HACETTEPE UNIVERSITY DEPARTMENT OF CHEMICAL ENGINEERING

X-Ray Diffraction (XRD)

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Outline

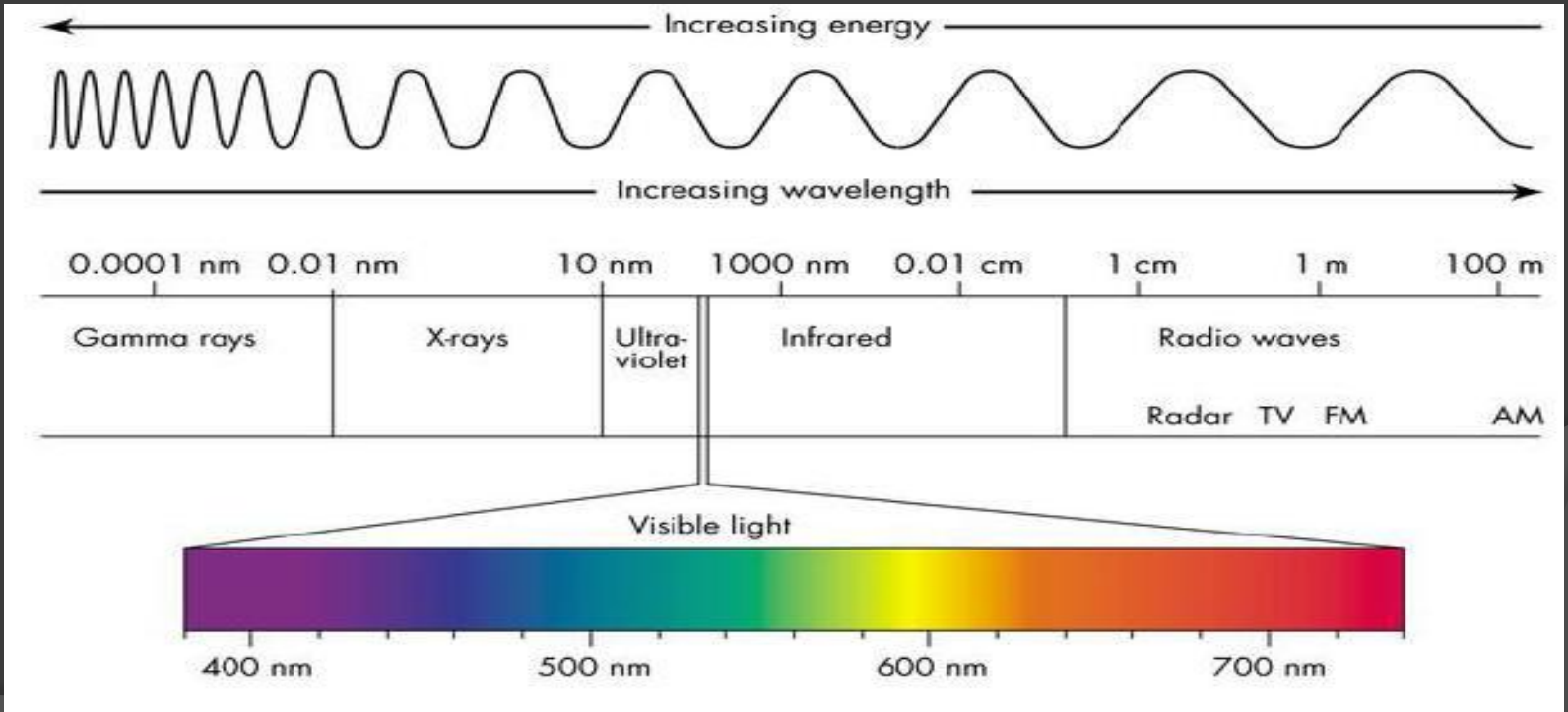
- Introduction
- What is X-ray & X-ray Diffraction
- History
- Components and Working Principles
- Power XRD and Single Crystal XRD
- Aplications
- Safety
- Summary

Introduction

- Motivation
  - X-ray diffraction is used to obtain structural information about crystalline solids.
  - Useful in biochemistry to solve the 3D structures of complex bio-molecules.
  - Bridge between physics, chemistry, and biology.
- X-ray diffraction is important for
  - Solid-state physics
  - Biophysics
  - Medical physics
  - Chemistry and Biochemistry

