Techniques of Materials Characterization
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Lecture - 41
Imaging in SEM (Continued) and X-ray Diffraction

Welcome everyone to the NPTEL online certification course on techniques of materials characterization. We are now in module 9, week 9. We have four more weeks to go and this week and onwards we will be discussing about X- ray diffraction. We have previously discussed finished various other imaging mostly microscopy techniques. So, we started with some general concepts of microscopy and then we discussed about optical microscopy.

Then we shifted to electron microscopy and there we discussed about first transmission electron microscopy, electron diffraction and then we discussed about scanning electron microscopy. And the last week, we were mostly dedicated to first analytical detectors, EDS and WDS detector. And then we discussed about imaging in SEM like imaging in various modes, topographic imaging, and topographic under topographic imaging.

We discussed about secondary electron imaging and then backscattered electron imaging in topographic mode. And then we discussed about the compositional mode of backscattered electron imaging. And this so only a very two or one or two topics was left from SEM from the discussion imaging and SEM. So, we will finish that and then we will start our discussion on X-ray diffraction from this week.

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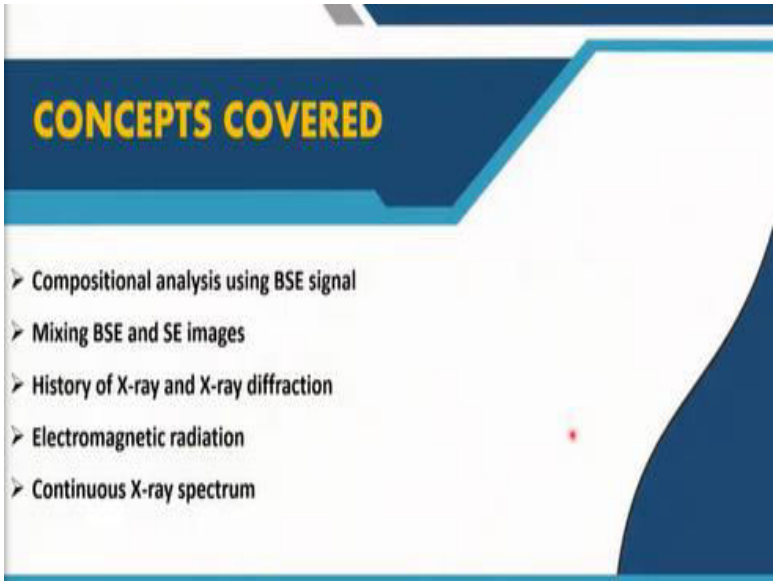

So, the concepts we will be covering here, first the composition analysis using backscattered electron signal and mixing the backscattered electron and secondary electron images. These are the two topics which were left from last week. And we will just finish it quickly and then we will discuss about X-ray and the first topic; like always, we will start with the history of X-ray and X-ray diffraction.

And then we will have a general discussion about electromagnetic radiation. And finally, we will end up with a question about the continuous X-ray spectrum how the X-rays are generated.

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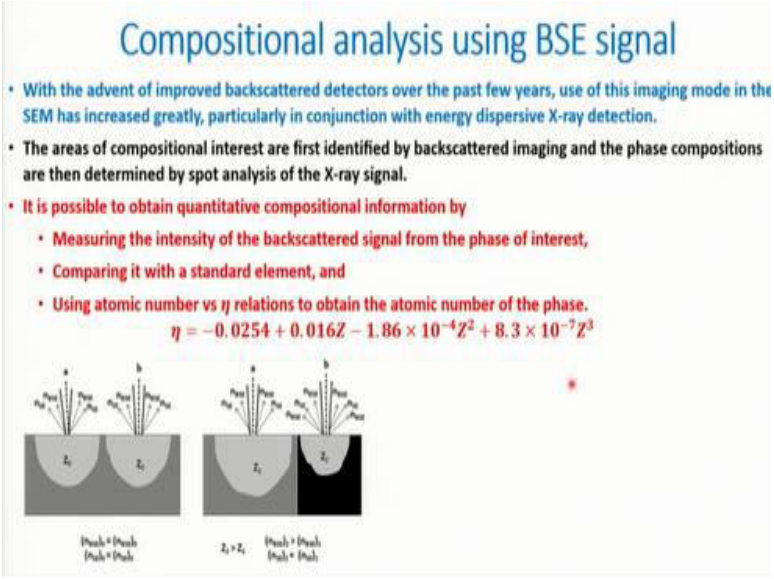

