Techniques in Materials Characterization

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Module No # 14
Lecture No # 56

Intensity of diffracted beam(Continued) & X-ray diffraction profile and analysis

Welcome everyone to this NPTEL online certification course on techniques of material characterization. And we are discussing about X-ray diffraction this is module 12 that this is the last module of this course and in this lecture, we will be discussing continue the discussing about the intensity of diffracted beam. And we will try to start discussion about X-ray diffraction from a real specimen and how to analyze that how to get information out of the regular X-ray and X-ray pattern which you normally use to see in X-ray diffraction profile.

So how to analyze that profile and how does it look like all of this we will be discussing in this module basically. So from the last module we were discussing about intensity diffractive beam and we have discussed about the structure factor and various other factors we discussed about the multiplicity factor and we discussed about we started discussing about temperature factor.

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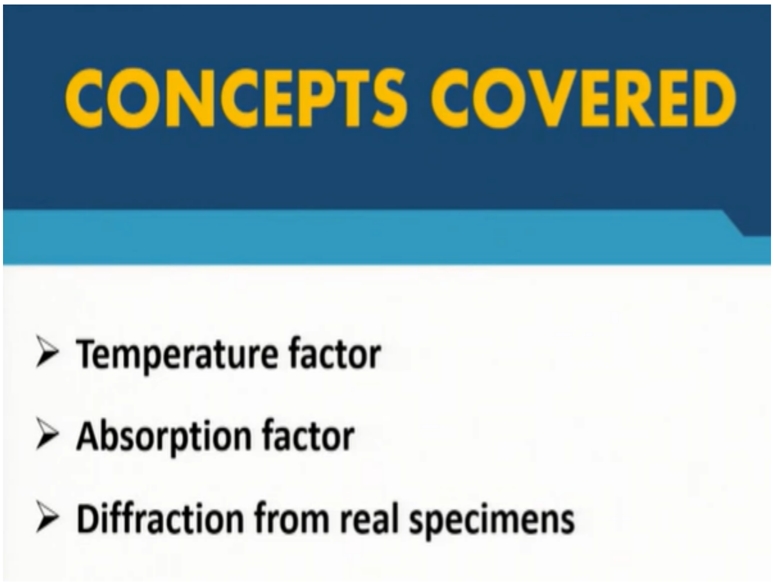

CONCEPTS COVERED

- Temperature factor
- Absorption factor
- Diffraction from real specimens

So we will be continuing with the temperature factor now and we will discuss about another factor that is called absorption factor. After finishing this 2 we will try to address this question of diffraction from a real specimen.

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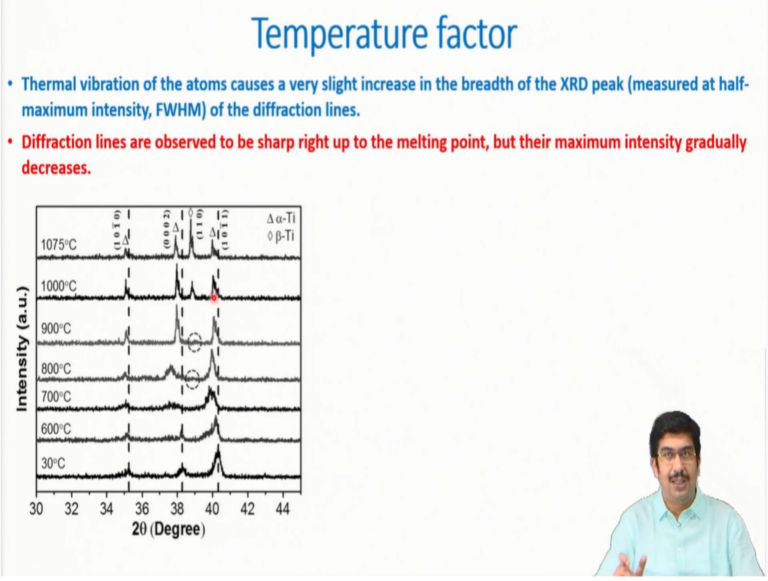

Temperature factor

- Thermal vibration of the atoms causes a very slight increase in the breadth of the XRD peak (measured at half-maximum intensity, FWHM) of the diffraction lines.
- Diffraction lines are observed to be sharp right up to the melting point, but their maximum intensity gradually decreases.

So temperature factor if you remember we discussed that temperature basically this factor comes because of thermal vibration of the atoms that causes the change in the d value which is again seeing the shift of the peaks in 2-theta axis. The peak is shifted that is one expansion of the unit cell is one reason for this thermal or one of the effect of the thermal vibration that is one. And then we have seen how this thermal vibration is basically causing the intensity decrease in intensity of the diffracted beam with temperature increase.

