**X-Ray Crystallography**
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**Lecture - 23**
**Tutorial 05**
**XRD - Laboratory Demonstration**

Today's class, I will just show you some demonstrations through the laboratory videos I will take you to our XRD lab. And then show the equipment details, how the X-ray diffraction equipment look like, and what are the primary components, and how one would perform an X-ray diffraction experiments in a polycrystalline material.

So, you will get a basic idea from this, though you will not be able to do other detailed experiments since it is only a part of this material characterization course I thought it would be very nice idea if you have a some detailed about how the equipment will look like and what are the basic details about XRD machines.

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So, if you look at the screen where I started showing this is the; a Bruker X-ray diffractometer. And you can see that it is all enclosed in a big box. You can see in it in a long shot and you also see our one of the scholars in our department is going to operate this machine for us and I will just show you the details one-by-one. So, this is X-ray diffractometer and you have the power console here and on and off and everything. So, it is a long shot. And what you are going to look at again? From now we have opened this equipment.

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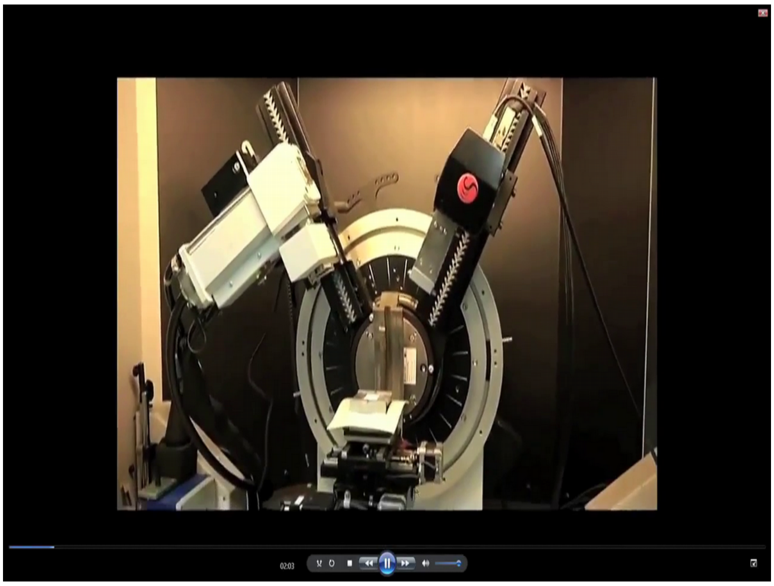

Now you are seeing the details of the diffractometer what you are seeing in the background I mean back side the big a circular disk is a goniometer; it is called a goniometer or and it is also referred as a diffractometer circle and then what you are now seeing is everything in a closer shot.

And do not worry you will be seeing this for very long time. So, you get the details this is the specimen stage where you have a specimens are kept whether it is a bulk sample or a powder sample a bulk sample is directly kept on this whereas, the powder sample is mixed with some medium and is kept on a glass slider something like that and this is the specimen stage we will talk about it much more detailed.

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So, what we need to know from this equipment is a basic component some of the basic components which you require is the X-ray source this is the X-ray source and from where we will talk about some slits and through which the X-ray beam comes out and then it is made fall on this specimen here. And this is a detector this is a detector again which has got lot of slits inside we will discuss about the role of slits. And then you have an X-ray detector in this case.

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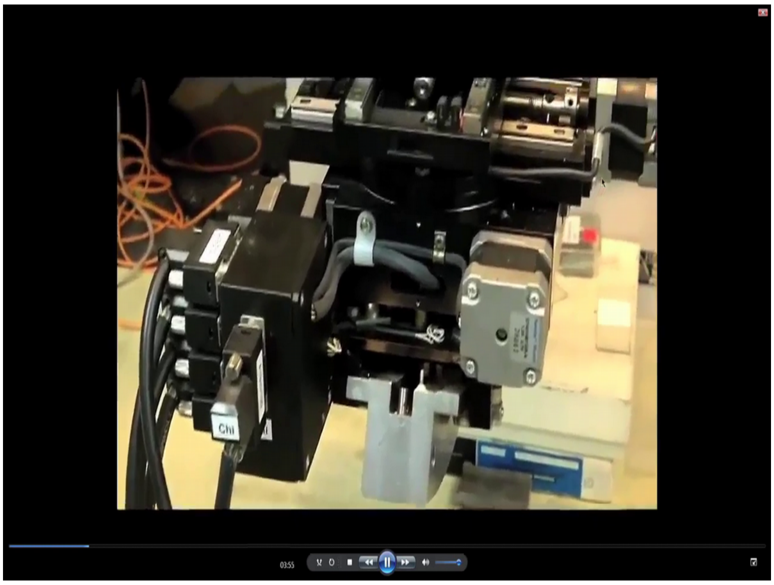

