ISSN Print: 2664-6552
ISSN Online: 2664-6560
Impact Factor: RJIF 5.5
IJCRD 2025; 7(1): 63-66
www.chemicaljournal.in
Received: 13-12-2024
Accepted: 18-01-2025

Kalpana Santosh Reddy
Ph.D. Scholar, Associate Professor, Department of Electronics and Communication, PIET, Parul Institute of Homoeopathy & Research, Gujarat, India

Dr. Santosh Reddy
Ph.D. Scholar, Professor & HOD of Homeopathic Pharmacy (PG), Parul Institute of Homoeopathy & Research, Gujarat, India

Corresponding Author:
Kalpana Santosh Reddy
Ph.D. Scholar, Associate Professor, Department of Electronics and Communication, PIET, Parul Institute of Homoeopathy & Research, Gujarat, India

X-ray diffraction: A non-destructive technique for crystalline structure analysis

Kalpana Santosh Reddy and Dr. Santosh Reddy

DOI: https://www.doi.org/10.33545/26646552.2025.v7.i1a.81

Abstract
X-ray Diffraction (XRD) is a powerful and widely used non-destructive analytical technique for characterizing the crystalline structure of materials. This article explores the fundamental principles of XRD, including Bragg's Law, which governs the relationship between diffraction angles and interplanar spacing.

The methodology covers sample preparation, X-ray generation, diffraction process, and data analysis. The operating system of an XRD machine is described in detail, including key components such as the X-ray source, goniometer, sample holder, and detector. The article highlights the merits of XRD, such as its ability to provide accurate phase identification and crystallographic information, as well as its limitations, including challenges with amorphous materials and complex data interpretation. Real-world applications in pharmaceuticals, material science, geology, nanotechnology, and archaeology are presented, demonstrating XRD's versatility.

Keywords: X-ray diffraction (XRD), crystallography, Bragg’s law, phase identification, non-destructive testing, material characterization, powder XRD, structural analysis

Introduction

An X-Ray Diffraction (X-RD) machine is an analytical instrument used to study the crystalline structure of materials. It operates based on the principle of X-ray diffraction, where X-rays interact with the atomic lattice of a material, producing a diffraction pattern that can be analysed to determine various structural properties.

Principle of Operation The working of an X-RD machine is based on Bragg’s Law:

Nλ=2dsin⁡θn\lambda = 2d \sin\thetanλ=2dsinθ

Where:

- nnn is an integer (Order of reflection)
- λ\lambdaλ is the wavelength of the X-rays
- ddd is the interplanar spacing of the crystal lattice
- θ\thetaθ is the angle of incidence of the X-ray beam

When a monochromatic X-ray beam is directed at a crystalline sample, it gets scattered by atomic planes. Constructive interference occurs when the scattered waves satisfy Bragg's Law, leading to a diffraction pattern characteristic of the sample's structure.

Components of an X-RD Machine

1. **X-ray Source:** Generates X-rays, usually using a Cu-Kα or Mo-Kα source.
2. **Goniometer:** A rotating sample holder that allows precise positioning of the sample.
3. **Sample Stage:** Holds the sample in place for measurement.
4. **Detector:** Captures the diffracted X-rays and converts them into electrical signals.
5. **Data Processing System:** Analyses diffraction patterns to determine crystal structure, phase composition, and other material properties.

Fig 1: XRD machine

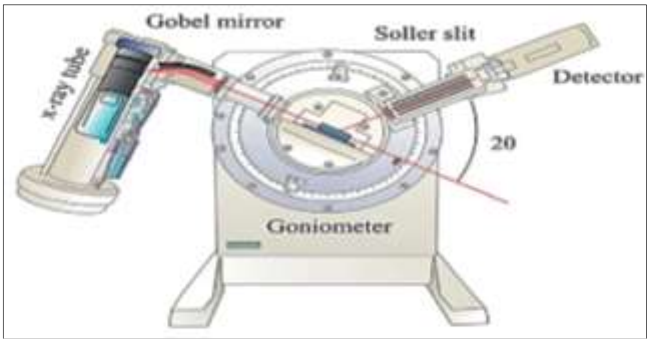

Fig 2: Schematic representation of XRD technique

Applications of X-RD

- **Material Science:** Identification of crystalline phases in metals, ceramics, and polymers.
- **Pharmaceuticals:** Characterization of drug polymorphs.
- **Geology & Mineralogy:** Analysis of rock and mineral compositions.
- **Nanotechnology:** Determination of nanoparticle size and structure.
- **Forensics:** Identification of unknown substances in forensic investigations.

