

General aspects of X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF)

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X-Ray Diffraction (XRD)

* **Measures crystalline materials** like ceramics, metals, intermetallics, minerals, inorganic compounds

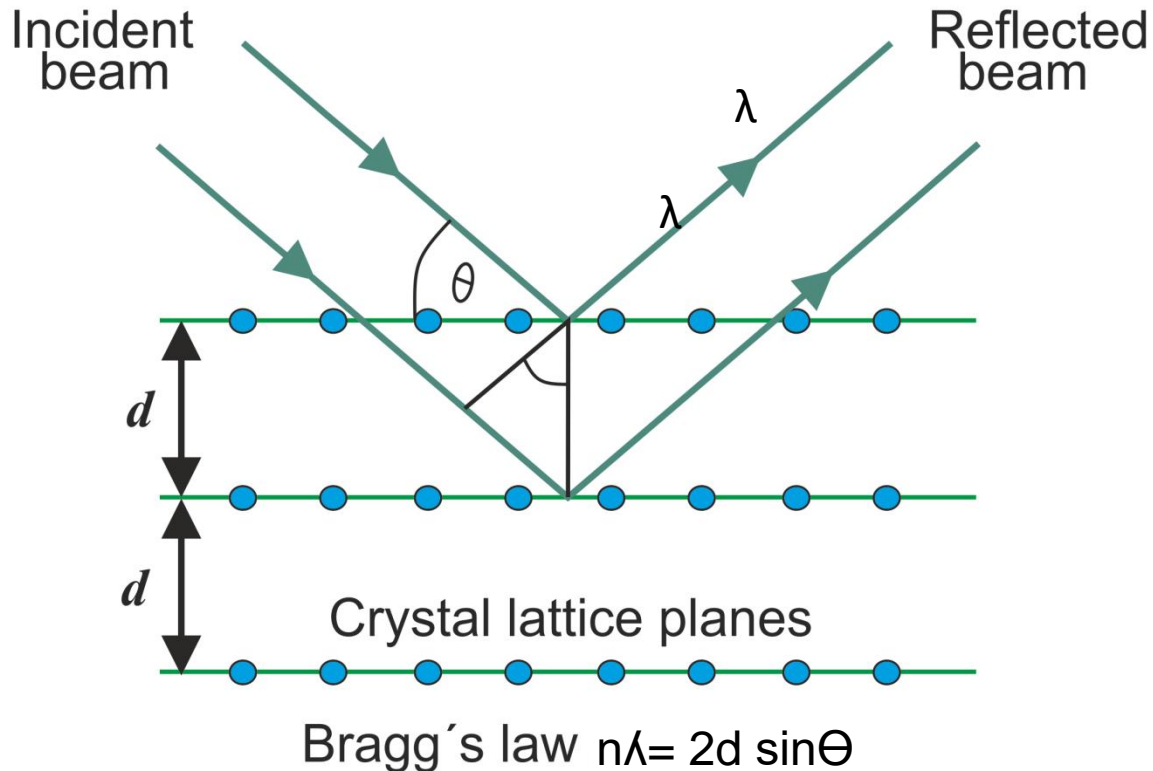


Author Riho Mõtlep

X-Ray Diffraction (XRD)

* X-ray beam hits a crystal, scattering the beam in a manner characterized by the atomic structure

* The beam reflected from the lower surface travels farther than the one reflected from the upper surface