Because the constructive interference that happens from planes from a parallel or from parallel atomic planes when an incident X-ray beam falls on this planes and Bragg's angle Bragg's condition is satisfied then the constructive interference is expected but that constructive interference does not peak or it does not happen in the prefect way. Or if there is an imperfection in the constructive interference or in the interference partial interference happens just because of this thermal vibration which we discussed in that.

And also we understood that the thermal vibration and that is basically the imperfection brings down the intensity of the diffractive beam overall. And this decrease is more seen at higher 2-theta angle because there it involve with low d value low inter clearance spacing planes. So this where what we discussed in the last class and what now we will be discussing couple of other factors or couple of other characteristics of this temperature factor or temperature effect.

So first of all, the thermal vibration as we just now we discussed that the thermal vibration basically changes the 2d in a unit cell it basically changes the inter planar spacing just because of this atomic vibration. The unit cell expands so as an effects to that what will happen is basically the peak can shift in 2-theta in the 2-theta axis the peak can shift. And this basically if as we will be understanding when we discuss about the diffraction of real specimen will understand this change in 2 theta means the peak width basically should increase in the process.

In the overall process the peak width should increase because if you imagine the temperature if not an uniform temperature increase then a different places in the specimen at different places different unit cell will have different 2d values. Different lattice parameter or different unit cell will expand differently that will cause different value for 2d. And that means for a particular plane the d spacing will be different in different places all of them if now gives rise to the diffraction condition if they satisfy 2 different 2 theta value different sin2ϴ values.

And that will ultimately be seen in the broadening of the peak that is what we call so this is basically is one of the effect but what happens is in thermal vibration? This thermal vibrations of the atoms along their mean position this causes a very slight increase in the breadth of the XRD peak which is measured at half maximum intensity also known as FWHM which we will discuss in this class if possible.

So this increase in the breadth of this XRD peak is very less so that means this peak shifting or peak broadening effect that we discussed because of the unit cell expansion and all is very minute effect or this thermal effect in increasing the peak width is a very minute effect. So mostly the way thermal vibration effects the intensity or diffracted beam intensity is basically they reduces this diffracted beam intensity.

But they did not cause much of a broadening of the peaks so diffraction lines all the way if we go up to melting point the diffraction in remains quite sharp as sharp as the used to be at room temperature for most of the materials. So that is the good news that otherwise that effect will be very difficult to sort of characterize or that effect the big broadening effect will be very difficult to measure just because of the thermal vibration.

So you can see this is one such experiment than on titanium alloys or 2 different titanium alloys and here you can see at room temperature this is one. So this is a phase transformation this is a classic case a phase transformation which I was discussing in last module gamma to alpha if you remember how the austenite ferrite phase transformation happens. And how different peaks evolves from which we can identify this transformation.

So here also you can see the same thing that we increase the temperature we are getting a completely different peak. Altogether that is coming out at a certain temperature at 1000 degree onward completely peak with which is not there in this low temperature X-ray diffraction profile basically alpha titanium hexagonal whereas beta titanium is body centered cubic. So that peak appears only at high temperature and from there we can find out temperature this transformation temperature we can find out which alpha how they are related these 2 phase.

How they are related? Lots of other things we can identify but the important point is if we look at any of these alpha peaks let us imagine we are just interested in any of this alpha peaks we can see that intensity is basically going down. If we imagine from here at room temperature all the way up to 1000 degree the intensity of the alpha peak is slowly going down. But the width of the alpha peak does not really change that much so that is what I was meaning that intensity.

Mostly thermal vibration effects in the overall intensity of the peaks rather than changing the peak position as such that is first thing.

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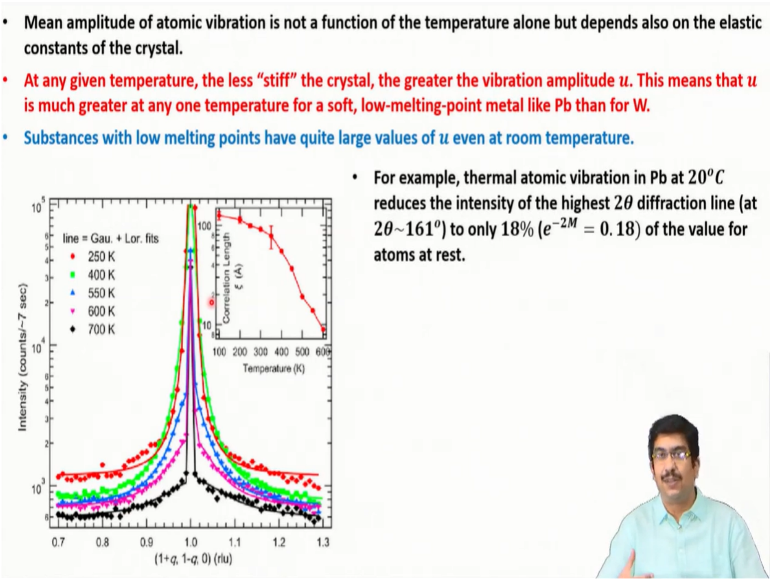