So, the first thing, continuing from the last week; the compositional analysis using BSE signal. So, we have seen that how the BSE signal is used for topographic imaging just because the yield of BSE or backscattered electron, yield. Similar to the secondary electron yield backscattered electron yield also depends on the specimen tilting with respect to the electron beam. And the difference between the; secondary electron topographic mode and backscattered electron topographic modes is that,

The backscattered electrons are far more having a directionality, the way they are generated that has a directional aspect. And that is why backscattered electron imaging is far less sensitive to compared to the secondary electron imaging. But backscattered electrons have an additional advantage that they depends their yield depends on the chemical nature of the elements or chemical nature of the phases that they are generated from.

And that same information can be utilized for a compositional analysis that means a quantitative analysis of the composition of any specimen. Just like in that way, the backscattered electrons are somewhere in between the normal secondary electrons which produced in a topographic imaging or compositional imaging mode. So, they can be used for imaging mode as well as they can be used just like a characteristic X-ray signal or Auger electron signal.

They can be used for a quantitative measurement of the composition of the specimen that you have what are the elements are present there and in which quantity they are present. Both can be identified using the backscattered electron. So, how to do that? This is a fairly recent development with a very improved backscattered electron detector. It is not that ET detector can do that, it is only with the use of this annular backscattered, solid state backscattered detector or even better the in-lense backscattered detector.

So, using all of this advanced backscattered detector, now in addition to the imaging mode we can also calculate the quantitative from the backscattered electron signals. And most often the way we do it is we use it in in combination with the energy dispersive X-ray detection. So, EDS signal and then in addition to the EDS signal we also use the backscattered electron. So, first what we do is that first we do a chemical analysis.

So, we first do a backscattered imaging on the area that we wanted to check the composition we want to check. First take an image, backscattered electron image and then what we do we know that these are the areas, which are having this. So, we must have let us say we have a light element like aluminium. We have an aluminium rich some area and we have say a heavier element like let us say iron. So, we have an aluminium and iron next to each other in some sense like here.

So, when the electron beam is falling with something like aluminium region of course, the number of electrons produced backscattered electron produced will be far less than when it is falling over iron rich region. So, from there we can identify if we know that composition if we know that I have aluminium and iron in the composition. I know this is the possibly the aluminium rich region and this will possibly the iron rich region.

Just simply from the contrast it develops, we know the contrast also depends on the signal, number of BSE electrons that is generated. From there we know this is aluminium region and this is iron rich region, then what we can do is that we can bring an EDS signal. We can capture the EDS signal from this corresponding region and from there we can find out the quantitative analysis. That is one way of doing it.

That is what in earlier days people used to do. Imaging using BSE signal first a larger area find out the compositional contrast from different phases and then bring the characteristic X-ray and find out the quantitative analysis and do the quantitative analysis from this region. But now, with the more advanced BSE signals, BSE detectors where we can straight away get the number of BSE signals and we can calibrate that.

We can find out we can do the entire analysis quantitative analysis using BSE signal. So, how to do that? First, we measure the intensity of the backscattered signal from certain phase of interest. So, we put the electron beam here and we measure how many number of backscattered electrons are coming out from this lighter phase. And then we again do the similar thing we put the primary electron beam here.

