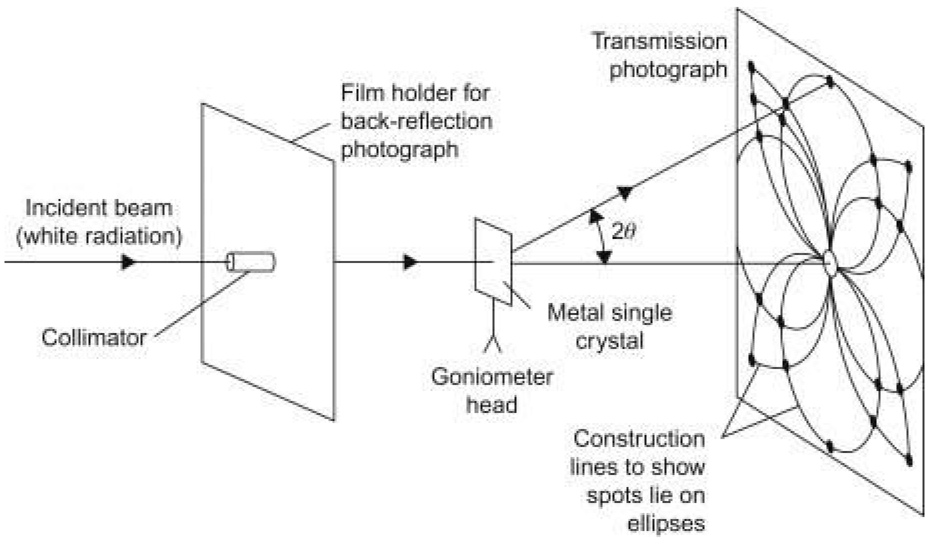
Project 1: Laue diffraction of X-rays and electrons HB

- Design an optical experiment to simulate Laue diffraction.
- Using an x-ray diffractometer set up an experiment to record a Laue diagram of a monocrystal.
- Bragg's theory of diffraction
- Evaluating a Laue diagram
- X-ray Film / X-ray camera:

Research:

X-ray diffraction:

Laue Diagram:



Experiment

https://www.youtube.com/watch?v=suVNYD1nCm4

https://www.youtube.com/watch?v=5NHhZ5Zk-oQ

Powder pattern

https://www.youtube.com/watch?v=ZYzKd2qMn1o

X-ray Diffraction

https://www.youtube.com/watch?v=lwV5WCBh9a0

Seeing things in a different light - How X-ray crystallography revealed the structure of everything

https://www.youtube.com/watch?v=gBxZVF3s4cU&t=746s

What is X-ray Diffraction?