- Mean amplitude of atomic vibration is not a function of the temperature alone but depends also on the elastic constants of the crystal.
- At any given temperature, the less "stiff" the crystal, the greater the vibration amplitude u. This means that u is much greater at any one temperature for a soft, low-melting-point metal like Pb than for W.
- Substances with low melting points have quite large values of u even at room temperature.
- For example, thermal atomic vibration in Pb at 20^oC reduces the intensity of the highest 2θ diffraction line (at $2\theta \sim 161^o$) to only 18% ($e^{-2M} = 0.18$) of the value for atoms at rest.

The second thing is that this mean amplitude of atomic vibration this is not only a function of temperature but also a function of bonding characteristics if the metal material which is reflected in terms of elastic stiffness or elastic constant of the material. So we know that elastic constant is basically a intrinsic property and intrinsic property means it only depends on the bonding characteristics or the inter atomic distance between the 2 atoms.

So that detects the elastic constants so that means the mean amplitude this atomic vibration is also an intrinsic as a result of this intrinsic properties or it is a result it is manifested through this intrinsic properties. It is related to the binding characteristics of the materials it is not only the temperature along. So what does it means is that at any given temperature if the bonding is stronger if the material is stiff if the crystal is stiff then this vibration amplitude will be lesser.

That means if the bonding is less then the atomic vibration the amplitude of this atomic vibration will also be higher. If the less stiff material for example if we go something like lead which is very low atomic number. And eventually very the bond strength is much lower compared to something like a heavy high number of high atomic element high atomic number element like tungsten.

Where it s much higher it is having an elastic constant which is much higher than something like a lead at least so if we compare these 2 different materials all together lead and versus Tungsten then this temperature effect or rather first we start with the atomic vibration. So the atomic vibration for lead the amount of this average displacement about the mean position. This will be much higher for let that means u which we discussed in the last class that u value will be much higher for lead as compared to the tungsten.

So again the peak the decreasing intensity with the temperature that will be much more prominent for something like lead as compared to Tungsten. If the material is stiff if the bonding is much stronger then this atomic average displacement atomic vibration will be less correspondingly the decrease in intensity due to the increase in temperature will also be less. That means in case of if you increase the atomic number if the bonding is very stronger that will increase this or that will keep help to keep or to retrain the intensity of the diffracted beam.

So other way around we can say that substances which has low melting points and low melting points of course is because they have low strength of the bonds are very low. So melting points substances will have large u value even at higher temperature. You can see this value for lead is given here 20°C which is very close to the room temperature this reduces the intensity of the 2-theta diffraction line at a very high 2 theta.

Of course very high 2-theta we already discussed that this temperature effect is more prominent for higher 2 theta peaks. So their this intensity is only at 20°C room temperature intensity is only 18% of the value of atoms at rest which is absolute 0 temperature close to theoretical value. But almost it has decreased almost like 82% from that value. So that kind of decrease we can expect with this.

So here also we can see that if you increase the temperature the intensity drastically decreases compared to something like at close to room temperature or at lower temperatures. So that is important point to remember is that not only the temperature. But also the characteristic bonding characteristic bonding strength all of these things is important when we calculate or when we consider the temperature effect on the diffracted intensity of X-ray beam.

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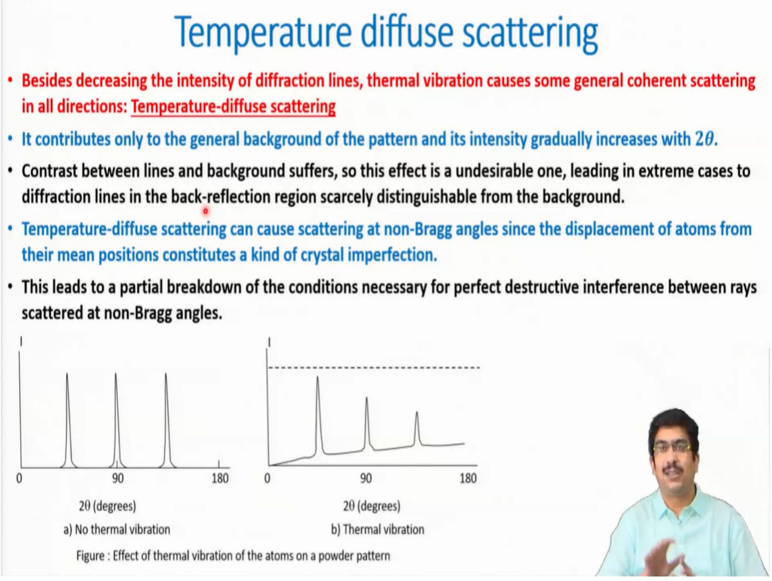