And we find out how many number of electron beams or electron backscattered electrons are coming out from here. And we know this initial the atomic number possibly we know the primary electron beam current, accelerating voltage and all of this we know. So, then what we do is that we compare that signal the number of backscattered electrons we compare it with a standard element. Standard element means let us say we have this aluminium here.

So, we take 100% aluminium. We have that library function, we recall that the number of electrons are number of backscattered electrons that 100% of aluminium. If a sample has 100% the aluminium and pretty much the same condition, acceleration voltage being current and all how many number of electrons are to be generated, backscattered electrons are to be generated from the standard elements.

So, we know that or we have that information a priory and then we compare this with this signal that we get from this two phase specimen. From there, we can find out this number or from this calibrated result or from this calibration, we can calculate that, what is the fraction of aluminium present within this entire region. So, we can get the compositional analysis, we can get the concentration of aluminium as well.

For that definitely we; can use this number of backscattered electron, we can also cross check this by using this relationship. We know if it is aluminum, we know that what should be the number of backscattered electron yield from here. So, again we can do this kind of quantitative analysis or we know the backscattered amount or backscattered electron yield standard and from there we can find out, back calculate.

And find out that what is the backscattered or what is that elemental concentration or what is the percentage of that particular element present within this specimen. So, this is how we can do the compositional analysis using BSE signals.

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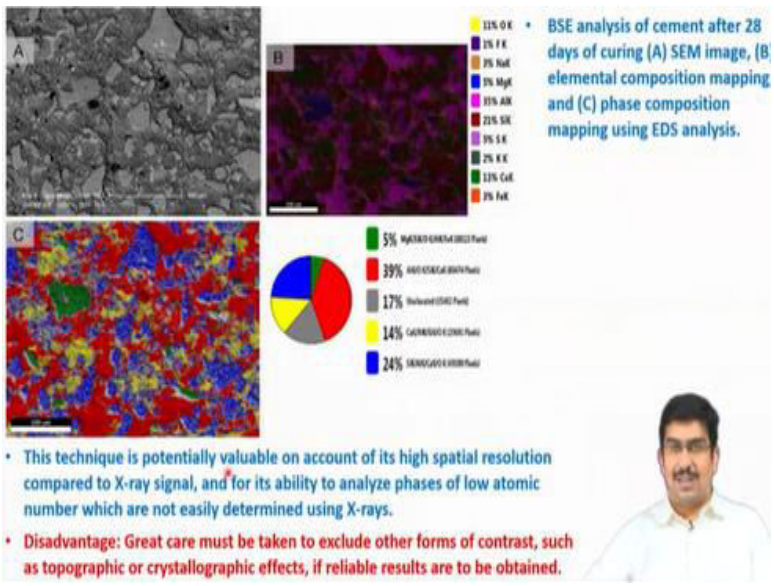

Now, let us take an example. So, this is a cement sample and this one is the SEM image in BSE mode that is backscattered mode compositional, pure compositional contrast mode we are getting and from this we calculate the elemental. So, this is the elemental analysis that we did from this BSE signal. So, this is the BSE image and here pixel by pixel, we do the composition analysis by comparing it with a standard sample.

And we find out the compositional map using the BSE signal and this colour coding tells us that exactly what is the quantitative analysis. So, this phase let us say a certain phase we have and what is the percentage of that phase within this microstructural area and where exactly more importantly with spatial resolution where exactly that phase out. If I go back to here and try to find out that what is the composition here I can do that.

I try to find out some other phase and try to find out what is the composition of this, I can do that. So, I know the distribution of the phases plus I know their composition. Both of this information I can get it just from the BSE signal using the BSE signal. And this is the corresponding regions were sorry this is the BSE signal I was a little wrong. So, this is the BSE signal and this is the corresponding phase composition mapping using EDS signal.

So, here I know the different, different phases same thing same kind of information I can get it from using the BSE signal here. Now, what is the advantage and disadvantage of this using the

BSE signal in comparison to something like EDS signal. And taking EDS signal here I am not going to some higher compositional higher techniques or more sophisticated techniques of compositional analysis in SEM like cathode luminescence like Auger electron, I am not going to that.