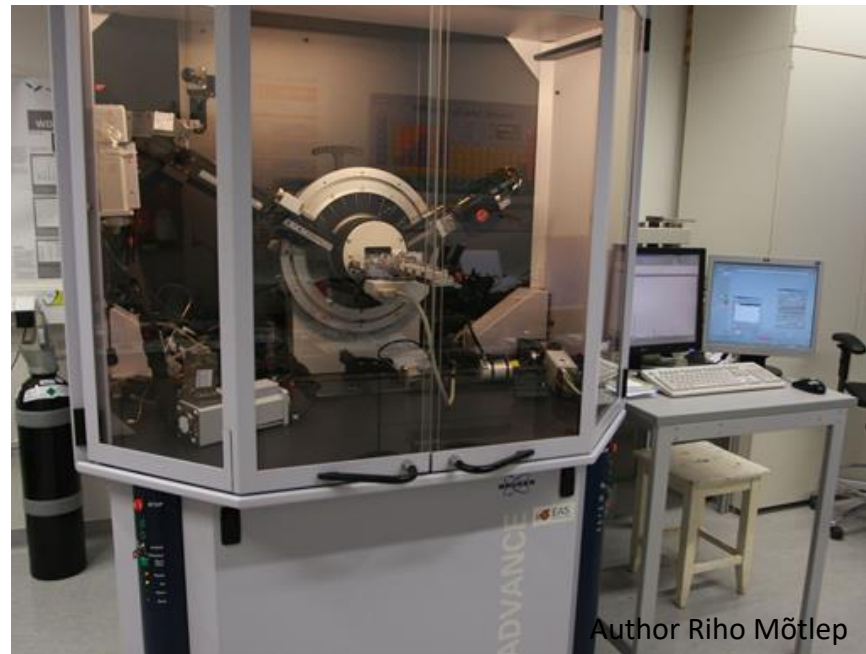
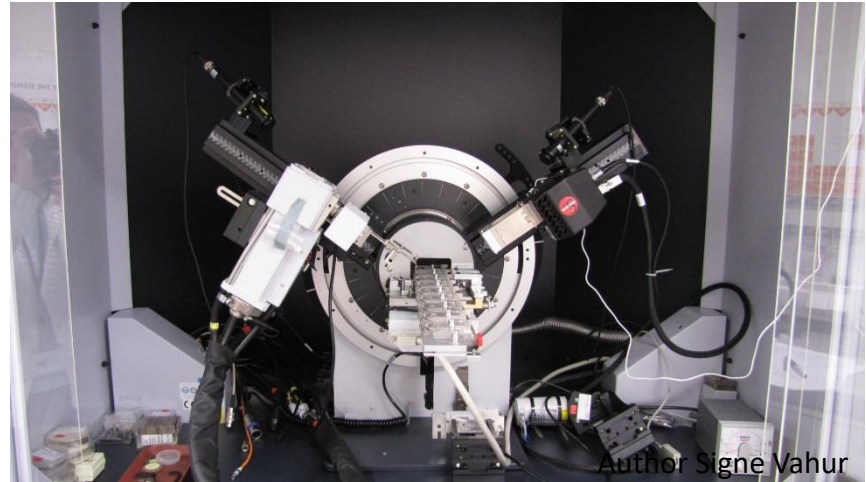
Advantages and Disadvantages of XRD

Advantages

- * Fast identification of materials
- * Easy sample preparation
- * Large database of known crystalline structures
- * Computer-aided material identification

Disadvantages

- * Impossible to identify directly amorphous materials
- * Quantitative analysis need grinding



Sample preparation

Semi-quantitative analysis:

* Pieces with smooth/even surfaces

(**NB: has to fit into sample holder**)

* Powders

* Min. sample needed for analysis **0.2 – 2 g**

Quantitative analysis:

* Homogenous powders

(grinding needed)

* Min. sample needed

for analysis **2-5 g**



Ball mill

samples (**~ 15 – 30 g**)

