X-Rays Diffraction Laboratory (XRDL)

X-rays were discovered and named by a German Physics Professor Wilhelm Konrad Roentgen in 1895. Roentgen observed that the invisible cathode rays caused a fluorescent effect on a small cardboard screen painted with barium platinocyanide when it was placed close to the aluminium window. Unable to identify, he temporarily termed "X-rays", using the mathematical designation ("X") for something unknown. Later, discovery of X-rays (or Roentgen-rays) earned him the first Nobel Prize in Physics in 1901. Following this achievement, Laue won the Nobel Prize in Physics in 1914 for his discovery of the diffraction of X-rays by crystals. However, it was a father, W. H. Bragg and a son, W. L Bragg, who discovered diffraction patterns of crystalline solids and awarded by a joint Nobel Prize in Physics in 1915.

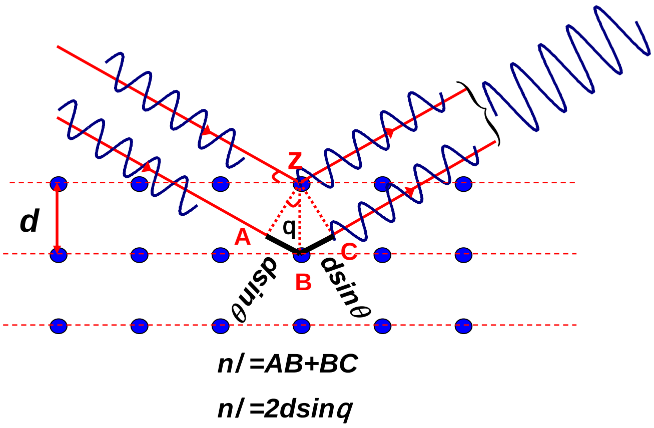

Figure 1. Bragg's Law: 2d sinθ=nλ

BASIC PRINCIPLES

Bragg's law (Figure 1), states that when the x-ray is incident onto a crystal surface, its angle of incidence, θ, will reflect back with a same angle of scattering, θ. And, when the path difference, *d* is equal to a whole number, *n*, of wavelength λ, a constructive interference will occur. This constructive interference is known as X-ray diffraction. Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal.

METU Central Lab has a Rigaku Ultima-IV (Figure 2), which is a multipurpose diffraction system. The system has a X-ray tube of Cu target and cooling water flow controlling the temperature changes of the X-ray tube. It utilizes a Curved Graphite Monochromator to create monochromatic X-rays. In addition, the system can be rearranged through sets of slits for different irradiation areas.

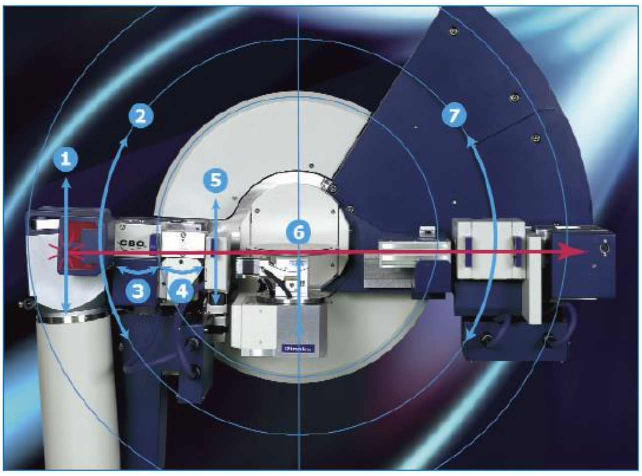

Figure 2. Rigaku Ultima-IV Optical Mechanism;

1: Source Height, 2: Source Angle, 3: CBO optic, 4: Crystal optics, 5: Slit height, 6: Sample surface, 7: Detector angle

The Ultima IV X-ray diffractometer can perform many different measurements incorporating Rigaku's patented cross beam optics (CBO) technology for permanently mounted, permanently aligned and user-selectable parallel and focusing geometries. CBO and automatic alignment combine for the ultimate in functionality for: thin-film diffraction, small angle scattering, and in-plane scattering. While "Bragg-Brentano Focusing Beam Geometry" method offers the advantages of high-resolution and high beam-intensity analysis for highly crystalline samples with flat surfaces, "Parallel Beam Geometry" is used for weakly crystallized and/or samples with rough surfaces as well as thin film measurements.

X-ray diffraction measurements of "thin" (1-1000 nm) films using conventional θ/2θ (2θ=2-90°) scanning methods generally produces a weak signal from the film and an intense signal from the substrate. Thin film measurements with a fixed grazing angle of incidence, popularly known as GIXRD, have been done in METU Central Lab with good sensitivity. In Figure 3, this method is compared with a standard θ/2θ scan for a thin film.

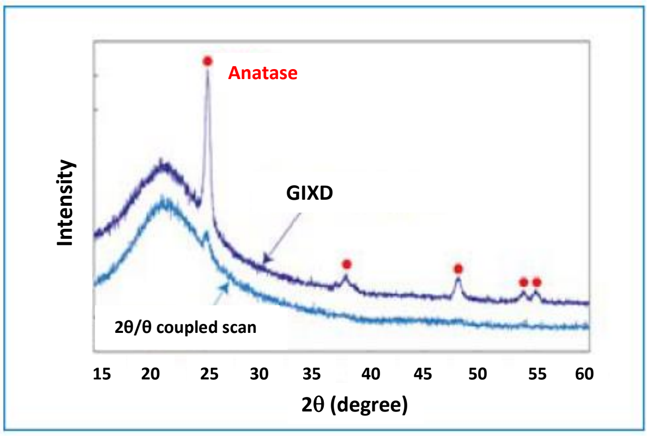

Figure 3. Grazing Incidence X-ray Diffraction (GIXD) Method

In addition, qualitative analysis of the phases can be done by using PDXL software, which has approximately 200000 diffractograms in its database, after XRD pattern is obtained.

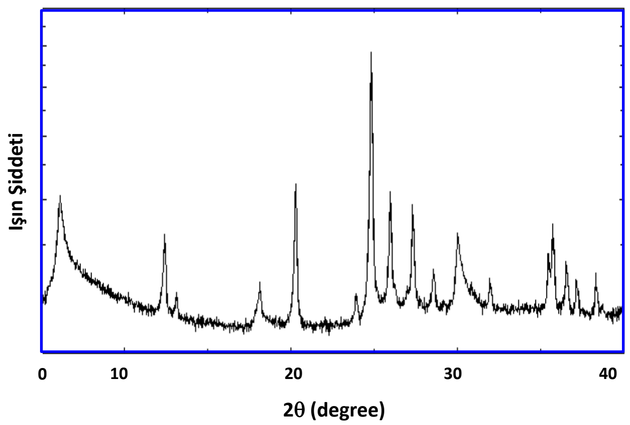

Figure 5. Diffractogram of a zeolite

APPLICATIONS

Rigaku Ultima-IV XRD has a wide range of applications such as:

Geological analysis of minerals and rocks

Metal and alloy analysis

Ceramic and cement analysis

Thin film analysis

Polymer analysis

Impurity and polymorphous phase determination in pharmaceutical industries

Characterization of archaeological findings

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