What is X-ray

- Beams of electromagnetic radiation
  * smaller wavelength than visible light,
  *higher energy
  *more penetrative



History of X-Ray Diffraction

1895 X-rays discovered by Roentgen 

1914 First diffraction pattern of a crystal made by Knipping and von Laue 

1915 Theory to determine crystal structure from diffraction pattern developed by Bragg. 

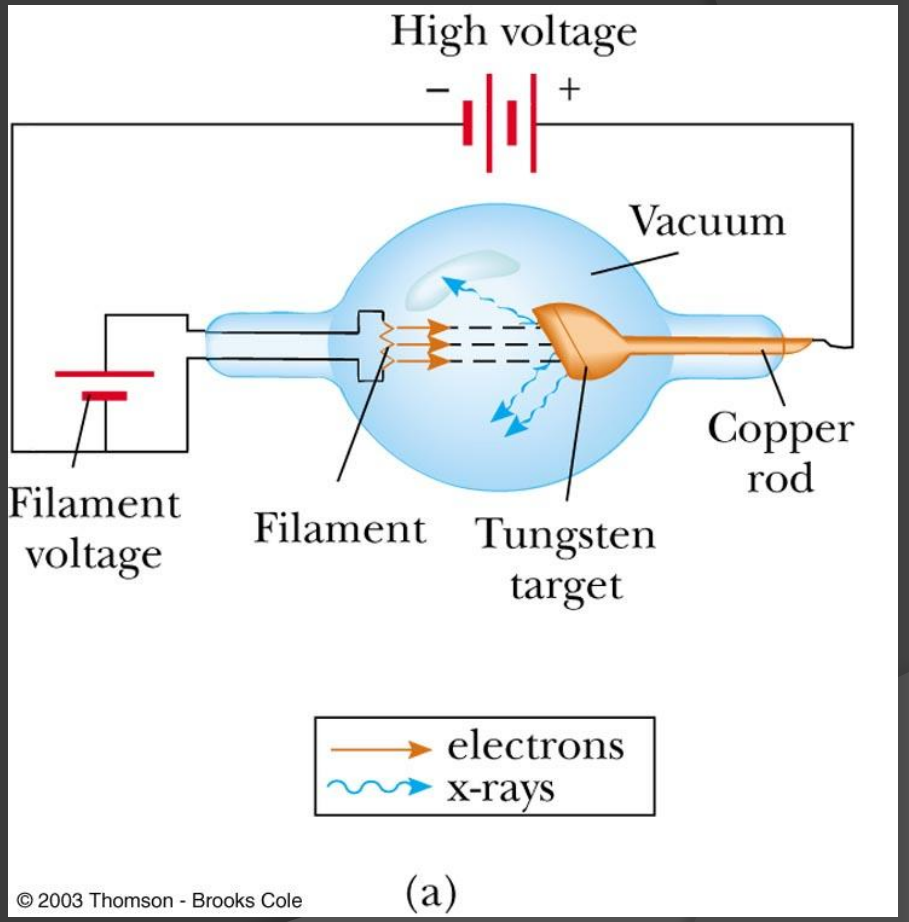
1953 DNA structure solved by Watson and Crick 

Now Diffraction improved by computer technology; methods used to determine atomic structures and in medical applications

The first X-ray

X-ray Production

- When high energy electrons strike an anode in a sealed vacuum, x-rays are generated. Anodes are often made of copper, iron or molybdenum.
- X-rays are electromagnetic radiation.
- They have enough energy to cause ionization.



What is X-ray Diffraction (XRD)

- Most useful in the characterisation of crystalline materials; Ceramics, metals, intermetallics, minerals, inorganic compounds
- rapid and nondestructive techniques
- Provide information on unit cell dimension

Components

- X-ray source
- Device for restricting wavelength range “goniometer”
- Sample holder
- Radiation detector
- Signal processor and readout