Agate mortar

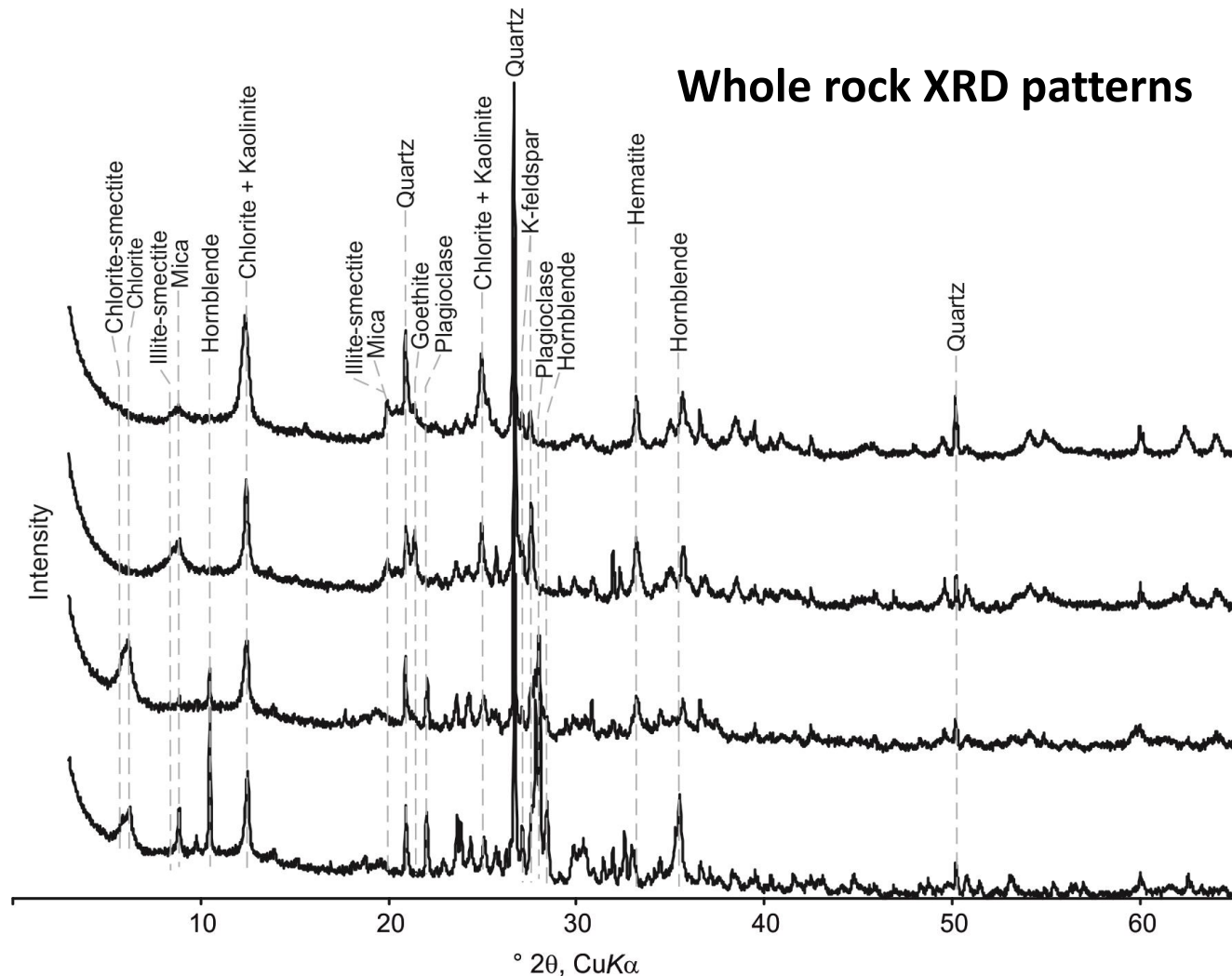
samples (**~ 0.2 – 5 g**)



Author Evelin Lumi



XRD – Crystal phase determination



- * Crystal phase analysis is possible by comparing the XRD patterns with library of known crystalline structures (minerals/phases)
- * Analysis of XRD patterns are complicated and need help from the specialist

X-Ray Fluorescence (XRF)

Handheld / Portable XRF

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Wavelength Dispersive X-Ray Fluorescence (WD-XRF)