So, the primary component is X-ray source a sample stage and a detector you can see that all these three or shown in this. And one important thing you have to understand is the X-ray source.

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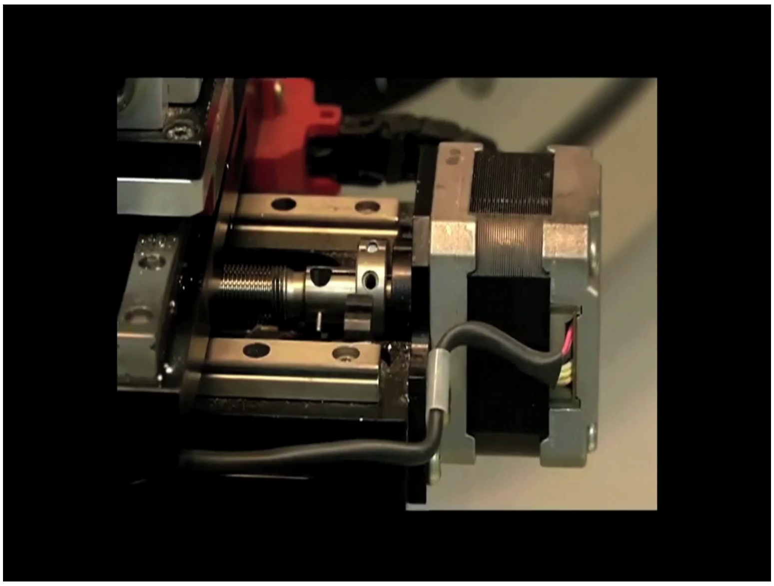

And a detector is kept intentionally on the goniometer circle you will just see that the X-ray source and the detector are going to be on the goniometer circle or you can say that diffractometer circle you just observe these details for some clarity these are all x y movement for the specimen stage. We will discuss about the importance of these movement, the specimen also can go in that semi circle fashion in this manner.

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So, what I will like to do is before our scholar perform the experiment let me also explain some of the basic components like you can see that what I would like to explain here is you just I will just stop the video here this is the goniometer circle your X-ray source as well as the detector are kept in the diffractometer circle or goniometer circle and the specimen is always kept in the centre here the centre stage. We will now just discuss the basic requirement of keeping the sample in the centre of this goniometer circle and there is a we will talk about X-ray optics before we do that experiment. So, what I will do is let me draw a line diagram on the blackboard in order to appreciate what you are now seeing.

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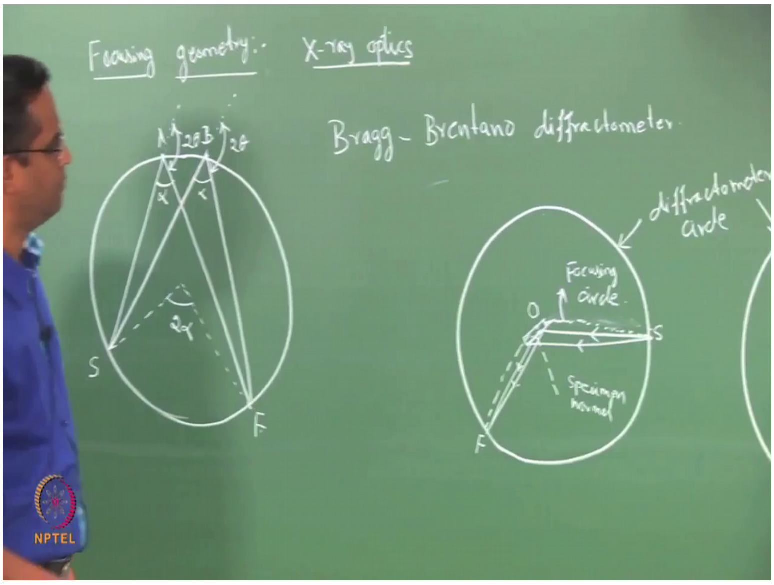