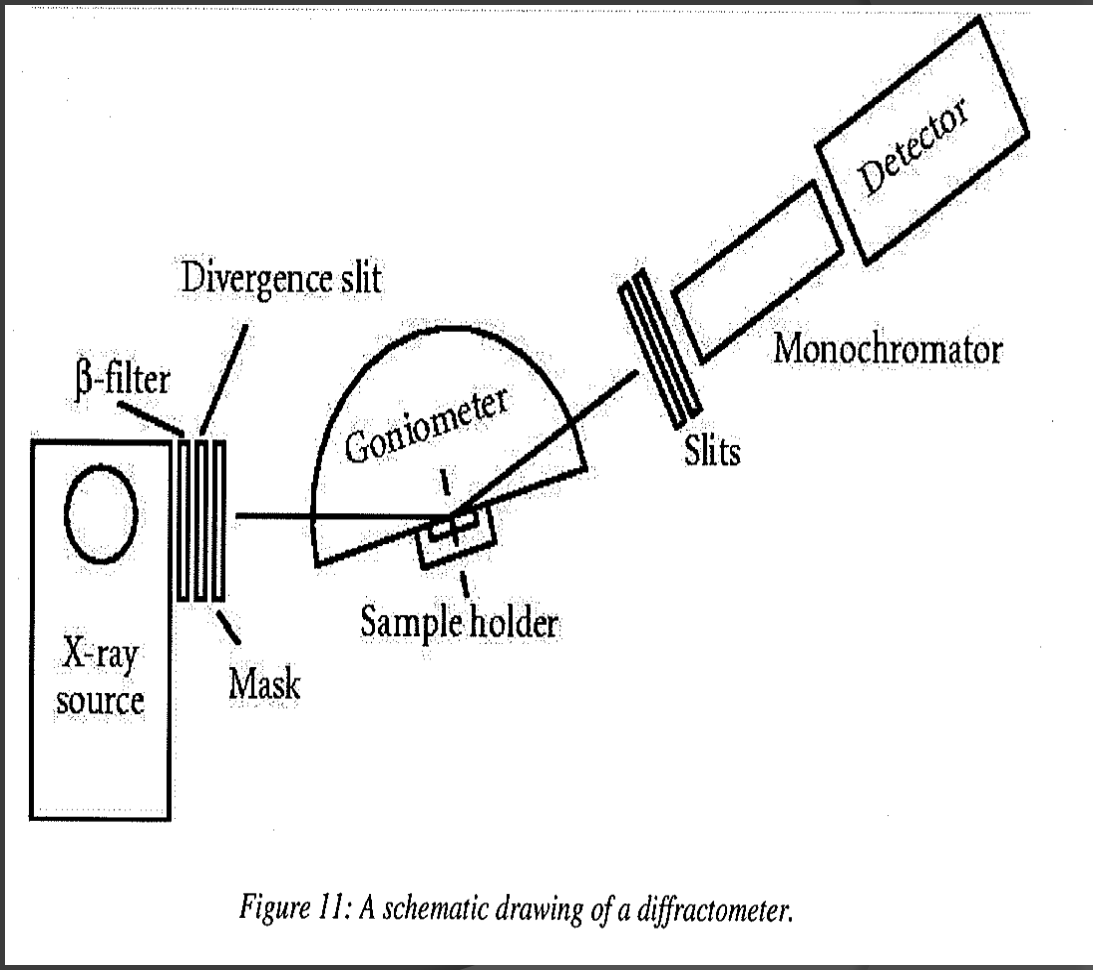
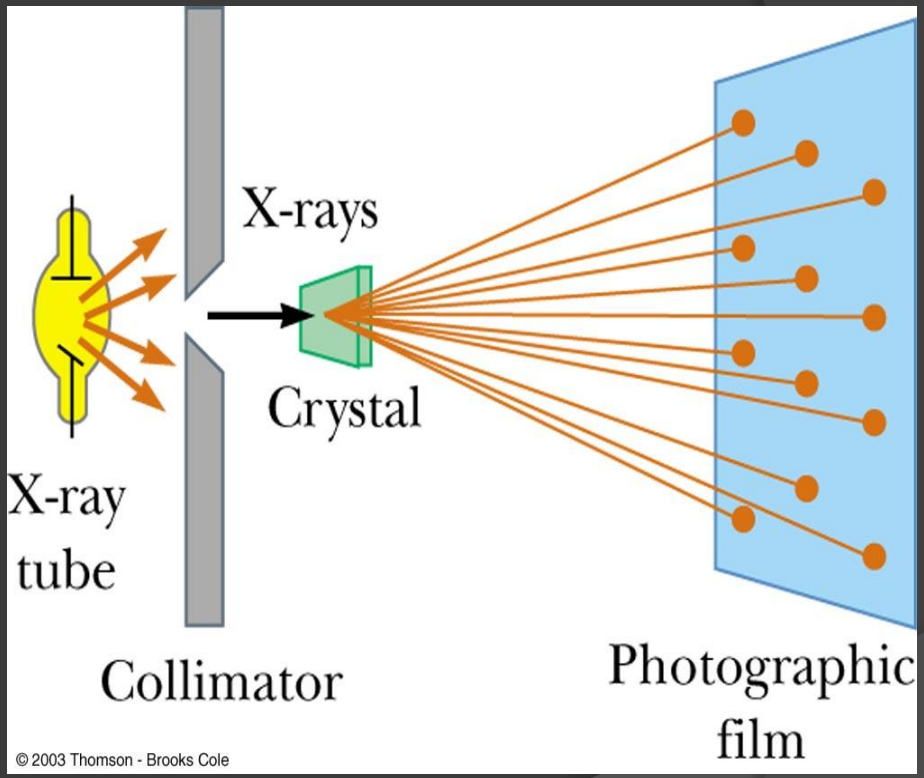


