The use of DIFFRACTION techniques at large scale facilities

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Outline

• Diffraction basics

• X-rays
  Micro-diffraction on a solar cell
  A high-resolution study of a membrane protein
  Time-resolved diffraction on a catalyst at work

• Neutrons
  Hydrogen bonding in cellulose
  Water and DNA fibres
  Magnetism on Mars
Diffraction basics

Radiation Type | Wavelength (m) | Approximate Scale of Wavelength
---|---|---
Radio | $10^3$ | Buildings
Microwave | $10^{-2}$ | Humans
Infrared | $10^{-5}$ | Butterflies
Visible | $0.5 \times 10^{-6}$ | Needle Point
Ultraviolet | $10^{-8}$ | Protozoans
X-ray | $10^{-10}$ | Molecules
Gamma ray | $10^{-12}$ | Atoms

Additional Note: Wavelengths and scales are approximate and vary depending on the specific type of radiation.
1 Ångstrøm = 0.00000000001 m
X-rays or neutrons

Wavelength

~1 Ångström
Fourier transformation

Jean Baptiste Joseph Fourier 1768 – 1830
MabLab IV Synchrotron

ESS (European Spallation Source)
High beam intensity can be achieved at 3\textsuperscript{rd} generation synchrotron sources (e.g. ESRF)
X-ray diffraction at synchrotrons

Advantages compared to in-house X-rays:

**Much higher intensity**
-> Crystals that diffracts weakly can be examined, e.g. macromolecules.
-> It is possible to sacrifice a lot of X-ray photons in the experimental setup in order to do new types of experiments!

**Tunable wavelength**
-> Can help us determine structures using anomalous scattering.
Exciting experiments at synchrotrons

Time resolved studies
Micro-diffraction
Difficult crystals
Three examples using X-rays

• Micro-diffraction on a solar cell
• A high-resolution membrane protein
• Time-resolved diffraction on a catalyst at work
Micro-diffraction

• The sample (crystal) can be micron (or even nanometer) sized.
• The inhomogeneity of larger samples can be examined.
• Samples that are only partly crystalline can be examined.
Micro diffraction
Microfocus and low divergence are achieved through an optical mirror and collimators.

Undulator
Si(111) double monochromator
Rh covered, condensing (ellipsoidal) mirror,
Capillary collimators further reduce beam size
Microdiffraction setup

The hutch contains instrumentation to:

- maximize the quality and quantity of data obtained from radiation sensitive microcrystals (whose lifetimes are limited by their small size)

- allow rapid changing of crystals to screen crystal quality.
A CCD area detector speeds data collection by intercepting a wide angle of diffracted X-rays.

A scintillation counter would be too slow, even for our small unit cell.
Cryostream

A cryostream slows the progression of radiation damage in the crystal.
Motorized arc
Scintillator and Beamstop
Microgoniometer

Mechanics allow 1\(\mu\)m accuracy in centering (minimal sphere of confusion).

To center the micro-crystal, a motorized finger translates (slides) the sample holder along a magnetic base.
High powered microscope.

A high powered microscope facilitates crystal “centering” by providing a clear view of the microscopic crystal.
10 micron
Organic solar cell.
Combined X-ray diffraction and fluorescence study.

High-resolution membrane protein
Aquaporin
membrane protein structure

Time resolved study
Zeolite catalysis of methanol to alkenes
Figure 1. a) Time- and space-resolved HEXRD data for the MTO process at 350°C and a flow rate of 30 ml min⁻¹. Mass spectrometry activity data are shown as line plots. b) The first 15 minutes of the reaction in more detail.
Neutron diffraction

Neutrons interact with the atomic nuclei.

1. Hydrogen atoms are clearly visible. The role of water and hydrogen bonding in biological systems can be investigated.

2. Neutrons can detect the magnetic moments of atoms. Magnetic properties of crystals can be investigated.

3. Neutrons are non-destructive and penetrating. Good for precious samples and for in-situ experiments.
Three research examples using neutrons

• Hydrogen bonding in cellulose
• Water and DNA fibres
• Magnetism on Mars
Hydrogen bonding in cellulose

Research by H. Chanzy (CERMAV-CNRS, Grenoble), Y. Nishiyama (Univ. Tokyo), P. Langan (Los Alamos National Laboratory), T. Forsyth (ILL)
Water and DNA fibres

Research by T. Forsyth (ILL/ Keele University), M. Shotton, L. Pope and W. Fuller (Keele University), P. Langan (Los Alamos National Laboratory)
How Mars lost its magnetism
Pyrrhotite
How Mars lost its magnetism
High pressure studies
X-rays vs neutrons

• X-rays are plentiful, neutrons are scarce.
• Neutron requires much larger crystals millimeters vs micrometers.
Neutrons offer unique possibilities for:
• Detection of H atoms.
• Magnetic properties.
• Precious samples
• In-situ studies.
The End

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