Lab scale X-ray facilities

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Overview

- Interaction of x-rays with matter
- Scattering techniques
- Spectroscopy
- Imaging
- Combination of techniques
- Conclusions

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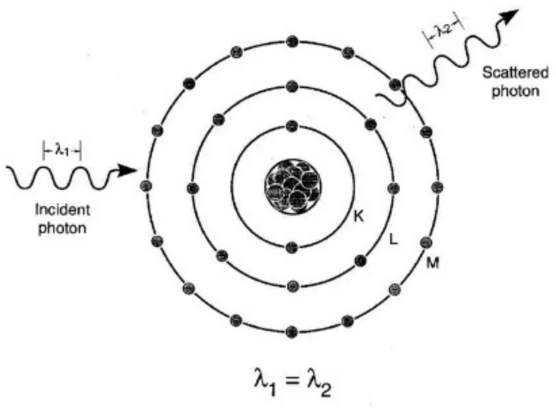
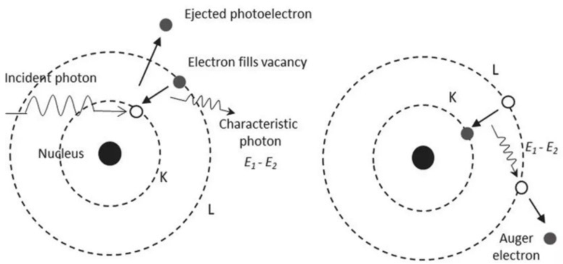
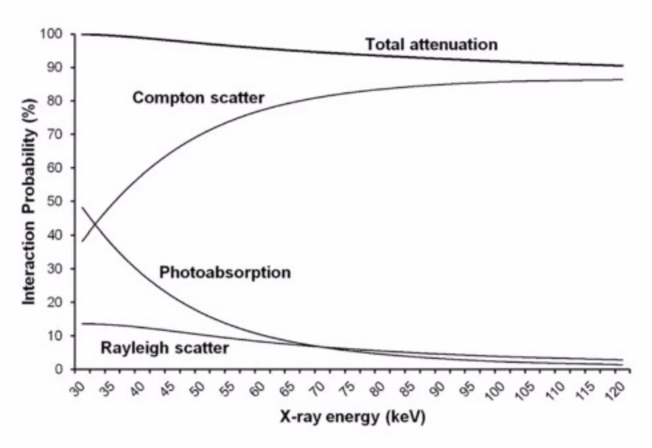
Interaction of x-rays with matter

Simplified view – not exhaustive







ELASTIC SCATTERING

- Energy of the scattered photon is the same as the incoming electron
- Rayleigh scatter

INELASTIC SCATTERING

- Energy of the scattered photon is different from the incoming photon
- Compton scattering and photoelectric effect are examples of inelastic scattering

X-RAY ATTENUATION

- X-ray are absorbed by an object because of the previously mentioned interactions

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X-ray scattering techniques

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- X-ray Diffraction (XRD)
  - Single Crystal XRD
  - Powder XRD
    - Residual Stress
    - Texture
    - High Resolution XRD

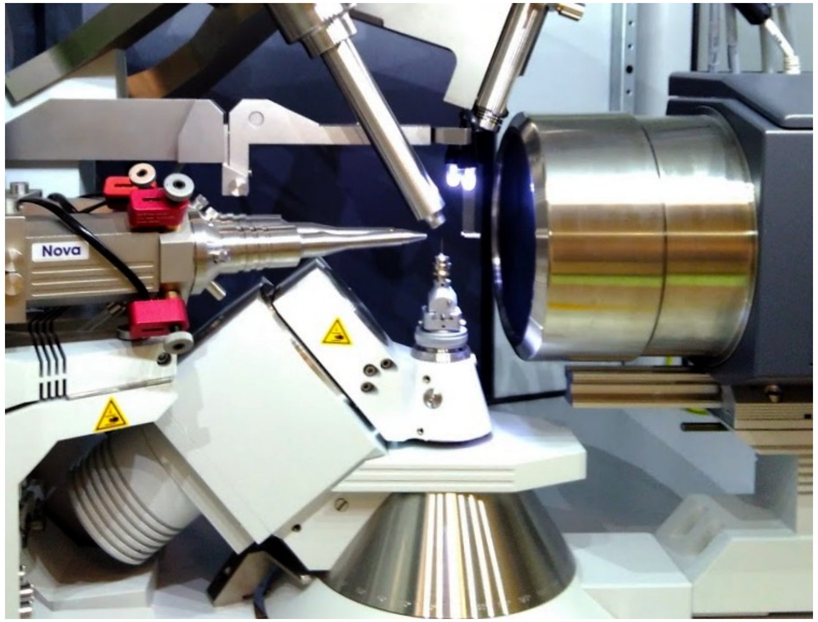
→ Require a crystalline (or polycrystalline sample)

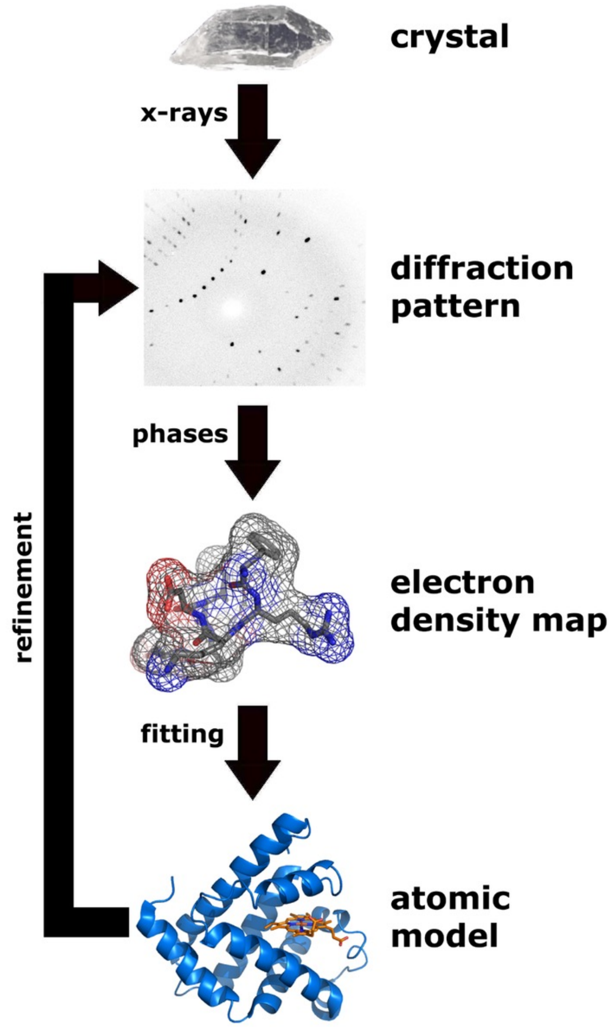
- Small Angle X-ray Scattering (SAXS)
- X-ray Reflectivity (XRR)
- Total scattering

→ All samples types

Single Crystal XRD

- Techniques to solve the structure of unknown compounds
- Require “large” single crystals
  (for lab scale, in the order of 100 – 500 µm)
- It is widely used in various fields of research, including chemistry, physics, materials science, and biology



