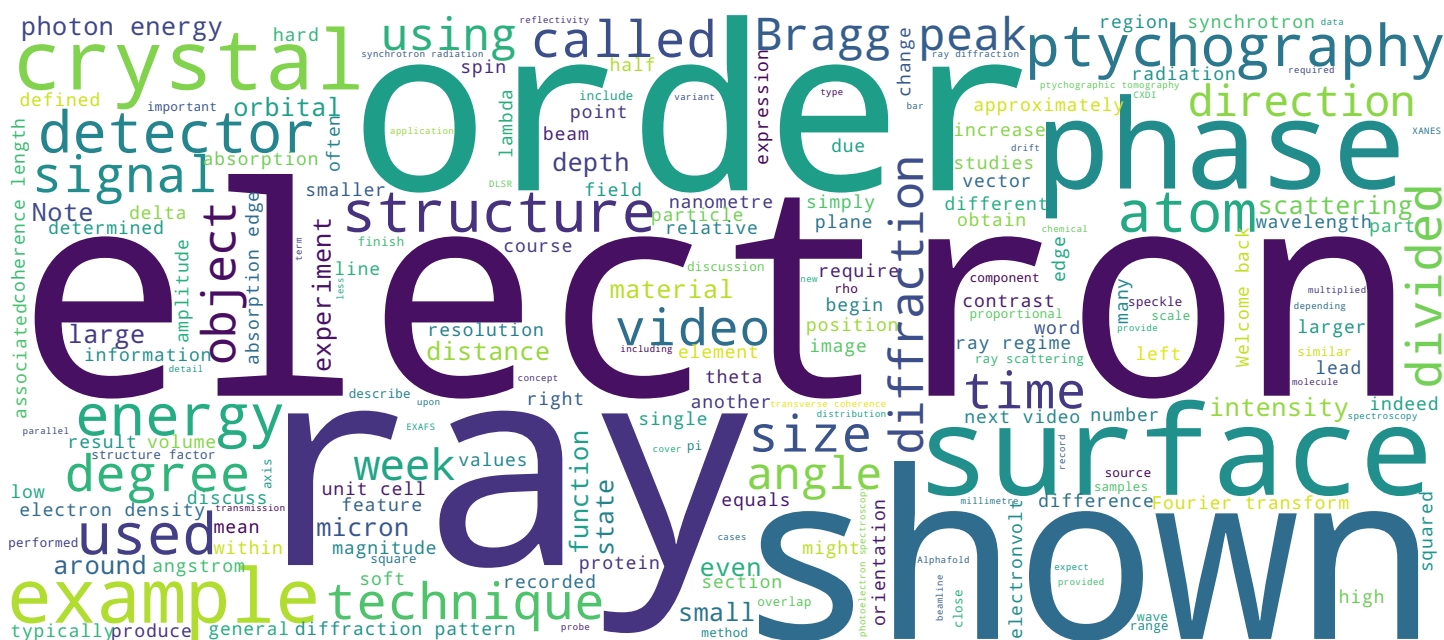


Synchrotrons and x-ray free-electron lasers

Techniques and applications

Prof. Philip Willmott



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Video



Contents and objectives of this video



- Basic description
- Parameters
 - Wavelength
 - Depth of field
 - Size of illumination spot
 - Amount of overlap

Welcome back to the sixth and final week of this introductory course on synchrotron radiation, and this, the last section, consisting of four videos. These will concentrate on ptychography and ptychographic tomography, higher dimensional imaging methods based on ptychography and small-angle X-ray scattering, and finally, X-ray photon correlation spectroscopy. We begin in this video with a basic description of ptychography and how it differs from more conventional coherent X-ray diffractive imaging. We discuss some experimental considerations, including the wavelength typically used, the depth of field achieved, the size of illumination, and the degree of overlap between successive images during an experiment. What I mean by this last parameter will become immediately clear.

