

X-Ray Diffraction XRD Facilities on Lab 7

XRD Facilities: Lab7

CENIMAT|i3N
FCT-UNL
Campus da Caparica
2829-516 Caparica
Portugal
www.cenimat.fct.unl.pt

Contact:

Joana Vaz Pinto
(jvp@fct.unl.pt)
Prof. Elvira Fortunato
(emf@fct.unl.pt)
Tel: +351212948562
Fax: +351212948558

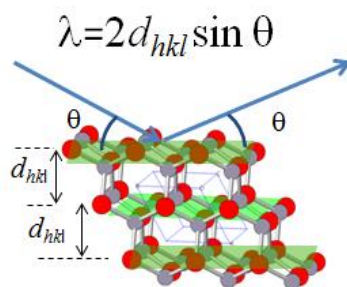
Applications

XRD allows the phase Identification and composition by qualitative and quantitative analysis of powdered, bulk and thin film materials. A high range of applications are possible in diverse areas such as material science, geology microelectronics, chemistry, cultural heritage, pharmaceuticals and forensic science among others.



XRD Principles

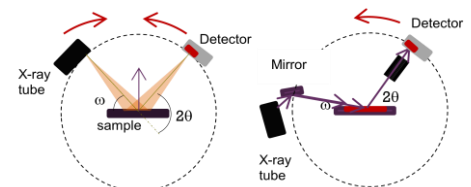
X-Ray diffraction is a powerful and non-destructive technique for identification and quantification of the crystalline phase composition of powdered and solid samples. It is based on the photon elastic scattering by the atoms of a given material. When the atoms are positioned in a periodic array, the scattered radiation undergoes destructive and constructive interference (diffraction) at specific angles and this phenomenon is described by the Bragg's Law:



The directions of the diffracted wave depend on the size and shape of the unit cell, and the intensities depend on the arrangement of the atoms in the crystal. Therefore, a diffraction pattern reveals the crystalline phases present (peak positions), the relative phase concentration (ratio of peak areas), amorphous content (background humps) and crystallite size and strain (peak widths).

Conventional Configuration

Electrons from a hot filament are accelerated to the anode material (usually Cu or Mo) in a HT tube, to produce the X-rays. The rays are directed to the sample, and the diffracted beam is collected by a photodetector. The movements of detector and X-ray source relative to the sample surface are controlled in a diffractometer and different kinds of geometries can be chosen.

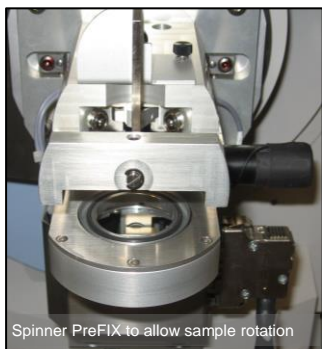


Bragg-Brentano and Parallel-Beam geometries

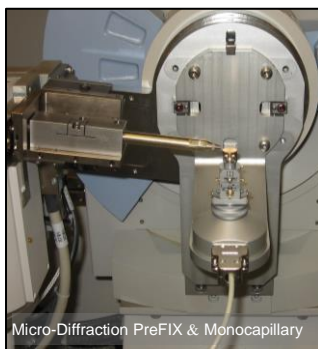
In the conventional Bragg-Brentano focusing geometry, the incidence angle is set equal to the diffracted angle (measured with respect to the sample surface) and data is usually plotted as Intensity vs. 2θ (related to interplanar spacing d_{hkl}) in a diffractogram. This configuration presents a high intense diffracted beam with very good 2θ resolution and is ideal for powder samples and polycrystalline films. Due to the symmetric angle geometry it mainly probes grains aligned parallel to the surface. However since the penetration depth depends on the incident angle, it has lower sensitivity for very thin films.



