Dr. S. Sankaran
Associate Professor
Department of Metallurgical and Materials Engineering
IIT Madras
Email: ssankaran@iitm.ac.in

Hello everyone welcome to this material characterization course. In the last class we have discussed about the x-ray intensity and how it is measured and what is the use of looking at this intensity profile and so on. In continuation to this we will look at the some of the important aspects of the intensity. In last class also I mentioned that there are two aspects important aspects about this x-ray intensity before we use this intensity calculation to determine the social structure or strain or texture or orientation and so on, which you will briefly go through in the coming classes.

But as I mentioned in the last class there are two things you have to keep in mind when you look at the x-ray intensity profile there is something called intensity max that is I max and then integrated intensity there are two aspects. So the integrated intensity is nothing but the area under the complete profile. So we are interested in calculating the integrated intensity and then I will we will discuss why this is so.

And I max is something which we can always relate it to Bragg law where no this $\lambda = 2D \sin\theta$ exact there are two things you have to again with respect to the Bragg law when we say that the set of planes are diffracting, so we assume that the planes are perfectly oriented with respect to the diffraction and in θ_B or Bragg law. So this is not going to be the same in reality, so there are two categories of θ we are going to discuss one is the diffracting plane which oriented exactly to the θ_B or Bragg angle are slightly different from θ_B.

So these are two aspects and they are going to have its own consequences in the x-ray intensity profile and this is what something which we are going to initiate the discussion today. So to understand the intensity profile I said that first of all we should know what are all the parameters which are affect the intensity in the last class we listed about what six parameters and couple of them you know that polarization factor and structure factor.

And then I said there are multiplicity factor and there is something called Lawrence polarization factor as well as temperature factor absorption factor and so on. We will not get into all this origin of these factors, but you should know some idea about these factors before we get into the intensity calculation and its start using this intensity x-ray profile for determining many things like the slow sector, a strain residual, stress and so on. So we will briefly go through what I discussed in the last class.

(Refer Slide Time: 03:45)
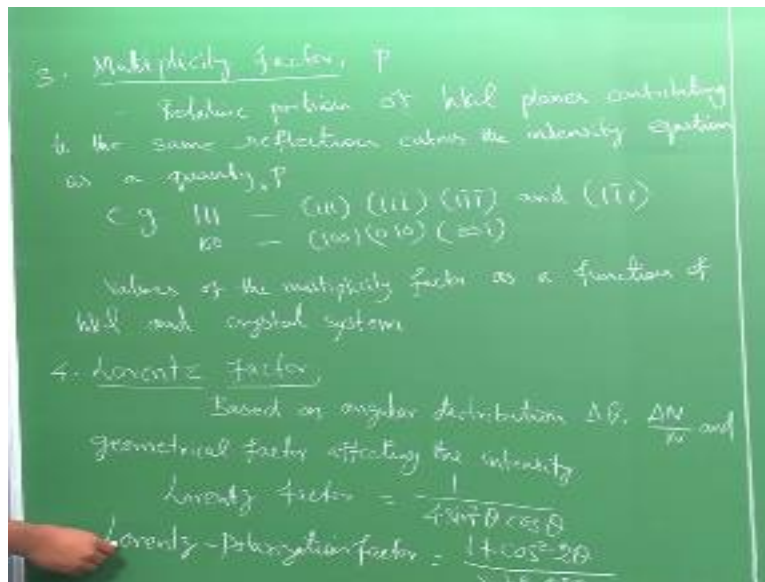
The first one which I am going to talk today is about multiplicity factor P. So what is multiplicity factor it is the relative portion of HKL planes contributing to the same reflection enters in the intensity equation as a quantity P. For example, if you take in a cubic system 111 for example

you can have these many variants and if you take 100 kind of a plane you have this many variants for example this is just for an example we can take.