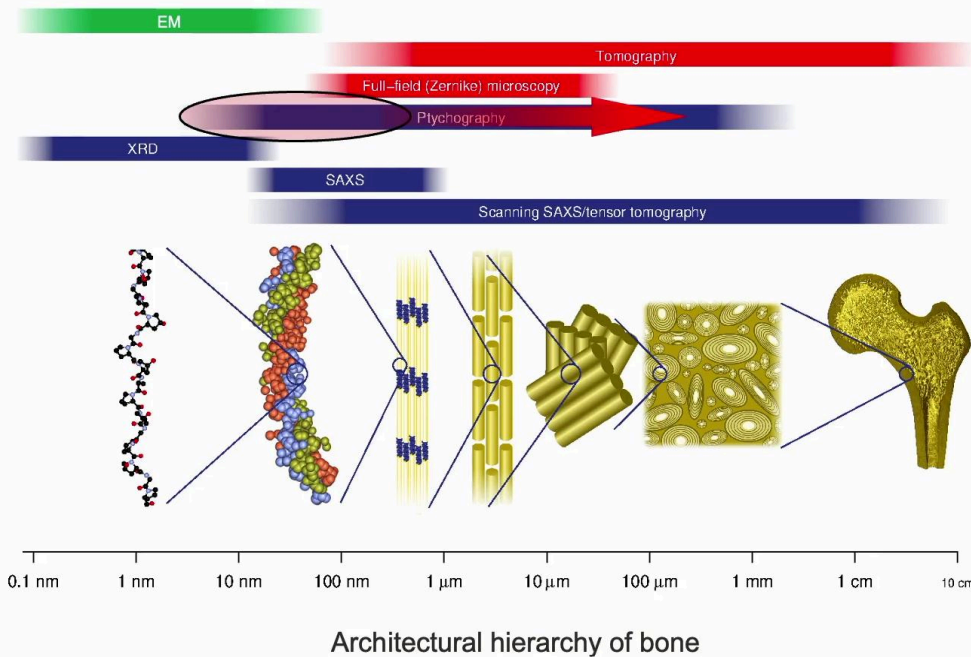
Notes

Summary



0m 04s

Role of ptychography



- Bridge resolution gap between **full-field tomographies** and **XRD/electron microscopy**
- Scanning aspect allows high resolution down to few nm on extended samples with macroscopic dimensions limited only by IT considerations (and absorption lengths, not normally a problem for HXR)
- Spatial resolution determined by
 - Largest scattering angle
 - Stability of sample movements
 - NOT by size of illumination or step size

