Experimental Physics – III
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Lecture – 59
X - Ray Diffraction and Crystal Structure (Contd.)

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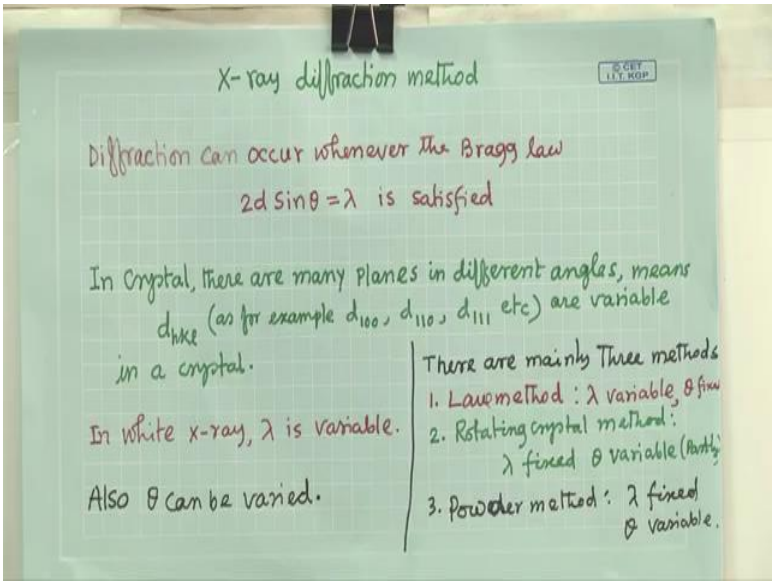

I will demonstrate X-ray diffraction experiment. X-ray diffraction method. Diffraction occurs can occur whenever the Bragg condition; Bragg law 2 d sin theta equal to lambda is satisfied. this is the main equation or law for X-ray diffraction 2 d sin theta equal to lambda.

Only we are considering first order n equal to 1. in crystal there are many planes in different angles, if you take a crystal. It will have it will have many planes in the crystal, so; that means, d is the planar distance. d is variable in a crystal, but this d are at different angles. In crystal, d of different value is available. In X-ray, white X-ray lambda is variable and theta here can be varied. Here you see three parameter one is d another is theta and lambda.

See in white X-ray lambda is variable. You can choose any lambda, you have option d in crystal there are many value of d, which that is how we tell the d h kl for different planes. d are different. In crystal, also there are many d available. You can choose a particular d.

d also, variable and theta is the angle of the incident X-ray with the surface of the sample. that we can; we can have provision to vary the angle theta.

Therefore, which one will be variable and which one will be fixed to satisfy these Bragg condition depending on that there are three methods. one is called Laue method Laue method. in Laue method lambda is variable; that means, white X-ray is taken and that white X-ray will fall on the sample and these angle theta is fixed; that means, sample is fixed sample is fixed its planes also are fixed incident X-ray are falling on it. angle with the sample planes are fixed, but theta lambda is variable all sources of lambda white X-ray is used.

Then lambda is variable. now, theta is also variable sorry theta is fixed theta is fixed. In this crystal there are many as I told, there are many planes are available. for different plane different lambda will be chosen different lambda by nature will be chosen. That this equation will be satisfied for different d and different lambda when theta is fixed. if experiment is done in this geometry then it is called Laue method and another method is called a rotating crystal method in rotating crystal method lambda is fixed means, white X-ray is not used this using the absorption filter one wavelength is chosen generally k alpha k alpha wavelength is chosen**.** Lambda is fixed.