I am going to the most elemental crude way of techniques of elemental concentration finding out elemental concentration in an SEM that is EDS. So, BSE signal of course, the first advantage that it gets is its very high spatial resolution compared to the EDS signal this we have discussed before. And that is because the sampling volume of BSE signal is much smaller than the sampling volume of this X-ray signal.

That is means, X-ray signal has a lower spatial resolution than compared to the BSE signal. So, that is the first advantage we can get this elemental composition determination with a higher spatial resolution if we use this BSE signal. Now, the disadvantage of BSE signal is that this contrast that comes from the number of BSE signal that is coming. If I have a multi phase material, the BSE signal yield not only depends on composition.

But it also can depend on the topography. So, these two effects how to deconvolute this effect that is a bigger question very big question. For example, if I take these two elements next to each other the number of BSE signals that is coming, what is the guarantee that this is purely due to the atomic number. This difference can also come on number of BSE signal, the difference contrast can also come because of the surface roughness.

Maybe surface roughness or maybe surface tilting is something that this area which is of higher which should produce high BSE number of BSE elements. But due to some contrast based or topographic problems, this is not producing that many BSE signal compared to that this region is possibly producing more of a topographic BSE it is producing more number of BSE signal. So, then what will happen that yield?

BSE yield will be not a true representation of their chemical identity. So, in order to do the BSE or this kind of compositional mapping quantitative composition mapping, we have to somehow

exclude any other form of contrast generation like topographic contrast or crystallographic effect. All of these things has to be completely neutralized and then we have to ensure that whatever the BSE signal is generated is purely because of the composition.

Because of the atomic number difference between two regions, if we do that, then this method will be very reliable.

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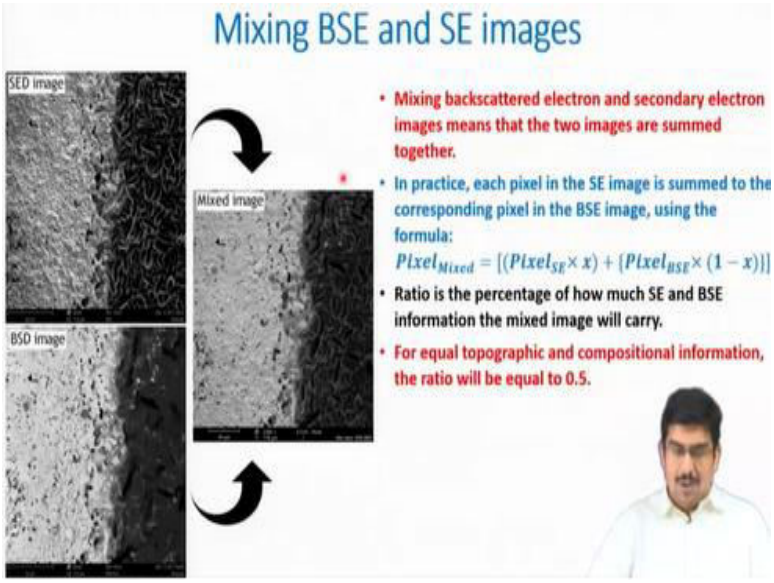

And the last one to discuss here is to mixing the BSE and SE image. So, this is possible because we are capturing nowadays everything as a digital image. So, we know pixel by pixel we have the information and we have already discussed this. So, what the pixel has it is x and y two coordinates plus it has this greyscale value depending on the number of signals that we collect. So, we have already seen that in case of a BSE image we get topographic contrast and compositional contrast.

And by a suitable combination addition or subtraction from different we have a multi component detector. That means, we have this A, B, C, D four different elements of a detector, annular detector by suitable addition and subtraction we can reduce the topographic contrast, enhance the composition contract or otherwise and vice versa, we can do that. One more step ahead, not in the detector level itself, but we can do this as a post processing.

