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Lecture-33
Materials Characterization
Fundamentals of X-ray diffraction

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Hello everyone welcome to this material characterization course. In the last class we just looked at the applications of x-ray diffraction and we looked at typically three cases one is the crystal structure determination and we also looked at how to look at the stress measurements that is a basic principles of stress measurements and also we looked at the phase identification in a phase mixture we looked at all those very fundamental ideas behind the application of this x-ray diffraction and it is how the intensity is calculated and so on.

And today's class I will just show you some demonstrations through the laboratory videos I will take you to our X-ray 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 apart of this material characterization course I thought it would be very nice idea if you have some detail about how the equipment will look like and what are the basic details about in x-ray dimensions.

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So if you look at the screen where I started showing this is the Brooker x-ray diffractometer and you can see that it is all enclosed in a big a box you can see in a long shot and you can 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 okay now we have opened this equipment now you are seeing the details of the diffractometer what you are seeing in the background I mean backside the big a circular disc is a goniometer it is called a goniometer are and it is also referred as a diffractometer circle. And then what you are now seeing is everything in a closer short.

And you do not worry you will be seeing this for very long time so you get the details so this is a specimen stage where your 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 in depth on a glass slide or something like that and this is a specimen stage we will talk about it much more detail.

So what we need to know from this equipment is the 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 made fall on this specimen here. So and 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 cas.

So the primary component is x-ray source a sample stage and a detector you can see that all these three are shown in this and one important thing you have to understand is the x-ray source and a detector is kept intentionally on the goniometer circle you 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 on XY moment for this specimen stage.

We will discuss about the importance of this moment okay, the specimen also can go in that semicircle fashion in this manner. So what I would like to do is before reverse collared perform the experiments 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 this video here this is the goniometer circle your x-ray source as well as the detector are kept in the diffractometer circle or in the goniometer circle.

And the specimen is always kept in the center here the center stage we will now just discuss the basic requirement of keeping the sample in the center of this goniometer circle and that 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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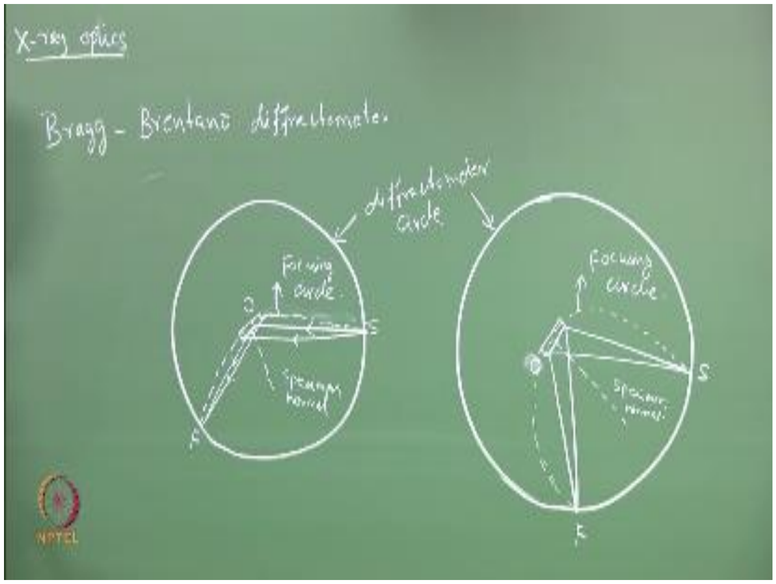

So I will draw a focusing geometry, so what I have drawn is a focusing geometry for x-ray optics suppose if you have the source here and then it goes to the sample and get reflected here and this is a another x-ray source which is falling on the sample in the point B and get focused here in the F. And you see that the angle between this is 2θ and this is α, and this is α and you can have the relation here, but the circumscribing the angle.

And what you have to keep in mind whenever you have the x-ray source which goes into a sample and get back to the a point F that is the, this is incident ray and this is diffracted ray or getting focused again at a point F. So you can say that this is 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 E for all the scanning for variable 2θ this is completely maintained for all the wherever you keep variable 2θ this geometry is maintained.

And we can write, so this is also called a Bragg-Brcntano diffractometer where this kind of geometry is used. And then I will draw one more schematic which will show the Para focusing of the goniometer as well as the what the sample you are going to scan during various angles of 2θ. So I will draw two more figure then I will get into the discussion.

So what I have drawn here is two types of 2θ angle how this Bragg Brentano geometry or I would say Bragg Brentano power focusing method which is being adopted in this particular diffractometer so you have this is a diffractometer circle or goniometer circle which I showed you in the video and this is your sample which I said that.