https://www.youtube.com/watch?v=QHMzFUo0NL8

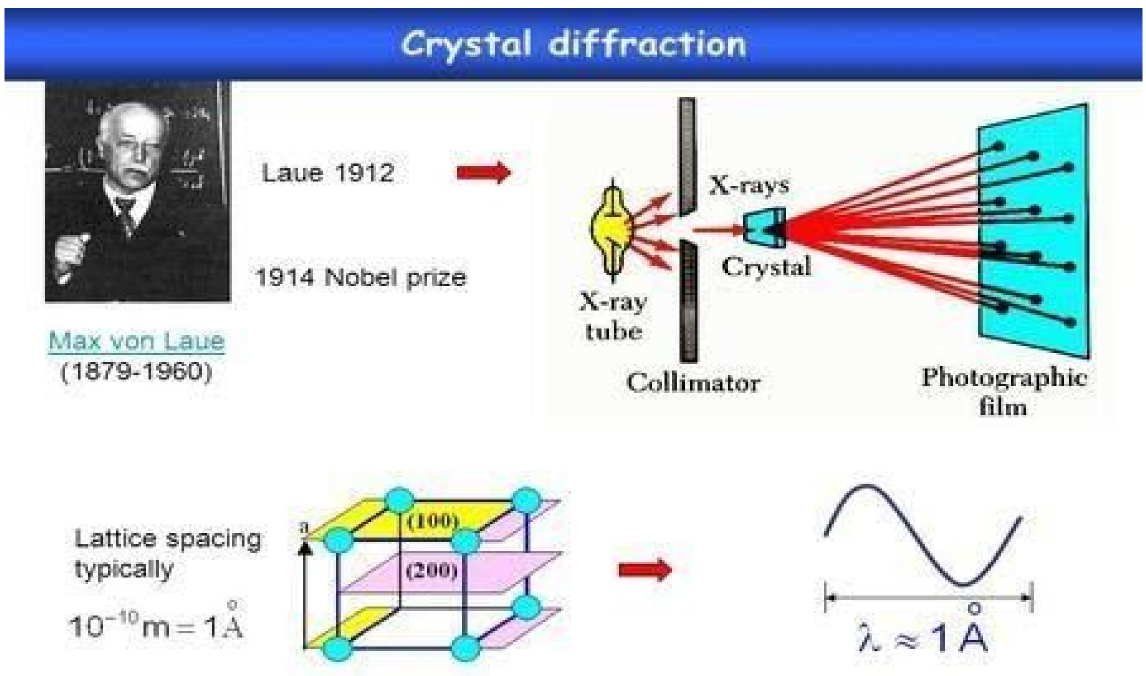
Optical simulation of debyescherrer

Example of monocrystals:

Need well defined polycrystalline material,

Individual particle size of material should be below 45 microns

X-ray diffraction techniques:



Bragg’s theory of diffraction

The structures of crystals and molecules are often being identified using x-ray diffraction studies, which are explained by Bragg’s Law. The law explains the relationship between an x-ray light shooting into and its reflection off from crystal surface.

Bragg's Law refers to the simple equation:

(eq 1) $n\lambda = 2d \sin\theta$

derived by the English physicists Sir W.H. Bragg and his son Sir W.L. Bragg in 1913 to explain why the cleavage faces of crystals appear to reflect X-ray beams at certain angles of incidence (theta, θ). The variable *d* is the distance between atomic layers in a crystal, and the variable lambda λ is the **wavelength** of the incident X-ray beam (see applet); n is an integer

This observation is an example of X-ray **wave interference** (Roentgenstrahlinterferenzen), commonly known as X-ray diffraction (XRD), and was direct evidence for the periodic atomic structure of crystals postulated for several centuries. The Braggs were awarded the Nobel Prize in physics in 1915 for their work in determining crystal structures beginning with NaCl, ZnS and diamond. Although Bragg's law was used to explain the interference pattern of X-rays scattered by crystals, diffraction has been developed to study the structure of all states of matter with any beam, e.g., ions, electrons, neutrons, and protons, with a wavelength similar to the distance between the atomic or molecular structures of interest.

Deriving Bragg's Law:

Bragg's Law can easily be derived by considering the conditions necessary to make the phases of the beams coincide when the incident angle equals and reflecting angle. The rays of the incident beam are always in phase and parallel up to the point at which the top beam strikes the top layer at atom z (Fig. 1). The second beam continues to the next layer where it is scattered by atom B. The second beam must travel the extra distance AB + BC if the two beams are to continue traveling adjacent and parallel. This extra distance must be an integral (n) multiple of the wavelength (λ) for the phases of the two beams to be the same:

(eq 2) $n\lambda = AB + BC$.

