

INDIAN INSTITUTE OF TECHNOLOGY ROORKEE
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NPTEL ONLINE CERTIFICATION COURSE
Structural Analysis of Nanomaterials
Lecture- 15
Orientation of Single Crystals
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Hello, our new lecture is on orientations of single crystals. So, before going to start just let us know that why we need the study about the orientations, identifications or maybe why you need this information's. So, as you know that many of the properties are poly crystalline materials have been explained by studies of isolated single crystals because single crystals are an isotropic in nature which requires the accurate knowledge of the orientations of the single crystal test specimen in order that

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Need for Orientation Identification:

- Many of the properties of polycrystalline materials have been explained by studies of isolated single crystals.
- Because single crystals are anisotropic, it requires accurate knowledge of the orientation of the single crystal test specimen in order that measurements may be made along known crystallographic directions or planes.
- By varying the crystal orientation, data on the property measured (e.g., yield strength, electrical resistivity, corrosion rate) is obtained as a function of crystal orientation.
- There is also an increasing production of single crystals, not for research studies, but for use as such in various devices, mainly electrical, optical and magnetic.
- Example: Si crystals for CPUs and RAM in computers and for semiconductor based consumer products; single-crystal nickel-based super alloy turbine blades which have very high creep resistance.
- These crystals must all be produced with particular orientations.



Measurements maybe made along known crystallographic directions or maybe the planes. By varying the crystal orientations, data on the property measured like yield strength, electrical resistivity, corrosion etc. is obtained as a function of crystal orientations. There is also an increasing production of single crystals as we know, not for research studies but for use as such in various devices mainly electrical, optical or maybe some kind of magnetic operations or maybe that application.

Example: if I give the example of an silicon crystals for CPU and RAM in computers just we are using tremendously and for semiconductor based consumer products, single-crystal nickel-based super alloy turbine blocks or maybe with blades which are very high grip resistance. These crystals must all be produces with particular orientations. So, before going to in depth study of that projection, just let us know what is the stereographic projections or maybe that angle true projections. So, generally the crystal

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Stereographic Projection-Angle True Projection:

- Crystals information can be illustrated from their planes and angles between planes.
- Stereographic projection helps in representing the angles between the faces of a crystal, and the symmetry relations between them.
- It represents planes as points on some representative surface, which maintains the angular relationship of the points to each other.

The stereographic projection is a projection of points from the surface of a sphere on to its equatorial plane.

If any point P on the surface of the sphere is joined to the south pole S and the line PS cuts the equatorial plane at f , then f is the stereographic projection of P .

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Information can be illustrated from their planes and angles between planes. These already we have gone through in our previous lecture. So, stereographic projections help in representing the angles between the faces of a crystal and the symmetry relations between them. It represents planes as points on some representative surface, which maintains the angular relationship of the points to each other.

So, this is the very vital point that angular relationship of the points to each other. See suppose this is the single crystals, so this is the planes, across that planes that we are just assuming that it is confined in a sphere. So, in that sphere the point will be lies on the sphere itself. So, where the points is perpendicular lies is known as the pole and then if we make a line from that pole to the pole point to the any pole, either it may be north or south and intersecting the projection plane is known as the if over here.

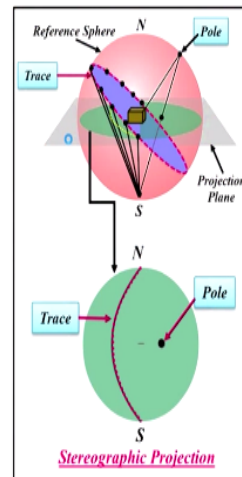
So, in this particular case C is the crystal plane as I already told and CP is the plane normal. So, this is the normal, this is actually 90° over there so, this is the 90° . So, now from here what we are going to do and this is the reference sphere. So, the stereographic projection is a projection of points from the surface of a sphere on to its equatorial plane. If any point P , this point on the surface of the sphere is joined to the South Pole S as I told already and the line this PS cuts the equatorial plane at F point. This is the F point. Then F is the stereographic projections of point P .

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Pole and Trace:

Stereographic Projection of Pole and Trace of a Crystal Plane:

1. Consider a plane in the unit cell and allow the plane to extend in space it will cut the reference sphere along a circle. This is the trace of the plane.
 2. Take the perpendicular to the crystal plane, it will go and intersect with the reference sphere; it will be the pole of the plane.
 3. Trace and pole of plane are at angle 90° with each other.
- The trace of a plane passing through the center of the sphere is a great circle i.e. circle of maximum diameter.
 - A plane not passing through the center will intersect the sphere in a small circle.



So, now we have to go more in depth that what is pole and what is trace? Suppose consider a plane in the unit cell and allow the plane to extend in space, it will cut the reference sphere along a circle. This is the trace of the plane. So, in this case is, you see that this is the circle; it is forming along the sphere. So, this is known as the trace and this is reference to this N or maybe this pole.

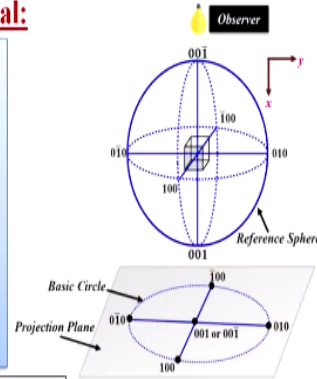
This is the North Pole and this is the South Pole and this is supposing your point P. So, irrespective of P, the trace is looks like this. Take the perpendicular to the crystal plane it will go and intersect with the reference sphere, it will be the pole of the plane. Trace and pole of the plane or at angle 90° with each other so, simple if you see the trace and if you see the trace and the pole so it is perfectly the 90° over there.

The trace of a plane passing through the center of the sphere is a great circle that is circle of maximum diameter. Yes, of course and if it is not passing through the center, or it will intersect the sphere in a very small circle. So, this is the pole over here and this is the trace because all the points lie on the trace itself.

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Construction of Stereogram for a Crystal:

1. The cube of a crystal is kept at the centre of the sphere.
2. All the plane normal are drawn from the cube surfaces, they cut the sphere on some particular points (poles).
3. We orient the crystal such that the pole to the (001) face is vertical and points to the North pole of the sphere.
4. Then, we draw a line from the point on the sphere directly to the South Pole of the sphere.
5. Poles are projected on to the equatorial plane/projection plane from north/south end of the reference sphere.