Temperature diffuse scattering

- Besides decreasing the intensity of diffraction lines, thermal vibration causes some general coherent scattering in all directions: Temperature-diffuse scattering
- It contributes only to the general background of the pattern and its intensity gradually increases with 2θ.
- Contrast between lines and background suffers, so this effect is a undesirable one, leading in extreme cases to diffraction lines in the back-reflection region scarcely distinguishable from the background.
- Temperature-diffuse scattering can cause scattering at non-Bragg angles since the displacement of atoms from their mean positions constitutes a kind of crystal imperfection.
- This leads to a partial breakdown of the conditions necessary for perfect destructive interference between rays scattered at non-Bragg angles.

a) No thermal vibration

b) Thermal vibration

Figure : Effect of thermal vibration of the atoms on a powder pattern

So now there is another effect of this temperature effect called the temperature diffuse scattering. So what happens is beside the decrease in the overall intensity of the peak the thermal vibration also causes something like increase in the background scattering, it causes a general coherence scattering which goes into the background in making the background of this intensity.

This does not take part in the diffraction phenomena diffraction this thermal diffuse scattering is nothing to do with the diffraction phenomena constructive interference. So that anyway is there and temperature increasing temperature is anyway affecting that constructive interference. Other than that, it produces some other coherent scattering effects in all possible direction which is basically increases the diffraction or basically increases the background for all possible 2-theta values.

At all possible direction at all possible 2-theta values it has nothing to do with the Bragg’s condition satisfaction of the Bragg’s condition. This background pattern also increase with 2-theta values. So as such the intensity of the peaks decreases with higher 2-theta values plus this temperature diffuse scattering that intensity also increases with the 2-theta value. So over all what happens is that the higher 2-theta values because of the thermal vibration 2 things happen are very high 2-theta values.

Number 1 the intensity or the beam or intensity of the diffracted beam anyway we will decrease plus the background also increase because of the temperature diffuse scattering. And at some ultimate cases very close to the back reflection 160 or 170° if those conditions it is very difficult to identify the peak from the background itself because of this 2 effects. Intensity of the diffracted beam decreases plus the background intensity also increases these 2 effects.

So if we see here that these are the 3 peaks if these are the 3 peaks which are of the same intensity due to if we ignore the thermal vibration effect or this temperature effect if we ignore if they have the same intensity high angle peaks of course very high angle peaks. If they have same intensity if we keep the temperature factor side then they will have this kind of a temperature this kind of an intensity profile because of 2 effects.

One their intensity will decrease if you see here if the intensity is constantly decreased due to the temperature, The thermal vibration or effect temperature continuously decreases due to thermal vibration effect at higher and higher at theta value plus this background continuously increases with 2 theta value. So both of them ultimately is affecting the absolute intensity of this peaks at higher 2 theta values.

So this is basically the another effect of temperature and why this is happening is basically the scattering at non-Bragg's angle is happening. This temperature diffusion scattering as I just explained the temperature diffuse scattering happens at non Bragg's angle and because of the displacement of atoms from their means position. And this is somewhat is similar to a crystal imperfection which we will be discussing in this module in the next few lectures we will be discussing.

So this basically causes a X-ray scattering at non-Bragg's angle and that will be sort of in some sense that destroys the condition necessary for perfect destructive interference between X-rays other than non-Bragg. So if you understand the constructive interference is completely it is related to the Bragg's condition satisfaction of the Bragg's condition. Other than those Bragg's condition other than this sin theta value ideally we should have complete destructive interference.

But because of this temperature diffuse scattering we get some intensity for those non-Bragg angle diffraction know Bragg angle values. There also we will get some limited intensity of the X-ray beam not everything will undergo destructive interference the perfect destructive interference will also be. So temperature effect basically number 1 not only it will take that perfect interference it will effect but also it will also effect the perfect destructive interference.

So one of this effect imperfection in the constructive interference it will decrease the intensity of the peak and imperfections from perfect destructive interference will increase a background intensity. So ultimately this is how the temperature will effect or both of them and that will be reflected in the overall intensity of the diffractive beam.