So these all will contribute to the intensity in some form that is what the factor that is taken care of by this factor, but this factor will depend upon the HKL as well as the crystal system it is not a general one average crystal system obviously you know that these things will vary depending upon the HKL and the type of crystal system. So the factor will depending upon this contribution the reflection from these contribution is called multiplicity factor P.

So now the next factor is Lorentz factor, so the next important factor is which is going to affect the intensity is Lorentz factor let us go in that sequence that is a third and this is fourth in total six factors which are going to discuss what I am trying to say here is I am just highlighting the importance we are not getting into the derivation of these factors and their equations but you should know the physical meaning of this then it is fine.

Based on the angular distribution Δθ what is Δθ just I just talked about we have for example if you take a crystal like this.

(Refer Slide Time: 09:52)
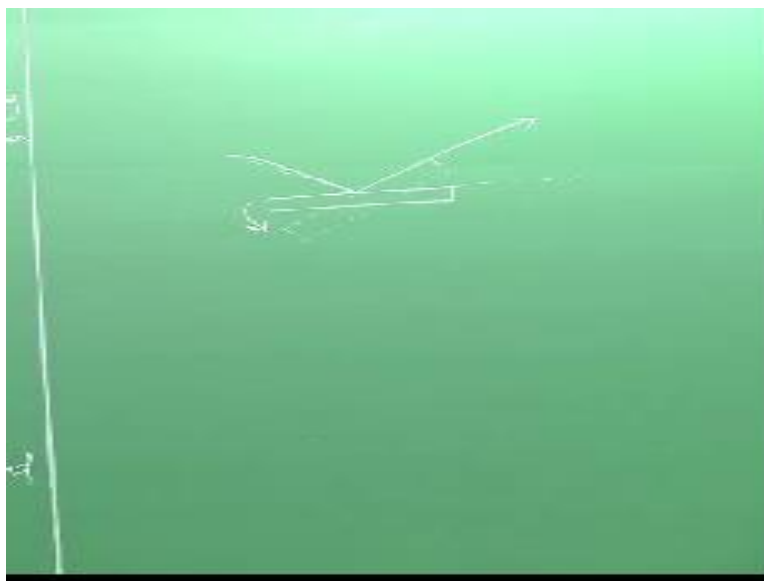
And this is your sample. So this is your sample and this is what incident ray and this your defected ray and so on. If you assume that this plane is oriented exactly to the Bragg condition that is θB and if I start rotating this crystal in this direction in such a way that it is slightly different from θB not exactly that θB that is a difference. So there is an angle up to which you can rotate this crystal and still get some contribution from that crystal to the intensity.

So there is a range of angle with you can do this that means you have setup planes which is oriented exactly to θB that is called exact bragged angle. And there are set of planes which are slightly away from this drag angle still contributing to the intensity. Why this is so why it is contributing that we are going to discuss little in another few minutes, but then to understand what is this Δθ this is the Δθ. The angle with which we are going to rotate but still we are going to contribute to the diffraction intensity is Δθ.

So what is Δn/n the fraction of crystal are the slides which are contributing to this event Δθ is Δn/n and some other geometrical factor affecting the all these effects put together a Lorentz factor is calculated I will just only write the final expression for the lack of time. So the Lorentz factor is written as $1/4\sin^2\theta\cos\theta$ and sometimes it is also combined with the polarization factor which we already know.

So Lorentz polarization factor is equal to $1+\cos^2 2\theta/\sin^2\theta\cos\theta$. So this factor also will take place in the intensity final expression. So that is about Lorentz factor we look at the other factors.