Some basic terminology to understand the Stereogram:

- A **face** is designated by miller indices in parentheses, e.g. (100), (111) etc.
- A **form** is a face plus its symmetric equivalents (in curly brackets) e.g. {100}, {111} etc.
- A **direction** in crystal space is given in square brackets e.g. [100], [111] etc.



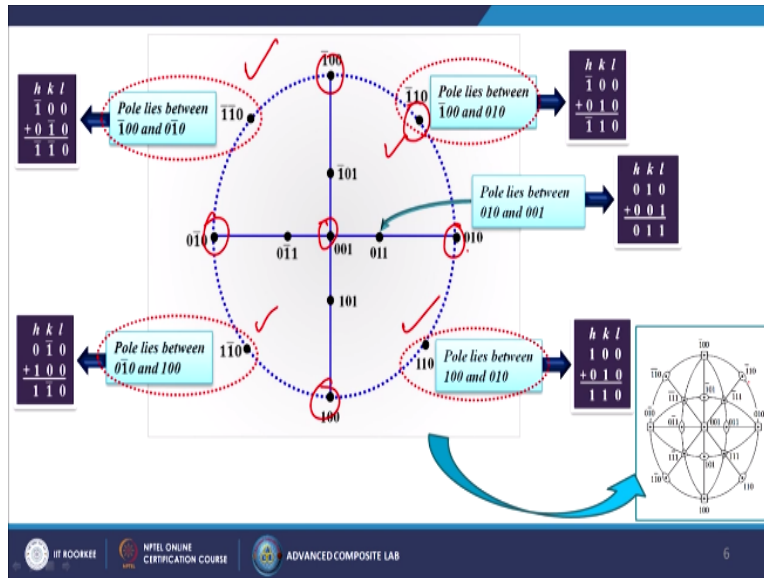
Now construction of a stereogram for a crystal: How we are going to do it? So, the cube of a crystal is kept at the centre of the sphere. In this image you can see that we have kept the crystal at the perfectly centre of this particular sphere. All the planes normal are drawn from the cube surfaces. They cut the sphere on some particular points or maybe the poles. So, here all the points it has cut over there.

We orient the crystal such that the pole to the 001 face. Where is 001? This is the 001. So, face is vertical and points to the North Pole of the sphere. So, you can see. Then we draw a line from the point on the sphere directly to the South Pole of the sphere itself. So, just we are drawing a line. Poles are projected on the equatorial plane or maybe the projection plane from north or south end of the reference sphere.

So, you can see, this is cutting in this particular point, in this particular point and in this particular point and in this particular point. So, if you see from the top because observer Eye is here so this is the observer Eye over there. So, simple this point that 001 bar and 001 it is into the same point and this is 1 bar 00 is here. This is 100 is here. This is 010 is here and 01 bar 0 is here. So, that means what?

Just we have seen from the top and we are plotting all the points. So, this is the basic circle and this is known as the projection plane. So, some basic terminology to understand the stereogram is a face is designated by miller indices in parenthesis in first bracket. Like first bracket 100 bracket closed then first bracket 111 bracket closed etc. A form generally we are giving it in to second bracket. So, is a face plus its symmetric equivalents in curly brackets like second bracket within 100 or maybe second bracket within 111 and a direction in crystal space is given in square brackets like tod brackets into in between that 100 or maybe 111 into tod brackets.

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So, now we have gone or maybe we have received these points, this is known to me, this is known to me, this is known to me, this is known to me. See suppose I am having some points which is in-between 010 and $\bar{1}00$. So, I want to calculate up these. So, what I am going to do? Just I will add all these value. So $\bar{1}00 + 010$ that means this point will be $\bar{1}10$. So, same thing like that so I will get all these points over there and then I can get this image that the points or maybe the positions up all the points over here. Now you can see over there some points are in square in bracket, some in oval shapes or maybe some in triangle, which I will come later.

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Standard Stereographic Projections of Cubic Crystal:

- A Standard stereographic projection shows the angular relationships between different poles for a given crystal orientation.
- It provide maps of low index direction and planes for identifying crystal orientations.
- Example: Standard Stereographic Projections of Cubic component $\{001\}$.
 - Forms $\{100\}$, $\{110\}$, $\{111\}$ have 6 faces (hexahedron), 12 faces (dodecahedron) and 8 faces (octahedron) in the stereogram respectively.
 - Rotation axes are indicated by the symbols as discussed in table.

Symmetry	angle	Identity
1-fold	360°	
2-fold	180°	
3-fold	120°	
4-fold	90°	
6-fold	60°	

So now standard stereographic projections of cubic crystals: So a standard stereographic projections shows the angular relationships between different poles for a given crystal orientations. Actually from 3D just we are converting all the points and the projections into 2D. So, it provides maps of low index directions and planes for identifying the crystal orientations.

Example: standard stereographic projections of cubic component. So, just here we are telling the form.

So, you can see that is in second bracket. So, in this particular case we are giving the forms. Second bracket 100 within second bracket 110 and within second bracket 111, have 6 faces that for 100 it is hexahedron, for 110 it is dodecahedron and 111 it is the octahedron, how in the stereogram respectively. See suppose when we are talking about the 100 family, so you can see there are total 1, 2, 3, 4, 5 and another points. So, total 6 points you can get it over there because 1 is back of that 1 and that is in square, square means it is the fourfold because each plane are 90° each other.

So, this is for the 100. So and when we are talking about the 110 family? So, you can see that there are total 1, 2, 3, 4, 5, 6, 7, 8 and another four points are there, which is the back of these particular backside of this particular form. So, that means it is $\bar{1}01$ opposite is $10\bar{1}$, then it is $0\bar{1}1$ which is $01\bar{1}$, it is 011 which is opposite is $0\bar{1}1$, it is 101 which opposite is $\bar{1}0\bar{1}$.

So, like this way, so that means it is in oval shapes that means, it is twofold. So, it is having 180° angles plane each other. So, this is for the 110 and when we are talking about 111 family. So, in this particular case we are getting total 8. So, that is why it is octahedron in nature and we are getting these shapes triangle that means it is having the threefold, which is the planes are 120° each other. So, this is for the 111 family.