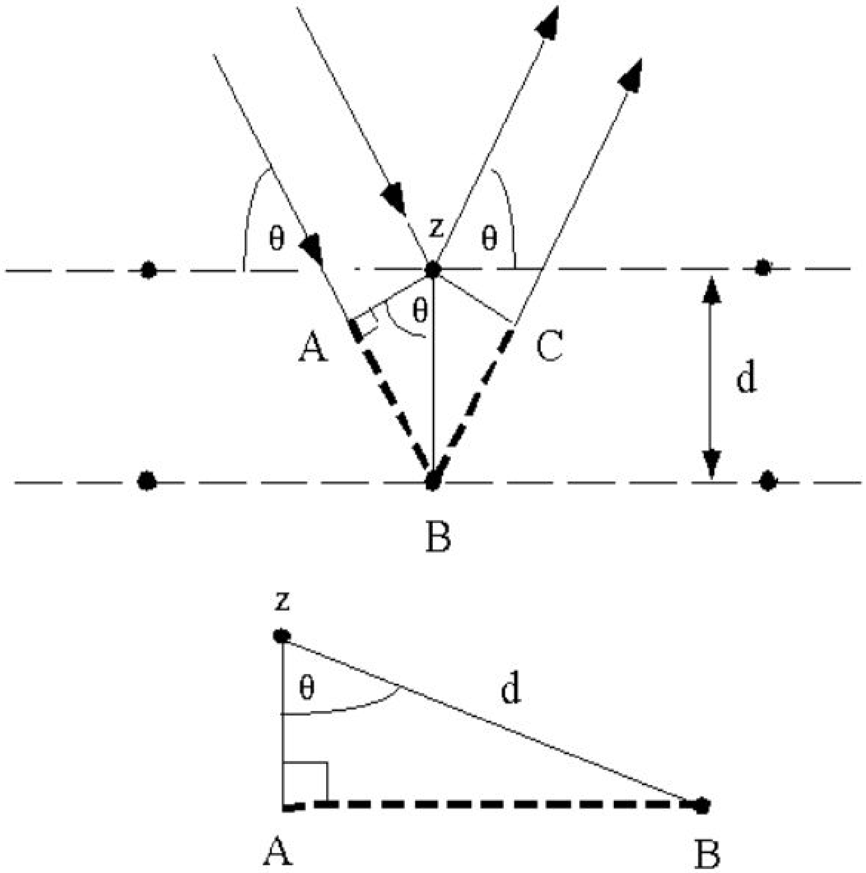


Fig. 1 Deriving Bragg's Law using the reflection geometry and applying trigonometry. The lower beam must travel the extra distance (AB + BC) to continue traveling parallel and adjacent to the top beam.

Recognizing d as the hypotenuse of the right triangle Abz, we can use trigonometry to relate d and θ to the distance (AB + BC). The distance AB is opposite θ so,

(eq 3) $AB = d \sin\theta$.

Because AB = BC eq. (2) becomes,

(eq 4) $n\lambda = 2AB$

Substituting eq. (3) in eq. (4) we have,

(eq 1) $n\lambda = 2\, d \sin\theta$

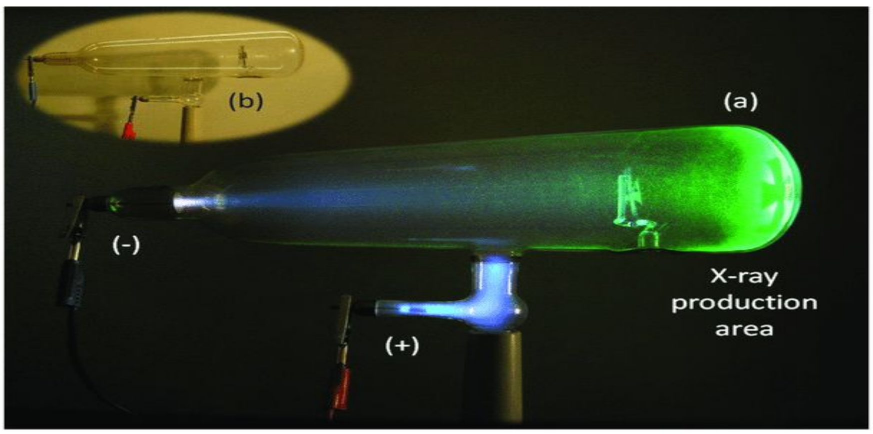
and Bragg's Law has been derived. The location of the surface does not change the derivation of Bragg's Law.

Players in the Discovery of X-ray Diffraction

Friedrich and Knipping first observed Roentgenstrahlinterferenzen in 1912 after a hint from their research advisor, Max von Laue, at the University of Munich. Bragg's Law greatly simplified von Laue's description of X-ray interference. The Braggs used crystals in the reflection geometry to analyze the intensity and wavelengths of X-rays (spectra) generated by different materials. Their apparatus for characterizing X-ray spectra was the Bragg spectrometer.

Laue knew that X-rays had wavelengths on the order of 1 Å. After learning that Paul Ewald's optical theories had approximated the distance between atoms in a crystal by the same length, Laue postulated that X-rays would diffract, by analogy to the diffraction of light from small periodic scratches drawn on a solid surface (an optical diffraction grating). In 1918 Ewald constructed a theory, in a form similar to his optical theory, quantitatively explaining the fundamental physical interactions associated with XRD. Elements of Ewald's eloquent theory continue to be useful for many applications in physics.