X-ray of particular lambda is falling on the sample. Now in sample this rotating crystal method means, this crystal are rotated in step by step. In that sense, their theta is not continuously variable. theta can be changed in different step in different step. Theta is variable lambda is fixed. lambda is fixed. For different plane, there will be different theta. Then again, you will get Bragg diffraction and you will get Bragg peaks

Next method is powder method. if you have a crystal single crystal. Now, you to make it make it powder the crystal now it is in form of powder means, small particles will be there. That particle is crystalline particles. Now in each crystal there are all source of planes are available. Now these crystals itself is oriented in randomly. What does it mean? In one particle if 1 1 1 plane is in this direction this is the 1 1 1 plane. direction is perpendicular to that.

Now, for this particle this is the 1 1 plane for next another particle is randomly oriented. 1 1 1 plane is in this direction other is. This 1 1 1 plane 1 1 1 plane as if this it is the continuously variable incident X-ray when it will fall on the powder sample this 1 1

plane 1 1 1 plane. The X-ray we will see 1 1 1 plane in all direction. that that is from different particles, but so in this case, you can see this by nature in powder sample these each plane not only 1 1 1 plane each plane 1 0 0 1 1 0 1 1 1 all plane. As your completely h kl plane or continuously variable, this they are angle with this incident X-ray will have the continuous theta variation

It is the completely variable. this is telling this powder method lambda is fixed theta is completely variable. Now, this lambda wavelength X-ray, we will see all planes with all possible angles. Only, when for you can expect that for theta is variable lambda is fixed. d for different d at different theta this equation will be satisfied.in rotating crystal method to vary the angle we use we just vary the change the angle of the of the sample this method is used generally in case of in case of bulk material in case of thin film. There we need to vary the angle using some mechanism using some goniometer. In powder crystal method, powder methods. We do not need to vary the angle change the angle of the sample because already in powder form this all planes have all source of angles variation in the sample.

By nature variation of angles are there this method is powder method. In our laboratory, I will demonstrate this powder method this is very popular method other two method I will not discuss, compared with this powder method is widely used and very useful and that is what I will discuss.

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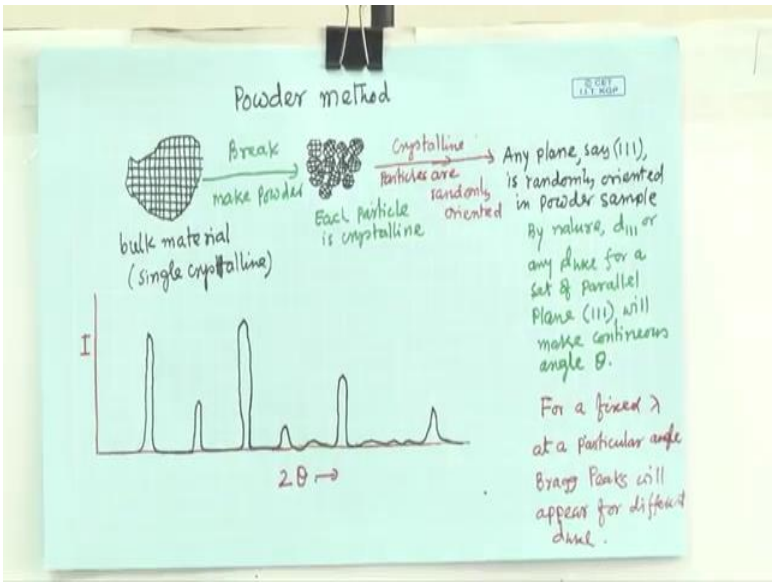

As I mentioned using powder method, we will. They say some bulk material I have to break this bulk material make powder in small particles.

These small particles, they have their crystallite they have all source of planes and these planes are in different particles are in different direction. thousands and thousands particles are there. You can expect that each plane will have these all vary all angle they are oriented in all angles. now, this X-ray will fall on these on these powder sample, where this Bragg condition will be satisfied and get the get the this kind of this kind of x r d pattern we tell x r d pattern X-ray diffraction pattern.