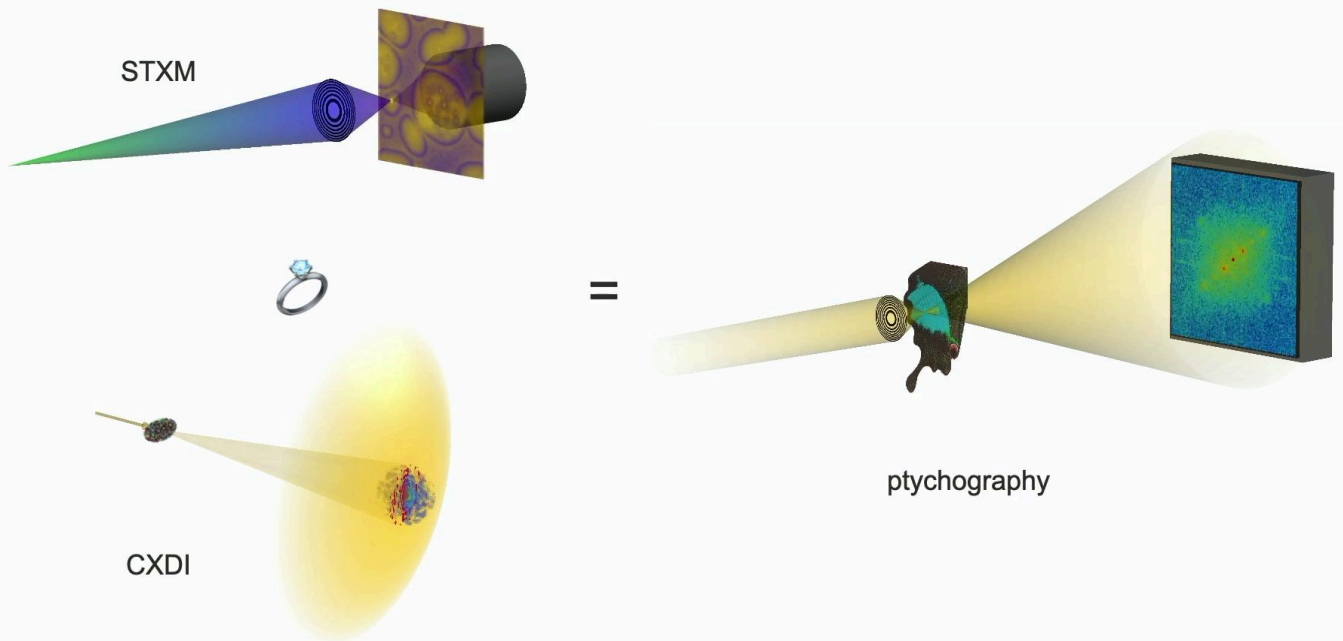
Ptychography is a burgeoning X-ray technique, although it was originally conceived for electron microscopy. It bridges nicely the resolution gap between full-field tomographies, which run out of steam below around 100 nanometres, and X-ray diffraction and electron microscopies that access nanometre and still smaller dimensions. Now, importantly, the scanning nature of ptychography also means that it can probe macroscopically-sized objects, the upper limit, in principle, being limited only by computing power and data-storage considerations. The spatial resolution of ptychography is determined primarily by the largest accessible scattering angle before the signal-to-noise ratio becomes too small, but also by the stability of sample movements. Note that it's not determined by the size of the illumination spot on the sample or by the step sizes in the scans.

Notes

Summary



The perfect marriage



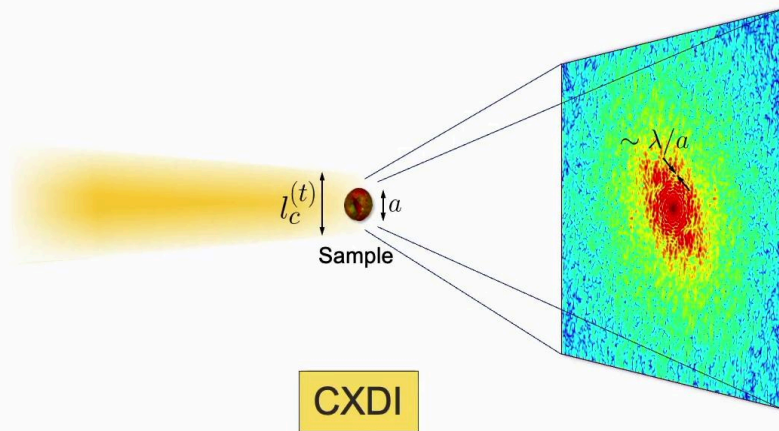
One can think of ptychography as being a marriage of scanning transmission X-ray microscopy, and coherent X-ray diffractive imaging. The experimental details differ subtly, however, from both. The focal spot size in ptychography is, at around a micron or thereabouts, much larger than in STXM, which attempts to achieve spot sizes of just a few nanometres to a few tens of nanometres. Now, as an aside, most STXM experiments are performed in the soft X-ray regime, most notably and famously around the water window between about 250 and 530 electronvolts, while the majority of ptychography experiments actually use hard X-rays.