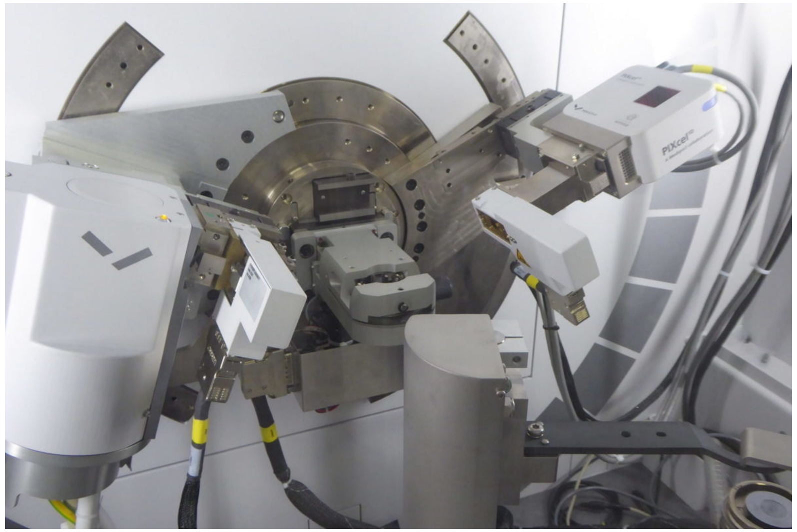


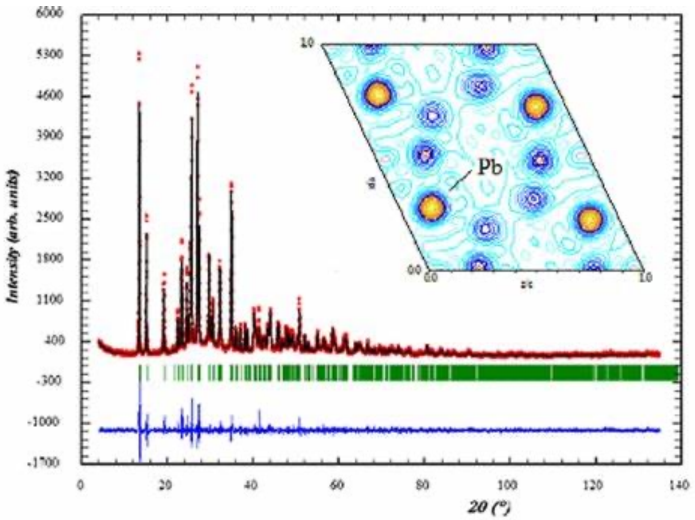
Powder X-ray Diffraction

- Technique mainly used to find out
  - What crystalline phases are present in my sample (Phase ID)
  - How much of each phase (Phase quantification)
  - Can be also used to solve structure of unknown compounds
  - Determine crystallite size and strain

- Sample is made of many “small” crystals (ideally between 1 and 10 µm)

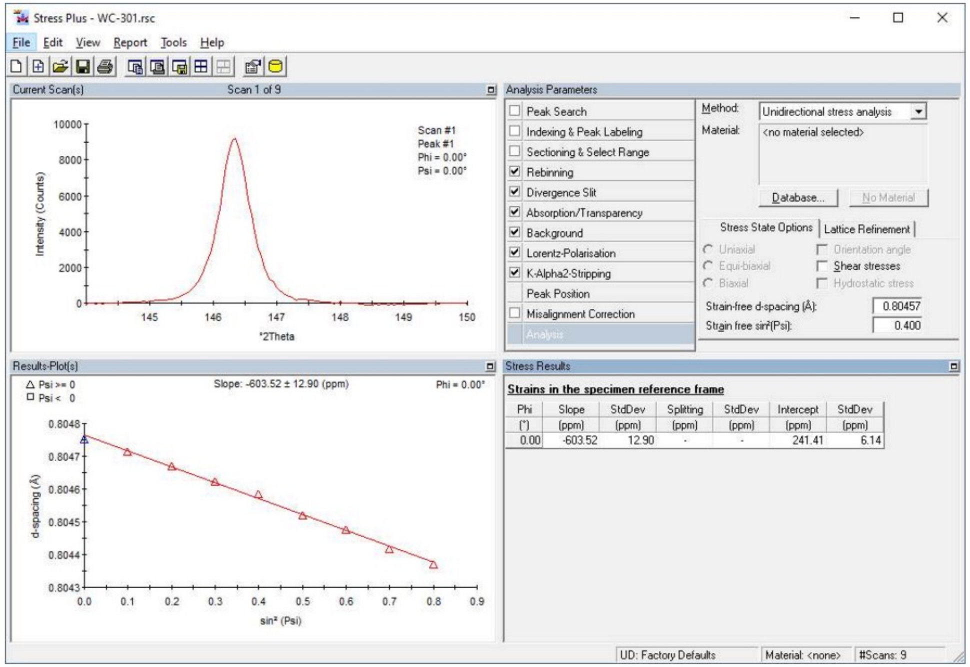
- Used in various fields of research, including mineralogy, geology, environmental science, material science, engineering, and biology and as QC tools in several industries, e.g. pharmaceutical, building materials

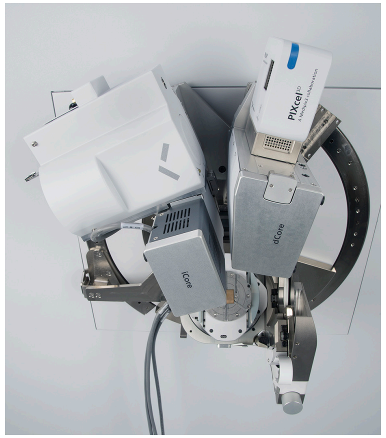




Residual Stress

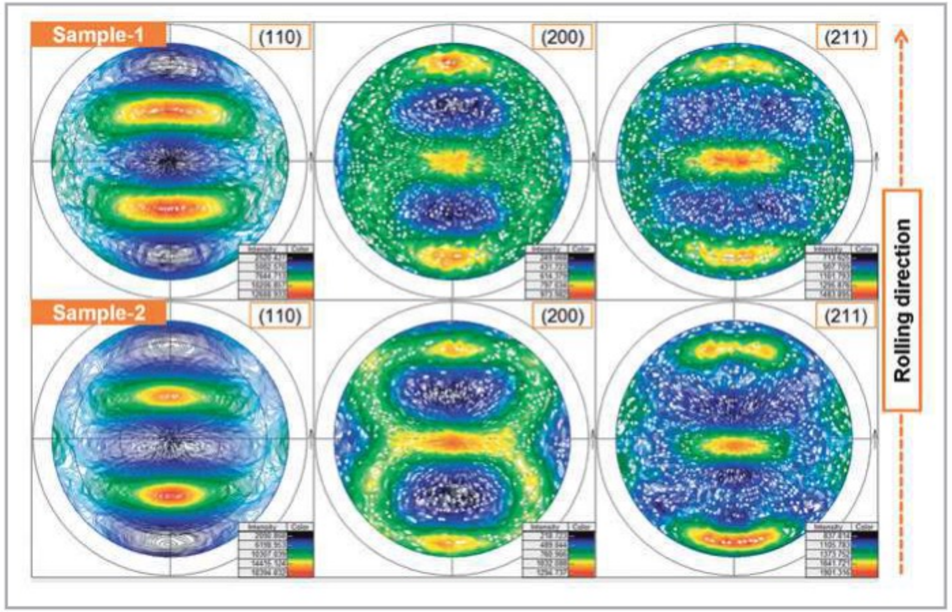
- Residual stresses: internal stresses present in the material after external loading forces have been removed
- Manufacturing processes such as machining, casting, alloying, tooling etc. can contribute to change in residual stress.
- Determining the distribution of residual stress is important because high residual stress can lead to premature catastrophic failure of a component.
- XRD can be used to accurately and non-destructively measure residual stress