As I explained that any plane say 1 1 1 is randomly oriented in powder sample by nature d 1 1 or d any dhkl for a set up parallel planes 1 1 1 will make continuous angle theta. for a fixed lambda at a particular angle Bragg peak will appear for different dhkl. What does it mean? Here we will get Bragg peak. Here I have shown many Bragg peaks. Each Bragg peak is from a particular plane set of plane.

this peak indicates a particular planes particular planes and as I mentioned that from this from this experiment, we find out this angle here I have written 2 theta one can write theta also, but 2 theta generally in powder method, we prefer 2 theta because I will tell in next transparency why we tell 2 theta.

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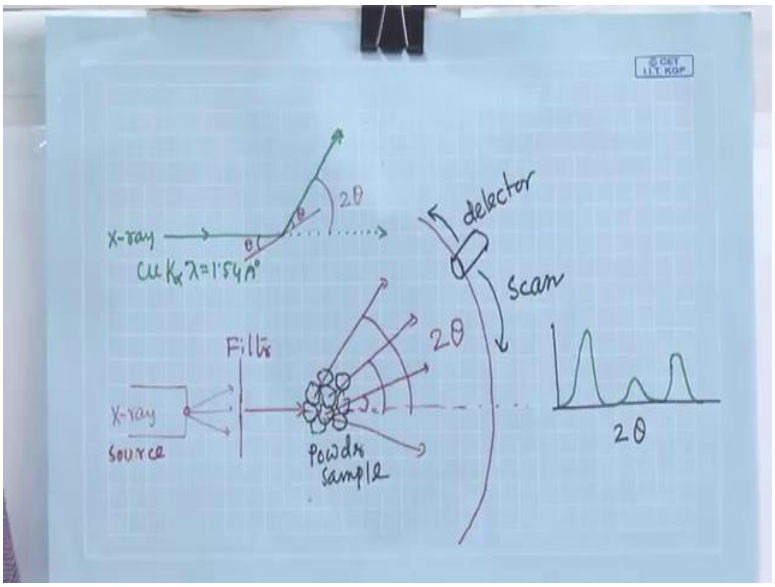

Here you can see this, you have a sample we have a sample here. just I have drawn a plane in that sample. X-ray is falling on it. This angle with this plane is theta.

Then it will be reflected or diffracted an angle theta. This 2 theta angle is with respect to the incident X-ray with respect to the incident x ray. The X-ray; the X-ray will be diffracted at different angle. Now as I told this we do not need to vary the angle of the sample, it is fixed it is fixed. Now only to catch these diffracted peak diffracted intensity of X-ray, we use detector we use detector and the detector we just we just scan over 0 to 90 degree or minus 90 to plus 90 degree we just scan.

Generally 0 to scan 0 to 90 degree. if you scan so this detector will tell you that this at which angle the diffracted beams are coming. That is why we plot here variable is 2 theta from 2 theta one can find theta dividing by 2. When it is scanned so it just noted down this variation of intensity of X-rays at different angle. We get peak when these Bragg condition is satisfied and from these peaks generally, we get the information about that crystal.

What are the information we get that I have discussed. If I will discuss more when we will analyze the data let me. now, for your experiment what do you need? We need X-ray source now, this X-ray source in powder method we need X-ray of particular wavelength. We will use filter. from after filtering. We will get K alpha K alpha that K alpha X-ray will fall on the sample powder sample. Now, it will be diffracted in different angles depending on the different planes. Now, we should have a detector at that detector should have option to scan from 0 to 90 degree.

Now, I will show you our diffractometer powder X-ray these are powder diffractometer.

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Here you can see this is the machine, we tell this X-ray machine and it is X-ray diffractometer.

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Here you can see that and in here, it is written power then X-ray shutter on off. from outside itself, we can see whether X-ray is on or not whether it is a X-ray is produced. Some shutter is there we just can close the close the exit of the X-ray using the shutter.

Now, we this is the when you will work in this machine. one has to be very careful because X-ray if it is exposed to human body for a long time, then there will be

damaged. It is the radiation one has to be careful when we are going to use this spectrometer.