Notes

Summary



CXDI vs ptychography



- Sample flooded with coherent radiation:

$$l_c^{(t)} > a$$

- Speckle and oversampling determined by sample size a
- Redundancy provided by
 - Positive electron density
 - Approximate maximum/minimum electron densities
 - Overall sample size (if known)

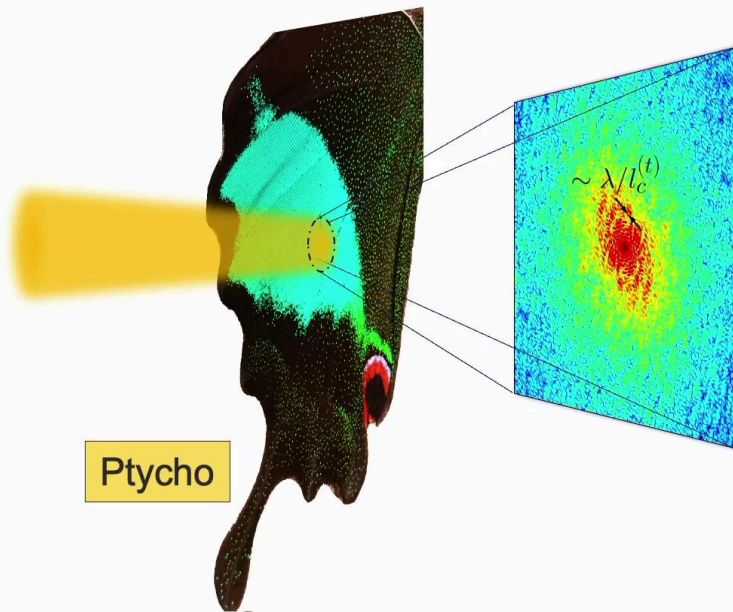
So ptychography can be thought of as scanning CXDI. However, in CXDI, it's essential that the sample is flooded with coherent radiation. In other words, the transverse coherence lengths need to be larger than the sample size a . Thus, in CXDI, the speckle and degree of oversampling are determined by a . Redundancy, which aids in resolving the phase problem, is achieved through simple assumptions, such as the need for the electron density to be positive, the expected maximum and values for that electron density, and in some cases, the approximately known overall shape and size.

Notes

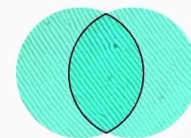
Summary



CXDI vs ptychography



- Extended sample
 - Larger than $l_c^{(t)}$
 - Part of sample illuminated with coherent radiation
- Speckle and oversampling determined by illumination size $l_c^{(t)}$
- Raster sample with step sizes $< l_c^{(t)}$
 - Marriage of CXDI and STXM
- Redundancy (real-space constraint) provided by
 - Overlap between adjacent recordings – solutions must be the same in real-space



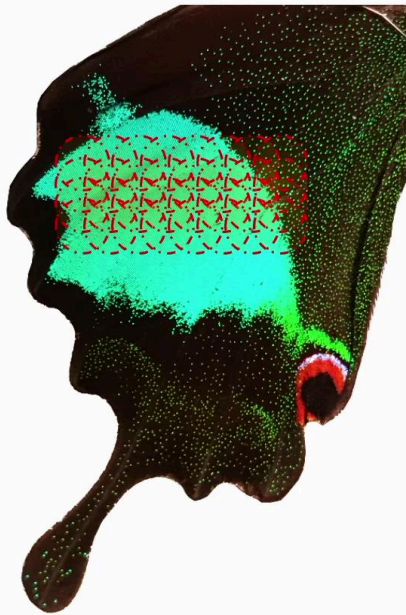
Now, in ptychography, the sample is significantly larger than the transverse coherence lengths, and thus, only a part of the sample defined by these coherence lengths will be illuminated by coherent radiation. It therefore follows that in ptychography, the speckle and oversampling are determined by the coherent illumination size. The sample is scanned or rastered using step sizes that are smaller than the transverse coherence lengths. The redundancy, in other words, the extra information exploited to help solve the phase problem derives from the overlap between the two illuminated areas. Logically, any real-space solution for one of the speckle patterns must be identical in the overlap region to the solution of its neighbour.