crookes tube



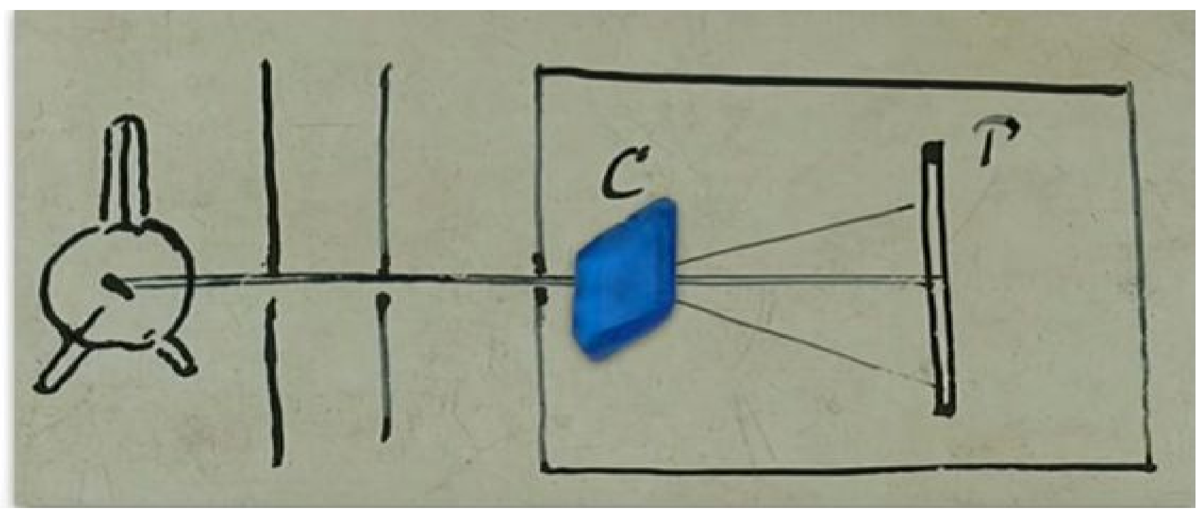
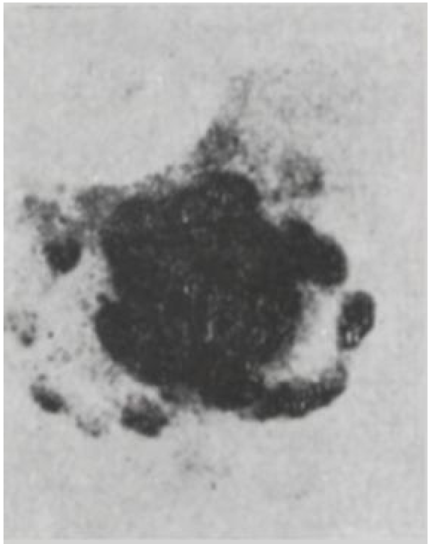
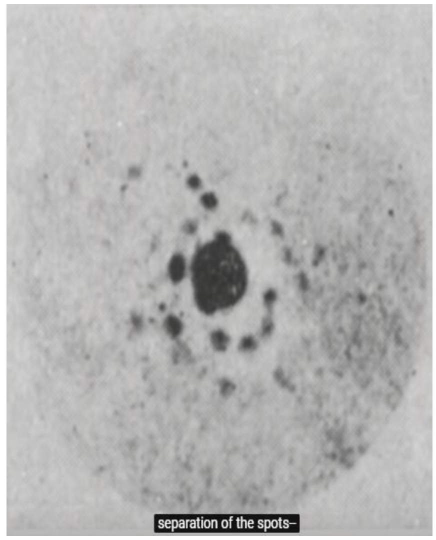


