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X-Ray Diffraction (XRD) Services

X-ray diffraction (XRD) is a powerful nondestructive technique for characterizing crystalline materials.

XRD has a very wide range of applications, across many sample types and materials. While most other analytical techniques provide elemental or molecular information from a sample, XRD is unique in providing a wide variety of information on structure, crystalline phase, preferred crystal orientation (texture), and other structural parameters such as crystallite size, percent crystallinity, strain, stress, and crystal defects.

In XRD analysis, a sample is exposed to a collimated X-ray beam of specific known wavelength. If the material is crystalline, it possesses a three dimensional ordering or "structure" with repeat units of atomic arrangements (unit cells). X-rays are diffracted by the repeating lattice spacings of crystalline materials, whereas they are simply scattered by amorphous materials. X-ray diffraction occurs at specific angles (2θ) with respect to the lattice spacings defined by Bragg's Law:

$$n\lambda = 2d \sin\theta$$

where n is an integer denoting the order of the reflection, λ is the X-ray wavelength, d is the lattice spacing of the crystal planes of interest, and θ is the diffraction angle. Any change or difference in lattice spacing results in a corresponding shift in the diffraction lines. Consequently, the X-ray diffraction pattern is a unique fingerprint of the specific periodic atomic arrangements in a given material. Diffraction patterns can be checked against large libraries of patterns from known materials in order to identify/quantify the different phases present in a sample.

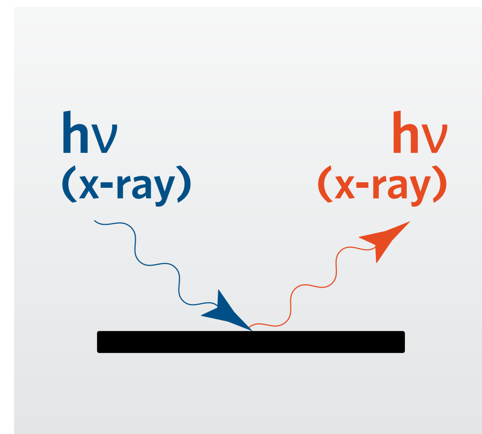

Figure 1: General schematic of how XRD works

Strengths

- Provides a wealth of unique information about crystalline materials
- Sampling depth for bulk materials of 10's µm to 100's µm, or near-surface analysis (50nm-1µm) using Grazing Incidence XRD
- Able to uniquely identify specific crystalline phases
- Measures lattice spacing/dimensions
- Full wafer analysis up to 300mm (not all of wafer accessible or at all diffraction angles)
- Minimal or no sample preparation requirements
- Non-destructive; ambient conditions typical but inert atmosphere/vacuum available
- Variable temperature measurements possible (R.T. – 1500C)

Limitations

- Typical detection limit ~1% w/w
- Cannot identify chemistry of amorphous components
- Limited information available with respect to depth (no depth profiling, but Grazing Incidence-XRD can be used to increase sensitivity for thin layers)
- Smallest spot size is ~25µm

Common Applications

While most other analytical techniques tend to provide elemental or molecular information, XRD provides crystalline information, resulting in many unique applications including:

- Identification/quantification of crystalline phase
- Measurement of average crystallite size, strain, or micro- strain effects
- Percent crystallinity versus amorphous component
- Texture (orientation): preferred orientation of crystallites
- Determination of lattice parameters to quantify solid-solution effects
- Residual Stress – lattice compression or expansion
- High Resolution XRD (HRXRD) – Epitaxial layer composition, strain in/out of plane
- SAXS – Small Angle X-ray Scattering for agglomerated particle size and spacing
- High Temperature X-ray Diffraction – in-situ phase change measurement

Industry Sectors and Technologies

- Aerospace & Defense
- Consumer Electronics
- Energy & Batteries
- Pharmaceuticals
- Semiconductors

Case Study: Phase ID

A typical XRD application is phase identification (phase-ID). Shown in Figure 2 is the diffraction pattern from a TiO_2 sample. The XRD results show that the sample contains both the Rutile (31.4%) and Anatase (68.6%) phases of TiO_2. Compositional results would (ideally) show a 2:1 ratio of O:Ti, but these different phases of TiO_2 have different physical and electronic properties so knowing which phases are present can be crucial.