We can mix these images, images captured in SE mode and images captured in BSE mode. So, SE mode as we said it gives you better topographic contrast with them higher or better spatial resolution. So, that is the advantage of SE image, better topographic contrast with a better spatial resolution than a BSE topographic image. But the BSE images are superior in the sense that they can give compositional contrast which the SE signal cannot give or does not give that well.

SE signal does give it but it does not give it in that good as the BSE signal. So, what if I can take advantage of both of; these two better things. From SE signal I get the topographic information and from BSE signal or BSE image I get the compositional input signal. And then I mix these two information pixel by pixel and construct a mixed image, artificial mixed image I construct which will represent both the topographic contrast of SE signal and the compositional contrast of the BSE signal.

And that will then be an ideal imaging mode which I cannot do it literally in the machine I cannot do it. I do not have any other means to do this in the machine itself, I can generate this artificially as a post processing method. So, this something very regularly people are trying with post processing software and this you do it basically in every pixel in the SE image is summed with the corresponding pixel in the BSE image.

And the formula that normally people use it is kind of again rule of mixture kind of formula, the pixel in the mixed pixel or the greyscale value of that mixed pixel is basically the pixel of secondary electron the greyscale plus the pixel of the BSE electron and they are multiplied with a suitable ratio or suitable percentage. And this ratio is basically artificially you define how better mixing you want or what proportion you want to mix the SE signal with BSE signal.

Usually this is unusual given input that people do very good this kind of image. So, this image is generated with something like a ratio of 0.5. That means you give equal importance to this SE signal and the BSE signal and then you generate this mixed image. So, this is this x is 0.5 here that is what you can possibly try to begin with and then slowly you can change and as per your need. So, you can see that these two images now if we just concentrate on this.

This is from a solar cell and this has two different material altogether. So, this is one and this is another so this topographic difference is very clear here. In the SE image and this is a BSE image and the BSE image shows very good compositional contrast, but topography is again very much missing. So, if we mix these two now, these mixed images showing very nice typography with very good compositional contrast.

And the most important one here, you can see that if you look at only the SE image with the typography, you basically mix these few nodules here which are not visible in topographic mode but which are very nicely visible in the compositional mode. But in the compositional mode you are not able to figure out exactly what shape they are whether they are spherical, whether they are irregular shaped and all.

And if you mix both of these now you know that they are possibly a little bit irregular, some of them are spherical, some of them irrregular and they are of certain elements of this one. So, this whatever element is this space, this is extending all the way up to the next one. So, this kind of information we can generate by mixing this SE and BSE signals here.

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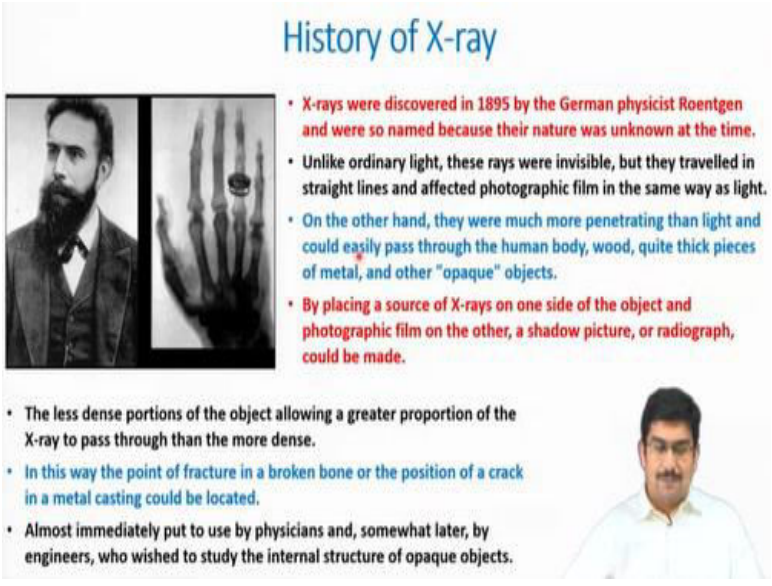