Figure 11: A schematic drawing of a diffractometer.

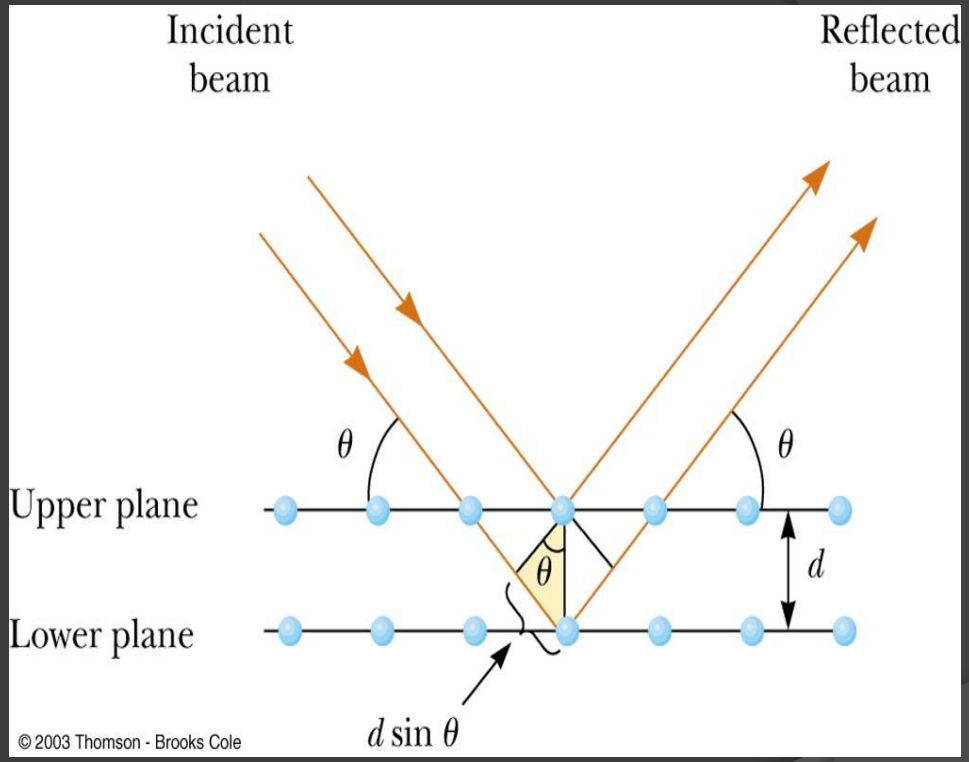
How XRD works

- A continuous beam of X-rays is incident on the crystal
- The diffracted radiation is very intense in certain directions
  - These directions correspond to constructive interference from waves reflected from the layers of the crystal
- The diffraction pattern is detected by photographic film



How Diffraction Works: Bragg's Law

- The beam reflected from the lower surface travels farther than the one reflected from the upper surface
- If the path difference equals some integral multiple of the wavelength, constructive interference occurs
- *Bragg's Law* gives the conditions for constructive interference