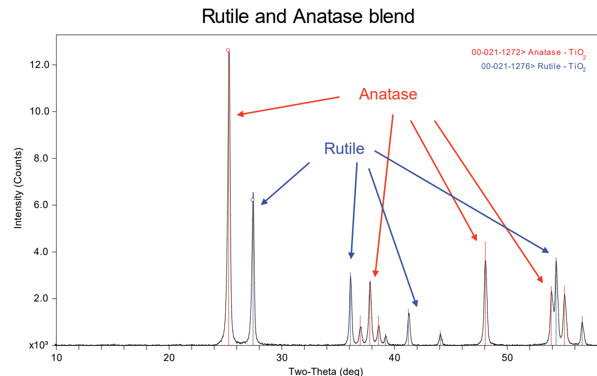

Figure 2: XRD Phase Analysis of titanium oxide (TiO_2). The TiO_2 consists of 31.4% Rutile and 68.6% Anatase phases.

Case Study: Amorphous vs Crystalline

In Figure 3 below, we have a complex diffraction pattern with a number of very interesting features. First we have a background that is slightly higher at low angles. This raised background originates from air scatter along the beam path that is more prevalent at lower incident angles. We have a number of diffraction peaks, where some are more narrow and some are more broad. While the sharper peaks on the right are due to crystalline materials, there is also a broad peak located toward the left of

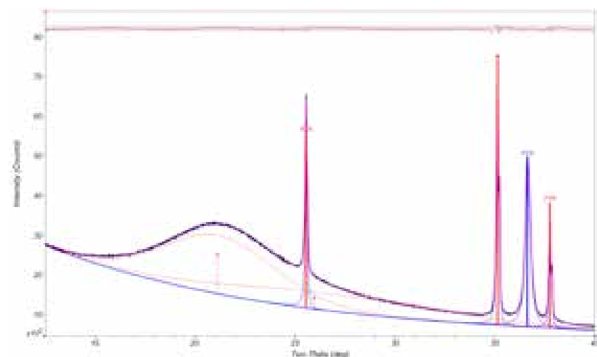

Figure 3: Crystallinity - Amorphous vs. Crystalline

the figure that represents amorphous content in the sample. Using the ratio of peak areas from the sharp peaks versus those from the broad peak(s) we can estimate how much of the sample is crystalline and how much is amorphous. This type of analysis may be used to determine whether a coating deposited on a representative substrate is amorphous or if it has crystallized due to annealing or some other experimental treatment.

Complementary Techniques

XRD is a good complement to bulk analysis techniques such as Glow Discharge Mass Spectrometry (GDMS), Inductively Coupled Plasma- Optical Emission Spectroscopy (ICP-OES) or Mass Spectrometry (ICP-MS), and and X-ray Fluorescence (XRF) Spectroscopy. While these other techniques provide compositional information, XRD provides crystalline information helping to provide a more complete characterization of the material. It should be noted that by itself XRD cannot provide unequivocal compositional or elemental information. XRD can be quite powerful when combined with other techniques used for thin film analysis, such as Rutherford Backscattering Spectroscopy (RBS), Auger Electron Spectroscopy (AES) and X-ray Photoelectron Spectroscopy (XPS). XRD can provide phase, orientation, and other crystalline information to complement the compositional results from RBS, Auger, or XPS. XRD tends to probe much deeper than these techniques, providing more bulk information unless grazing incidence (GI-XRD) is used to enhance the surface sensitivity of the technique.

XRD at EAG

EAG has at least ten XRD systems located within our network of labs. Most of our instruments are equipped with optical modules that can be easily exchanged depending on the particular analysis requirement. This allows us to provide high-quality analysis of powders, coatings, thin films, slurries, fabricated parts, or even high- resolution analysis of epitaxial films. Two systems have microbeam X-ray sources, allowing for the analysis of very small samples or specific locations. These tools also have area detectors allowing for complete texture analyses.

At Eurofins EAG, our XRD team has collectively many decades of XRD expertise. We have a collaborative group coming from different backgrounds, industries and research specialties. With our recent addition of a Small Spot XRD tool along with our extensive instrument base, we offer a wide variety of analytical options that can be tailored specifically to our clients' needs.

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