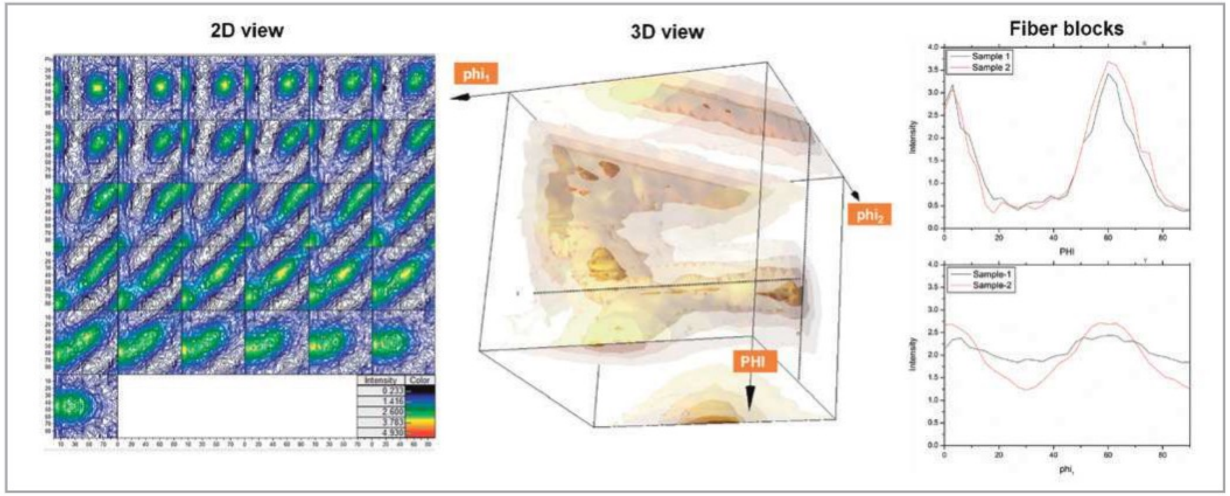




Texture

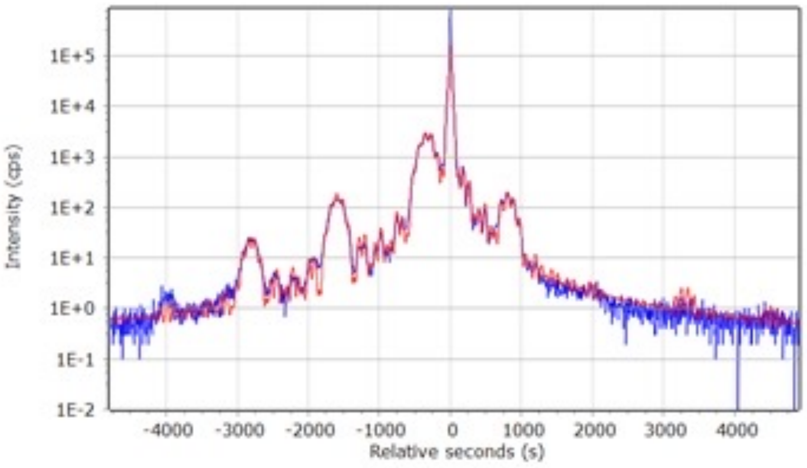
- The texture of a material refers to the relative orientation of its grains, which can influence its mechanical and physical properties
- XRD can be used to measure the diffracted intensity of X-rays by a specimen under various geometrical conditions
- The intensity of the diffracted X-rays is related to the non-homogeneous distribution of crystallites within the sample, which can be used to determine the crystallographic texture

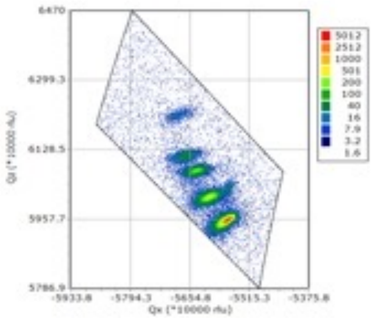


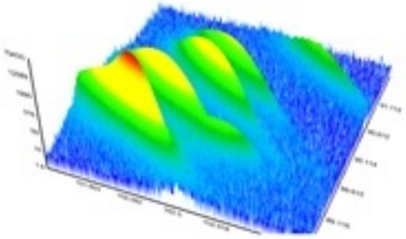


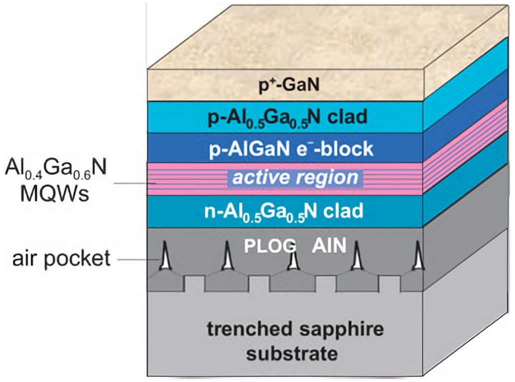
High Resolution X-Ray Diffraction

- Modern semiconductor device structures are epitaxially grown onto a substrate made from silicon, silicon-germanium, III-V and II-VI compounds.
- These films are nearly-perfect crystalline films containing a relatively low dislocation density.
- High-resolution X-ray diffraction (HRXRD) is used for the non-destructive analysis of mostly layered, nearly-perfect crystalline structured materials
- By using HRXRD, is possible to determine parameters such as layer thickness, composition, strain, relaxation and structural quality
- Rocking curves and reciprocal space maps are some of the techniques used



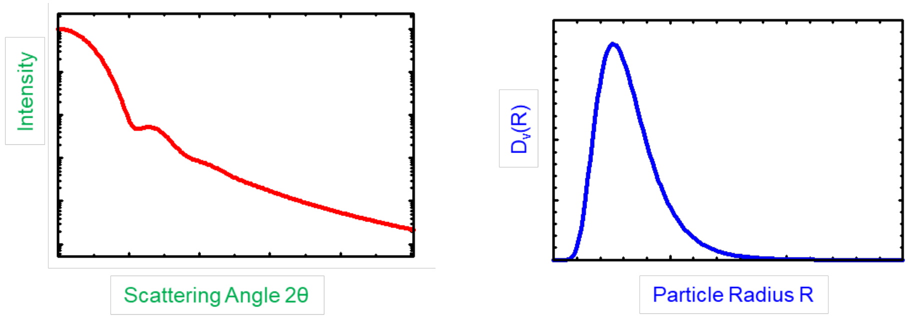


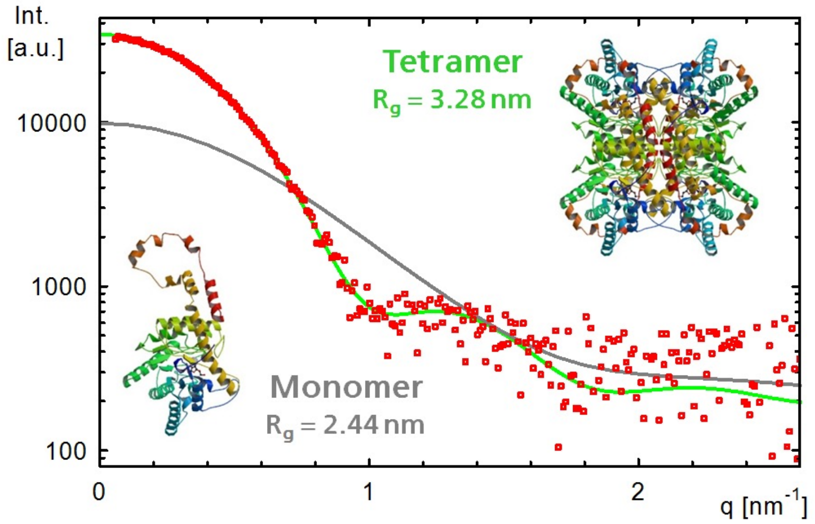




SAXS

- Small-angle X-ray scattering (SAXS) is a technique that measures the intensities of X-rays scattered at small angles (typically in the range of 0.1 deg to 5 deg)
- A SAXS signal is observed whenever a material contains structural features on the length scale of nanometers, typically in the range of 1-100 nm
- Typical samples that can be studied by SAXS include liquid nanoparticle dispersions / colloids, nano powders, biomacromolecules…
- From SAXS is possible to obtain information e.g. nanoparticle size distribution, particle shape and particle structure (e.g. core-shell)