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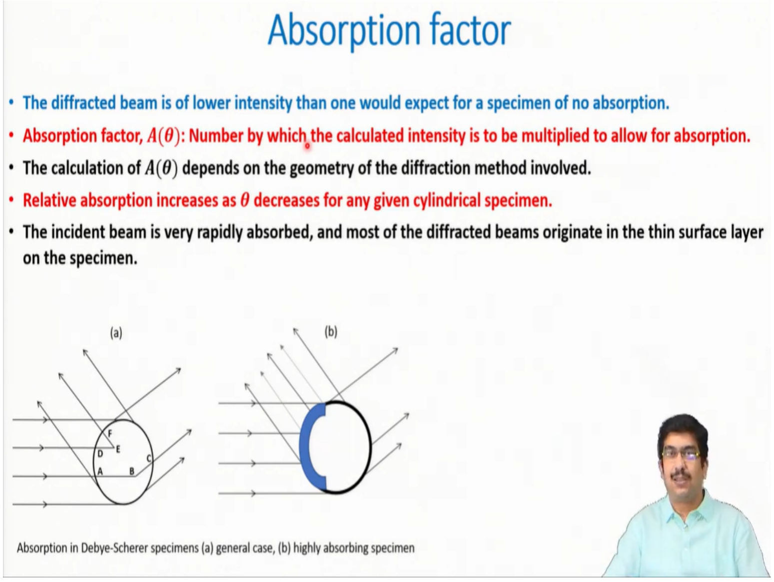

Absorption factor

- The diffracted beam is of lower intensity than one would expect for a specimen of no absorption.
- Absorption factor, $A(\theta)$: Number by which the calculated intensity is to be multiplied to allow for absorption.
- The calculation of $A(\theta)$ depends on the geometry of the diffraction method involved.
- Relative absorption increases as θ decreases for any given cylindrical specimen.
- The incident beam is very rapidly absorbed, and most of the diffracted beams originate in the thin surface layer on the specimen.

Absorption in Debye-Scherer specimens (a) general case, (b) highly absorbing specimen

So, with this we are coming to the last factor that we will be discussing about intensity of the diffracted beam and that is the absorption factor. So, absorption factor is basically related to absorption as the name suggests it is related to extra absorption and X-ray absorption we have already discussed in the first few lectures on X-ray. There we discussed how the absorption happened through absorption and all of this.

So, we are not going into that but what happens is that the diffracted beam is of lower intensity than what we expect if the specimen is exist without any absorption. So, if the absorption is not there within the specimen, then the diffracted beam intensity will not decrease because of this factor. But since limited amount some amount of absorption will always happen in the before the incident extra beam hitting the specimen and the diffracted beam reaching the detector.

In the entire process there will be some amount of X-ray absorption that will definitely happen and that will decrease the intensity of the diffracted even further. So, the way this one is included in the calculation of diffracted beam intensity we call it absorption factor and this absorption factor is expressed in terms of A(Θ). This is a number by which the calculated intensity is ultimately to be multiplied.

So, this is very similar to the temperature factor we will discuss how these 2 are related absorption factor and temperature factor. But basically, multiplicity factor, temperature factor, absorption factor all of this ultimately gives me the number through which the diffracted intensity should be multiplied. And to take care of individual all, of these effects now the calculations of this absorption factor this related to the geometry of the diffraction method involved.

We are not going into geometry of the diffraction we largely vaguely saying we can have various type of geometries. For example, Laue diffraction, Debye Scherrer diffraction method diffraction method powder diffraction method various type of diffraction experiments are there. Where the geometry ultimately it is all about the Bragg's condition satisfaction and all. The geometry of the diffraction means the X-ray source the relative position of X-ray source and specimen and the detector.

So this and what type of detector we use what type of source we use based on this X-ray diffraction geometry will vary. We are not going into that but this absorption factor for all other factor this does not matter much. But for absorption factor this really depends on what type of geometry we are using for example if we use or if we think of something Debye Scherrer method which we are not discussing.

But if you are interested we can go through the book reference book and I can read about it what is Debye Scherrer method. So in Debye Scherrer method what happens is that we use in the specimen in form of the cylinder basically something like this. So here from this cylindrical specimen what we can understand is that absorption increases as theta decreases as the absorb as the angle of scattering decreases the absorption increases for this kind of a cylindrical specimen.

Now why is it so? This is because if we understand or if we see this kind of a material this cylindrical material. So the incident beam is entering and then it is getting diffracted for different scattering angle different 2 theta value it is getting diffracted. Now for different type of 2 theta values the incident X-ray and the diffracted X-ray the amount of or the path they have to travel within the specimen that varies depending on theta.