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Zone of Crystallographic Planes:

- **Zone:** A set of planes whose intersection lines are all parallel to each other.
- The inter-section line is called Zone axis.
- The planes of a zone axis $[uvw]$ must satisfy the Weiss Zone Law:

$$hu + kv + lw = 0$$
- The direction of zone axis for $(h_1k_1l_1)$ and $(h_2k_2l_2)$ sets of planes:

$$u = k_1l_2 - l_1k_2$$

$$v = l_1h_2 - h_1l_2$$

$$w = h_1k_2 - k_1h_2$$
- ❖ The poles of planes of a zone will all lie on the same great circle on the projection, and the axis of the zone will be at 90° from this great circle.

(100) (111) (011) ($\bar{1}11$) ($\bar{1}00$) are in same zone with zone axis $[0\bar{1}1]$

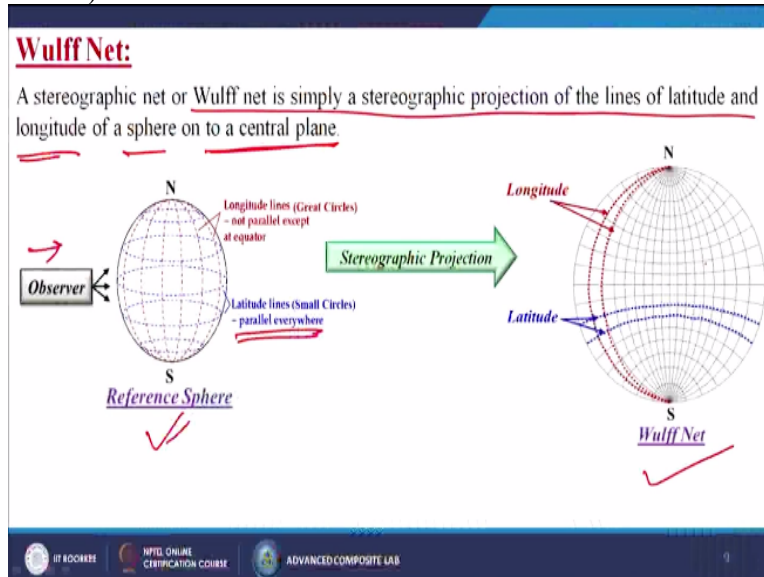
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So, now we are going to discuss about the zone of crystallographic planes. So, what is zone? Zone is nothing but a set of planes whose intersection lines are all parallel to each other. So, in this case we are having different planes over there. So, intersection lines so this is known as the zone axis which is generally denoted by UVW or maybe some other planes. So, the intersection line is called the zone axis.

The planes of the zone axis which is within the tod bracket UVW must satisfy the Weiss zone law which tells us $Hu + kV + LW = 0$. So, the direction of zone axis for $h_1k_1l_1$ and $h_2k_2l_2$ set of planes we can calculate the value of UVW like this way. $U = k_1l_2 - l_1k_2$, $V = l_1h_2 - h_1l_2$ and $w =$

h1k2 - k1h2. So, if we know the value of h1k1l1 or may be h2k2l2 so easily we can calculate the value of UV and W. The poles of planes of a zone will all lie on the same great circle on the projections and the axis of the zone will be at 90° from this great circle. So, in this particular image you can see that point 01̄1 is the zone axis, for which point 100, 111, 011, 1̄11 and then 1̄100 so, same thing as been over here.

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So, now we are going to discuss about one interesting thing that is called the wulff net. So, wulff net is generally, if you remember the longitudinal and the latitude of art then we can simply get the information's of any point that where it is lying based on the longitudinal value or maybe the latitude value. So, a stereographic net or wulff net is simply a stereographic projection of the lines of latitude and longitude of a sphere on to a central plane.

So, observer is over here, so from this place we are looking over there. So, this perpendicular line is known as the longitude lines which is called the great circles not parallel except at the equator and this oriental lines is called the latitude lines or small circles parallel everywhere. So, this is the reference sphere. So, from these we are getting the stereographic projections. So, this is our longitude line and this is our latitude lines in the wulff net itself.

So, same thing we are making the replica and from that we are going to trace the exact place of a particular point so, generally wulff net is used for measurement

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Wulff net is used for:

1. Measurement of angle between points on stereogram.
2. Construction of stereogram for different crystal systems.

Steps:

1. Tracing sheet is superimposed on the Wulff net & pinned together at the center.
2. Draw the primitive circle, draw N-S axis & E-W axis (reference axes).
3. Wulff net is kept constant and by rotating the tracing sheet above operations can be performed.

Tracing sheet superimposed on Wulff net → **After the Wulff net is removed**

Measurement of angle between poles 1 and 2:

- Rotate the tracing sheet to put poles on same great circle of Wulff net.
- The poles must be on same longitudinal line.

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Of angle between points on stereogram: construction of stereogram for different crystal systems. How we are going to do? So steps 1. Tracing sheet is superimposed on the wulff net and pinned together at the center itself. So, first we are putting our tracing sheet on the wulff net itself. Draw the primitives circle, draw north and south pole axis and east and west axis which is the reference axes over there.

Wulff net is kept constant and by rotating the tracing sheet above operations can be performed. How we are going to do? I will tell you. So, now rotate the tracing sheet to put poles on same great circle of the wulff nets itself. The poles must be on same longitudinal line. So, how we are going to do? Suppose we are going to measure the point irrespective of point 1. So, simple point1 and then just we are putting the tracing sheet and then we are trying to rotate and simple by rotations because here you can see that each one gap or maybe the deviations is 1° , so automatically here we are having 9 deviations over there. So, 9 into 10 that mean 90 and then 1 unit so, 91° just we are going to do the rotations. (Refer Slide Time: 16:14)

Relation between Pole and Trace of a Plane:

- Trace becomes a great circle in the stereographic projection.
- Every point on this great circle is 90° from the pole of the plane.

Method of Finding the Trace of a Pole:

- Trace becomes a great circle in the stereographic projection.
- Every point on this great circle is 90° from the pole of the plane.

Steps:

1. Rotate the projection until the pole falls on the equator of the underlying Wulff net.
2. Trace that meridian which cuts the equator 90° from the pole.

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So, relation between pole and trace of a plane: Trace becomes a great circle in the stereographic projections. Every point on this great circle is 90° from the pole of the plane itself. So, already we have gone through. So, this is the trace from whole it is absolutely 90° . Now method of finding the trace of a pole: Trace becomes a great circle in the stereographic projections which we have already gone through.

Every point on this great circle is 90° from the pole of the plane. So, now the steps rotate the projection until the pole falls on the equator of the underlying wulff net. So, you are having any point over there. So, just first you have to rotate your tracing paper, so that these P point or maybe P 2 prime will fall on to a, some particular projection lines. Then trace the meridian which cuts the equator 90° from the pole. So, now you have to find these lines, so automatically this will be become your trace over the, assort by this way you can calculate the pole and trace of a particular plane.