(Refer Slide Time: 13:52)
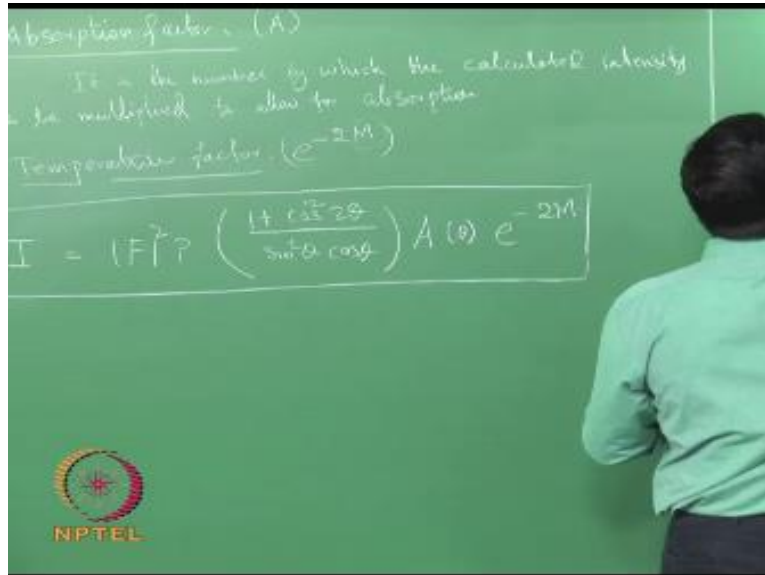
So it is simply a number by which a calculated intensity to be multiplied to allow for an absorption it is a material property some of the rays are absorbed. In fact we talked about this absorption in the very beginning of this course like in the when we discussed about the fundamentals like when the electromagnetic radiation interacts with matter one of the action is also absorption.

Other than you have many numbers of signals which comes out of the sample the absorption is also one of the event. So to account for that there is some factor to be multiplied so that is A absorption factor A and then finally the temperature factor. So we can simply relate this temperature factor what will happen when the temperature increases you are increasing the vibration of the atoms.

Obviously when you increase the vibration of the atoms this will also have some consequence in the distance of the I mean inter atomic planes D okay. So and depending upon the what kind of vibration and its amplitude thermal vibration and its geometry and so on this expression is given the final expression is e^{-2m} where m is the factor depending upon the amplitude of thermal vibration as well as the scattering angle 2θ.

So very briefly you can remember that m is the factor which depends upon the amplitude of thermal vibration as well as the scattering angle to theta so this is a temperature factor. Now you will try to write the final expression a full expression complete expression for an x-ray intensity which you observe in the typical x-ray diffraction experiment. So this is the typical intensity expression, so now you know all the factors which is involved and you also know some little bit about the meaning of each one F is a structure factor you have elaborately discussed that multiplicity factor Lorentz polarization factor absorption factor then temperature factor.

So all these parameters need to be taken into account before we start using this intensity of x-ray photon which comes out of the sample. So this is about the brief introduction about an intensity of an X-ray. Now we will now see get into a details of some very important aspects of the specimen influence on the intensity of the x-ray these are all very fundamental aspects but in a material science applications what we do normally we use x-ray photons as a probe to determine a crystal structure.

And we also use this to arrive at the residual stress profile and crystal orientation very important texture and so on. So since most of us are interested in material science to look at the crystal structure determination as well as the grain size determination today we are all interested in the grain refinement we are looking at very small grains.

And we the only probing tool which feed we quite often uses the x-rays for a grain size measurement or we will also say sometimes particle size measurement we will get into that details little later why what we call it is a particle what we call it as a crystallite what we call as grain and so on, there is we need to have some clarity on this. But before getting into that how this the crystal size crystallite size or a grain size is going to influence the x-ray intensity profile that we will see today little more detail.

So when we talk about the crystallite size in if you look at the Bragg derivation what we have gone through before what we said is we have just we have drawn a parallel planes of atoms and then we said that x-ray is just impinging on the surface and then it get diffracted. And then we

talked about the path difference between a subsequent layers I mean the which is distance with D that is V spacing.

And when it is the post differences with the which is integral multiple of λ then we said that it will contribute to the diffraction this what we just said and we derived a couple of variations one are based on the scalar equation another is the form of vector equations. So when we talk about a diffraction we always said that we be get the diffraction because of the constructive interference and that is a consequences of a periodicity of the lattice you have a complete three-dimensional periodicity.