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

How Diffraction Works: Schematic

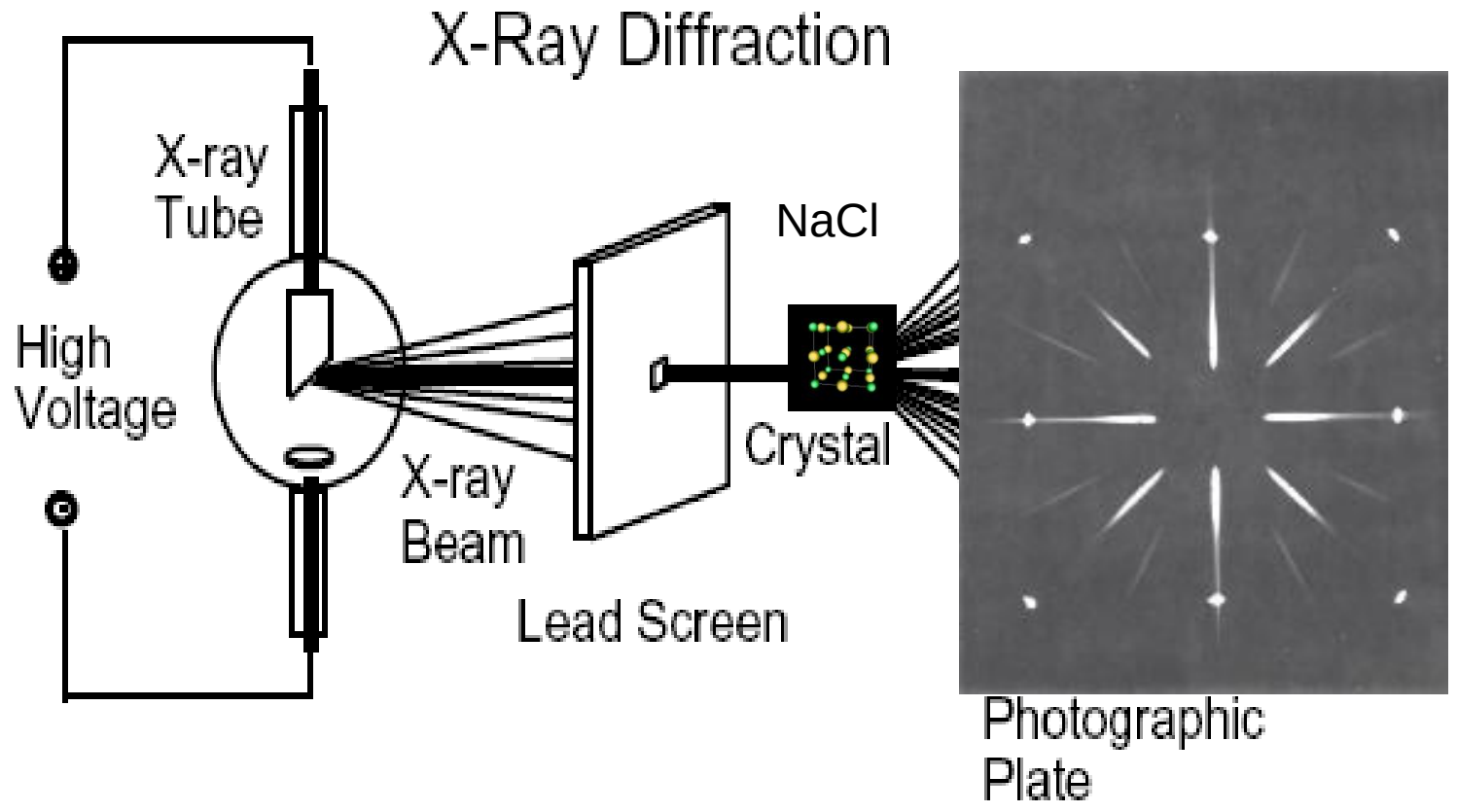
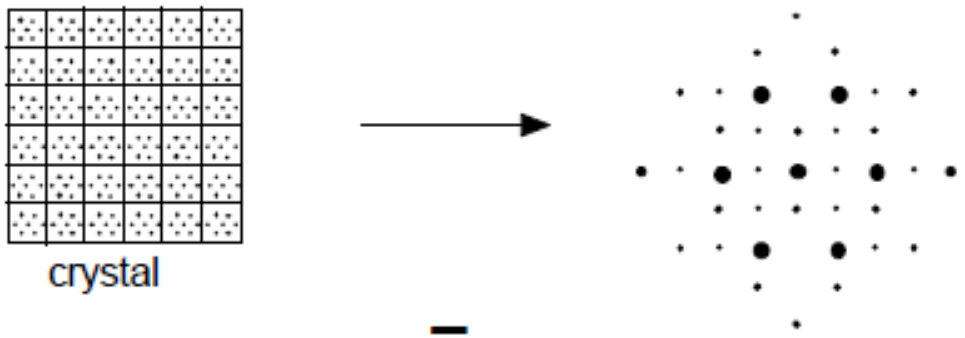