So, with that we close our discussion about SEM and now we are moving towards the X-ray part. So, one thing about the first thing about X-ray if we; compared to X-ray and electron microscopy. Because we have studied electron microscopy and we kept on telling that electron

microscopy are a very popular one because of its diversity. The different kind of signals it can give the different modes you can use it; you can get imaging.

You can get diffraction, you can get compositional analysis, you can use imaging in different various if you take SEM, you can have secondary, backscattered, if you go for transmission mode you can use it for like dark field, bright field, have more lots of different modes of imaging itself. So, this is a very, very diverse one electron microscope and that is why it is very much used by almost all fields of science and technology.

And everything is happening in one single equipment itself, it is possible to get. X-rays are equally capable for that matter if I have to put it in this way. And in some sense in some fields, they are even better than electron microscope they could be even better than electron microscope. Like for example diffraction, at least in diffraction we come to know that in a few days or within a few lectures that how good the X-rays can be in terms of determination of crystal structure at least in terms of determination of lattice parameters and so on.

But X-rays are equally capable of doing certain things which electron microscopes can do. They also can be used for diffraction experiment, they also can be used for imaging purpose, they also can be used for a chemical analysis purpose and so on and so forth. The point is, till today, there is not one single instrument available to do all of this. All the people are claiming certain people are claiming certain companies are saying that at least they are able to mix the imaging one and the diffraction one so on.

But it is not very much reliable. Till now, different types of X-ray machines are used for different purpose. X-ray diffraction machines are different, the X-ray imaging one is different, X-ray imaging is basically what we know is a CT computerized tomography, x-ray micro CT not one that is which is very widely used for medical purpose and sometimes this is again used for material science as well.

So, scientific purpose research purposes this imaging mode is also becoming pretty popular. The micro CT mode or the CT mode that is computerized tomography mode that is nothing but a

kind of imaging only. And then you have something called radiography that also is quite popular. This is all you can virtually say this is an imaging mode of X-ray signal. And then you have XPS, which is a spectroscopy mode.

And that spectroscopy mode is capable of giving you elemental composition and many more information related to bonding and so on. But still now, point is none of these techniques with three or four different techniques, X-ray based techniques till today they could not be combined in one single equipment. That is why electron microscopy in some sense is superior in that sense that it can give you chemical identity whatever scales it is.

Of course, the scale is not as good as x-ray, but it has added advantage that it can give that signal and comes up with a spatial resolution. Like you can get in a microstructure you can pinpoint this region and get that chemical analysis done from there. X-ray signal it is little difficult to do still now. Micro XRD and all those things and they are increasingly becoming important they are popular, but it is still difficult.

So, the bottom line is X- rays in some sense are equally capable or sometimes even better than electron microscope. But still now, they are lacking in the sense that they do not come in; one single equipment. And as often a time they cannot give you information with better spatial resolution like electron microscopy can do. But with that, we will discuss or we will start our discussion about X-ray diffraction. And we will restrict ourselves mostly to X-ray diffraction.

Although we will; very briefly discuss about the X-ray imaging part that is X-ray tomography, computerized tomography part. But we will very briefly discuss but mostly we will restrict ourselves to X-ray diffraction and we will certainly not go into experience that is the chemical analysis part of X-ray. So, X-rays were discovered in 1895 by a German physicist Rontgen and he named X-ray because the nature of this new radiation was not known at the time.

He could not understand what exactly he has discovered and the story basically almost all of us have possibly heard in popular science books or in our school physics level physics book, we have heard about the story. This is the original Rontgen himself his image and what he really

understood or what he realized is that this is very similar to light signals visible light. But different in that way is that this is invisible with a naked eye, we cannot see these rays so unlike the light.