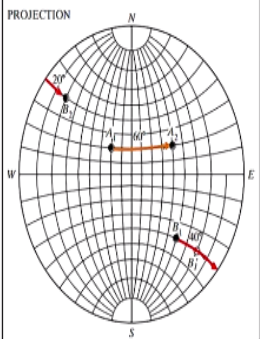
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Rotation of Pole about an Axis:

1. Rotation of Pole about N-S Axis:

Problem: Rotate the poles A_1 and B_1 by 60° about the N-S axis (direction of motion being from W to E).

- Rotate the point on the same latitude by 60° from W to E.
- **Pole A_1 :**
 - After rotation by 60° A_1 moves to A_2 .
- **Pole B_1 :**
 - B_1 is rotated only 40° to reach the edge of the projection then it moves 20° in from the diametrically opposite end staying on the same latitude to reach B_2 .



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Now next one is the rotation of pole about an axis. So, first rotation of pole about north to south or maybe north south axis: Problem: rotate the poles A_1 and B_1 by 60° about the north south axis (direction of motion being from west to east). So, that means from west to east you have to rotate it and this A_1 point and B_1 point, you have to rotate by 60° . So, rotate the point on the same latitude by 60° from west to east. Pole A_1 : after rotation by 60° A_1 moves to A_2 . It is very straight forward.

So, 60° rotations it is coming to A_2 point. For B_1 , B_1 is rotated only 40° to reach the edge of the projection and then it moves 20° in form the diametrically opposite end staying on the same latitude to reach B_2 . Yes of course because it is already there 20° so I have to move 40° over here then again from back side it will come another 20° , so B_2 formation will be taking place here.

So, that is the point.

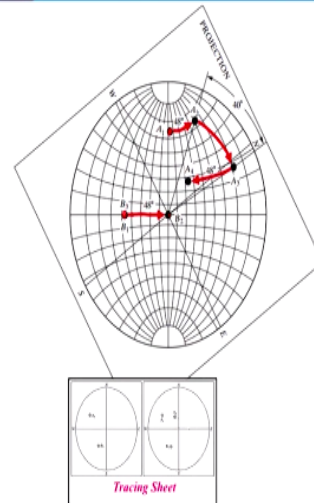
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2. Rotation of Pole about an Inclined Axis:

Problem: Rotate A_1 about B_1 by 40° in a clockwise direction.

Steps:

1. Rotate the projection to bring B_1 to the equator of a Wulff net.
2. Rotate B_1 by 48° about the N-S axis to bring B_1 to the point B_2 at the center of the net; at the same time A_1 must go to A_2 along a parallel of latitude.
3. Rotate A_2 by 40° to move A_2 to A_3 along a circular path centered on B_2 .
4. Reverse the step-2 by a 48° reverse rotation about the N-S axis to bring B_2 to B_3 and A_3 to A_4 .



Now second one, rotation of pole about an inclined axis. So, what is the problem? Rotate A_1 about B_1 by 40° in a clock wise direction. So, our A_1 point is over here, our B_1 point is over here. So, now we have to rotate A_1 references B_1 with or maybe the by 40° angle. So, what we have to do? Because B_1 first we have to put into the center, so that no projections is going to be changed. So for making this B_1 up to B_2 , we have to rotate 48° first. So, we are rotating 48° so B_1 is becoming B_2 , in this case A_1 becoming A_2 .

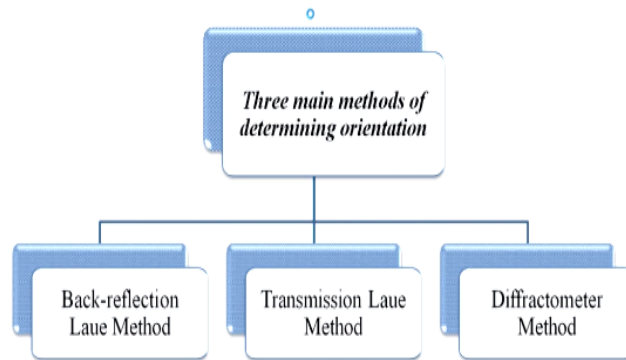
So, now our problem is that we have to rotate it by 40° . So, again we are doing the rotations of 40° in this particular case because it is into the center. If I change the positions of the A_1 or A_2 or maybe A_2 to A_3 , so here nothing is going to be changed. So, that is why we are keeping it into the same term. So, now we are rotating it 40° but initially we have given the rotations of 48° . So, again we have to back 48° once again.

So, that again it will become over here and we will get the point A_4 which is the final point. So, irrespective of B_1 new point formation will be A_4 which is the rotations by 40° so, same thing as been written over here.

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Methods for Determining Crystal Orientation:



Now what are the methods for determining the crystal orientations? So, there are mainly three basic or maybe the main methods, first one is called the back reflection Laue method, second is called the transmission Laue method and the third one is called the diffractometer method. (Refer Slide Time: 20:32)

Back-reflection Laue Method:

- The Laue pattern of a single crystal consists of a set of diffraction spots on the film and the positions of these spots depend on the orientation of the crystal.
- The back-reflection method is the more widely used because.
 - ✓ **Back-reflection:** no special preparation of the specimen (any thickness).
 - ✓ **Transmission Method:** relatively thin specimens of low absorption.
- Since the orientation of the specimen is to be determined from the location of the Laue spots on the film, it is necessary to orient the specimen relative to the film in some known manner.

Type of Specimen	Orientation of Specimen Relative to the Film
Wire or Rod	Mounted with their axis parallel to one edge of the rectangular film, a fiducial mark on the side nearest the film.
Sheet or Plate	Mounted with their plane parallel to the plane of the film and one edge of the sheet plate parallel to an edge of the film.
Irregularly Shaped	Fiducial marks on their surface to fix their orientation relative to that of the film.