Figure 2. A schematic of X-ray diffraction.

http://mrsec.wisc.edu/edetc/modules/xray/X-raystm.pdf

Diffraction from a crystal



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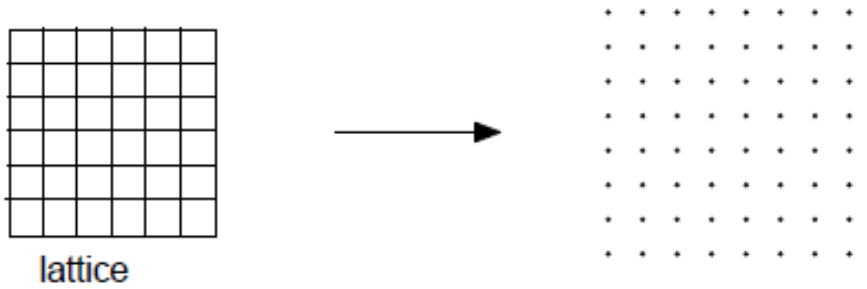
Diffraction from a lattice



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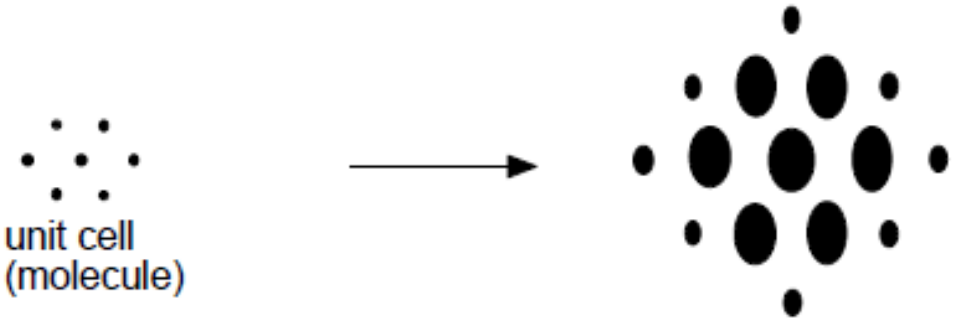
Diffraction from a unit cell



Single Crystal X-ray Diffraction

- Used to determine
  - crystal structure
  - orientation
  - degree of crystalline perfection/imperfections (twinning, mozaicity, etc.)
- Sample is illuminated with monochromatic radiation
  - Easier to index and solve the crystal structure because it diffraction peak is uniquely resolved

X-ray Powder Diffraction

- More appropriately called polycrystalline X-ray diffraction, because it can also be used for sintered samples, metal foils, coatings and films, finished parts, etc.
- Used to determine
  - phase composition (commonly called phase ID)- what phases are present?
  - quantitative phase analysis- how much of each phase is present?
  - unit cell lattice parameters, crystal structure
  - average crystallite size of nanocrystalline samples
  - crystallite microstrain and texture
  - residual stress (really residual strain)

Applications of X-Ray Diffraction

- Determination of Crystal structure
- Phase identification / transition
- Grain size / micro-strain
- Texture/stress(i.e.polymer , fiber)
- Determination of thin film composition
- Industry Identification of archeological materials