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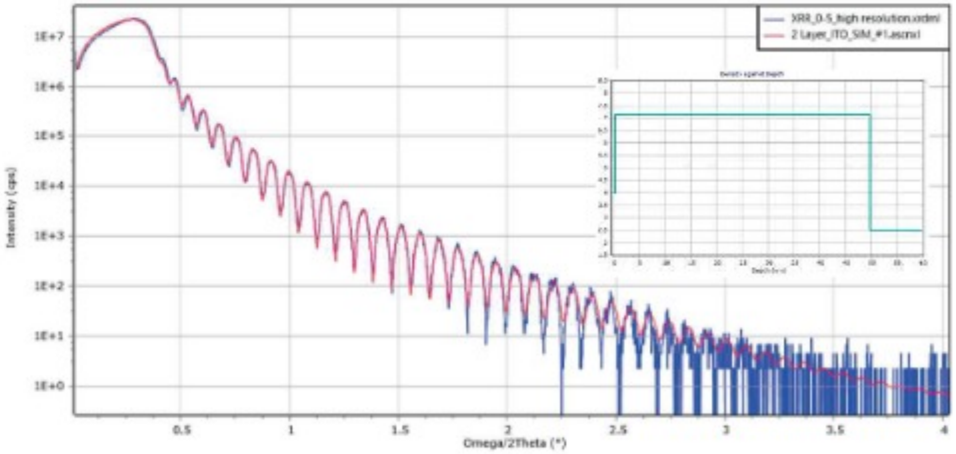
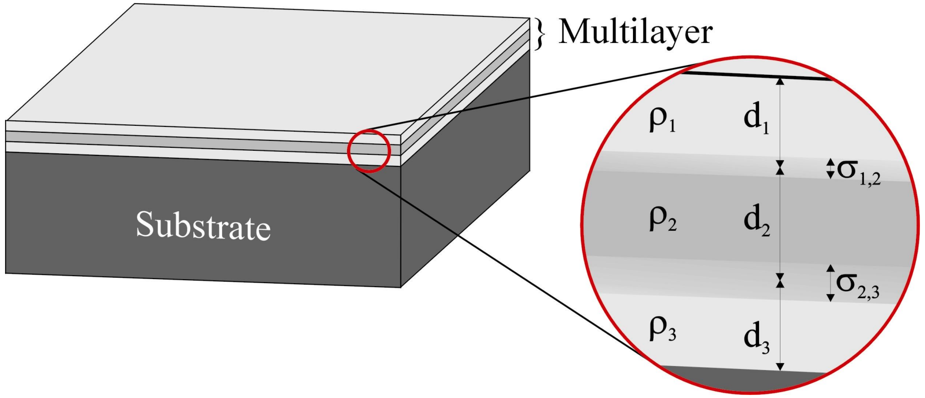
X-ray Reflectivity

- X-ray reflectometry (XRR) is a technique for investigating thin layered structures, surfaces and interfaces using the effect of total external reflection of X-rays.
- Reflectometry is used to characterize single and multi-layer structures and coatings in magnetic, semiconducting and optical materials, among others.
- With XRR is possible to obtain layer parameters such as thickness and density, interface and surface roughness.

Layers:

Layer No.	Type	Material	Density (g/cm³)	Thickness (nm)	Roughness (nm)
2	Single	In2O3 [DensityOnly]	4.011	0.1873	1.2006
1	Single	ITO [DensityOnly]	7.122	49.592	0.4039
Sub.	Substrate	SiO2 [DensityOnly]	2.5	600000.0	0.5481





Total scattering

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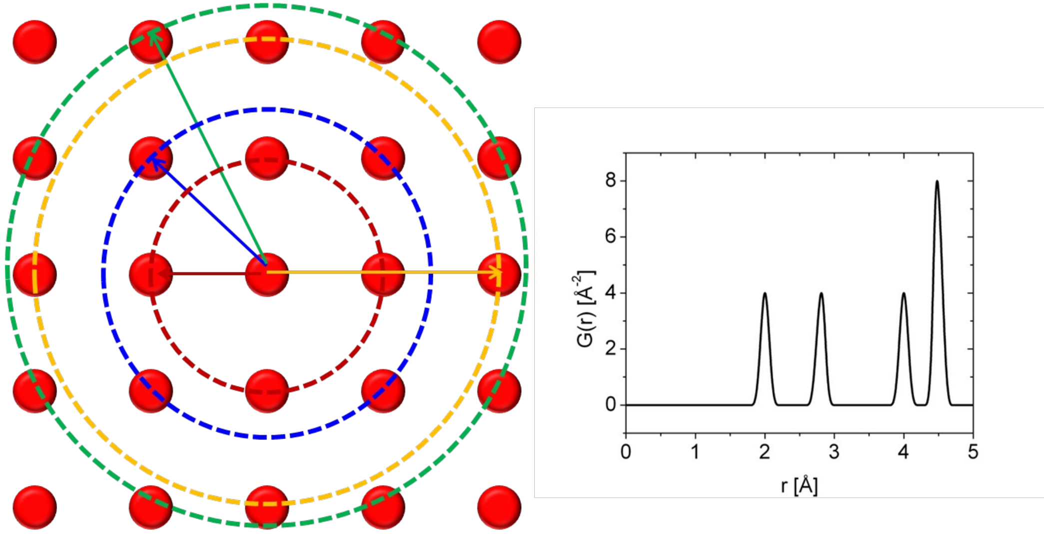
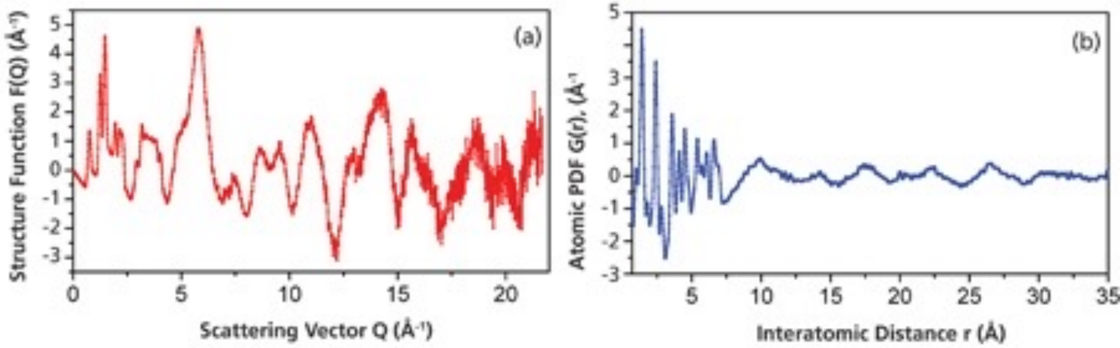
- Total scattering or pair distribution function analysis (PDF) is an analytical technique that can provide structural information from disordered materials by using the complete powder XRD patter
- From the Bragg peaks in an X-ray diffractogram the long range order of the atoms can be deduced.
- The short range order, i.e. the local atomic structure, is present in the broad, less well- defined features in the diffractogram. This local structure is described quantitatively by the atomic pair distribution function.





X-ray spectroscopy techniques

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- X-ray Fluorescence (XRF)
  - Wavelength Dispersive XRF (WD-XRF)
  - Energy Dispersive XRF (ED-XRF)
- X-ray Absorption Spectroscopy (XAS)
  - XANES (X-ray Absorption Near Edge Structure)
  - EXAFS (Extended X-ray Absorption Fine Structure)

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X-ray Fluorescence spectroscopy (XRF)