So when we talk about a periodicity and its importance to the constructive interference the same consequence is valid for a destructive consequence also or destructive interference also. So we have two important we have to understand the important of this point like the destructive interference due to the periodicity.

So what we understand when we take a plane A which is refracting in one direction and then we say that plane B which is below from the plane a differing the distance D will also contribute the diffract provided if the phase difference is in the order of one wavelength or we will say that the atoms in plane A will diffract the plane B or atoms B out of phase by one wavelength if you assume that λ=2dsinθ.

We just assume that λ=2dsinθ then these two plane the diffraction wave will differ in there I mean path difference or we will say that it will be out of phase by a one wavelength. So if you consider the orientation which we talked about all this statements are valid provided the diffraction angle θB is exact Bragg angle, if it is not an exact Bragg angle if it is slightly away in a positive manner or a negative manner plus or minus then this destructive interference is not going to take place.

Because even the planes which are very slightly away from the Bragg angle they are going to contribute to the diffraction intensity that is what we have just seen just two minutes before just I said Δθ. So when suppose if you assume that all the surface layers they are going to contribute to

their were diffraction intensity that means they are all within the range of $\Delta\theta$ the plane which is containing atoms which are going to scatter the x-ray from the, and I mean the path difference I would say from the top layer the layer which is going to differ half the wavelength is going to be lying much more deeper inside the crystal.

If you have the layers of atom which are going to vary in their θ $\Delta\theta$ that is only slightly differ from the Δ or I would say try two different from θB the plane which is going to diffract the x-rays which will differ in their path difference by half wavelength why only with the half-length wavelength it is going to completely cancel out the amplitude which is scattered from the top layer.

When you have this kind of a situation that the plane which we talked about which is going to cause the destructive interference which is going to do lie very deeper, very deeper. And if you assume that your crystallite size itself becomes very small then such a plane will not exist that is the plane which is containing atoms which are going to cause the destructive interference will not exist.

So you have to, this is the significance of the crystallite size and its consequence towards the diffraction intensity for that we need to I mean arrive at some expression what causes this and then I will just draw a schematic which will explain this much more clearly.
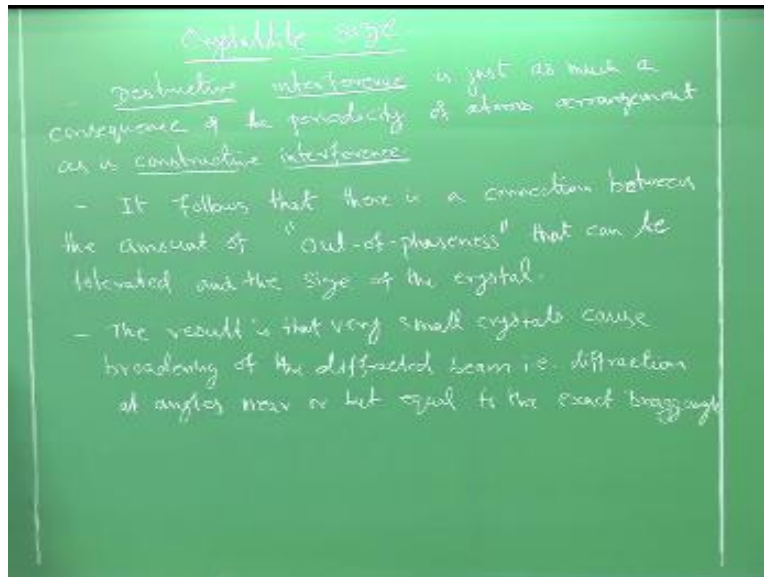
So before I really draw that schematic LLP reinforce this statement what I just spoke about. So the destructive interference is just as much as the consequence of periodicity of atoms arrangement as is the constructive interference. And because of this existence of planes are crystal systems where you have the concept of $\Delta\theta$ a range of angle by which the diffraction can still contribute to the intensity in that context it follows that there is a connection between the amount of out of phaseness that can be tolerated and the size of the crystal.