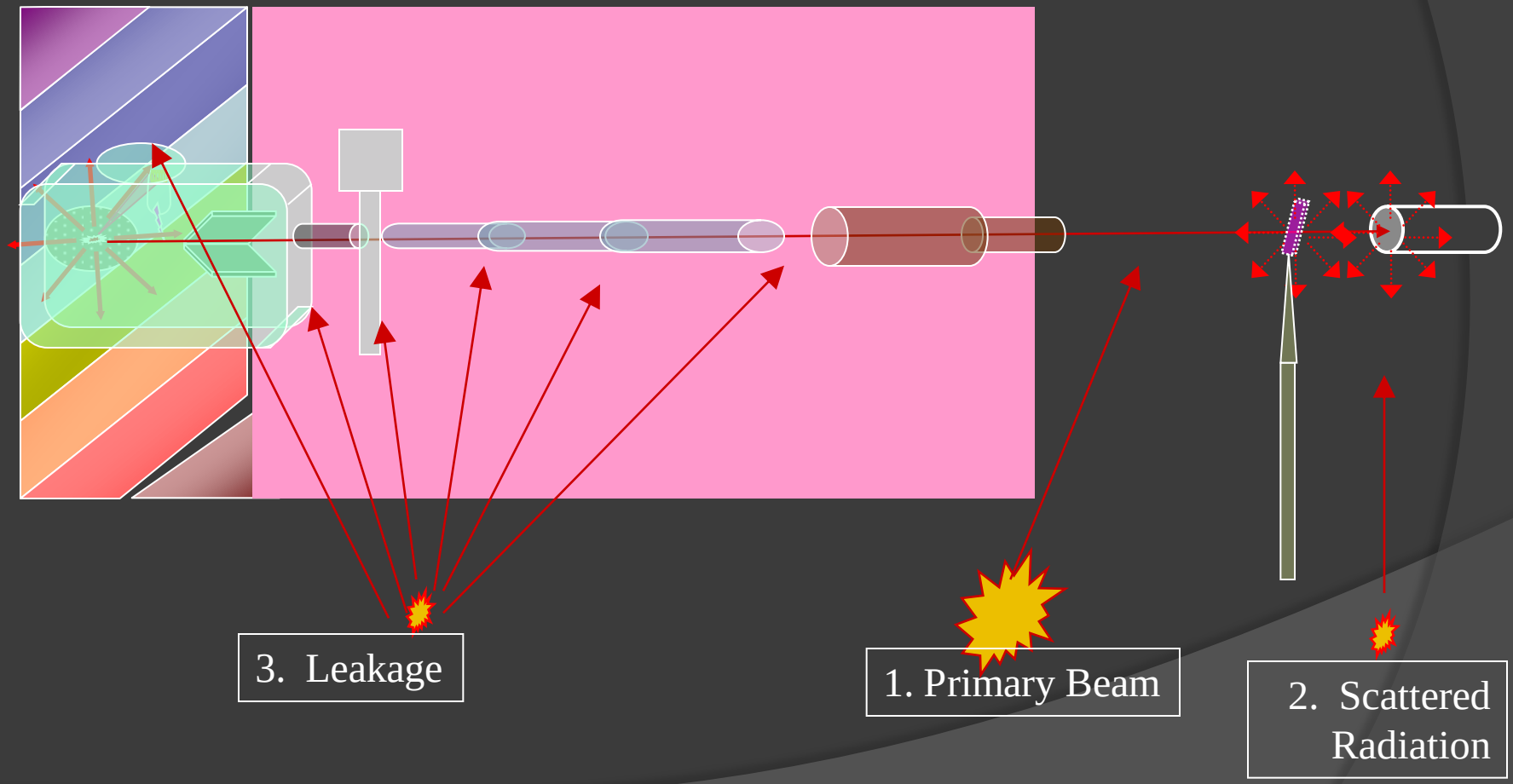
Advantages of XRD

- Fast identification of materials,
- Easy sample preparation,
- Computer-aided material identification,
- Large library of known crystalline structures.

Safety in XRD

- Exposure types
  - Short-term high-dose
  - Long-term low-dose
- Invisible, odorless, colorless
  (most exposures undetectable)
- Lab users must understand radiation safety issues and pass an exam to use lab
- Safeguards present in lab do not substitute for knowledge and following safe procedures

What are the dangerous areas?



Summary & Conclusion

- X-ray diffraction is a technique for analyzing structures of biological molecules
- X-ray beam hits a crystal, scattering the beam in a manner characterized by the atomic structure
- Even complex structures can be analyzed by x-ray diffraction, such as DNA and proteins
- This will provide useful in the future for combining knowledge from physics, chemistry, and biology

References

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- *prism.mit.edu/**xray**/Basicsof**XRD**.ppt*

Thank you