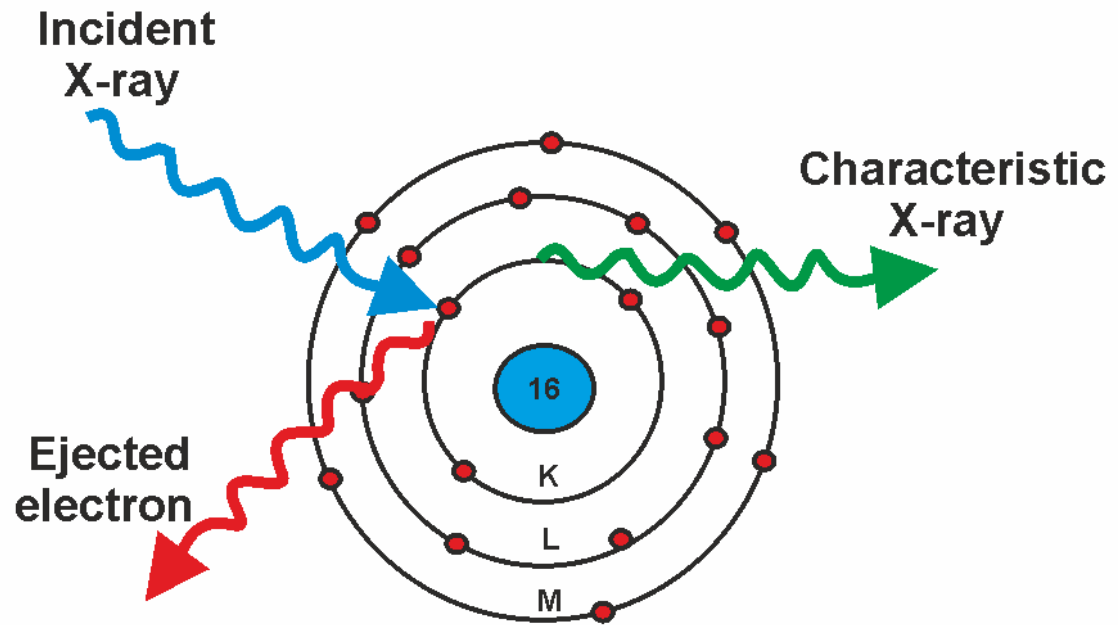
Energy Dispersive X-Ray Fluorescence (ED-XRF)

*XRF is a method for measuring the **elemental composition** of studied sample

X-Ray Fluorescence (XRF)

* Measures emission of characteristic secondary X-rays from a material that has been excited by bombarding with high energy X-Rays.

* Characteristic X-Rays are unique for each element



Advantages and Disadvantages of XRF

Wavelength Dispersive X-Ray Fluorescence (WD-XRF)

Advantages

- * Fast elemental analysis
(from seconds up to 30 min)
- * Elemental mapping
- * Computer-aided material identification

Disadvantages

- * Qualitative limit is 10-20 ppm
- * Quantitative analysis need grinding
- * Impossible to measure **elements** lighter than **Boron**
- *

Energy Dispersive X-Ray Fluorescence (ED-XRF)

Advantages

- * Instant elemental analysis
- * Cost-efficient
- * In-situ analyzes
- * No sample preparation
- * Easy to use

Disadvantages

- * Short analytical and application range
- * Typically measures starting from **Mg to U**

Sample preparation (WD-XRF)

Semi quantitative:

Smooth/even surface (sample) is fitted into sample holder (**NB has to fit into sample holder**)

Quantitative:

1) **The pressed pellet method** – grinded sample is pressed into pellet using mechanical or hydraulic press (**min. sample** weight needed for analyze **6 -15g**)

- * method is convenient and relatively easy
- * By grinding the particle size effect can be eliminated only to a certain extent
- * Mineralogical composition has to be identical to the standard