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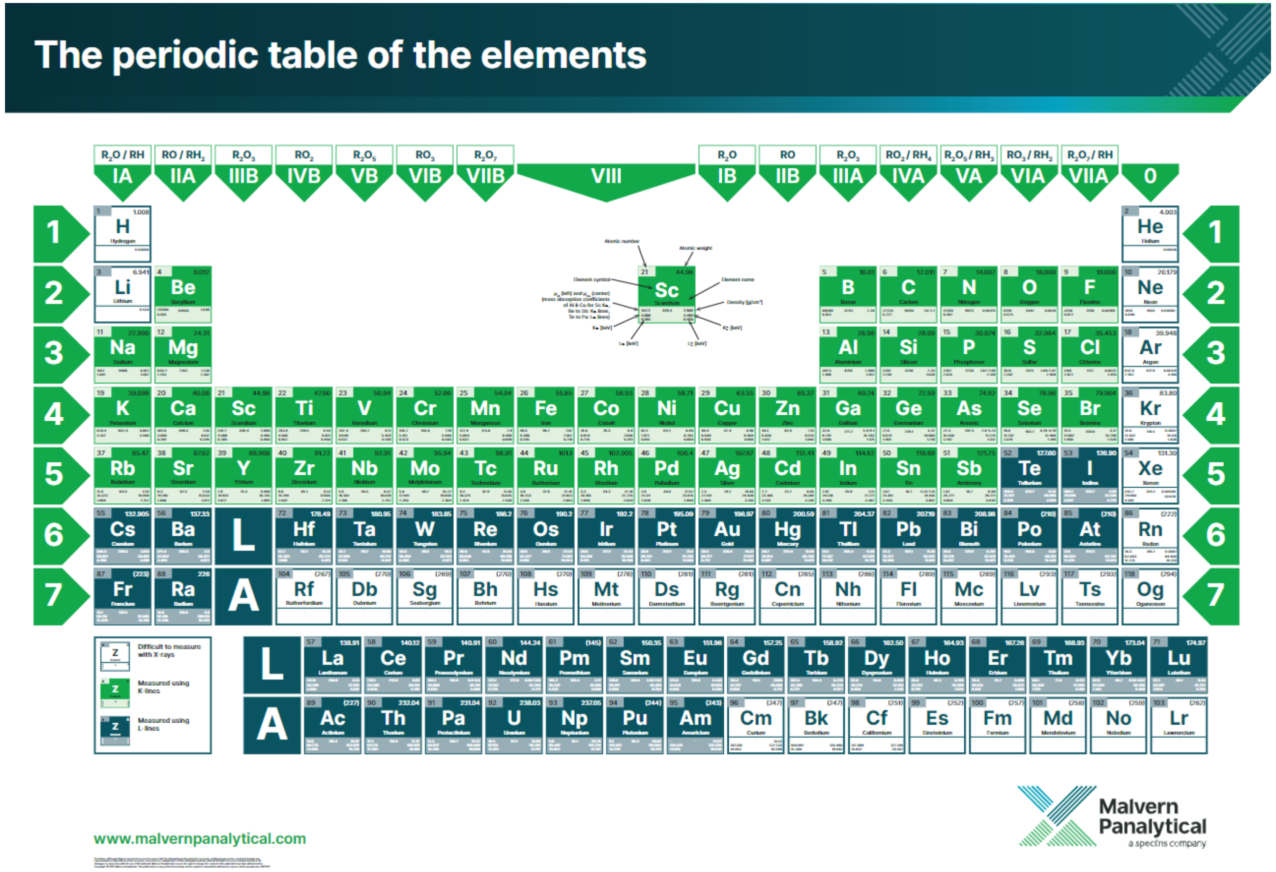
- Analytical elemental method to determine the chemical composition of a sample
- Types of materials that can be analyzed
  - Solids
  - Liquids
  - Powders
  - Filters
  - Fusion beads