So if we imagine something like a low 2-theta almost like a forward scattering low 2-theta diffraction in that case this the incident beam and the diffractive beam they have to spend more or they have to cover or they have to travel and more path or more amount of path within this specimen itself. So that means they will be having more chances to get absorbed by the specimen here itself.

Compared to that if we consider low theta value low theta diffraction or low 2-theta scattering almost close to the back scattering here other way around. If we consider something like high 2-theta so this is a back scattering here if we consider something like a high 2 theta value here. For high 2-theta value this incident X-ray and the diffracted X-ray both will be spending a much little path within this specimen here compared to something like a forward scattering there in a low 2-theta value low 2-theta scattering.

This has to spend or this has to undergo larger path here the path is ABC for low 2 theta scattering and here the path is DEF this much is DE + EF this is the path. So this path is much lower than this AB + BC path of course so that means here it self we can understand that the amount of absorption the chances of this incident X-ray and diffracted X-ray to get absorbed within the specimen.

This has a relationship within the angle of scattering forward scattering which is lower 2-theta value this absorption will be higher and for backward scattering close to very higher 2-theta value these chances of absorption will be much lower. And this will of course depend on the linear absorption coefficient of the material. So if we imagine something like a highly absorption specimen something like a Tungsten.

So there this entire diffraction phenomena or entire absorption the entire diffraction is basically will happen for this very small very small region very lost to the surface. All other beams which are entering the specimen they will be all absorbed there will be nothing left for diffraction itself. So entire diffraction phenomena will happen from the region very close to the surface. There also so what we can understand that the forward scattering.

So forward scattering means it has to come all the way here and then get scattered. So the amount of forward scattered beam the intensity of the forward scattered beam will be much lower compared to the intensity of the back scattered beam which is means at high 2-theta values. So forward scattering is basically limited here very close to the surface these 2 extreme cases somewhere within this the forward scattering condition is not at all valid.

So overall what we understand the same thing is written the back scattered high 2 theta scattering and high 2 theta angle speaks the scatting the absorption factor is much lower compared to low 2 theta values. So this difference in high and low 2 theta reflection of course decreases as the linear absorption coefficient decreases. So if we imagine something lighter element which does not have linear absorption coefficient true absorption is lower and so on and so forth.

So with a decrease in linear absorption coefficient this overall absorption factor will decrease and then the difference in absorption factor will decrease and then the difference in absorption between a high 2-theta peak and low 2-theta peak will also be decreasing. But it will be still there so if depends not only on the 2 theta angle it also depends on the linear absorption coefficient.

For linear absorption just like temperature factor not only it depends on the temperature itself it also depends on the 2-theta here not only the 2-theta value it will also depend on this linear absorption coefficient. If the linear absorption coefficient is lower lesser absorption overall, then the difference between low 2-theta and high theta values those peaks will also be lower.

If the linear absorption coefficient is very high something like a tungsten then there will be a considerable different between the intensity of low 2-theta and high 2-theta peaks due to the absorption factor.

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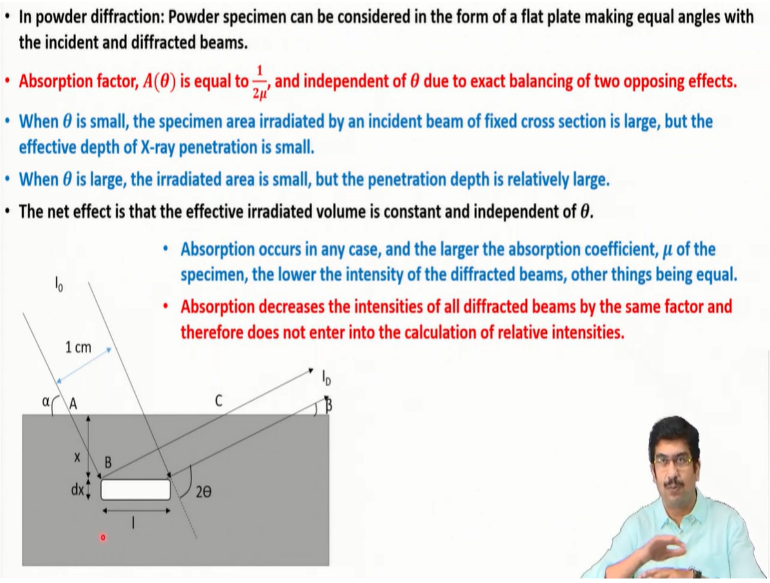

- In powder diffraction: Powder specimen can be considered in the form of a flat plate making equal angles with the incident and diffracted beams.
- Absorption factor, $A(\theta)$ is equal to $\frac{1}{2\mu}$, and independent of θ due to exact balancing of two opposing effects.
- When θ is small, the specimen area irradiated by an incident beam of fixed cross section is large, but the effective depth of X-ray penetration is small.
- When θ is large, the irradiated area is small, but the penetration depth is relatively large.
- The net effect is that the effective irradiated volume is constant and independent of θ.
- Absorption occurs in any case, and the larger the absorption coefficient, μ of the specimen, the lower the intensity of the diffracted beams, other things being equal.
- Absorption decreases the intensities of all diffracted beams by the same factor and therefore does not enter into the calculation of relative intensities.