Standard FlatStage PreFIX



Spinner PreFIX to allow sample rotation



Micro-Diffraction PreFIX & Monocapillary



MRI Heating Chamber for *in-situ* XRD

Technical specifications

X'Pert PRO MPD

Multi-Purpose Diffractometer in a Theta-Theta configuration:

- Radius of 240 mm
- Min. step in ω and 2θ of 0.001°

X'Pert Tube: Ceramic X-ray tube with Cu anode ($\lambda=1.540593 \text{ \AA}$, $K_{\alpha 1}$), with linear and point focus possibilities:

- Max. HT: 60 kV
- Max Curr: 55mA

PreFIX Modules: easily dismantled and replaced without the need for re-alignment:

- Flat Sample Stage
- Spinner for Reflection Transmission Experiments
- Microdiffraction and monocapillary
 - 0.1 mm exit beam
 - Alignment Microscope
- MRI Non-ambient Chamber
 - Pt foil, 10 mm width
 - T from 30°C to 1200°C
 - Manual height adjust.

X'Celerator Detector: ultrafast X-ray 1D detector based on RTMS (Real Time Multiple Strip) Technology.

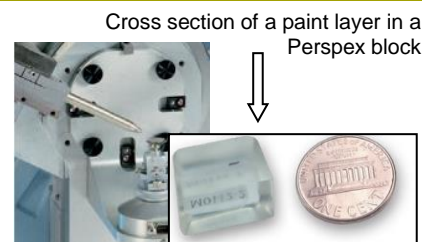
X-ray Mirror, Gutman type, for parallel beam configuration :

- Axial Diverg. 0.04°
- Reflectivity of 65%

Additional features

Microdiffraction

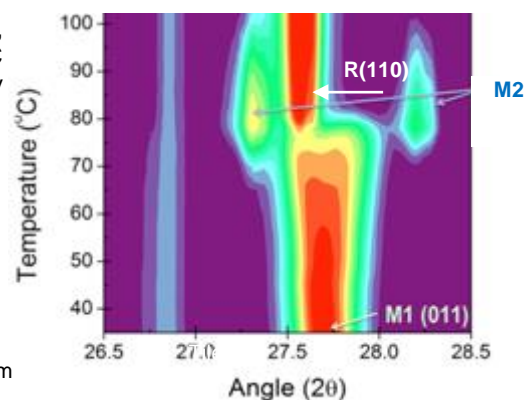
Micro X-ray diffraction uses a **capillary focusing optics** to collect X-rays from the divergent X-ray source and to direct them to a focused beam at the sample surface with diameters of tens of μm . The increase of intensity in a small spot allows an enhanced spatial resolution for small feature analysis such as for cultural heritage samples.



MRI Non-ambient Chamber

PreFIX with a Pt heater element, able to heat samples up to 1200°C and simultaneously collect X-ray diffraction data for *in-situ* analysis:

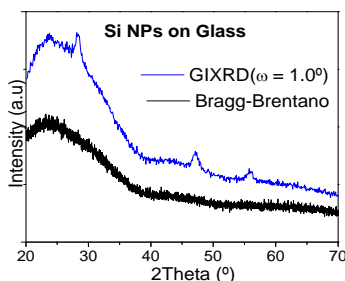
- Temperature induced phase transitions
- Time resolved experiments for reaction kinetics



Picture: VO_2 phase transition at 78°C from Monoclinic to Rutile type structure

Parallel Beam Geometry for Thin Films

Grazing Incidence GIXRD



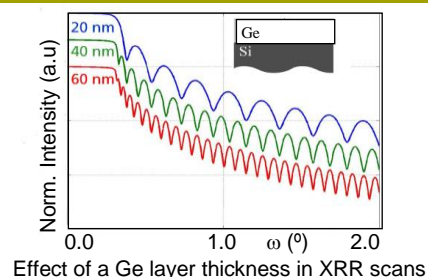
At small incident angle ($\omega < 3^\circ$) the beam travels a bigger distance on the upmost layers of interest, reinforcing its diffraction pattern, while the signal from the substrate is reduced.

- depth control diffraction
- analysis of tilted planes
- Suitable for very thin films

X-Ray Reflectivity (XRR)

XRR is used with ω scans to analyze amorphous or crystalline thin films allowing to extract information on:

- Thickness
- Density
- critical angle
- refraction index
- Porosity
- Roughness



Effect of a Ge layer thickness in XRR scans