- Precise: 0.1 – 0.3% relative
- Accurate: LLDs 0.1 ppm to 100%



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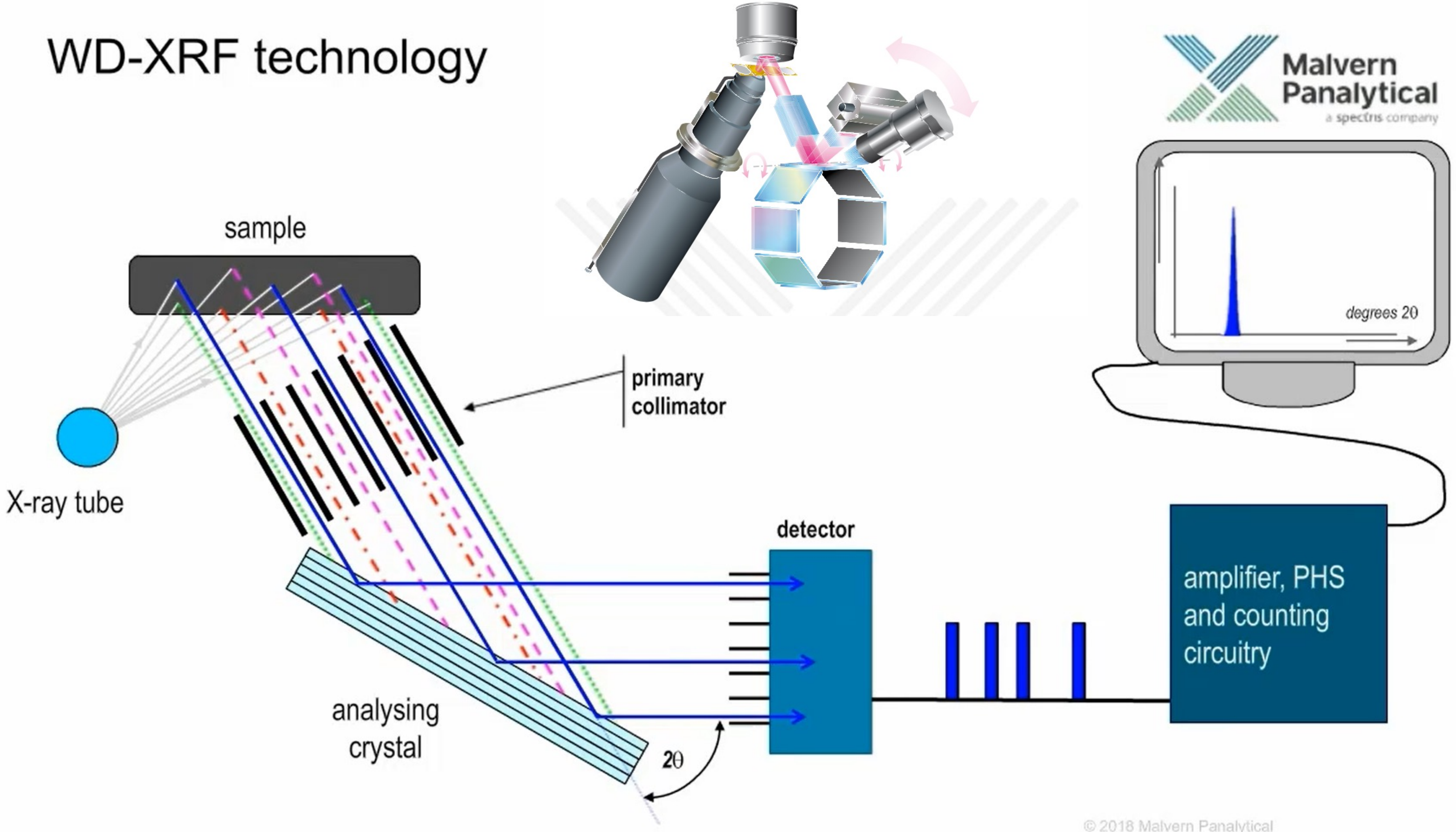
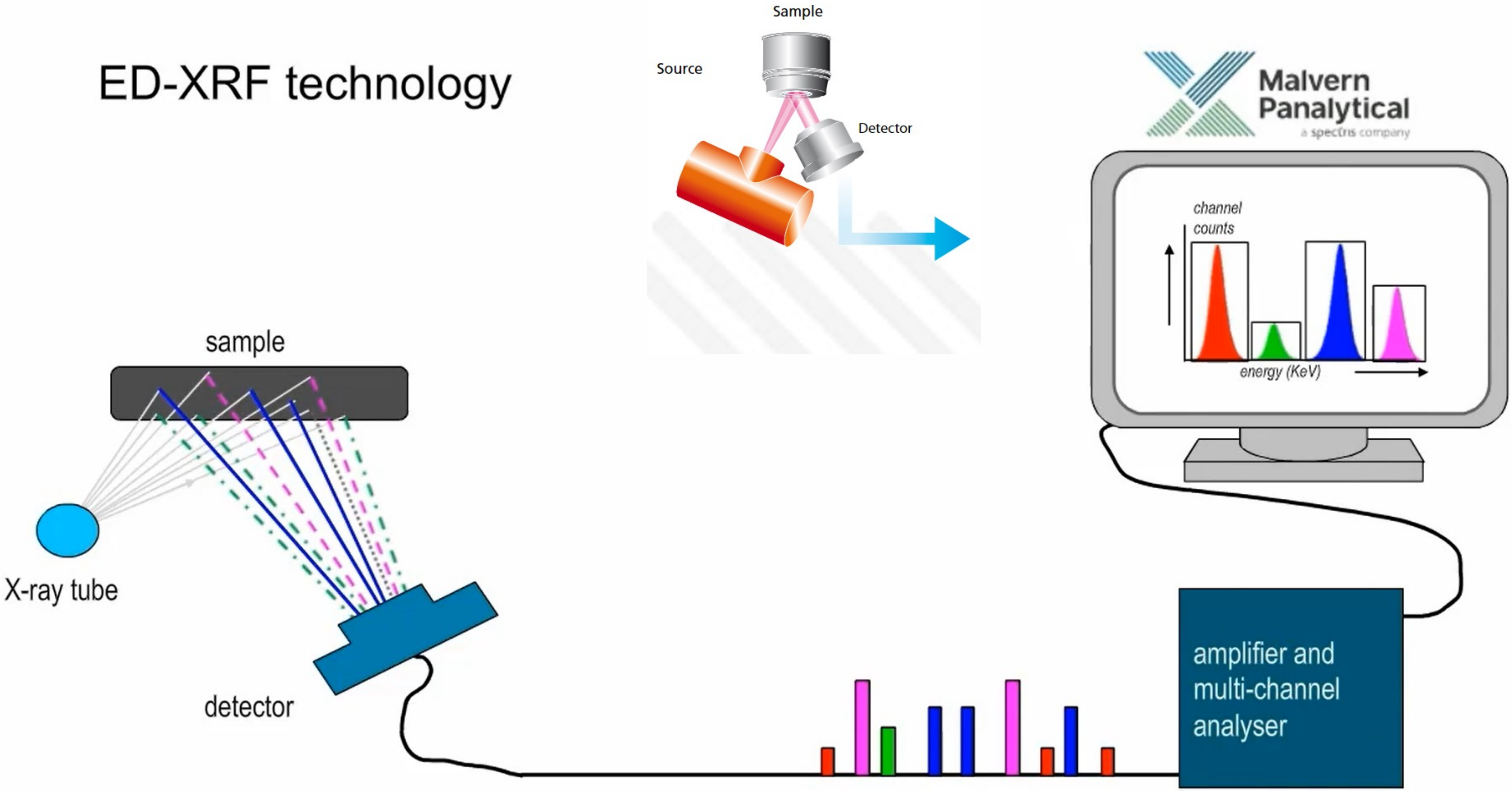
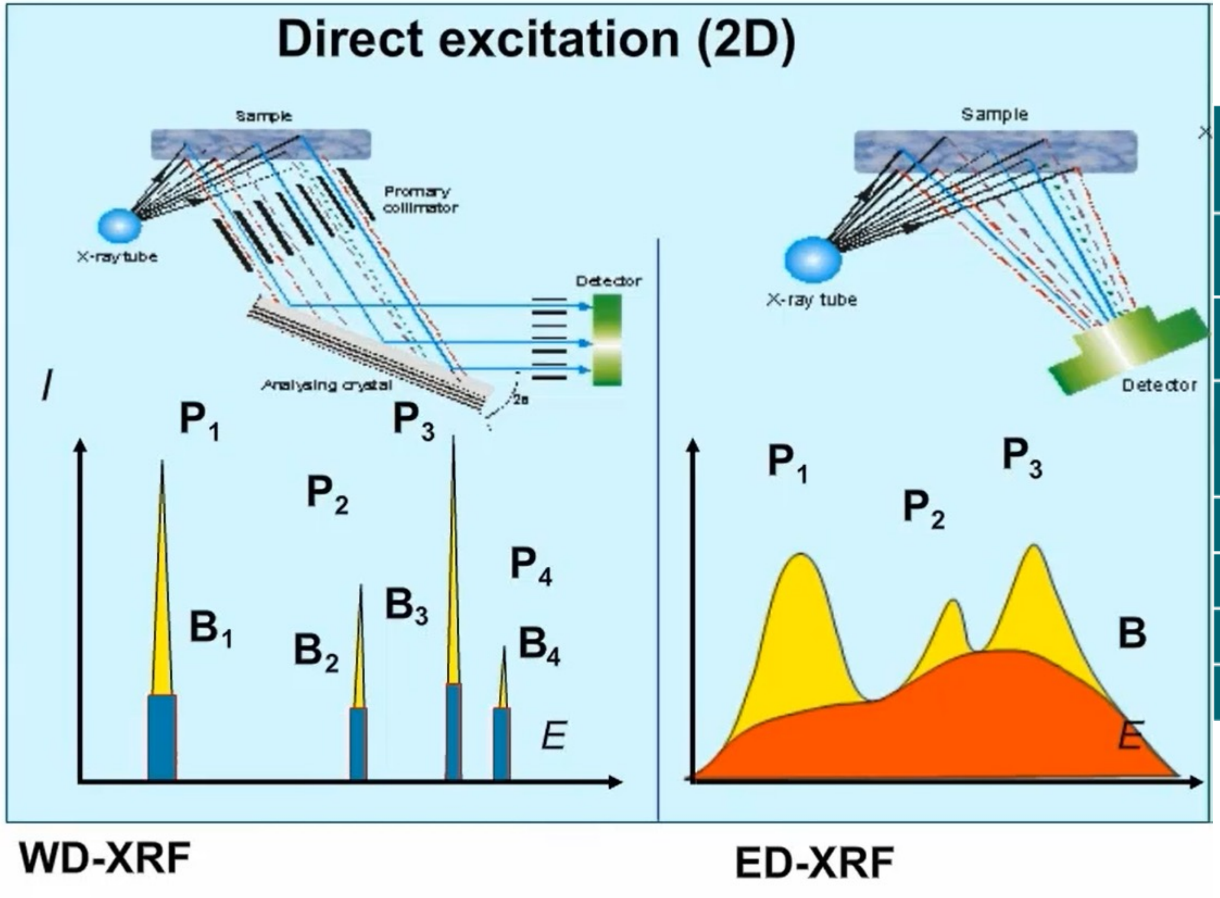
WD-XRF technology



ED-XRF technology



Comparison between WD and ED XRF



	EDXRF	WDXRF
Elemental range	Na .. U (sodium .. uranium)	Be .. U (beryllium .. uranium)
Detection limit	Less optimal for light elements Good for heavy elements	Good for Be and all heavier elements
Sensitivity	Less optimal for light elements Good for heavy elements	Reasonable for light elements Good for heavy elements
Resolution	Less optimal for light elements Good for heavy elements	Good for light elements Less optimal for heavy elements
Costs	Relatively inexpensive	Relatively expensive
Power consumption	5 .. 1000 W	200 .. 4000 W
Measurement	Simultaneous	Sequential/simultaneous
Critical moving parts	No	Crystal, goniometer

Where is XRF used?