Now this is Debye Scherrer method but more than division method we should discuss about powder diffraction method that is what we normally use. So in powder diffraction method we normally consider this powders like a flat plate. So it is kept the powders are kept like a flat plate or normally it is kept in a group or a plate in a group we put the powder. So we can imagine this as a flat plate which is making an equal angle with a incident and the diffracted beam and that angle is basically the theta angle.

So this here what happens is that the absorption factor A(Θ) this is equal to 1/2μ that means this absorption factor is independent if theta value in case of a powder diffraction specimen. In case of Debye Scherrer method it is obviously decreases or obviously depends on the absorption factor depends on both the linear absorption coefficient as well as the 2-theta. Here for powder diffraction method this fortunately does not depend on the theta value.

So it only depends on the linear absorption coefficient so the absorption the amount of absorption is exactly the same for low 2-theta for back reflection condition. Forward reflection condition low 2-theta peak and high 2-theta peak which is back reflection condition. It is only depends on the linear absorption coefficient of the material that is used there. Now why is it so what happens is for a flat plate like this if we have if we imagine first let us imagine a low 2-theta condition.

So this beam is falling somewhere over here what it will does is this beam will the amount of are this beam is saying the cross section. Basically the beam for a fixed size of the beam if the beam size of the fixed then this incident beam the fixed the depth of this X-ray penetration this will be small. That means if the angle of incident is shallow then the depth of penetration will also be small for a very fixed cross section of the beam.

Then exactly reverse will if theta is large if the angle of incident is large here then the depth of penetration will also be larger. Effectively what will happen is that the effective irradiative volume this cross section so in a shallow angle the cross section will increase over which the cross section will increase but the depth of penetration will be small if the beam is falling at a higher angle.

Then this cross section this irradiated area this are will be small but the penetration depth will be large. Altogether the irradiated volume will remain constant for all d to the value it will be independent of the 2-theta. In case of device error this is what changing the irradiated volume was changing.

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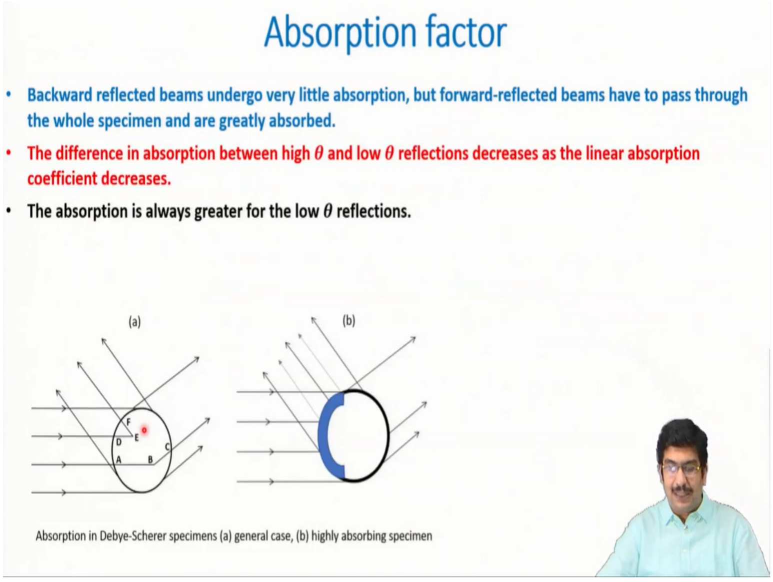

Absorption factor

- Backward reflected beams undergo very little absorption, but forward-reflected beams have to pass through the whole specimen and are greatly absorbed.
- The difference in absorption between high θ and low θ reflections decreases as the linear absorption coefficient decreases.
- The absorption is always greater for the low θ reflections.

Absorption in Debye-Scherer specimens (a) general case, (b) highly absorbing specimen

If you see this here the irradiated volume was changing for forward reflection versus backward reflection. Here for powder diffraction this irradiated volume remains constant for any kind of theta values. So this ultimately the absorption factors becomes independent of theta and only depends on the linear absorption coefficient of the material that is it all other conditions being remains the same.