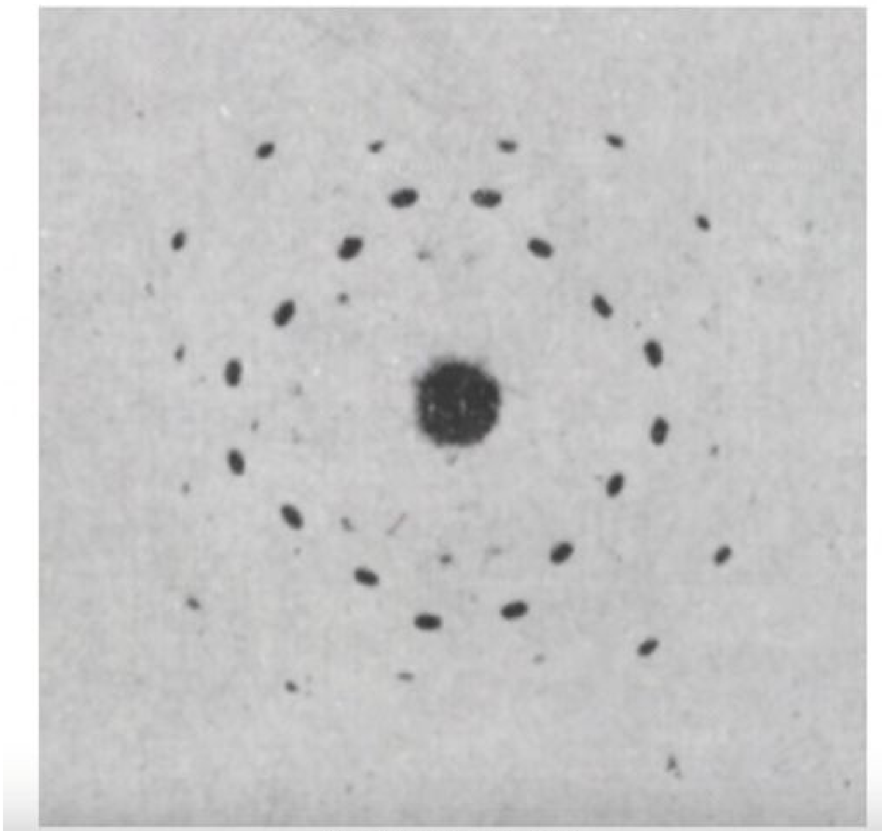
image produced in experiment, second image is after they moved photographic plate back a bit





Copper sulfate:

Diffraction from ZincBlende (ZnS)



(Friedrich and Knipping)

Hints at four fold symmetery, suggests cube inside crystal

The Bragg's Law

n λ = $d_{hkl}\, sin\, \Theta$ + d_{hkl} sin Θ

= 2. d_{hkl} sin Θ

For n = order of reflection

λ = wavelength

For crystal structures that have cubical symmetry

d_{hkl} = $a/\sqrt{(h^2 + k^2 + I^2)}$

Diffraction occurs whenever Bragg's Law is satisfied. With monochromatic radiation, an arbitrary setting of a single crystal in an x-ray beam will generally produce any diffracted beams. there would therefore be very little info in a single crystal diffraction pattern from using monochromatic radiation.

This problem can be overcome by continuously varying I or q, over a range of values to satisfy Bragg's law. Practically this is done by:

- Using a range of x-ray wavelengths (i.e. white radiation), or
- by rotating the crystal or, using a powder or polycrystalline specimen.

XRD Methods:

Rotating crystal method

- In the rotating crystal method, a single crystal is mounted with an axis normal to a monochromatic x-ray beam. A cylindrical film is placed around it and the crystal is rotated about the chosen axis.
- As the crystal rotates, set of lattice planes will at some point make the correct Bragg angle for the monochromatic incident beam, and at that point a diffracted beam will be formed
- The reflected beams are located on the surface of imaginary cones. When the film is laid out flat, the diffraction spots lie on horizontal lines

Laue method:

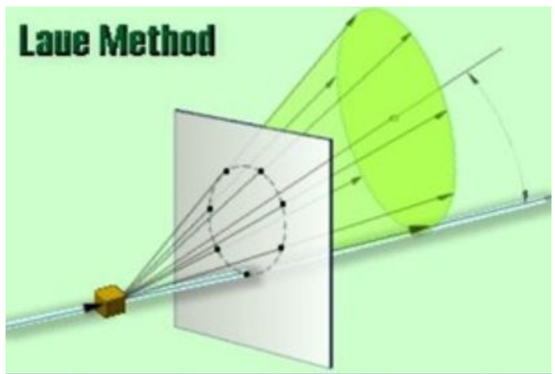
The laue method is mainly used to determine the orientation of large single crystals. White radiation is reflected from, or transmitted through, a fixed crystal.

The diffracted beams form arrays of spots, that lie on curves on the film. The bragg angle is fixed for every set of planes in the crystal. Each set of planes picks out and diffracts the particular wavelength from the white radiation that satisfies the Bragg law for the values of d and q involved. Each curve therefore corresponds to a different wavelength. The spots lying on any one curve are reflections from planes belonging to one zone. Laue reflections from planes of the same zone all lie on the surface of an imaginary cone whose axis is the zone axis.