But they are similar to lights in the sense that they travel in straight lines, just like light and they could also affect the photographic films like light rays. So, in that sense he found the similarity, but the difference is these are invisible to our human eye. And another difference he very quickly realized that unlike light, these new rays or these X-rays have a much better penetrating power that means they have a higher energy than the visible light.

They could penetrate through human body, they could penetrate to wood, quite thick piece of metal, other opaque objects which are visible light just get reflected from them. The visible light cannot penetrate through this opaque object. So, opaque named, this name opaque comes in terms of light penetration. Now that opaque objects become transparent, the moment we change to from visible light to the X-ray signal.

So, what he understood also is that if you place a source of X-ray one side of the object and you place a photographic film on the other side object, you find a shadow picture just like an image that you could capture using a light signal. If you place a transparent light transparent image and you good get capture an image of that light, transparent object or could capture an image in the reflection mode. Similarly, you can capture an image using the X-ray source as well.

And this picture is called radiograph. So, this is first ever radiograph recorded and this one is the hands of Rontgen's wife that was the first radiograph that was captured with their engagement with their marriage ring in this hand. So, immediately, Rontgen understood that you can basically see this our flesh and all the veins are transparent to the x-rays but not our bones, bones are not transparent. So, immediately this was picked up by the medical practitioners.

This becomes a very useful tool for them and what happens here the less dense portion of the object that allows a greater proportion of X-ray. So, there is a difference in something called X-ray absorption. So, their density or the material effects the absorption of X-rays that is why this

is happening the flesh and the veins and all other parts they are lesser dense. And that is why they cannot absorb X-ray and they become transparent to the X-ray signal.

X-ray signal can penetrate through those parts, but the bones are much more denser and that is why the bones could not be penetrated by X-ray we could capture this signal on the other side of the bone. So, this becomes a very important immediately the medical practitioners. They were jumping onto this and they started using this radiographic technique to capture the exact point of fracture on a broken bone or the position of a crack in a metal casting and so on.

So, this is again; this all of these things was started and still today the same thing. So, X-ray plates or X-ray scans are still used the radiographs are still used by medical practitioners, radiographs are still used by people working in industrial practices. They have people who are casting bigger components and this is a very popular non destructive testing of materials. Bigger components in the component level to find out cracks, internal cracks and defects in them and in the component. Little later people also started using it to study the internal structure of opaque objects.

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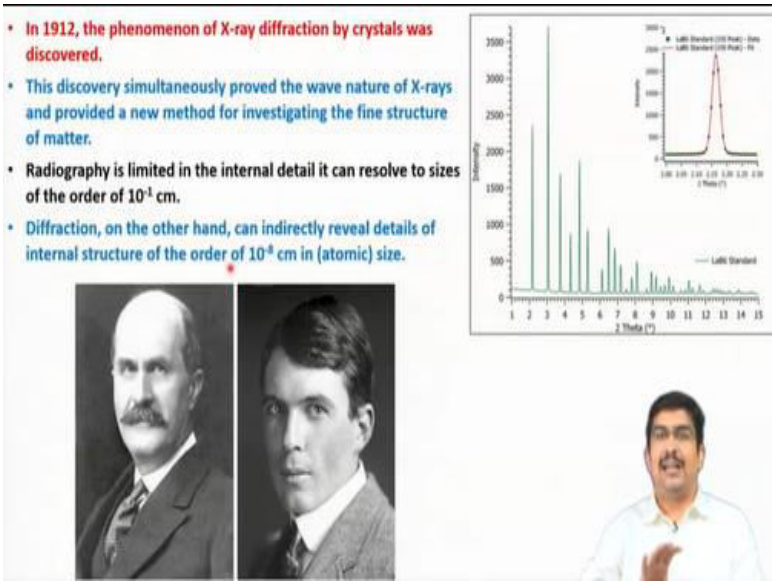

So, how it happened is that basically in 1912, two great physicists again, the only physicist I think, I believe these are the only instance where a father and the son got Nobel Prize for the same invention and for the same year. Father and son have got others also in other instances, but

not for the same year and not for the same invention most importantly. So, they have got it for the same invention senior and junior Bragg.