So these two there are we are talking about two circles here one is diffractometer circle goniometer circle the other is a focusing circle so what is this focusing circle depending upon the 2θ it is going to vary so you see that you see you are as I said that your specimen detector and the source should be in that circle.

So assume that this is a focusing circle and this is your diffractometer circle depending upon the 2θ 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 the incoming source and this is a diffracted beam which is focused and then you see that the specimen is kept in the circle on the perimeter of the circle of focusing.

So depending upon the 2θ you can see that the 2θ is becoming smaller then you see that the focusing circle is also becoming smaller as the 2θ becomes bigger then you see that it is the you are focusing circling also so this geometry is preserved and that geometry is called Bragg Brentano geometry and this kind of focusing is called Para focusing and which is being adopted 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.

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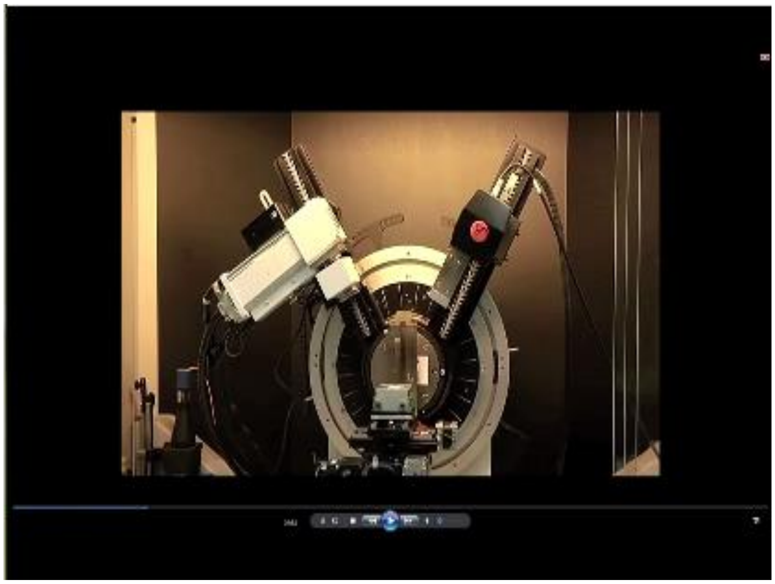

Now if you look at the video again what I will do is now you can see the done emitter circle and then that we finish that before I go done meter circle and this is the source and this is a detector and this is a sample which is kept in the center of the done emitter circle in order to maintain this focusing circle which will be here depending upon it is going to vary right now it is small because now the 2θ, 2θ is here this 2θ is this is sample you draw a straight line and this is the diffracted beam.

So this angle is 2θ so that is going to vary then you see that the focusing circles 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 okay this is what we are now she seeing herein the screen is the x-ray tube what is the typical x-ray tube one would look at okay.

So now you have the better view this is an x- ray tube wherein 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 that this is how the x-ray tube will look like at least you have some idea and which will go into 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 the power X ray comes out beryllium windows are transparent to excess so 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 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 you 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 this divergence is by through slits and we have a typical slits which are popular and then which are kept inside this and then it is being made to fall or 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 α and you will in general if you if without the slits you will also come across k β and so on so the other radiations are blocked by this filters for example kβ is suppressed you cannot eliminate completely but it can be substantially suppressed as compared to K α radiation the filters are here and this is a detector it could be a semiconductor device as well.

So 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 experiment is being performed in this machine and please remember this stages is a rotating stage it can rotate 360^0 and also it can move same in on a semicircle in the in the stage which I have mentioned now you can see that a typical stage movement in order to save time.

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 focusing circle is maintained here, too not to mention that I mean preserve that brag vented know geometry focusing geometry you can see that the stage is moving yeah know that it is measuring the beam with various angles to θ angles.

Now we will see that the stage will also move in a this the same a semi-circle fashion in order to one of the primary requirement are the use of this the stage movement is you will be exposing the sample in all possible and diffracting planes for example if you are interested in particular plane or if it is a powder sample the statistical important statistically important data you will be able to generate if you are able to rotate this stage in all the three directions like this see that the stamp sample is coming towards the front and x and y movement and you will you will be able to appreciate that and we will see that the sample will move in this direction it will rotate in this direction that also you can see.

Yeah what you are now seeing is a top view the sample is now completely rotated you can see in that in that semicircle stage it has move, now it is coming back you can see that and these rotations are completely exploited in the in the case of a texture measurements which we are not showing we are trying to just show 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 α silicon oxide powder and you will see that the kind of diffraction data which will be obtained from this experiment you can see that the specimen rotation completely set 360degreeokay this is just for work for your information the x-ray tube which I we showed earlier.

Now it has opened up this is where a node material it could be you can change this target material based upon the specimen you are analyzing it could be a chromium or you know chromium target copper target and so on depending upon the type of sample you analyze and what the other disappointing part is okay this is a close-up view of this anode just for your information.

