Materials characterisation techniques

High resolution electron microscopy

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X-ray diffraction

High resolution electron microscopy

Technique

High Resolution Electron Microscopy is used for obtaining atomic structure images in materials, with the aim of revealing both crystalline and atomic imperfections. Moreover, the addition of special spectrometers to these microscopes allows identification of both light (energy loss spectrometers) and heavy elements (emitted X-ray spectrometers) in nanometric-scale areas of the observed samples. High resolution electron microscopes also allow recording of electron diffraction patterns originated by sample illumination with a convergent electron beam focused onto nanometric-scale areas. Careful analysis of these patterns allows refinement of the crystalline network parameters, thus asserting the spatial group of the examined structure.

Capabilities

- Examination of interfaces in multilayer structures of semiconductor materials.
- Examination of nanometric particles for catalytic uses or for fabrication of gas sensors.
- Determination of tensions and modified network parameters in epitaxially grown layers.
- Elemental analysis of nanometric precipitates at interfaces and grain boundaries.
- Structural study of new materials such as nanotubes, superconductor materials, quantum wells in semiconductor materials and others.

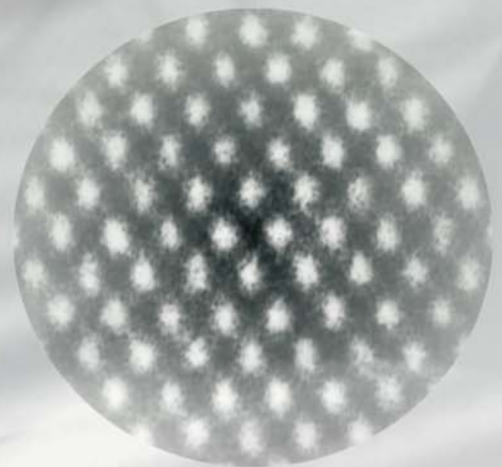
Instrumentation

- Transmission Electron Microscope, TEM 300KV Philips CM30 with EDX spectrometer, Link LZ5.
- Transmission Electron Microscope, TEM 200KV Philips with field emission gun, JEOL JEM 2010F with GATAN energy loss spectrometer.

Specific applications

- Analysis of complex structures in copper and aluminium alloys.
- Thickness determination in ranges down to two- or three-atomic layers in complex semiconductor structures.
- Nanometer size silicide particle assessment in metal matrixes.

Transmission electron microscope,
TEM 300KV Philips CM30,
with EDX spectrometer, Link LZ5



Electron diffraction pattern obtained
under convergent beam on a silicon monocrystal

High resolution electron microscope image
where bi-atomic columns of crystalline gallium
indium arsenide may be observed along
the 110 crystallographic direction

X-ray diffraction

Technique

X-ray diffraction is essentially a structural characterisation technique for solid samples of crystalline nature. The wavelength of the X-ray radiation with which measured specimens are illuminated in this technique is within the range of most inter-atomic distances, and therefore, the sample itself acts as a diffraction network which selectively deviates (diffracts) the incoming X-rays into specific directions with various intensities. This means that the diffraction signal emitted by a crystalline solid is a fingerprint of its constituent structure. In diffraction experiments, mono- or polycrystalline samples mounted onto goniometric stages are analysed monitoring the angular dependence of the diffracted beams with respect to the sample and incident X-ray relative orientation. The experimental set-up is relatively simple and the range of applications wide and diverse.

Capabilities

The most usual applications are:

- Structural determinations. Resolution and crystal structure refinement.
- Qualitative and quantitative analysis of crystalline phases.
- Polymorphism analysis.
- Isomorphism studies and isostructurality. Solid solutions.
- Characterisation of phase transitions and solid state reactions.
- Estimates of particle size and microdeformations.
- Deformation analysis. Determination of residual tensions.
- Study of preferential orientations and texture analysis.
- Depth-dependent studies by grazing incidence.
- Characterisation of epitaxial layers. Layer-substrate relationships.
- Determination of structural parameters in multilayers.
- Reflectometry.

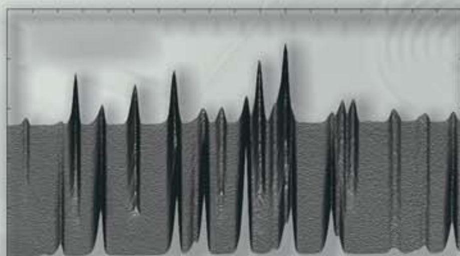
Instrumentation

- Powder diffractometer of $\theta/2\theta$ Bragg-Brentano geometry, Siemens D-500. Automated sample loader. Temperature chambers. Accessory for grazing incidence.
- Multi-application diffractometer, Philips MRD. Interchangeable optics for high (with Bartel's monochromator and analyser crystal) and low resolution (parallel). Texture goniometer.
- Powder diffractometer with Debye-Sherrer geometry, INEL CPS-120. Accessories for capillary and flat samples. Cryostat. Furnace for samples mounted in the capillary.
- Four circle single crystal diffractometer with kappa geometry, Enraf-Nonius CAD-4. Low temperature accessory.
- Single crystal diffractometer with bidimensional detector Image Plate MAR345.

Specific applications

- Characterisation of polymorphes in pharmaceutical products.
- Detection of cement types (Portland, aluminous, etc.) present in concrete.
- Quality control in thin layers and coatings for electronic devices, wear-resistant tools, etc.

Powder diffractometer, Siemens D-500 (S1) with automated sample loader



Thermo-diffractometrical study. Time-dependent polymorphic transition at 393 K, from II to I form in a pirazetam sample (pharmaceutical product)

Diffractometer, Siemens D-500 (S2), with temperature chamber, TTK, and device for humidity controlled measurements

Powder diffractometer, INEL CPS-120, fitted with a liquid nitrogen cryostat

Texture goniometer in the diffractometer Philips MRD

Texture study in a silicon carbide layer. Pole figure evaluation

X-ray diffraction laboratory: Philips MRD and INEL CPS-120

Four-circle goniometer for the single crystal diffractometer, Enraf-Nonius CAD-4

Single crystal diffractometer with the Image Plate detector. Diffraction photogram