So that means finally the absorption factor this basically decreases all the diffracted beam intensity of all the diffracted beams will decrease by the same factor and it does not finally enter into the calculations of relative intensity. So incase of powder specimen we can safely remove the absorption factor when we remove the final diffracted intensity at all.

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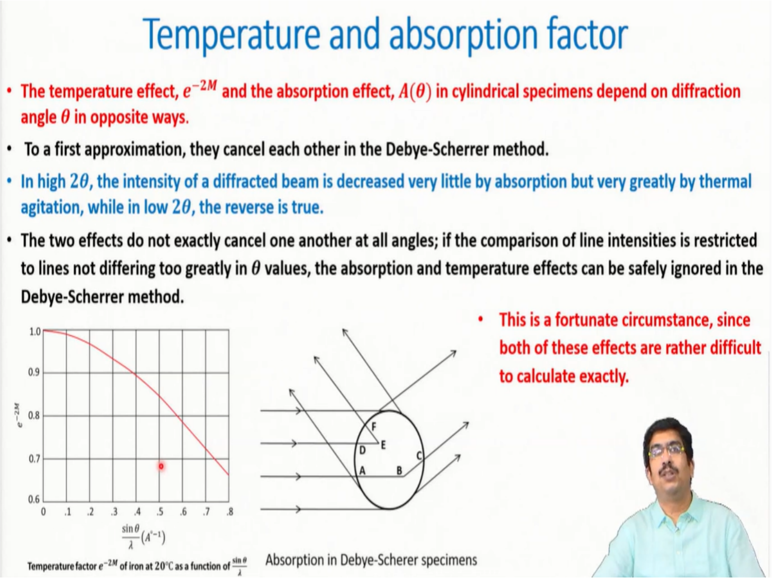

Temperature and absorption factor

- The temperature effect, e^{-2M} and the absorption effect, $A(\theta)$ in cylindrical specimens depend on diffraction angle θ in opposite ways.
- To a first approximation, they cancel each other in the Debye-Scherrer method.
- In high 2θ, the intensity of a diffracted beam is decreased very little by absorption but very greatly by thermal agitation, while in low 2θ, the reverse is true.
- The two effects do not exactly cancel one another at all angles; if the comparison of line intensities is restricted to lines not differing too greatly in θ values, the absorption and temperature effects can be safely ignored in the Debye-Scherrer method.
- This is a fortunate circumstance, since both of these effects are rather difficult to calculate exactly.

Temperature factor e^{-2M} of iron at 20°C as a function of $\frac{\sin\theta}{\lambda}$

Absorption in Debye-Scherer specimens

Now let us see the relationship between the temperature and absorption factor. So temperature factor e^{2m} and absorption factor which is A(Θ) incase of a cylindrical specimen that means in case of Debye Scherrer method this will depend on the diffraction angle in opposite way F. If you remember so this is a temperature factor decreases with your diffraction angle.

So the temperature in factor decreases with diffraction angle and diffracted intensity decreases as the 2 theta value is increasing for high 2-theta peaks temperature factor will decrease the intensity. On the other hand side the absorption factor increases the diffracted beam intensity or it is more effective on the lower 2-theta beams rather than the higher 2-theta diffracted beams. So absorption factor will cause more intensity for low 2-theta diffraction beam compared to the high 2 theta diffraction beam.

So in that what it does mean is that the temperature factor and absorption factor goes in 2 opposite directions they have opposite relationship with the 3 theta beam. In high 2-theta the intensity of the diffracted beam will decrease very little by absorption but much higher by thermal agitation. While in lower 2-theta the diffracted beam intensity will be mostly decreased by absorption effect but not so much by the temperature effect.

So these 2 effects we can safely say for cylindrical specimen as such these 2 effects basically cancels each other may not be exactly cancelling each other but they are sort of cancelled each other. Only for powder specimen anywhere the diffraction factor is the absorption factor is independent of theta. So their entire decrease in diffracted intensity with 2-theta is because of the temperature factor.

So for powder specimen temperature factor will be there absorption factor will not be there and for a this spherical the cylindrical specimen Debye Scherrer method temperature factor and absorption factor will be cancelling each other basically. So finally in the calculation of diffracted intensity both of these factors will not come because and this is a very fortunate situation.

Because the exact calculation of whether the either the temperature factor or absorption factor is quite difficult for any kind of a material. So for Debye Scherrer method we do not need to consider temperature factor or absorption factor separately they can be cancelling each other they neither of them will enter in the final diffracted beam intensity. And for powder specimen anyway we have to consider the temperature factor because absorption factor is 0 or absorption factor is not there for this powder specimen.

So we will be stopping here and in the next lecture we will discussing about the diffraction intensity or this x-X-ray diffraction profile of a real specimen and how to analyze so bye.