What I talked about the plane which are really going to do the job of the phasing out the previous intensity amplitudes there is a cut-off here you can see there is a cut-off and a short connection between the amount of out of business and the size of the crystal. The result is that very small crystals cause broadening of the diffracted beam that is diffraction at angles near but equal to the exact Bragg angle.

So this is the first consequence of a peak broadening, because of the crystal size why the crystal size is important are they the peak broadening is due to the small crystal size because of this effect.

(Refer Slide Time: 33:19)
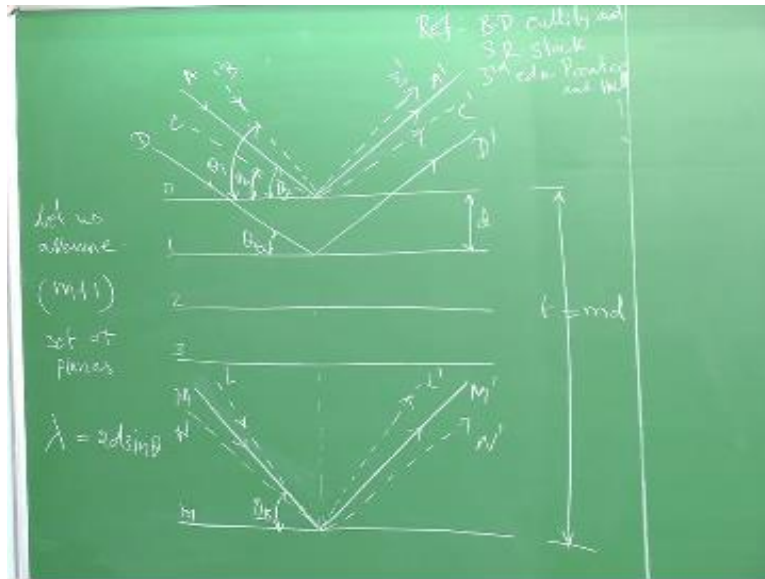
So we will now prove this with the small schematic, so let us assume that in this schematic there is m plus one set of planes, let us assume you have m plus one set of planes. And then we say that the diffraction follows this. So that means you have the ray x-ray coming A' this diffraction D D' you can put.

So this is exactly 1 λ path difference and as I said you have a Δθ range here where a B' I mean x-ray be and the B' if you look at it that is slightly different from θB. And similarly you have to see a C' which is also a slightly different from θB that means it is not exact Bragg angle but then still they will be contributing to the θ1 and θ2.

And similarly you can consider the same thing here M' is θB where you have L' and N'prime. So this extension is shown here just to accommodate all the planes which is in the sample of a thickness T with the D spacing D just so that the whole schematic represent a bulk sample. So now we can talk about this the path difference and its consequence.
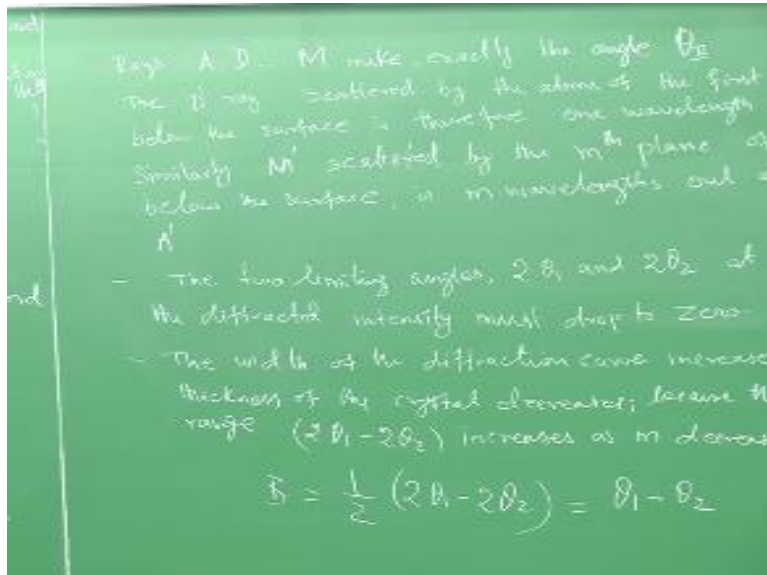
So the point is like I just mentioned with respect to the schematic AB and M raised make exactly the angle θB. So the D' or D dash ray scattered by the atom of the first lattice planes below the surface is therefore one wavelength out of phase this is a one wavelength out of phase we can write it like this. And similarly you see that the M' is scattered by the nth plane of atoms below the surface is M wavelengths out of phase with respect to A' with respect to this C'.

