Powder X-Ray Diffraction (PXRD)

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PXRD is a heavily used analytical technique in multiple scientific fields including metallurgy, pharmaceuticals, dyes, ceramics and many more. The main use for this technique is the fingerprint identification of crystalline materials and the distinction of crystalline materials from amorphous materials.[1,2] Moreover, this technique can be used to determine the crystallite size using the Scherrer equation[3] in which the crystallite size is determined based on the broadening of the diffraction peaks within the diffraction pattern. The most common expression of the Scherrer equation can be seen below:

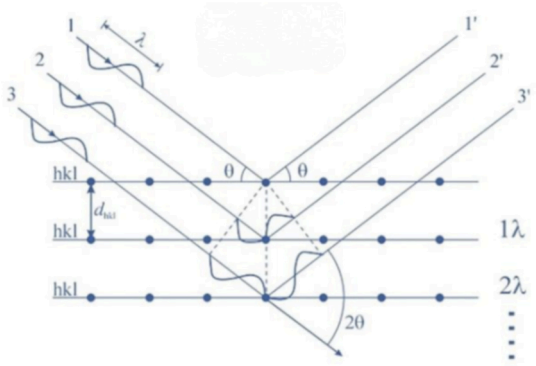
$$D = \frac{K\lambda}{\beta cos\vartheta}$$

where D is the crystallite size, K is a shape factor, λ is the wavelength and θ is the angle of the diffracted peak.

Furthermore, when single crystals of sufficient size and quality cannot be grown, PXRD can be employed to determine the crystal structure and, thus, enabling the elucidation of structural properties when other techniques cannot be used.[4,5] Additionally, the stability of the constituent crystalline phases can be assessed making it the benchmark technique for many industries that are product phase dependant, such as the pharmaceutical, cosmetics and materials industries.

Figure 1: Examples of Diffraction in Nature

But what is diffraction? Diffraction is the interaction of waves with an opening or an object. Given that X-Rays, with a wavelength approximately the size or smaller of a chemical bond, are also waves, they can be employed to characterise the reciprocal structure of crystalline materials. By employing the Bragg's Law,[6] named after the Bragg father and son physicist duo, a correlation between wavelength and interatomic distance is made and thus can give an insight to the structure of a material. The Bragg's law can be seen below along with an example of how the X-rays interact with the different crystalline lattice planes within the material:



$$\lambda = 2dsin\vartheta$$

Bragg's Law

where λ is the X-Ray wavelength (ex, ~1.54 Å for Cu, ~0.71 Å for Mo, ~1.79 Å for Co, etc), d is the interplanar spacing for each family of diffraction planes and θ is the corresponding angle of diffraction.

PXRD is a non-destructive technique as the sample can be recovered post-analysis for potential further characterisation. There are two most commonly used PXRD configurations, the transmission and the reflection modes. In transmission mode, the sample is usually packed into a capillary or placed between two layers of a non diffracting Kapton® film. The X-ray beam passes through (i.e. transmits) the sample. In reflection mode, the sample is usually packed onto a sample holder. The X-ray beam doesn't transmit through the sample but gets diffracted from the sample surface.

Applications of PXRD technique:

- Phase identification
- Crystalline vs amorphous identification
- Crystallite size determination
- Crystal structure determination
- Polymorphic stability
- Phase quantitative analysis
- Time-resolved structure analysis of crystalline and amorphous phases
- Variable-temperature and variable-humidity studies of both crystalline and amorphous phases

References

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