Notes

Summary



Experimental considerations in ptychography



- HXR: $\sim 0.5 - 2 \text{ \AA}$
- SXR: near absorption edges e.g., magnetic materials
 - L-edges 600 – 900 eV
- Size of illumination $\sim \mu\text{m}$
- Depth of field* $T \sim 5(\Delta x)^2/\lambda$
 - Δx = desired resolution
 - $\Rightarrow T \sim 1 - 10 \mu\text{m}$ for HXR and 10-nm resn.
- Optimal areal overlap[†] between adjacent illuminations $\sim 60 - 80\%$
- Nested iteration to determine (imperfect) incident wavefront[‡]
- DLSRs: increase in coherent flux $\sim 10^3$!!

*M. Holler *et al.*, Sci. Rep. **4** 3857 (2014)

†O. Bunk *et al.*, Ultramicroscopy **108** 481 (2008)

‡P. Thibault *et al.*, Ultramicroscopy **109** 338 (2009)

Regarding the experimental conditions appropriate for ptychography, the large majority of studies are performed in the hard X-ray regime, though recently it has been demonstrated in the soft X-ray regime in studies of magnetic materials around their L-edges at a few hundred electronvolts. The size of the illumination is of the order of a micron. The depth of field, that is, the depth of the sample in the direction of illumination that remains in focus, is given by the expression five Delta squared divided by Lambda, where Delta is the desired resolution. Hence, for hard X-rays, one can expect a depth of field of a few microns for a desired resolution of 10 nanometres. This scales proportionately with the photon energy, and hence, soft X-ray ptychography studies have a more limited depth of field. What you should also note is that while the depth of field is inversely proportional to the energy, the absorption length scales with the inverse third power of the photon energy. So at some point, as one decreases the photon energy, the absorption length becomes shorter than the depth of field. As a consequence, ptychography in the soft X-ray regime is often in reflection mode. The optimum aerial overlap between adjacent illuminations is typically 60-80%.

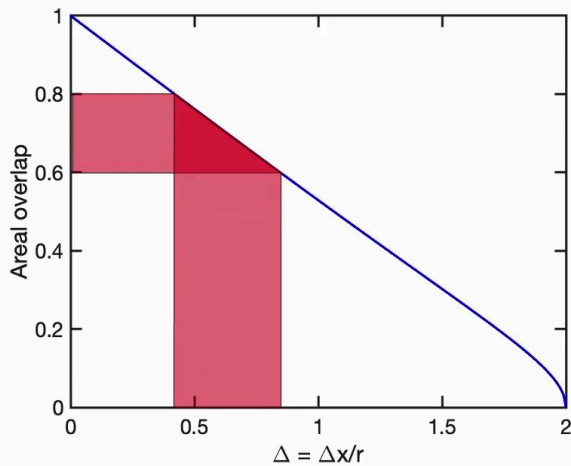
Notes

Summary

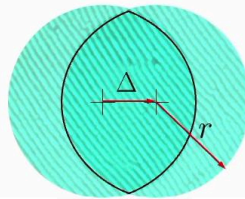


4m 36s

Experimental considerations in ptychography



$$\text{Overlap} = \frac{2}{\pi} \arccos\left(\frac{\Delta}{2}\right) - \frac{\Delta}{2\pi} \sqrt{1 - (\Delta/2)^2}$$



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This optimum aerial overlap between adjacent illuminations of 60-80% leads to linear translations delta of around 50-75 % of the illumination radius, according to the equation provided here, whereby delta is the ratio of the translation to the coherent illumination radius. Just to stress an important point, don't confuse delta, the translation of the illumination length, with delta X, the resolution obtained through inverse Fourier transforming the speckle pattern.