So similarly we can write we can think of this be B' and CC' they are not exactly the Bragg angle and they will they will be scattered by M+1 plane of atoms below the surface and then they will have NM+1 wavelengths out of phase in this case and similarly C and C' will have M-1 plane of atoms below the surface they will be differ by I mean they will be out of phase X-1 wavelengths with respect to this A'.

So we can write similar things for that so we can say that the two limiting angles 2θ1 and 2θ2 at which, so you have two limiting angles at which the diffracted intensity must drop to zero because of this slightly different from the brow angle. So we can also write that, so the width of the diffraction curve will increases as the thickness of the crystal decreases this also we can keep that in mind.

Now we can now write the expression for the B this is because the angular range θ1 - θ2 increases as M decreases. So that is one of the major consequence of the broadening, now we can write B = ½ (2θ1-2θ2) θ1-θ2.

(Refer Slide Time: 48:13)
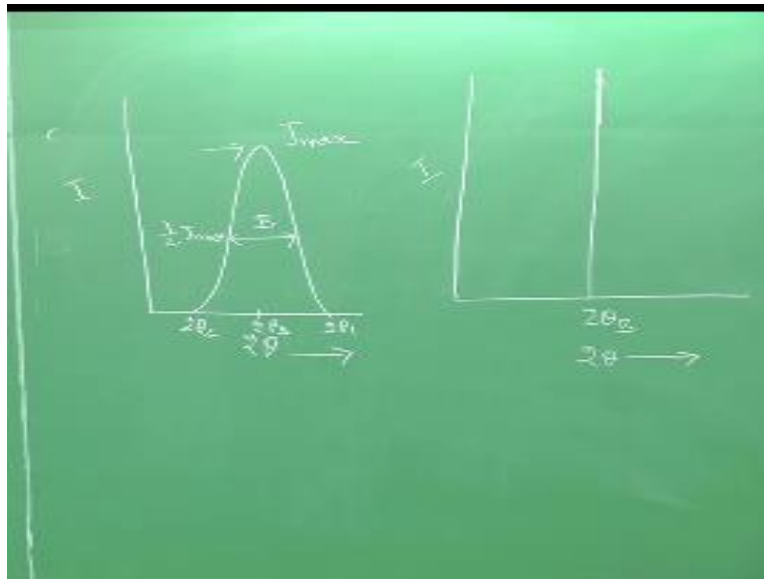
Now we will just, so you have the two type of pattern you will get as an effect of crystal size this is a full width of maximum you can see that B which is in nothing but an angular range between I mean plus or minus θB exact condition and we can now relate this we can we can write a path difference for the two curves but what to raise what we just talked about and then we can arrive at an expression to find out the type of the slide size I will continue that derivation in the next class.

So this particular schematic tells you that and it clearly demonstrates that plane or the x-rays which are not necessarily diffracting with θB will also will contribute to the diffraction intensity or slightly different from the Bragg angle exact brand angle will also will contribute to the diffraction intensity, that is the significance of this schematic and then how that is able to explain

the line broadening or a peak broadening in an x-ray diffraction spectrum in the case of a small grain or a small slide size that we will continue in the next class thank you.