So, back reflection Laue method: the Laue pattern of a single crystal consists of a set of diffraction spots on the film and the positions of these spots depend on the orientation of the crystal. The back reflection method is the more widely used because back reflection no special preparation of the specimen is required. So, any thickness can be done. Transmission method relatively thin specimens of low absorption so, that is why it is widely used. Since the orientation of the specimen is to be determined from the location of the Laue spots on the film, it is necessary to orient the specimen relative to the film in some known manner. So, what are the types of specimens? If it is wire or rod so, orientation of specimen relative to the film will be mounted with their axis parallel to one edge of the rectangular film and a fiducial mark on the side nearest the film. I will show you in the next slide. Sheet or plate mounted with


their plane parallel to the plane of the film and one edge of the sheet or plate parallel to an edge of the film and the third one is called the irregularly shaped, fiducial marks on their surface to fix their orientations relative to that of the film.

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Determination of Orientation of the Crystal from the Position of Back-reflection Laue Spots:

1. Measurement of the (x, y) coordinates of the diffraction spots on the film.
2. Determination of the orientation of the plane normal (in terms of its angular coordinates) causing each spot by using Geringer chart.
3. Plotting of the stereographic projections of the poles of the plane, and measuring the angles between them.
4. Identification and indexing of planes by comparing their measured angles with a list of known interplanar angles for the crystal involved.

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So, now first is the determination of orientations of the crystal from the position of back reflection Laue spots: so first measurement of the x, y coordinates of the diffraction spots on the film? Determination of the orientation of the plane normal in terms of its angular coordinates causing each spot by using Geringer chart. Plotting of the stereographic projections of these poles of the plane and measuring the angles between them. Identification and indexing of planes by comparing their measured angles with a list of known interplanar angles for the crystal involved.

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1. Position of Diffraction Spots on the Film:

- All planes of one zone diffract beams which lie on the surface of a cone whose axis is the zone axis and whose semi-apex angle is equal to the angle ϕ at which the zone axis is inclined to the transmitted beam.
- Diffraction plane is rotated about the zone axis **AB, HK**.
- In Back-reflection Laue method, diffraction spots on the film, due to the planes of a single zone in the crystal, lie on
 - ✓ hyperbola (when $45^\circ < \phi < 90^\circ$)
 - ✓ straight line (when $\phi = 90^\circ$)

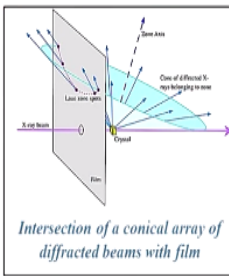
AB is the locus of plane normal intersections with the film

HK is the locus of diffracted beam intersections

CN= Plane Normal;
S= Position of a diffraction spot




Location of back-reflection Laue spot



Intersection of a conical array of diffracted beams with film

The orientation of the plane normal in space can be described by its angular coordinates γ and δ .


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So, first position of diffraction spots on the film: all planes of one zone diffract beams which lie on the surface of a cone whose axis is the zone axis and whose semi-apex angle = the angle ϕ at

which the zone axis is inclined to the transmitted beam. So, this is the ϕ and this is the zone axis and this is diffraction beam. So, diffraction plane is rotated about the zone axis AB and HK. So one, suppose plane is over here, so the diffraction point is going over here then the plane is rotating like this way, so automatically there will be a line or maybe the ellipse will be formed in these particular zone on the film itself. So, in back reflection Laue method diffraction spots on the film due to the planes of a single zone in the crystal lie on hyperbola when ϕ is more than 45° and less than 90° and straight line when the ϕ is, $= 90^\circ$. So, in this particular case the AB is the locus of plane normal intersections with the films. So, this is the AB. HK is the locus of diffracted beam intersections, so this is HK. CN is the plane normal, so in this case C and your n will be the normal. So, this one and S is the position of a diffraction spot. So, this is the S. so, same thing it has been elaborated over here so, intersections of a conical array of diffracted beams with film. So, simple as I told that how it is forming an ellipse over there. So, the orientation of the plane normal in space can be described by its angular coordinates that is γ and delta. So, γ is over here, this is the γ and δ is over here so delta.

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2. Determination of γ and δ from the Measured Coordinates (x, y) of the Diffraction Spot S on the Film:

Spot S on the Film:

Greninger Chart Method:

- Graphical method of finding γ and δ from the measured coordinates x and y of the diffraction spot S on the film.
- Greninger developed a chart which, when placed on the film, gives directly the γ and δ coordinates corresponding to any diffraction spot.
- From figure,

$$x = OS \sin \mu, \quad y = OS \cos \mu \quad \text{and} \quad OS = OC \tan 2\sigma$$

$$\tan 2\mu = \frac{FN}{FO} = \frac{CF \tan \delta}{CF \sin \gamma} = \frac{\tan \delta}{\sin \gamma}$$

$$\tan \sigma = \frac{ON}{OC} = \left(\frac{FN}{\sin \mu} \right) \left(\frac{1}{CF \cos \gamma} \right) = \left(\frac{CF \tan \delta}{\sin \mu} \right) \left(\frac{1}{CF \cos \gamma} \right) = \frac{\tan \delta}{\sin \mu \cos \gamma}$$

$$OS = OC \tan 2\sigma, \quad \tan \sigma = \frac{\tan \delta}{\sin \mu \cos \gamma}$$

- With these equations, the position (in terms of x and y) of any diffraction spot can be plotted for given values of γ and δ and any desired specimen-film distance D.

Now second one, determination of γ and δ from the measure coordinates x, y of the diffraction spot S on the film: so, how we are going to calculate these all these points. So, by Greninger chart method. So, generally the graphical method of finding γ and delta, here it is the γ and here it is the delta. So, from the measured coordinates x and y of the diffraction spot S on the film, Greninger developed a chart which, when placed on the film gives directly the γ and δ coordinates corresponding to any diffraction spot.

So, simple we have to fit it into the Greninger chart itself. So from figure, how we has calculated? So, from figure $x = OS \sin \mu$ of this point, $y = OS \cos \mu$ and what is OS? So that is $OC \tan 2\sigma$. So, $\tan 2\mu = FN$ by $FO = CF \tan \delta$ by $CF \sin \delta$, so CF will be cancelled which is nothing but the $\tan \delta$ by $\sin \gamma$. So, in this case $\tan 2\mu = FN$ by $FO = CF \tan \delta$ by $CF \sin \gamma$. So, CF will be cancelled so $\tan \delta$ by $\sin \gamma$. So, $\tan \sigma = ON$ by $OC = FN$ by $\sin \mu$ INTO 1 by $CF \cos \gamma =$

CF tan δ by sin μ into 1 by CF cos γ . So, CF CF will be cancelled so tan δ by sin μ into cos γ . So, in this case OS = OC tan 2σ , tan $\sigma = \tan \delta$ by sin μ cos γ . With these equations the positions in terms of x and y, this x and y of any diffraction spot can be plotted for given value of γ and δ and any desired specimen-film distance D. so, in these case these OC is equal to the D which is nothing but the specimen film distance over there.

