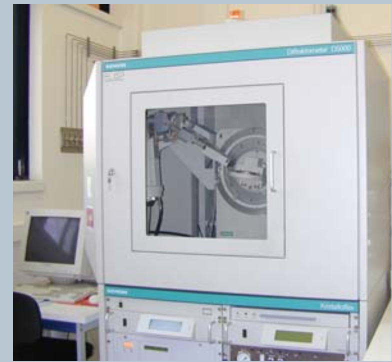
IMOMEC
Institute for Materials Research
University of Hasselt
Associated Lab IMEC

X-ray Diffraction Equipment

X-ray diffraction (XRD) is a non-destructive technique to reveal crystallographic information about materials such as bulk materials, thin films and powders.
With powder XRD the crystal structure of materials (original or on purpose in powder form) is gathered. The XRD studies upon bulk materials and films allow besides the definition of the crystal structure also other crystallopgraphic information as preferential orientation, residual stress, …Combination of the XRD equipment with a temperature chamber under a controlled atmosphere allows the study of phase transformations, oxidation reactions,….
Besides the above mentioned modes based upon the diffraction of X-rays on crystalline materials, also X-ray reflectometry (XRR) measurements can be performed on the same equipment. Such XRR measurements are used to determine the thickness, roughness and density of films. The XRR technique is applicable on crystalline and amorphous layers.

System description and specifications

IMOMEC has a Siemens D5000 and a Bruker D8 Discover diffractometer.



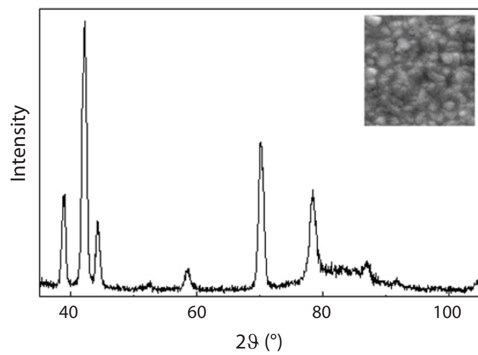
The Siemens D5000 diffractometer is a high-resolution ϑ-2ϑ powder diffractometer with a focusing Bragg-Brentano geometry and is equipped with a primary Ge(111) monochromator which allows the selection of a monochromatic Cu-$K_{\alpha 1}$ radiation. This diffractometer is suitable for powder applications which require high resolution, such as microstructural characterization in terms of size and strain, crystal structure determination and quantitative phase determination.

The Bruker D8 Discover diffractometer has several optics allowing the realization of different experiment types under optimal conditions. This XRD diffractometer has a parallel beam geometry using a Göbel mirror (line focus, Cu radiation) or a poly capillary (point focus, Cr radiation) in the primary beam and a collimator in the diffracted beam. The diffracted beam is measured with a scintillator or position sensitive detector. This geometry allows measurements on irregular and non flat samples. The standard multifunctional eulerian cradle allows reflectometry, texture and stress measurements. On the cradle also a capillary sample stage can be mounted for powder studies where the Bragg-Brentano geometry is limited, such as crystal structure determination or quantitative phase determination on very small amounts of material (a few tens mg), or for samples with a strong preferential orientation, very transparent materials (e.g. polymers or alumina) or for samples which have to be protected against atmospheric influences. On the cradle also an eucentric goniometer head can be mounted for accurate measurements of the orientation of single crystals. There is also the option to mount a 2-bounce Ge(022) monochromator (~0.006° divergence) so that high-resolution applications are possible. Combination of the XRD equipment with a temperature chamber (up to 1200°C) under a controlled atmosphere (oxygen, nitrogen, air,…) allows the study of phase transformations, oxidation reactions,… upon powders, thin films and thick coatings.

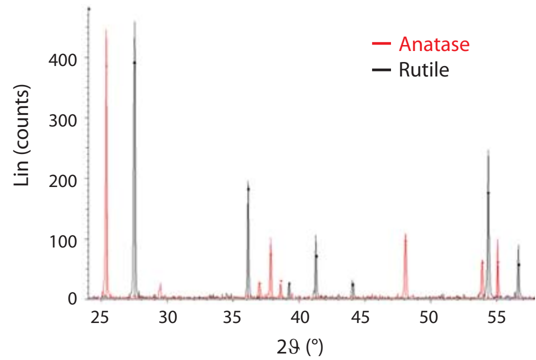
Measurement example

Some typical applications are :

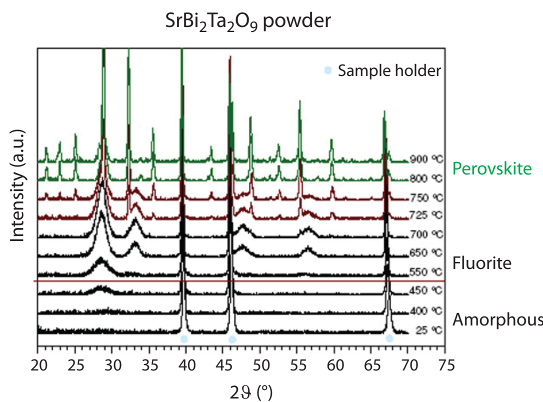
- Identification of crystalline materials (qualitative / quantitative)
- Determination of unit cell parameters (e.g. parameters are related to doping level in oxides)
- Determination of crystallite size and strain in materials using peak profiles
- In-situ phase formation within HT-XRD equipment (e.g. thermal decomposition of precursor materials)
- Determination of miscut in single crystals
- Stress measurement
- Determination of preferential orientation in materials
- Determination of thicknesses, density upon thin film and multilayer structures by means of XRR



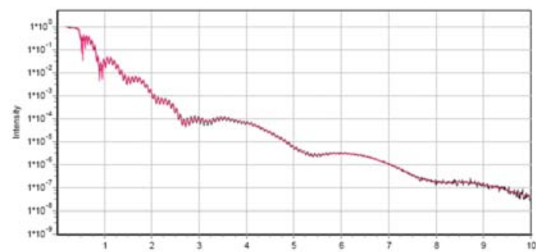
ϑ-2ϑ X-ray diffractogram of AlN film with SEM image of surface morphology in inset (1x1 μm² scanned area in SEM)



ϑ-2ϑ X-ray diffractogram of mixed TiO2 powder with anatase and rutile phases



HT-XRD experiment : in-situ crystallization study during heat treatment of $SrBi_2Ta_2O_9$ powder



Sample

N	R	Material	Thickness	Roughness	Profile	Grading	Density T	Density B
1	1	Si	95.56	1.001	Linear	10	2.55598	2.63511
2	1	TiN	10.40	0.942	Linear	10	5.85494	5.92828
3	1	HfO2	3.41	0.339	Linear	10	10.64580	11.98980
4	1	SiO2	2.00	0.309	No Gradient	10	2.45298	2.54345
SUB	1	Si	0.00	0.000	No Gradient	0	2.32910	2.32910

Experimental + simulated curve of XRR measurement upon multilayer structure Si / TiN / HfO_2 / SiO_2 upon Si substrate (thicknesses all given in nm)

imec
aspire invent achieve

IMEC
Kapeldreef 75
B-3001 Leuven
Belgium

Ph. +32 16 28 18 80
Fax. +32 16 28 16 37
Katrien.Marent@imec.be

universiteit hasselt
INSTITUUT VOOR MATERIAALONDERZOEK

Institute for Materials Research - Hasselt University
Division IMOMEC, IMEC vzw
Wetenschapspark 1
B-3590 Diepenbeek
Belgium

Ph. +32 11 26 88 83
Fax. +32 11 26 88 99
jan.dhaen@uhasselt.be