So, I will draw a focussing geometry. So, what I have drawn is a focussing geometry for X-ray optics suppose if you have the source here and then it goes to the sample and get reflected here and this is a another X-ray source which is falling on the sample in the point b and get focussed here in f and you see that the angle between this is 2 theta and this is alpha and this is alpha. And you can have the relation here with the circle inscribing the angle and what you have to keep in mind whenever you have the X-ray source which goes in to the a sample and gets back to the a point f that is the this is incident ray and this is diffracted ray are getting focussed again at a point f. So, you can say that a source of X-ray and this is your detector and this is your sample for example.

So, if you this geometry is maintained for all the scanning for all the scanning for variable 2 theta this is completely maintained for all the wherever you keep variable 2 theta this geometry is maintained and we can write. So, this is also called a Bragg Brentano diffractometer where this kind of a geometry is used and then I will draw one more schematic which will show the Para-focussing of the goniometer as well as the what the sample you are going to scan during various angles of 2 theta. So, I will draw 2 more figure then I will get into the discussion.

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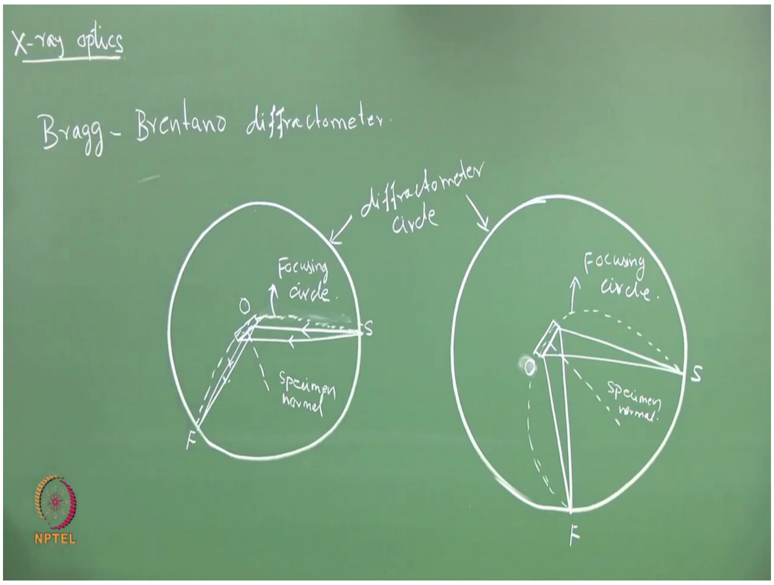


So, what I have drawn here is 2 types of 2 theta angle how this Bragg Brentano geometry or I would say Bragg Brentano far focussing method which is being adopted in this particular diffractometer. So, you have this is a diffractometer circle or goniometer circle which I have showed in the video and this is your sample which I said that. So, these 2 there are we are talking 2 circles here one is diffractometer circle or goniometer circle the other is a focussing circle.

So, what is this focussing circle depending upon the 2 theta it is going to vary. So, you see that you see your; as I said that your specimen detector and the source should be inner circle. So, assume that this is a focussing circle and this is your diffractometer circle depending upon the 2 theta it is going to vary this way or this way. So, in this case you see that this is the specimen and the specimen normal and this is a incoming source and this is a diffracted beam which is focussed and then you see that the specimen is kept in the circle on the perimeter of the circle of focussing.

So, depending upon the 2 theta you can see that the 2 theta is becoming smaller then you see that the focussing circle is also becoming smaller as the theta 2 theta becoming bigger then you see that it is bigger focussing also. So, this geometry is preserved and that geometry is called Bragg Brentano geometry and this kind of a focussing is called Para-focussing and which is being adapted in this particular diffractometer.