(Refer Slide Time: 26:59)

■ The hyperbolas running from left to right are curves of constant γ , and any one of these curves is the locus of diffraction spots from planes of a zone whose axis is tilted away from the plane of the film by the indicated angle γ .

In order to know, after processing, the orientation the film had during the x-ray exposure, the upper right-hand corner of the film (viewed from the crystal) is cut away.

When the film is read, this cut corner must therefore be at the upper left.

Steps:

1. The Greninger chart is placed over the film with its center coinciding with the film center and with the edges of chart and film parallel.
2. The γ and δ coordinates corresponding to any diffraction spot are then read directly.

The γ , δ coordinates corresponding to diffraction spots on the lower half of the film are obtained simply by reversing the Greninger chart end for end.

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So, the hyperbolas running from left to right are curves of constant γ and any one of these curves is the locus of diffraction spots from planes of a zone whose axis is tilted away from the plane of the film by indicated angle γ . So, like this we can get $\gamma = 0^\circ, 10^\circ, 20^\circ$ then in this case we are getting the value of δ 20 then δ 10 and the $\delta 0$ and this is the 2D. So, in order to know after processing the orientations the film had during the x-ray exposure, the upper right hand corner of the film viewed from the crystal is cut away.

When the film is read, this cut corner must therefore be at the upper left. So, this is the cut corner, just to make the mark that which side I am getting the point and how I am going to and what is the crystal position or maybe which side I am keeping the crystals. Now steps, the Greninger chart is place over the film with its center coinciding with the film center, so this is the case and with the edges of chart and film parallel also. In this case film and your Greninger chart is parallel.

The γ and δ coordinates corresponding to any diffraction spot are then read directly. So, we can easily get the positions of the diffraction spot. So, the γ and δ coordinates corresponding to diffraction spots on the lower half of the film are obtains simply by reversing the Greninger chart end for end.

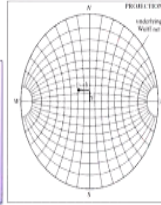
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3. Plotting of the Stereographic Projections of the Poles of the Plane:

Knowing the γ and δ coordinates of any plane normal, the pole of the plane can be plotted on a stereographic projection.

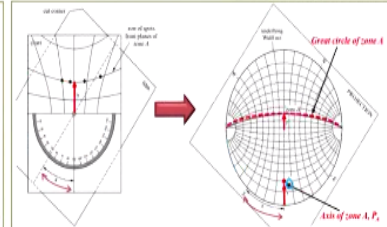
Note:

- Underlying Wulff net must be oriented so that its meridians run from side to side.
- The reason for this is the fact that diffraction spots which lie on curves of constant γ come from planes of a zone, and the poles of these planes must therefore lie on a great circle on the projection.



Plotting the Axis of a Zone of Planes on the Projection:

1. Rotate the film such that the hyperbola of spots is lined up with a horizontal hyperbola on the chart.
2. Measure γ and ϵ and map them on the tracing sheet using Wulff net to get the projection.



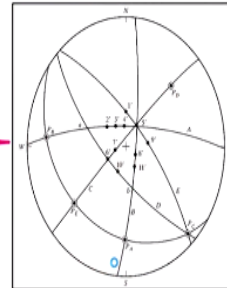
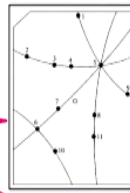
Next one is called the plotting of the stereographic projections of the poles of the plane. So, knowing the γ and δ coordinates of any plane normal the pole of the plane can be plotted on stereographic projections. So, these we have got from the, our last slides, so note underline Wulff net must be oriented, so that its meridians run from side to side. The reasons for this is the affect that diffractions spots which lie on the curves of constant γ come from planes of a zone and the poles of these planes must therefore lie o a great circle of the projection. So, this is the vital point over there.

So now, how we are going to plotting? So plotting the axis of a zone of planes on the projections, first rotate the films such that the hyperbola of spots is lined up with a horizontal hyperbola of the chart itself. So, it will match over there. So this is the rows posts generally what we are getting. So, simple we are rotating it so that it will match on that perfectly on line. Now measure γ and ϵ , ϵ is at what $^\circ$ just I have rotating that film so, them on the tracing sheet using wulff net to get the projections over there. So, simple we will get on the great circles all the points are lying, so this is the axis of zone which is nothing but know as the PA and this is the angle of rotations. (Refer Slide Time: 30:13)

4. Identification and Indexing of Planes:

This procedure can be illustrated by taking the example of **Aluminium crystal**.

1. Selected diffraction spots of back-reflection Laue pattern of an **aluminium crystal**, are traced and numbered for reference.
2. The poles of the planes causing these numbered spots are plotted stereographically.
3. With the aid of a Wulff net, great circles are drawn through the various sets of poles corresponding to the various hyperbolae of spots on the film.
4. The angles between important poles (zone intersections and zone axes) are measured and the poles are identified by comparing of these measured angles with those calculated for cubic crystals. The method is essentially one of trial and error.

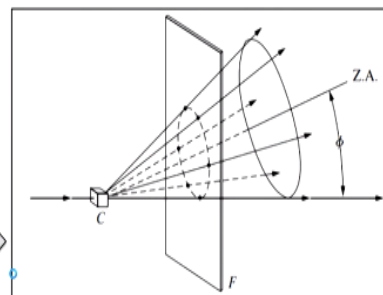


Next is that identifications and indexing of planes. So, this procedure can be illustrated by taking the example of aluminium crystal. So, first selected diffraction spots of back-reflection Laue pattern of an aluminium crystal are traced and numbered for reference. So, we have numbered all the spots over there. The poles of the planes causing these numbered spots are plotted stereographically so all the poles has been plotted over there. With the aid of a wulff net great circles are drawn through the various sets of poles corresponding to the various hyperbolas of spots on the film.