Advantages of X-RD

- Non-destructive technique.
- Highly accurate for crystalline materials.
- Can analyse small quantities of material.

Limitations of X-RD

- Cannot be used for amorphous (Non-crystalline) materials.
- Requires careful sample preparation.
- Limited penetration depth.

Aim and Objectives

Aim

The aim of this article is to provide a comprehensive overview of X-ray Diffraction (XRD) as a non-destructive technique for crystalline structure analysis, covering its principles, methodology, operating system, and applications in various scientific and industrial fields.

Objectives

- To explain the fundamental principles and working mechanism of XRD.
- To outline the methodology involved in XRD analysis, including sample preparation, diffraction, and data interpretation.
- To describe the operating system of an XRD machine, highlighting its key components.
- To present the advantages and limitations of XRD.
- To provide real-world applications demonstrating the practical utility of XRD in material science, pharmaceuticals, geology, and nanotechnology.

Methodology: X-ray Diffraction analysis involves a series of systematic steps that enable the accurate characterization of crystalline materials.

Sample Preparation

- The material is ground into a fine powder to ensure homogeneity and consistent diffraction patterns.
- For thin films or single crystals, the sample is mounted on a specialized stage.
- Proper sample mounting reduces errors and enhances accuracy.

X-ray Generation

- The XRD machine generates X-rays using a sealed X-ray tube or a rotating anode.
- Electrons strike a metal target (commonly copper or molybdenum), emitting characteristic X-rays.
- The X-ray beam is filtered and collimated before interacting with the sample.

Diffraction Process

- The X-rays interact with the crystalline planes of the sample, producing a diffraction pattern.
- According to Bragg's Law, the relationship between the wavelength, diffraction angle, and interplanar spacing is given by:

Nλ=2dsin θn\lambda = 2d\sin\thetanλ=2dsinθ

Where:

- nnn = Order of reflection.
- λ\lambdaλ = X-ray wavelength.
- ddd = Interplanar spacing.
- θ\thetaθ = Diffraction angle.

Data Collection and Analysis

- The detector records the diffraction pattern as a series of peaks.
- The position and intensity of the peaks correspond to the crystal structure and phase composition.
- Advanced software (e.g., X'Pert High Score, Jade, or Bruker TOPAS) is used for:

1. Phase identification (Matching diffraction patterns with reference databases).
2. Quantitative phase analysis.
3. Crystallite size estimation using the Scherer equation.

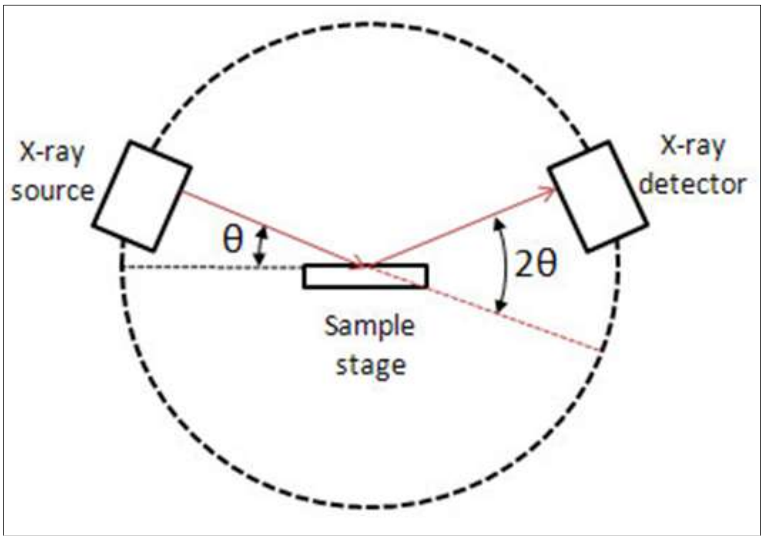

Fig 3: Schematic representation of XRD working principle

Operating System of an XRD Machine: An XRD machine consists of core components that work together to generate, detect, and interpret X-ray diffraction patterns.