So, with this background now you go back and look at the video you will be able to appreciate what I am trying to say now if you look at the video again what I will do is now you can see the goniometer circle and then let me finish that. Before I go goniometer circle and this is the source and this is the detector and this is the sample which is kept in the centre of the goniometer circle. In order to maintain the focussing circle which will be here depending upon its going to vary right now it is small because now the 2 theta; theta is here this 2 theta 2 theta is this sample you draw a straight line and this is the diffracted beam. So, this angle is 2 theta. So, that is going to vary then you see that the focussing circle is also vary.

So, now we will look at much more detail about this about this is about the basic components of the diffractometer we will now show some of the important components here this is what now we are seeing here in the screen is the X-ray tube what is the typical X-ray tube one would look at . So, now, you have the better view this is an X-ray tube where you have the target material which is inside and later we will also open this and then show you some of the details of the X-ray tube this is how the X-ray tube will look like at least you have some idea and which will go in to that source what we have just shown. So, of course this the whole tube is under the cooling system and then you have the beryllium windows through which your X-ray comes out beryllium windows are transparent to X-rays to that detail I would like to finish.

So, have a close look at this X-ray tube. So, now, we will move on to an experiment now before even go to the experiment let me stop here.

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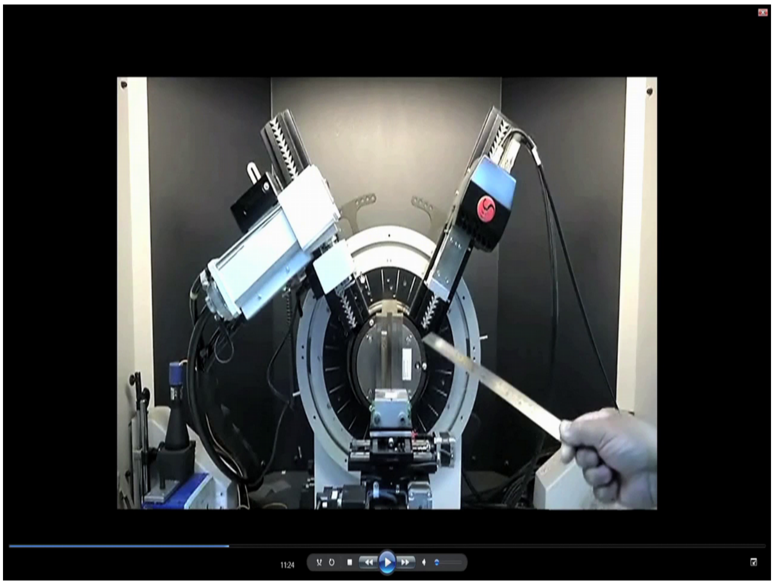

And then explain little more details about this. So, I said that the X-ray source is here and it is going to come out through this exit window and we remember the X-rays which are coming out of this window is completely divergent beam and it is divergent beam in all directions. So, it need to be controlled the one of the way to control these divergent is by through slits we have a typical slits which are popular and then which are kept inside this and then it is being made to fall at collimated beam is made fall on to this specimen and similarly the detector also will have a lot of slits and filters please remember we are interested in taking only a particular X-ray beam for example, k alpha and you will in generally if you without the slits you will also come across k beta and so on.

So, the other radiations are blocked by this filters for example, k beta is suppressed you cannot eliminate completely it can be substantially suppressed as compared to k alpha radiation the filters are here and this is a detector it could be a semiconductor devise as well. Now, I think that is the basic idea you should have about this X-ray diffractometer and then now we can even look at how a experiment is being performed in this machine.

And please remember this stages is a rotating stage it can rotate 360 degrees and also it can move same in a on a semi circle in the in the stage which I have mentioned now you can see that a typical stage movement in order to save I am just cutting some of the shots. So, that you will be able to see this you can see that the source and detector are moving, but still that focussing circle is maintained here to in order to mention that I mean preserve that Bragg Brentano geometry Para-focussing geometry we can see that the stage is moving.

Now it is measuring the beam with various angles 2 theta angles. Now we will see that the stage will also move in the semi circle fashion in order to one of the primary requirement or the use of this the stage movement is you will be exposing the sample in all possible diffracting planes. For example, if you are interested in particular plane or if it is a powder sample there the statistical importance statistically important data you will be able to generate if you are able to rotate the stage in all the three directions like this see that sample is coming towards front and x and y movement and you will be able to appreciate that.