So, we have drawn all the hyperbola over there. The angles between important poles, here important poles are 5 because you can see that maximum poles have coincided in this particular point high prime. So, the angels between the important poles zone intersection and zone axes are measured and the poles are identified by comparing of these measured angles with those calculated for cubic crystal. The method is essentially one of trial and error method. (Refer Slide Time: 31:24)

Transmission Laue Method:

- For a specimen of sufficiently low absorption, a transmission Laue pattern can be obtained and used, in much the same way as a back-reflection Laue pattern, to reveal the orientation of the crystal.
- In Transmission Laue method, the diffraction spots on the film, due to the planes of a single zone in the crystal, lie on
 - ellipse (for small values of ϕ)
 - parabola (when $\phi = 45^\circ$)
 - hyperbola (when $\phi > 45^\circ$)
 - straight line (when $\phi = 90^\circ$).



C = Crystal
 Z.A. = Zone Axis
 F = Film
 ϕ = Semi-apex Angle of Cone

Next second that is transmission Laue method: so, for a specimen of sufficiently low absorption, transmissions Laue pattern can be obtained and used, in much the same way as back-reflection Laue pattern reveal the orientation of the crystal. In transmission Laue method the diffraction spots on the film, due to the planes of a single zone in the crystal lie on if it is ellipse, so for small values are ϕ , parabola when $= 45^\circ$, hyperbola when ϕ is more than 45° , straight line when $\phi = 90^\circ$.

So, in this case particular case you can see that C is the crystal over there. So, crystal planes ZA so this line is the, your zone axis. F is the film over there and ϕ is the semi-apex angle of cone, so this is the ϕ over there.

(Refer Slide Time: 32:22)

Continued...

Consider diffraction from a plane of a zone whose axis lies in the yz-plane at an angle ϕ to the incident beam.

Diffraction plane is rotated about the zone axis

- ❖ APEBWA is the locus of pole of the diffraction plane (Great Circle).
- ❖ DROD is the locus of the diffraction spot on the film (Ellipse).

Any particular orientation of the plane is characterized by particular values of ϕ and δ , the angular coordinates of its pole.

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So, now consider the diffraction from a plane of zone whose axis lies on the yz-plane at an angle ϕ to the incident beam. So, in this particular case we have already gone through so, this is the zone axis over there. So, this is the incident beam is going over there and then AP where A then your P then your E then B and WA is the locus of the pole of the diffraction plane which is known as the great circle and in this particular case D R O and D, this circles which is blue in color, dot it is the locus of the diffraction spots on the film which is elliptical in nature. So, any particular orientation of the plane is characterized by particular values of ϕ and δ the angular coordinates of its pole.

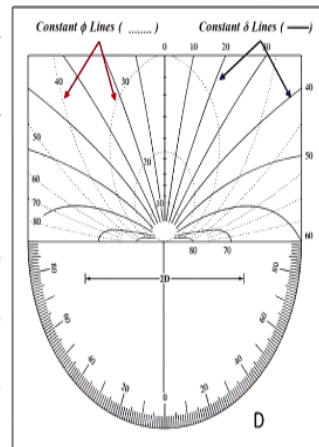
So, anyhow we are going to measure the angular coordinates of any points or maybe the pole.

Next is called the Leonhardt chart. So, Leonhardt chart

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Leonhardt Chart:

- Leonhardt chart is used to determine the angular coordinates of the diffraction spots.
- This chart is exactly analogous to the Greninger chart for solving back-reflection patterns and is used in precisely the same way.
- It consists of a grid composed of two sets of lines:
 - a) Lines of constant ϕ correspond to the meridians on a Wulff net,
 - b) Lines of constant δ correspond to latitude lines.
- The dashed lines are lines of constant ϕ , and the solid lines are lines of constant δ .

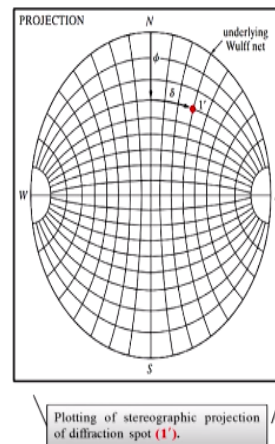
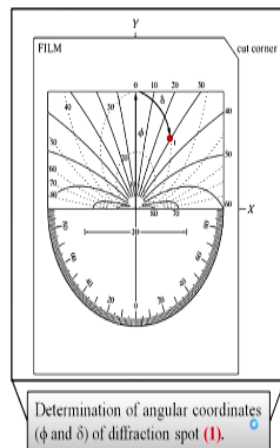


Is used to determine the angular coordinates of the diffractions spots, these chart is exactly analogous to the Greninger chart for solving the back-reflection patterns and is used in precisely the same way. Actually there are so many methods by which we can easily measure the crystal structure or the crystal orientation of any particular materials. So, it consists of a grid composed of a two sets of lines, lines of constant ϕ correspondent to the meridians on a wulff net, Lines of constant δ correspondent to latitude lines.

In this case you can see that one is constant ϕ lines, which is in dotted in nature and another one is the constant δ lines which is in dark line over there. So, this is in 20, 30 like that and this is 10, 20, 30 like that. So, this is the constant ϕ lines over there and this dark is the constant δ lines. So, the dashed lines are lines of constant ϕ and the solid lines are lines of constant delta.

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Plotting of Stereographic Projection:



So, plotting of the stereographic projections, so suppose, I am going to get one point over there. So, determination of angular coordinate's ϕ and δ of diffraction spots one so I am going to calculate of this one. So, simple I am putting into the wulff net and simple I am getting the

longitude and latitude of that particular point, so, plotting of stereographic projection of diffraction font O prime in this particular zone, if I know the ϕ and δ value.
 (Refer Slide Time: 35:07)

Plotting the Axis of a Zone of Planes on the Projection:

1. Rotate the film such that the hyperbola of spots is lined up with a horizontal hyperbola on the chart.
2. Measure ϕ and ϵ and map them on the tracing sheet using Wulff net to get the projection.

The poles of diffraction planes are indexed in the same way as back-reflection patterns.

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Plotting the axis of a zone of planes on the projection, so, rotate the film such that the hyperbola of spots is lined up with a horizontal hyperbola on the chart itself. So, just I have to rotate the film. Measure ϕ and ϵ and because ϵ we note that how much $^\circ$ of rotations I am doing over there to match the horizontal hyperbola. So we can get the ϵ value over there, we can get the ϕ value in this particular point.

So, ϕ and ϵ and map them on the tracing sheet using the wulff net to get the projection over there. So, simple the poles of diffraction planes are indexed in the same way as back-reflection patterns.