X-ray Source

- Generates the X-ray beam using a sealed tube or rotating anode.
- Common target materials include:
- Copper (Cu) → Wavelength: 1.54 Å (Most common for material analysis)
- Molybdenum (Mo) → Wavelength: 0.71 Å (for high-resolution studies).

Goniometer

- A precision instrument that rotates the sample and detector.
- Controls the diffraction angle 2θ2\theta2θ and ensures accurate alignment.

Sample Holder/Stage

- Holds the sample in a fixed position.
- Specialized stages are used for thin films, powders, or single crystals.

Detector

- Captures the diffracted X-rays and records the diffraction pattern.
- Modern detectors (e.g., silicon strip detectors, CCD detectors) provide high sensitivity and resolution.

Control and Analysis Software

- Instrument Control Software: Automates XRD machine operations.
- Data Analysis Software: Processes diffraction patterns for phase identification and structural analysis.

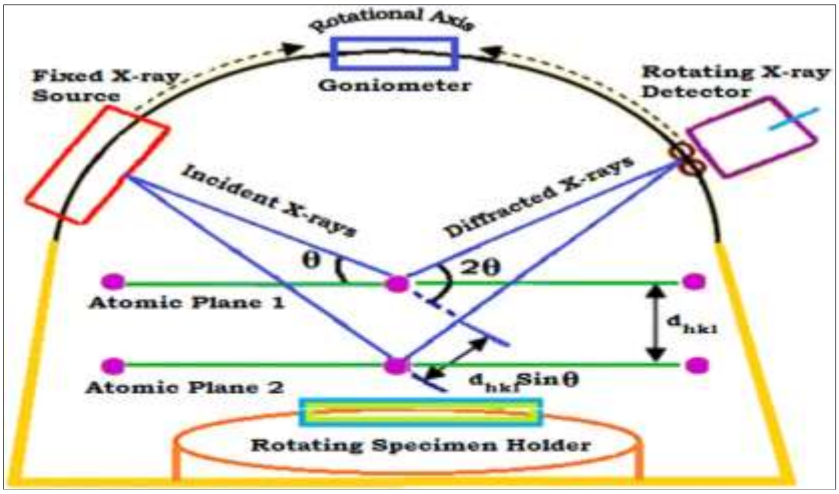

Fig 4: Schematic representation of Bragg banteno configuration

Conflict of interest

No such.

Acknowledgement

Author would like to Thanks to Parul University, PIET and Dean of the Faculty.

References

1. Cullity BD, Stock SR. Elements of X-ray Diffraction. 3rd ed. Upper Saddle River (NJ): Addison-Wesley; c2001.
2. Klug HP, Alexander LE. X-ray Diffraction Procedures: For Polycrystalline and Amorphous Materials. 2nd ed. New York (NY): John Wiley & Sons; c1974.

3. Pecharsky VK, Zavalij PY. Fundamentals of Powder Diffraction and Structural Characterization of Materials. 2nd ed. New York (NY): Springer; c2009.
4. International Centre for Diffraction Data. Powder Diffraction File. Release 2020. Newtown Square (PA): ICDD; c2020.
5. Jenkins R, Snyder RL. Introduction to X-ray Powder Diffractometry. New York (NY): Wiley-Interscience; c1996.
6. Young RA. The Rietveld Method. New York (NY): Oxford University Press; c1995.
7. Warren BE. X-ray Diffraction. New York (NY): Dover Publications; c1990.
8. Buerger MJ. X-ray Crystallography: An Introduction to the Theory and Practice of Single-Crystal Structure Analysis. New York (NY): John Wiley & Sons; c1942.
9. Bish DL, Post JE. Quantitative Mineral Analysis Using the Rietveld Method. Am Mineral. 1993;78(9-10):932-940.
10. Cheary RW, Coelho A. Fundamental Parameters Line Profile Fitting in Powder Diffraction. J Appl Crystallogr. 1992;25(2):109-121.