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it is a door I will open it this door if I open this door and I will open this backside also to show you to show you just I can open it. Here you can see, when I open door immediately I am able to see here, we put sample we put sample this is the sample holder

This just glass slide we use for putting our sample say we have an aluminum powder here you can see we have aluminum powder.

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This aluminum powder if I want to do experiment on this aluminum powder or sometimes, we give to student that aluminum powder and copper powder to test to find out the composition ratio of copper and aluminum in the sample.

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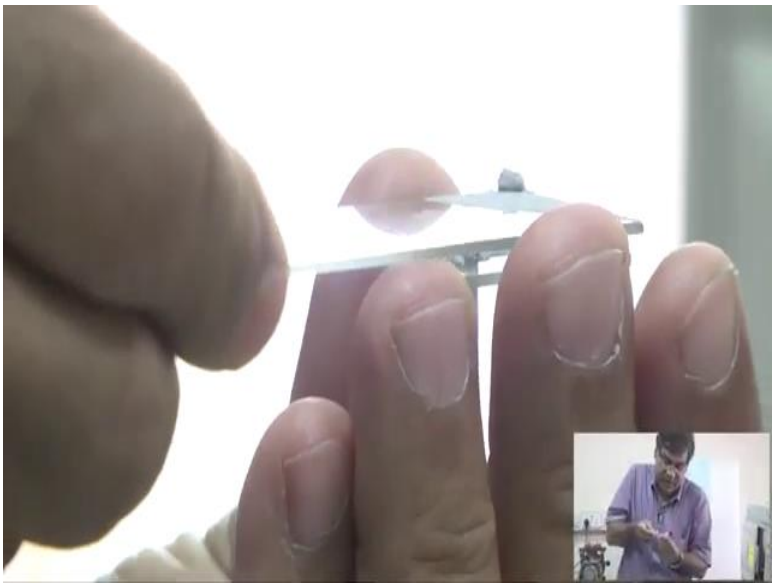

This you can see powder this is the sample powder, we put slight amount of powder on this on this. just paste it spread over it. The sample that sample now I will put in sample holder I will put in sample holder here I am putting now, sample is there. Now, here you can see here there is an X-ray generator; X-ray generator here I will show you from backside. From this X-ray generator this X-ray will come and there is a filter inside.

Monochromatic X-ray or X-ray this is the copper this is the copper targets.

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Copper K alpha this wavelength is 1.54 angstrom. that K alpha X-ray will come and fall on the fall on the sample. now, so as I told these we do not need to vary the angle of the sample we do not need to vary the angle of the sample, this is fixed this is fixed and with these with these this is you can see this one handle kind of things one handle kind of things and on that handle on that handle there is a detector here.

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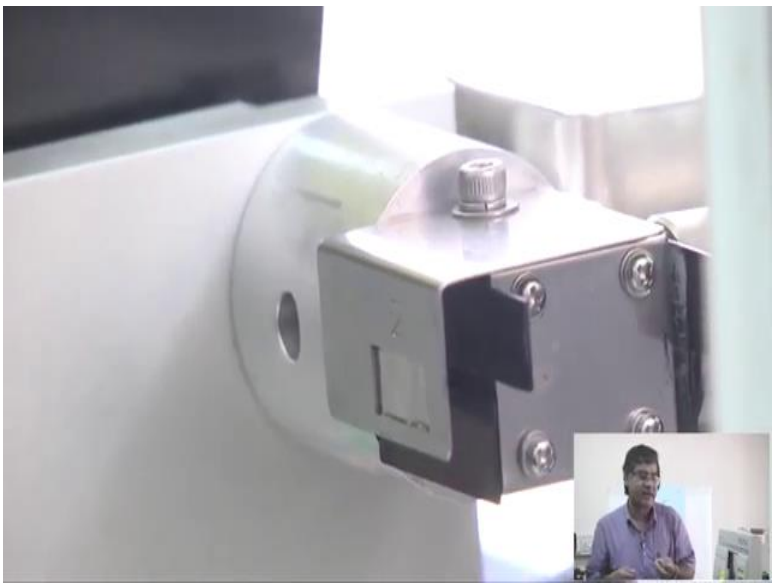