There are two practical variants of the laue method, the back reflection and the transmission Laue method.

Back reflection Laue

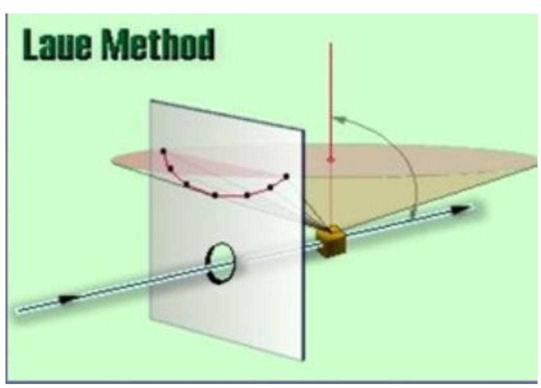
In the back reflection method, the film is placed between the x-ray source and the crystal. the beams which are diffracted in a backward direction are recorded. One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an hyperbola.



Transmission Laue

In the transmission Laue method, the film is placed behind the crystal to record beams which are transmitted through the crystal.

One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an ellipse.



Powder method:

The powder method is used to determine the value of the lattice parameters accurately. Lattice parameters are the magnitudes of the unit vectors a, b and c which define the unit cell for the crystal.

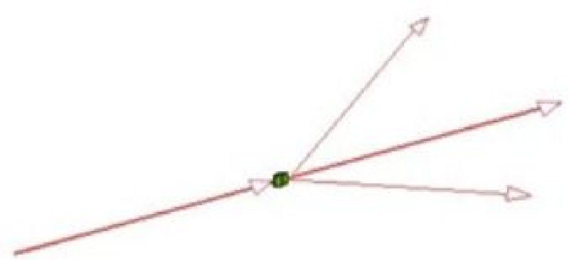
If a monochromatic x-ray beam is directed at a single crystal, then only one or two diffracted beams may result. If the sample consists of some tens of randomly orientated single crystals, the diffracted beams are seen to lie on the surface of several cones. The cones may emerge in all directions, forwards and backwards.

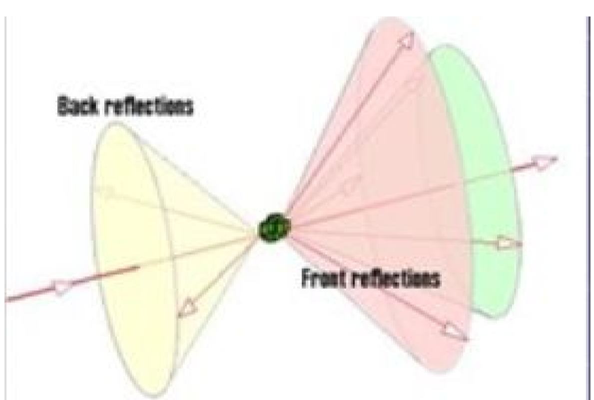
A sample of some hundreds of crystals (i.e. a powdered sample) show that the diffracted beams form continuous cones.

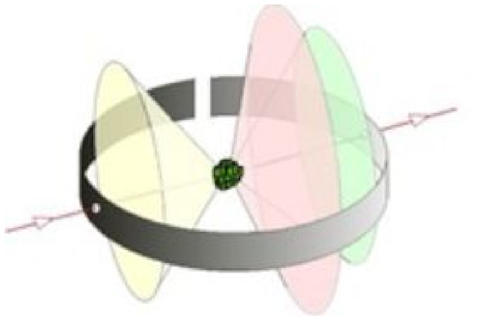
A circle of film is used to record the diffraction pattern as shown. Each cone intersect the film giving lines. The lines are seen as arcs in the film.

For every set of crystal planes, by chance one or more crystals will be in the correct orientation to give the correct Bragg angle to satisfy Bragg's equation. Every crystal plane is thus capable of diffraction. Each diffraction line is made up of a large number of small spots, each diffraction line is made up of a large number of small spots, each from a separate crystal. Each spot is so small as to give the appearance of a continuous line. If the crystal is not ground finely enough, the diffraction lines appear speckled.