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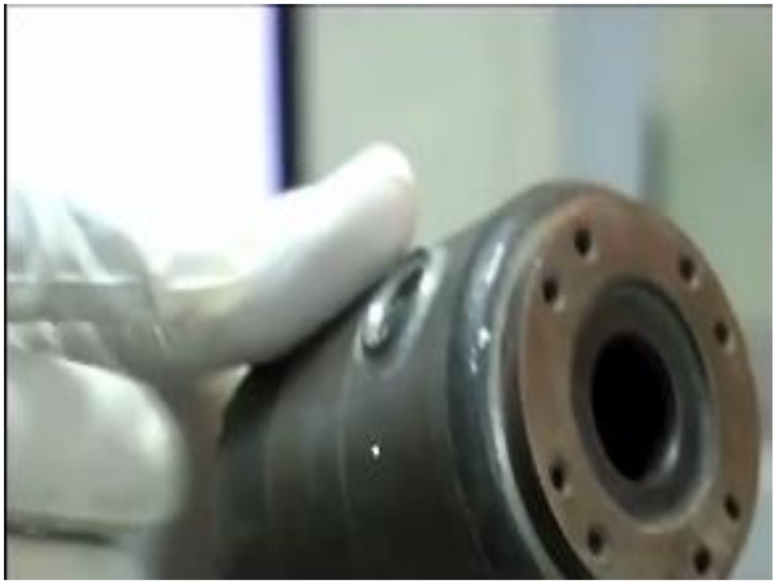

And this is the beryllium window which we talked about through which takes rays will come out this is a dismantle part not just turn it and important here but just to for an information how the exit tube parts are shown. So we will now quickly do a one full set of complete experiment and then generate an x-ray diffraction pattern and then we will see what we get.

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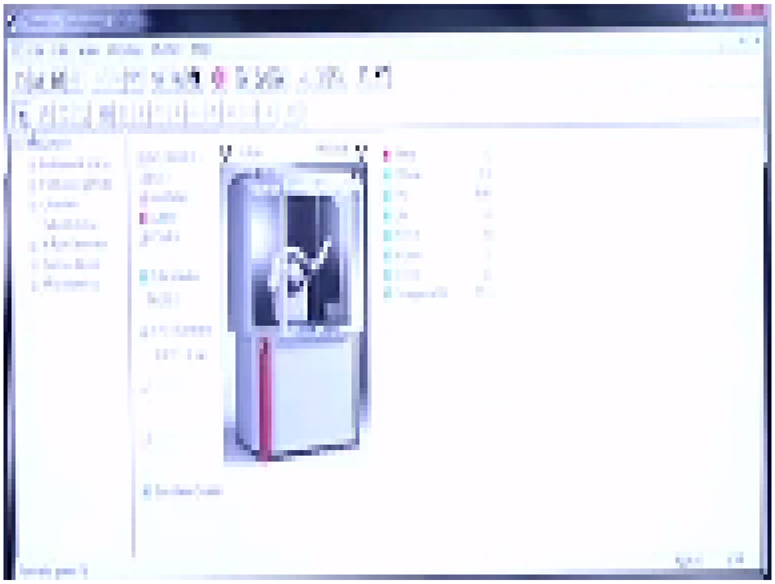

So the data collected from the instrument is completely analyzed by the interface software and we will show you how what kind of data you get and then how it is how the background everything is filtered by the software and then you can have a look at it so let the first experiment starts with all the possible to θ angles we will not hold this video for the complete experiment we will quickly go to that final result.

So you see that the sample is kept here and then it is being measured at various to θ angles I will skip that step because you have known you now you know that how this diffract meter functions will quickly forward this video in order to save some time okay. 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 this to theta corresponding peak belong to a characteristic peaks of silicon α silicon oxide which I just said powder a poly crystalline diffraction pattern typical polycrystalline diffraction pattern will appear like this and you can compare this to theta value and go to a JC pds database to identify particular crystal system of course you have large number of software parameters to derive the data from I mean information from this basic data and this is a typical x-ray diffraction spectrum you will get I will finish this I will go to the another important aspect of the system.