Now, we will see that the sample will move in this direction it will rotate in this direction that also you can see; what you are now seeing is a top view the sample is now completely rotated you can see in that semi circle stage it has moved. Now it is coming back you can see that and this rotations are completely exploited in the in the case of texture measurements which we are not showing we are trying to just shown the instrumentation detail and what are the possible you know tilt angles and so on. And we will simply look at the basic diffraction data which comes out of this sample the sample which is being used as I think alpha silicon oxide powder and you will see that kind of diffraction data which will be obtained from this experiment.

You can see that specimen rotation completely it is a 360 degree this is just for your information the X-ray tube which I we showed earlier.

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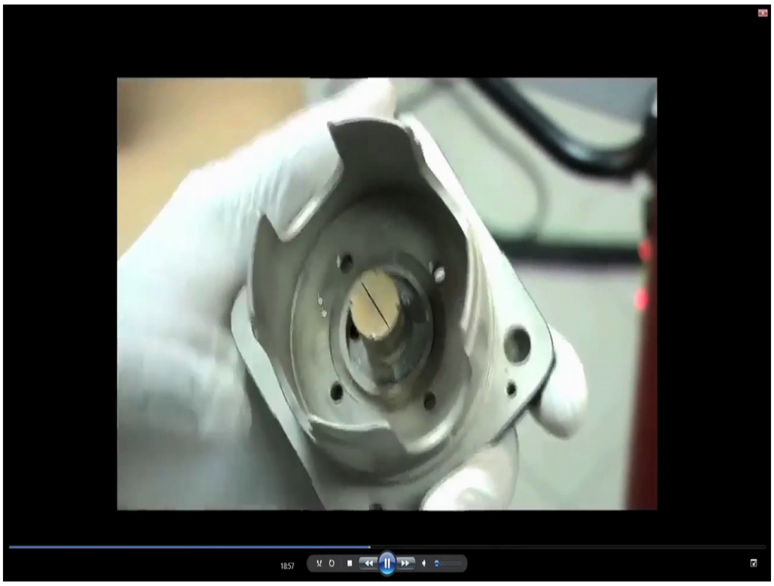

Now it is opened up this is your anode material it could be you can change this target material based upon the specimen you are analysing it could be a chromium or you know chromium target copper target and so on depending upon the type of sample you analyse and what the dismantle part is this is a close up view of this anode just for your information and this is that beryllium.

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Which we talk about through which the X-rays will come out this is a dismantle part not just any important here, but just to for an information how the X-ray tube parts are shown.

So, we will now quickly do a one full set of complete experiment and then generate an X-ray diffraction pattern and then we will see what we get. So, the data collected from the instrument is completely analysed by the interface software and we will show you how what kind of data you get and then how it is how the background everything is filtered by the software and then you can have a look at it. So, let the fresh experiment starts with all the possible 2 theta angle we will not hold this video for the complete experiment we will go to that final result. So, you see that the sample is kept here and then it is being measured at various 2 theta angles I will skip that step, because we now that how this diffractometer functions I will quickly forward this video in order to save some time.

Now, you are seeing that how you are dynamically getting that signal we are viewing through software you see that typical X-ray diffraction peaks are coming on the screen. So, all these 2 theta corresponding peak belong to a characteristic peak of silicon alpha silicon oxide which I just said powder a polycrystalline diffraction pattern typical polycrystalline diffraction pattern will appear like this and you can compare this 2 theta value and go to a GCPDS data base to identify particular crystal system of course, you have large number of software parameter to derive the data from I mean information from this basic data and this is a typical X-ray diffraction spectrum you will get.

So, with that what I think is you have some basic idea if not very detailed idea how the X-ray diffractometer look like and then how the source and the specimens are kept and how the detectors are kept and what is the basic data you get out of this equipment and I hope these equipments and this small demonstrations gave you some basic insight about an X-ray diffraction laboratory if you are not able to access these laboratory at least you know now how these equipment look like and how what are the basic operations behind this.

We have not done elaborate experiment here due to constraint of time, but I hope you had some basic idea and now you can connect what the what we do I mean theoretically what we study in the books and then what practically you can do in the laboratory when you have an opportunity look at these equipments in some other labs. Now you will have some idea how it is turned and how the basic data is generated. So, that was my intention of doing this laboratory demonstration.

Thank you.