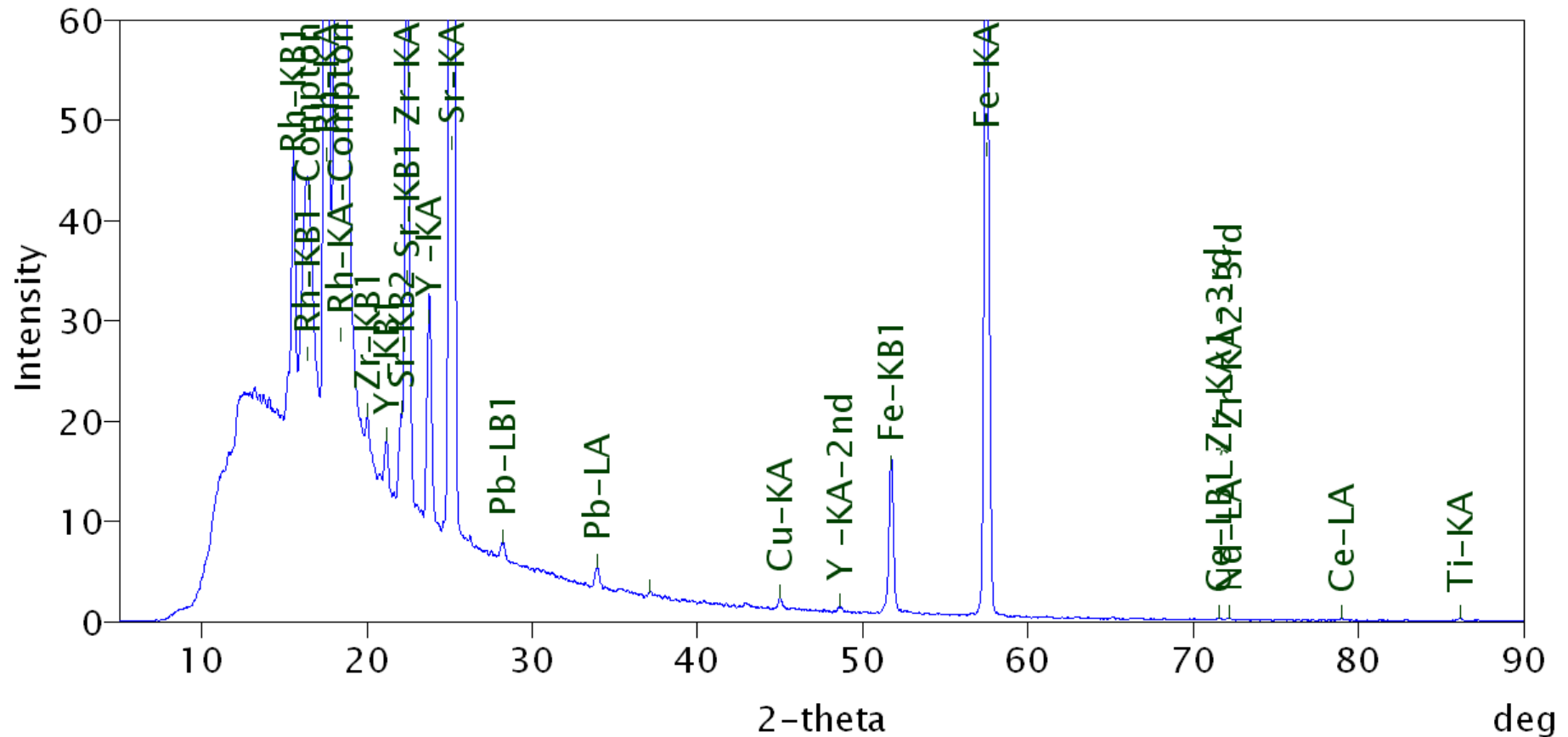
2) **The fused bead method** - previously weighted sample together with lithium borate is mixed in platinum dish and subjected to high temperatures. The fusion melt is then casted into a casting mould and cooled (**min. sample** weight needed for analyze **1-5g**)

- * quite a time-consuming process
- * The effective particle size is almost atomic
- * mineralogy effects are eliminated

For more detailed overview please look the video from following site:

<https://sisu.ut.ee/heritage-analysis/book/42-xrf>

Interpretation of WD-XRF Spectra



- * Computer-aided material identification
- * Interpretation is easier compared to XRD

X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) in Art and Archaeology Studies

*Cellulose-based materials

Paper

*paper consists of two main components: cellulose and additives

*Cellulose is partially crystalline and the technique used for its characterization consists in determining the crystallinity value that decreases owing to the ageing.

*Pigments; paints

pigments, as crystalline materials

*Stone; mortars

recognizing composition

degradation or conservation state

in some cases provenance

*Metals, alloys corrosion Products

In the case of small samples, they can be analysed directly in the diffractometer itself.

For the objects of major size some micro- samples are drawn in order to perform analyses.