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Diffraction Method:

- In the diffractometer method monochromatic radiation is used.
- A single crystal will diffract only when its orientation is such that a certain set of diffraction planes is inclined to the incident beam at an angle θ which satisfies Bragg's law for that set of planes and the characteristic radiation employed
- Three possible rotation axes in a diffractometer are:
 1. Diffractometer Axis
 2. Tangent to the specimen surface (AA')
 3. Normal to the specimen surface (BB')

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Now is the last one which is called the diffractometer method: so in the diffractometer method monochromatic radiation is used. A single crystal will diffract only when its orientation is such

that a certain set of diffraction planes is inclined to the incident beam at an angle Θ which satisfies the Bragg's law for that set of planes and the characteristic radiation employed. So, simple our machine X or D equipment is following this methods or maybe we are getting the results by this methods itself but actually in the backend that whatever the formula or maybe that, whatever the projection is going on that is followed by those methods which I have discussed earlier.

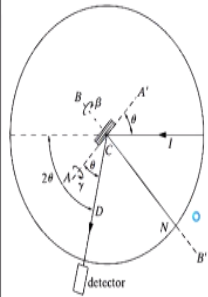
Three possible rotation axes in a diffractometer arc: diffractometer axes, tangent to the specimen that is AA prime so AA prime, m=normal to the specimen surface that is BB prime over there. (Refer Slide Time: 36:52)

Continued...

Suppose the orientation of a cubic crystal is to be determined. For such crystals it is convenient to use $\{111\}$: there are four sets of these and their diffracting power is usually high.

Steps:

1. Calculate the 2θ value for 111 reflection (or, if desired, the 222 reflection) from the known spacing of the $\{111\}$ planes and the known wavelength of the radiation used.
2. Fix the detector in this 2θ position.
3. Rotate the specimen holder about the diffractometer axis until its surface, and the rotation axis AA' , is equally inclined to the incident beam and the diffracted beam.
4. The specimen holder is then fixed in this position, no further rotation about the diffractometer axis being required.
5. Then, by rotation about the axis BB' , one edge of the specimen, or a line drawn on it is made parallel to the diffractometer axis.



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So, suppose the orientation of a cubic crystal is to be determined for such crystals it is convenient to use from 111, there are four sets of these and their diffracting power is usually high. How to do? First calculate the 2Θ value for 111 reflection or if desired the 222 reflection from the known spacing of from 111 planes and the known wavelength of the radiation used. Fix the detector in this 2Θ positions. Then rotate the specimen holder about the diffractometer axis until its surface and the rotation axis AA prime is equally inclined to the incident beam and the diffracted beam.

So, you have to rotate. The specimen holder is then fixed in this position; no further rotation about the diffractometer axis is being required. Then by rotation about the axis BB prime, so just you have to rotate this one, one edge of the specimen or a line drawn on its, it is made parallel to the diffractometer axis. So, one is the rotation of this one another one is the rotation of this one.

So, like this way the diffractometer is working.

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6. The crystal is slowly rotated about the axes AA' and BB' until an indication of a reflection is observed on the rate meter.

Computerized diffractometers allow this search to be automated; the computer systematically checks different combinations of rotations about axes AA' and BB' until a peak is found.

7. Once the position of the crystal for diffraction has been found, the normal to one set of $\{111\}$ planes coincides with the line CN .

8. The pole of these diffracting planes may now be plotted stereographically.

The projection is made on a plane parallel to the specimen surface, and with the NS -axis of the projection parallel to the reference edge. Two $\{111\}$ poles are enough to fix the orientation of the crystal, a third should be located as a check.

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Now the crystal is slowly rotated about the axes AA' and BB' until an indication of a reflection is observed on the rate meter. Computerized diffractometer allow this search to be automated. The computer systematically checks different combinations of rotations about the axes AA' and BB' until a peak is found. Once the position of the crystal for diffraction has been found, the normal to one set of $\{111\}$ planes coincide with the line CN over there. So, like this way we can plot all the points over there.

The pole of these diffracting planes may now be plotted stereographically. So, we are plotting all the elliptical lines over there. So, the projection is made on a plane parallel to the specimen surface with the NS -axis of the projection parallel to the reference edge. Two $\{111\}$ poles are enough to fix the orientation of the crystal; a third should be located as a check. Now we are already discussed about the X or D and the stereographic projections. Now what is the advantage for this diffractometer method? In the

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Advantages of Diffractometer Method:

- In the hands of an experienced operator, the diffractometer method is faster than either Laue method.
- It can yield results of greater accuracy if narrow slits are used to reduce the divergence of the incident beam.

Limitations of Diffractometer Method:

- The diffractometer method furnishes no permanent record of the orientation determination, whereas Laue patterns may be filed away for future reference.
- The diffractometer method does not readily disclose the state of perfection of the crystal, whereas a Laue pattern yields this kind of information at a glance and in many investigations the relative perfection of a single crystal is of as much interest as its orientation.

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Hands of an experienced operator, the diffractometer method is faster than either Laue method. It can yield results of greater accuracy if narrow slits are used to reduce the divergence of the incident beam but of course there is certain limitation. What are those? The diffractometer method furnishes no permanent record of the orientation determination, whereas Laue patterns may be filled away for future reference.

The diffractometer method does not readily disclose the state of perfection of the crystal, whereas a Laue pattern yields this kind of information at a glance and in many investigations the relative perfection of a single crystal is of as much interest of its orientations. Now we have reached to the last slide of this particular lecture. So, now we are going to summarize the whole lecture. So, in this particular lecture we have studied about the
(Refer Slide Time: 40:23)

Summary:

- ✓ Studies of isolated single crystal requires accurate knowledge of the orientation of the single crystal test specimen as they are anisotropic.
- ✓ Three main methods of determining orientation of single crystal discussed are:
 1. Back-reflection Laue Method
 2. Transmission Laue Method
 3. Diffractometer Method
- ✓ Transmission method is used for relatively thin specimens of low absorption, whereas no special sample preparation is required for back-reflection method.
- ✓ The diffractometer method is faster than either Laue method but it does not furnish a permanent record for future reference.

Isolated single crystal requires the accurate knowledge of the orientation of the single crystal test specimen as they are anisotropic in nature. Three main methods of determining the orientation of single crystals which we have discussed in this particular lecture, first one is called the back-reflection Laue method, second one is called the transmission Laue method and third one is called the diffractometer method.

Transmission method is used for relatively thin specimens of low absorptions, whereas no special sample preparation is required for the back-reflection method. The diffractometer method is faster than either Laue method but it does not furnish a permanent record for future reference.

Thank you.

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