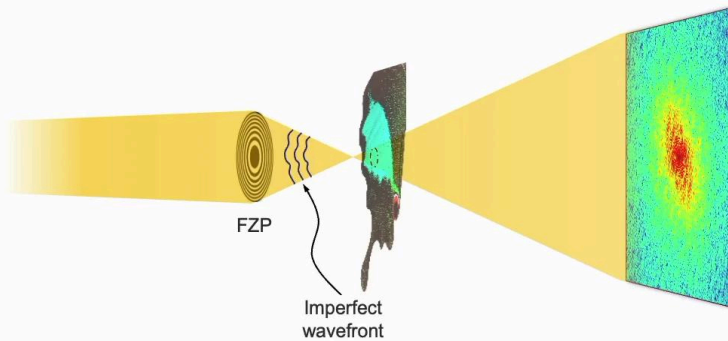
Notes

Summary



6m 09s

Experimental considerations in ptychography



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Lastly, because it's impossible to avoid entirely X-ray optical components such as monochromators and focusing elements such as Fresnel zone plates, the wavefront will deviate by an unknown amount from a perfect plane wave or indeed, a perfectly converging or diverging spherical wave. Instead of simply accepting this unknown, modern algorithms used to solve the phase problem also include a nested iteration to describe the form of the wavefront. Note also that DLSRs with their increased coherent fraction and the latest developments in undulator technology, also increasing the absolute flux by another order of magnitude, can lead to an increase in the number of coherent photons on the sample by over three orders of magnitude. And on top of this, the use of multilayered monochromators with their 100-fold higher bandwidth, and possibly also the use of reflective optics such as KB mirrors instead of Fresnel zone plates, will potentially lead to fluxes on the sample a million times higher than previously possible.

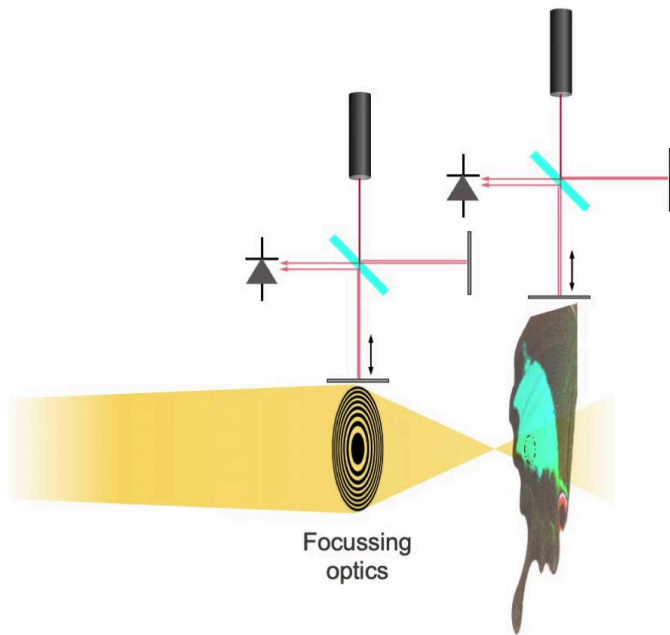
Notes

Summary



6m 49s

Sample manipulation



- Sample illumination accuracy better than desired spatial resolution
 - Optics (FZP, OSA, pinhole, etc) – fixed
 - Sample – controlled movements
 - Both x- and y-directions
- Interferometric control using lasers
- Avoid long-term drift
 - Especially problematic in cryogenically cooled samples
- In case of ptychographic tomography (PXCT, see next video) rotation control also required
 - Axis wobble

*M. Holler *et al.*, Sci. Rep. 4 3857 (2014)

It's imperative that for a successful experiment, the sample illumination accuracy be better than the desired spatial resolution. This sets upper limits on vibrations and drifts of optical components, while the sample manipulator must also have compatible specifications. This is achieved using laser interferometers. Long-term drift is especially problematic in cryogenically cooled samples. As thermal expansion coefficients are often of the order of 10 to the -6 per degree Kelvin, even a drift of 1 degree on a one-centimeter component will induce a 10-nanometer expansion or contraction. Moreover, in the case of ptychographic tomography discussed in the next video, extremely precise rotation control is also required.

Notes

Summary



8m 02s

In the next video...



Okay, in the next video, we cover ptychographic tomography and its variant laminography used for flat samples in which one characteristic dimension is much smaller than the other two.

Notes

Summary



8m 56s