This is a detector. It is the X-ray detector if it can measure the intensity of the x ray. As I for any detector, in this case, X-ray will fall and then it will produce equivalent number

of carriers and due to that carriers, we will get current or voltage. that current or voltage is proportional to the intensity of x ray. That way this detector tell us the intensity of X-ray. now, the detector it is on this on this lever.

Now that detector have option here, we use stepper motor. it will it will vary with some step you can specify that step 0.1 degree or 0.2 degree or 0.5 degree or 1 degree step. It will from 0 to it will in different steps it will change it will the angle will change 2 theta is changed. It is this angle is with respect to the incident X-ray. now, theta the detector are moving. That angle we are telling with respect to the incident angle of the X-ray this that was up to 990 degree generally we scan

Then you will get this type of as I mentioned then your it is interfaced with the computer. This is a computer screen you will see this type of this type of pattern diffraction pattern we will see. Now using a diffraction pattern one has to analyze and find out the find out the different parameters of our interest. That I will discuss how to analyze the X-ray and get the information. Let me from side you cannot see all in better way.

I will try to show the all components from backside. I have opened the box. From backside, you can see that.

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As I showed you that, this is the sample holder.

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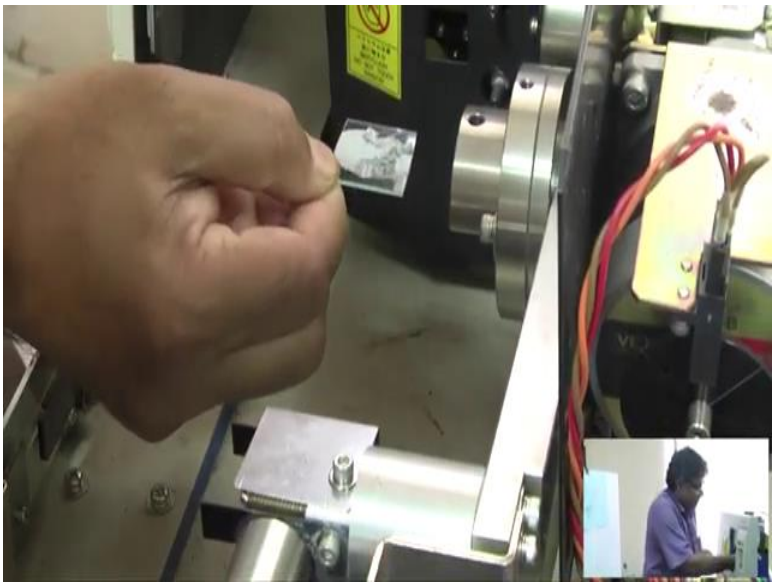

This sample here I have put sample. this powder sample soon a glass plate. You put in the sample holder. Now, this is fixed, but the detector this is the detector X-ray detector, which can detect the intensity of X-rays. This is on this liver on this arm. this arm you can see this is the free to move free to move from 0 degree to 90 degree.

We use stepper motor. backside you can see that there is a stepper motor. The stepper motor is rotate this detector with respect to these axis passing through the through the center of the sample. This rotational axis is axis of the sample holder. Now, this is the X-ray source this is the X-ray source. from this X-ray source, the there is a filter inside you cannot see. Now, this X-ray filter X-ray K alpha this is the copper targets. that will come and fall on this and this detector will start to start this rotation of the detector which some particular step.

From computer everything is controlled. now, this detector will rotate at different steps. These we are tell in the change of 2 theta. at different 2 theta it will collect the X-ray then its intensity. Then that pattern will appear in the pattern will appear in the on the on the screen of computer. This I have taken the aluminum sample powder.

