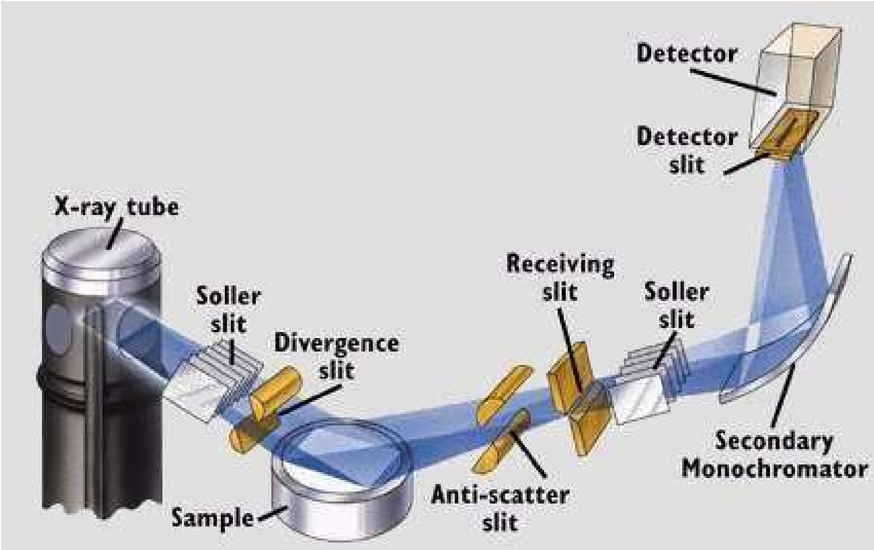

XRD set-up

The XRD-facilities of FAME

The group has three PANalytical X'Pert diffractometers

	Radiation	λ(Å)	geometry
XRD-1	Cu-Kα	1.5418	θ-θ
XRD-2	Cu-Kα	1.5418	θ-θ
XRD-3	Co-Kα	1.7902	θ-2θ

These three diffractometers can be equipped with different interchangeable sample stages:

	XRD-1	XRD-2	XRD-3
Flat sample spinner	x	x	x
Capillary spinner		x	x
Simple alignment shaft	x	x	x
TTK 450	x		
XRK 900	x		
Multi-purpose stage	x	x	x

We have several different sample-stages to make it possible to measure a variety of different samples. Among these stages are two sample-chambers with temperature control and there is a possibility to do in-situ measurements on battery materials and measurement on magnetic materials. X-Ray powder diffraction techniques can solve various problems. These non-destructive techniques can be used for different analyses, often as the only alternative for destructive methods. Only a small sample is usually necessary for most determinations, sometimes a few milligrams are already enough. The main condition is that the material is (partly) crystalline. Many materials fulfil this requirement :

- metals
- organic and inorganic compounds
- minerals
- synthetic materials (plastics, polymers)

The power of X-ray powder diffraction (XRPD)

Each crystalline material diffracts X-rays in a unique way, so resulting in a material-characteristic diffraction pattern. If the material consists of a large amount of crystallites (powder or polycrystalline sample), an XRPD pattern is obtained, consisting of diffracted intensities vs diffraction angle. An XRPD pattern can be obtained in very short time and the analysis of XRPD patterns has many possible applications. Below are some examples:

Qualitative and quantitative identification of materials

We use large databases of powder patterns to analyse our measurements. By comparing a XRPD pattern of a certain material (pure or a mixture) with the databases, the composition of the material can be determined, answering questions like:

- What does my material consists of? (qualitative analysis)
- What is the (precise) composition? (semi-quantitative analysis)

Examples:

- Analysis of kidney stones

The various types of kidney stones that exist can be completely different from a chemical point of view. An XRD analysis is highly efficient to establish the precise constituency of a kidney stone, even in case of very small stones.
We offer a very fast turn-around time for hospitals that require kidney-stone analysis at short notice.

- Composition of and additives in concrete

- Type of asbestos (chrysotile, crocidolite)

Non-destructive surface/thin layer analysis

If the X-rays incides at very small angles (so-called grazing incidence), only the surface material will diffract. This enables to establish differences between bulk vs surface material.

Example:

- Thin (corrosion) layers

Layer thicknesses < 0.1 um can be analysed without damaging the substrate. Samples sizes upto a few cm can be analysed without any additional destructive preparation

Polymorph determination

Many materials can crystallize in different crystal modifications, so-called polymorphs, because of differences in crystallization conditions (e.g. temperature, pressure, solvent used etc). Polymorphs may have completely different properties and it is often essential to know whether the correct polymorph has been formed, e.g. in industrial processes, to support patent applications etc.

Examples:

- Titanium dioxide (TiO_2), rutile or anatase

- Silicon oxide (SiO_2), quartz, cristobalite and tridymite

- Carbon (C), diamond and graphite

- Cocoa butter

Physical properties of materials

A powder diffraction pattern of a material does not depend only on the underlying crystal structure(s). An analysis of the peak profiles and peak widths may reveal also other important physical and material characteristics, for example,

- Crystallinity

- Crystallite size
- Micro and macro strain as a result of mechanical forces
- Crystallite morphology (texture)

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