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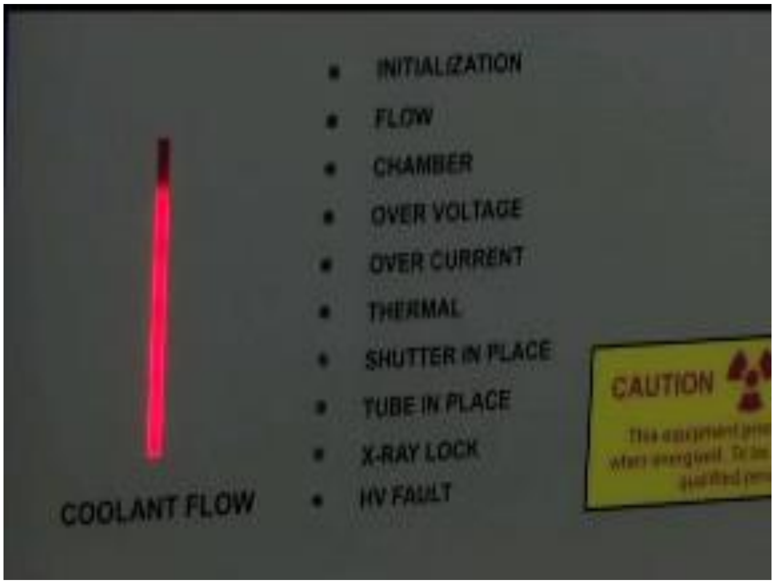

Whatever we are now seen as a powder diffract meter you see that another important source I would like to show is this the equipment which I am going to show you is another very important x-ray machine.

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Which measures the residual stresses in an RA stress measurement system basic which is industrially important?

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Then you can see that this is the equipment typical equipment you have a look at it and then we will discuss about the function functionality of this equipment so this is an extended arm where you have this I will probably stop here, and unlike the Bragg ventano geometry what we have just witnessed before which is not maintained here and here this is an x-ray source x-ray source are here it is not a diverged beam here it is a parallel beam parallel beam and the x-ray source straightaway comes there and then you see that there is a typical connecting rod which is being measured which is clamped through a stand here and the x-ray beams are simply falling on this connecting rod.

And then you have the two detectors besides this in fact the you are looking at the side view so the detector will move on this the curved of off circle stage like this so the x-rays will come straight on the sample and then get diffracted into the two that I mean detectors which is kept side by side to this source so the difference between the previous diffract meter and this is here the x-rays are a parallel beam and you have the direct collection of the diffracted beam and then you try to analyze the x-ray data.

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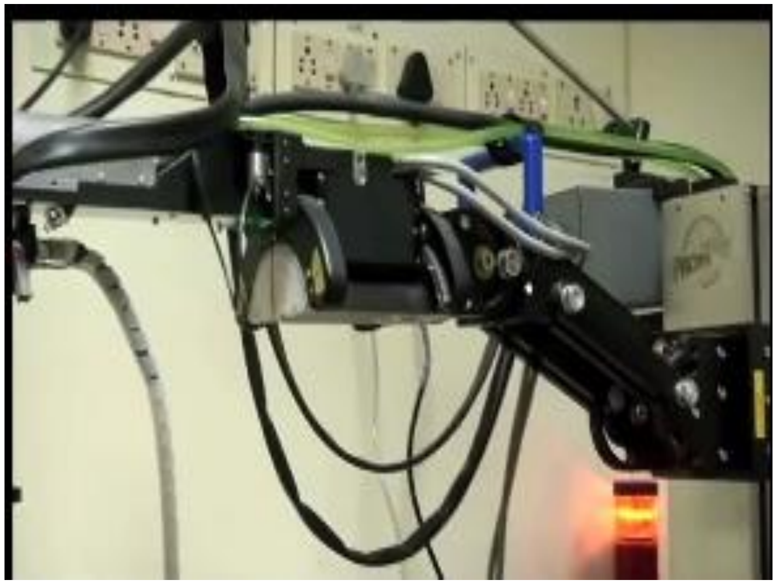

So now you can have a close look at this arrangement this is one of the unique facility of this our laboratory a stress measurements using x-ray diffraction okay you have no better clarity here yes the source which I am talking about this x-ray source and the two detectors are kept side by side the one advantage with the this kind of setup is you do not have any restriction on the specimen size any specimen which can be fixed into this stage can be brought you can see that.

Now how the scanning is done the x-ray source can rotate and that off circle and then you can see that this is a source now very clearly source and the detectors are side by side the source in the center and two detectors yeah now it scans for all the two theta measurements here. So this is a advantage of this particular mission of any component a big component can be scanned and then you will get the data.

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So with that what I think is you have some basic idea if not very detailed idea how the x-ray diffract meter 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 we have also demonstrated to you the stress measurement equipment how it scans there is a big difference between these two equipments and most importantly this the x-ray optics behind these two equipments are also quite different 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 this laboratory.

At least you know now how this equipment look like and how what are the basic operations behind this we have not done in elaborate experiment here due to the constraint of the 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 it in the laboratory when you have an opportunity to look at these equipments in some other labs. Now you will have some idea how it is done and how the basic data is generated, so that was my intention of doing this laboratory demonstration. Thank you.

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