QC and process control

Lubricating oils

Polymers & Plastics

Pharmaceuticals & Cosmetics

Mining & Minerals

Oils & Fuels

Metals

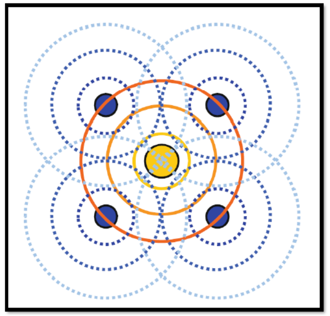
Food & Environment

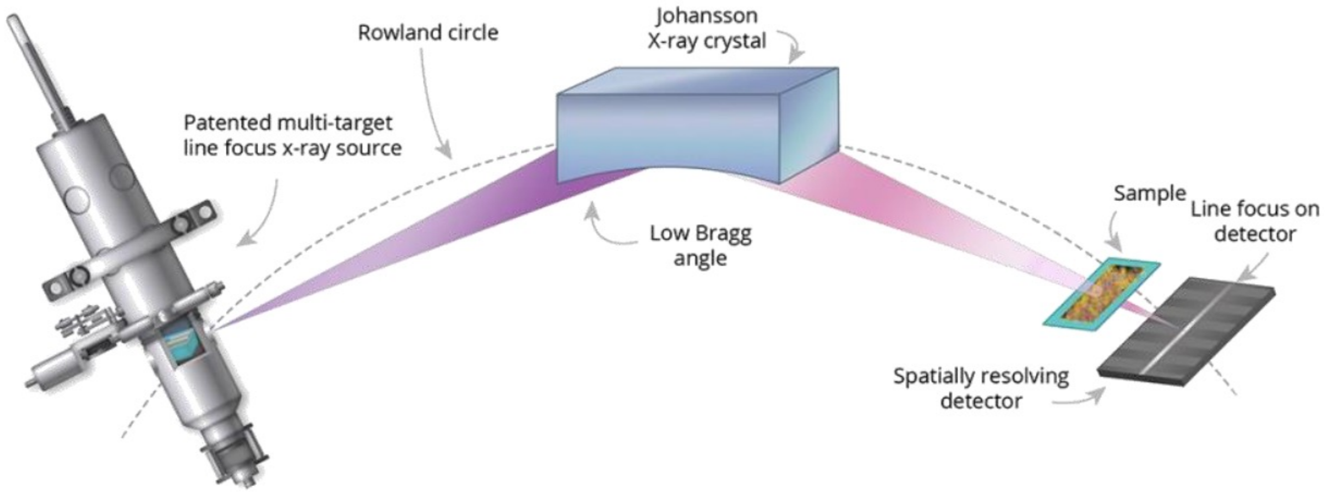
Building materials

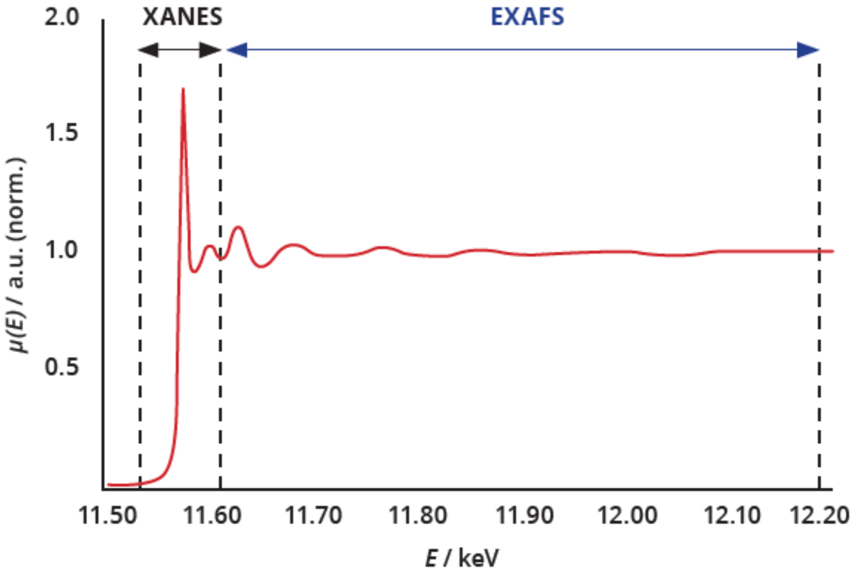
Advanced materials

X-ray Absorption Spectroscopy

- XAS is method for obtaining element-specific information with unique applications in material science, providing valuable insights into the local atomic structure and chemical state of materials







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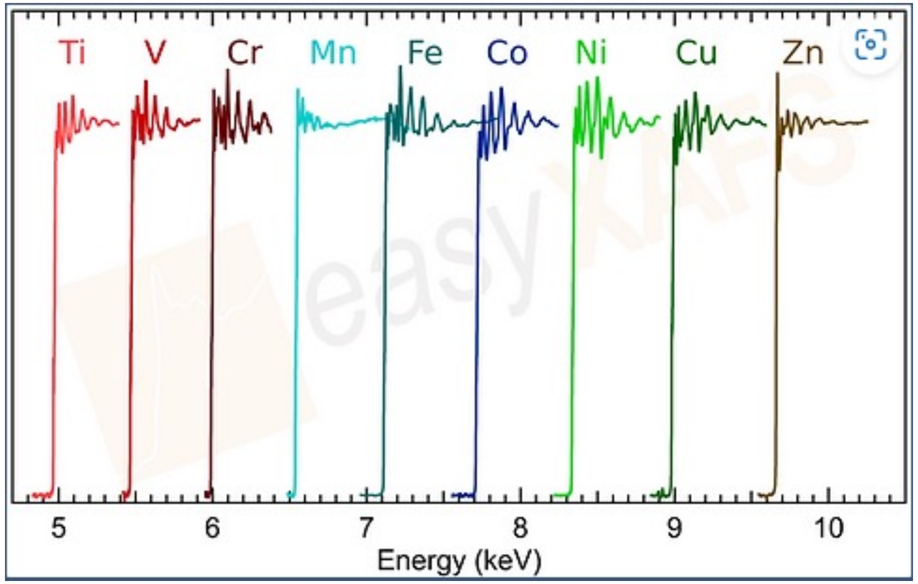
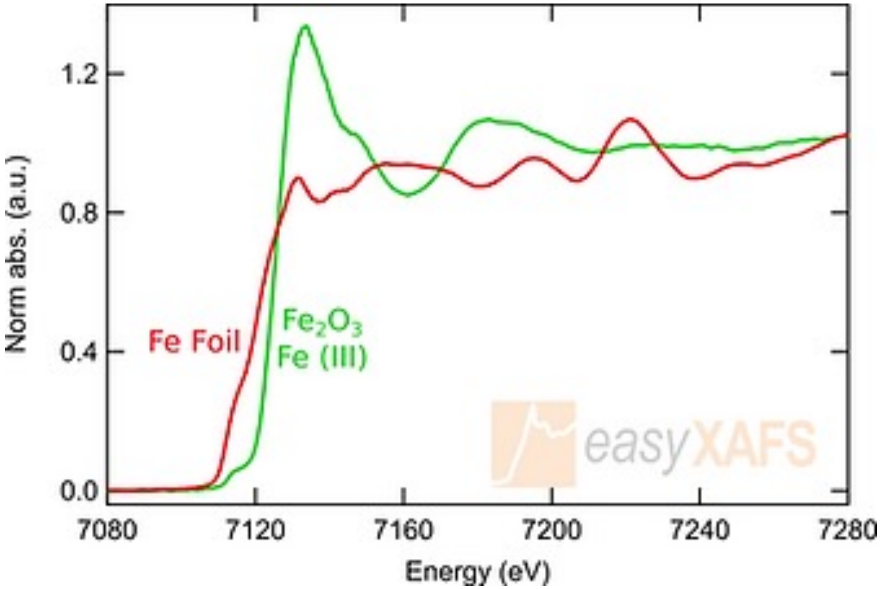
X-ray Absorption Spectroscopy

Advantages

- Element specific by tuning energy
- Sensitive to oxidation state
- No long range order needed, can be used on any sample type
  - Sensitive to local structure of the element being probed