Sometimes any other sample in powder form we have to take. This is the geometry suitable for powder method only. In some better diffractometer, where this rotation of this sample as well as rotation of the detector both facilities available. in that case we can study the, we can study the thin film or bulk material, but in this diffractometer only

even if you want to study thin film or bulk material if you want to know the crystallinity of the material.

You have to break this bulk material or thin film and make it powder and that powder you have to use on this on the on the sample holder and do the experiment. This as I told the X-ray machine have one part is X-ray generation for that we need chilled water cooling water.

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Here you can see some pipe some pipe has come from outside.

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This is the chiller is outside. from outside this water pipe chilled water will come and this is connected with this instrument this is connected with this instrument.

That chilled water is coming to the instrument. the chiller is outside. Again that that chilling part is here inside around the around the X-ray tube. it is a very small machine, it is the low power machine it Is a for teaching laboratory. We can it is I think it is its kilowatt is around 6 kilowatt this power is 6 kilowatt no 18 kilowatt is a one third of that one. 6 kilowatt so accordingly voltage and current that specification is there. Inside if you see inside there is a not, you see this looks very simple only this X-ray generator.

This is the standard generator. Now, we need to sample holder. Now with respect to the sample holder, there will be a detector the detector should rotate with respect to the axis passing through this sample holder. For rotating the detector, we use stepper motor. that stepper motor some gears are there here some gears are there one can see.

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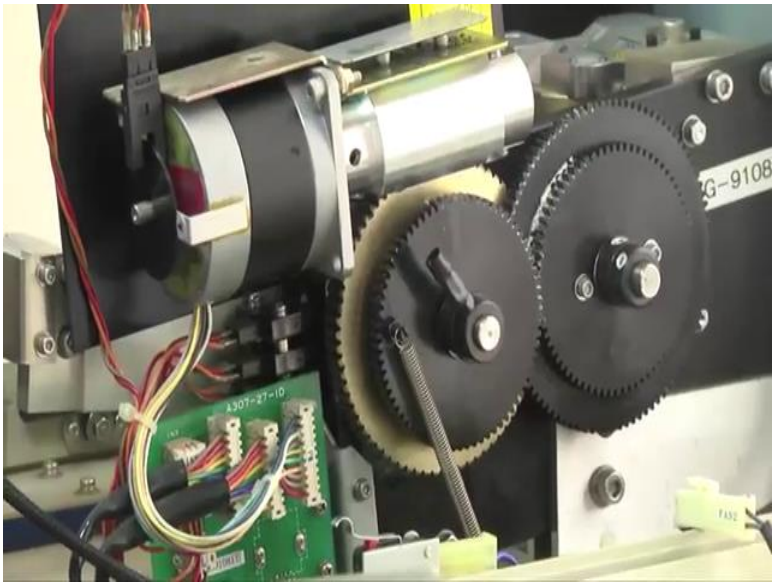

That that stepper motor that is also standard that stepper motor is connected with this detector holder and from computer programming one can choose the step of this change of the angle 2 theta or change of the angle of the detector and from 0 to 90 degree, we scan and on screen, we will get the X-ray diffraction pattern.

Now they have to X-ray diffraction pattern, we have to analyze for getting the information as I told that if aluminum powder and copper powder this mixture if I give

you then what is the ratio of this two phase two phase one is aluminum and another is copper. What is their ratio in this mixture that one can find out that is generally this experiment we give to our student. In addition, one can find out what is the crystal structure of aluminum. What is the crystal structure of copper? In addition, many more things one can analyze from the from the x r d pattern.

That is more important than doing this experiment generally; if we if you give samples our operator will give you, the data the x r d pattern the data they will give you. Now, it is our task to analyze these data and find out different parameters. That I will discuss it possible I will discuss later on. I will stop here.

Thank you.