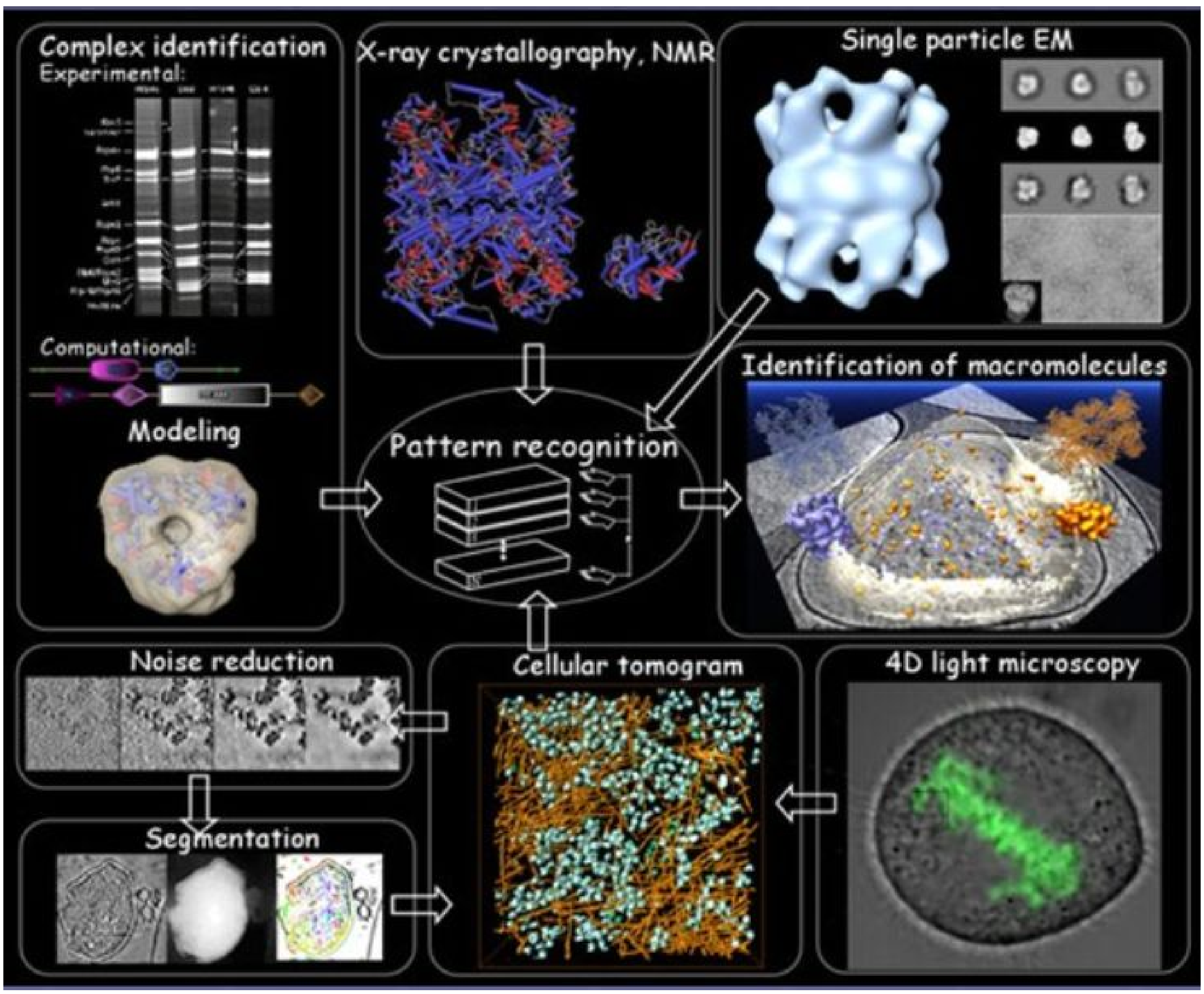
This arrangement is achieved practically in the debye scherrer.







Steps of analysis



References:

Sciencedirect.com. (2020). *X-Ray Diffraction - an overview | ScienceDirect Topics*. [online] Available at: https://www.sciencedirect.com/topics/materials-science/x-ray-diffraction [Accessed 27 Jan. 2020].

X-ray diffraction B.E Warren

http://skuld.bmsc.washington.edu/~merritt/bc530/bragg/

Plan:

Try to simulate, demonstrate the concept of x-ray diffraction (using lasers,

Thn use