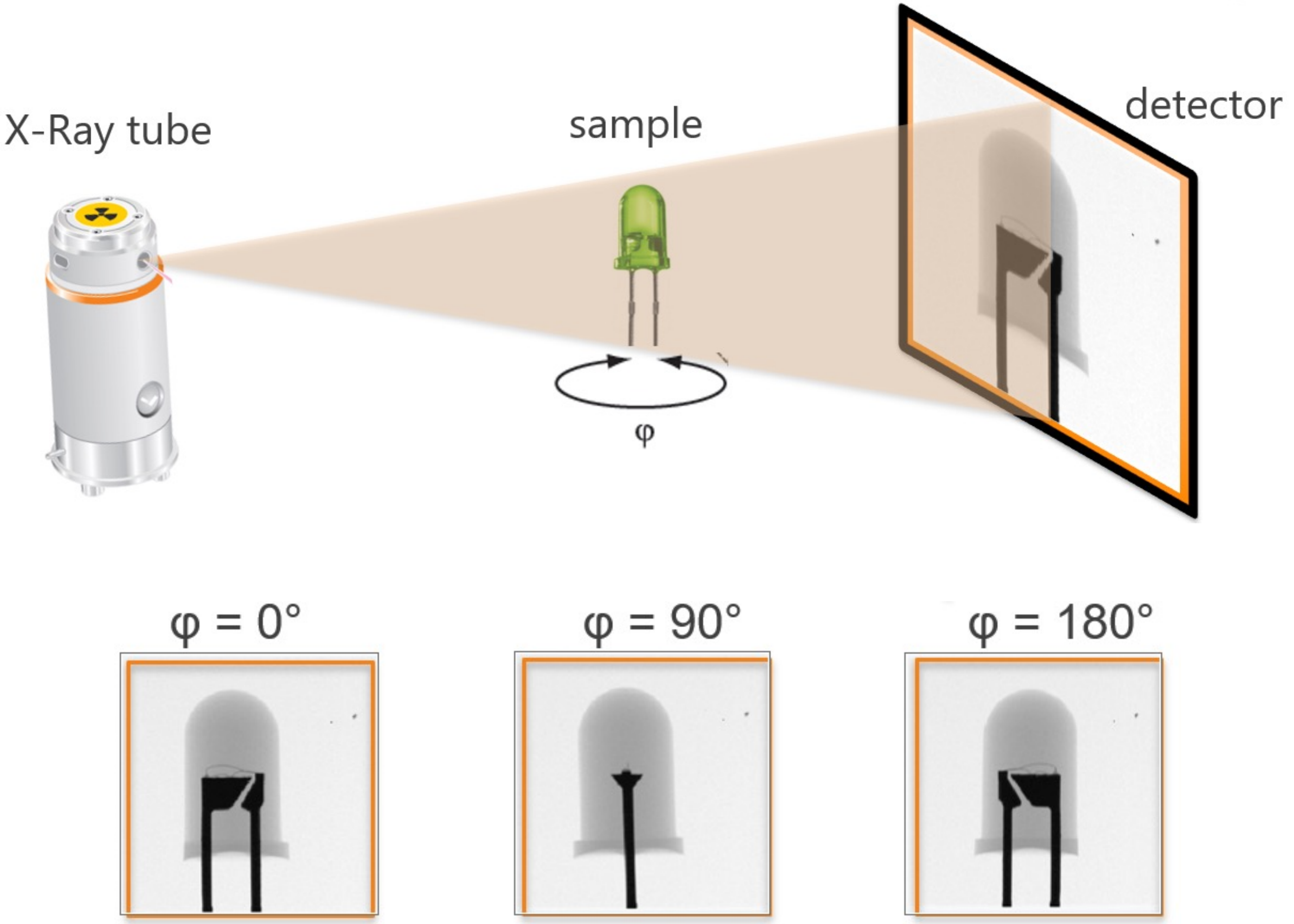




Absorption techniques

Imaging

- Imaging (radiography)
- Tomography



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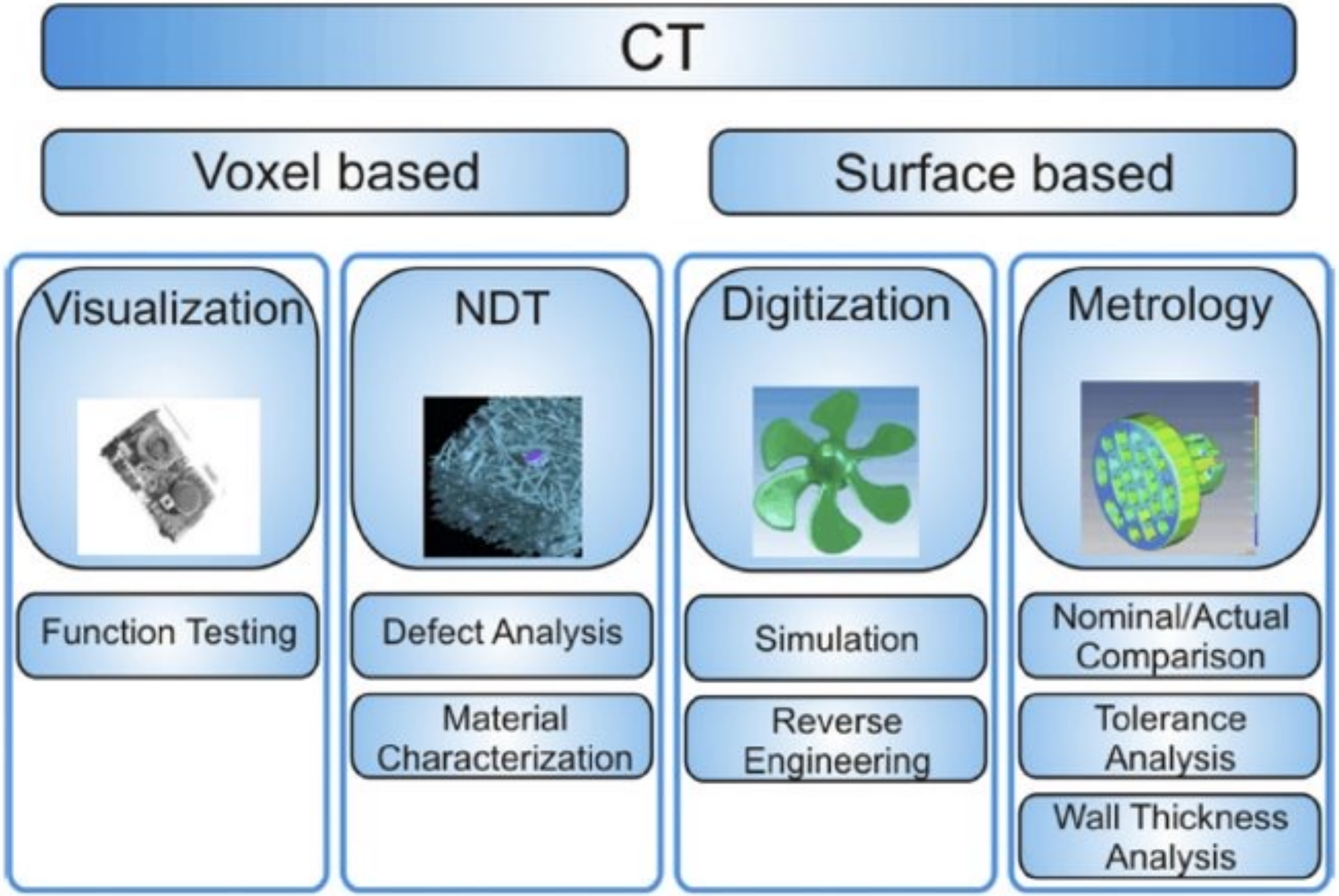
Computed Tomography

Application-property relation

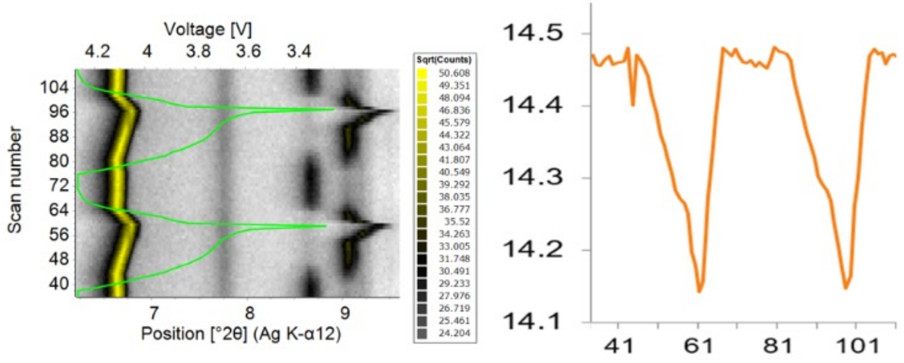


Exotic

Combination of techniques

- XRD-CT
  - Combination of the information obtained in CT with crystallographic grain microstructure

- In-situ/ in operando
  - Possibility to perform many of the techniques at different ambient condition or same conditions as when material is functionally used



Conclusions

- Short introduction to many (not all) the techniques that can be performed using conventional X-ray instrumentations in a laboratory
- Many of these techniques used to be possible only in large scale facilities but thanks to progress in source, optics, detectors are available also in the lab facilities
- When requirements in terms of speed or resolution cannot be met, and a large scale facility is needed, having data already collected on samples using conventional lab instrumentation helps in getting beamtime
- Lab scale facilities are also an excellent tool to train the future users/scientists of the large scale facilities

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