And they discovered that what we have already discussed many times is the Bragg's equation. So, this Bragg's equation the phenomenon of X-ray diffraction by crystal in 1912 they have discovered and they also through this discovery it was also proved the wave nature of X-rays. And then this provided way to investigate the final structure or internal structure of matters. Because radiography although it also reveals the internal structure of materials.

But now the internal details it can reveal resolve that is limited to the order of 10 raised to the power(- 1) in the millimetre range.

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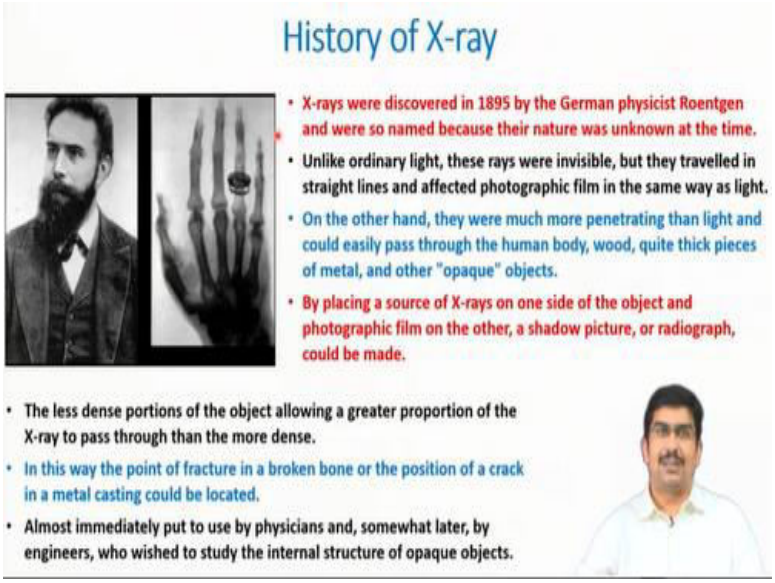

Pretty much in the millimetre range the radiograph that you are seeing here, the resolution the spatial resolution of this radiograph is in a millimetre range. Until today that is the limit that people go. But diffraction is one phenomena which is actually the resolution is in the atomic level because the atoms are responsible for that phenomena. The diffraction phenomena happens on that atomic level, individual atoms are responsible for diffraction or individual atomic planes.

That arrangement of atoms they are responsible for causing the diffraction phenomena. The resolution are spatial resolution if you if you think it is that term, the spatial resolution of x-ray

diffraction or the diffraction phenomena is in the atomic level by virtue of the way depression happens it is the spatial resolution is almost in the atomic level. So, the internal structure the way the atoms are arranged in an atomic plane inside the material.

For the first time people were able to understand the internal structure. And from there itself, the entire study of crystallography got a complete different shape. Before that, people were doing the crystallographic study of crystallography and all from measuring the external shape their angles and developing single crystals and all those things. And it was pretty much a theoretical subject and less of an experimental subject.

But after this X-ray diffraction phenomenon was observed was obtained or was discovered. Now this crystallography got a tool with which it becomes completely an experimental subject. And this is the kind of just we will discuss about this in great detail. This is the kind of graph that you very often see in an x-ray diffraction scan, the 2 theta versus intensity here the 2 theta is that, n lambda = 2d sin theta, that theta angle here and this corresponding to the intensity.

So, you basically measure the intensity of the incident X-ray beam which is coming with fixed lambda and you measure you vary the sin theta and measure the intensity. And from this you determine basically the internal structure you determine how the atoms are arranged, what is the symmetry and so on.

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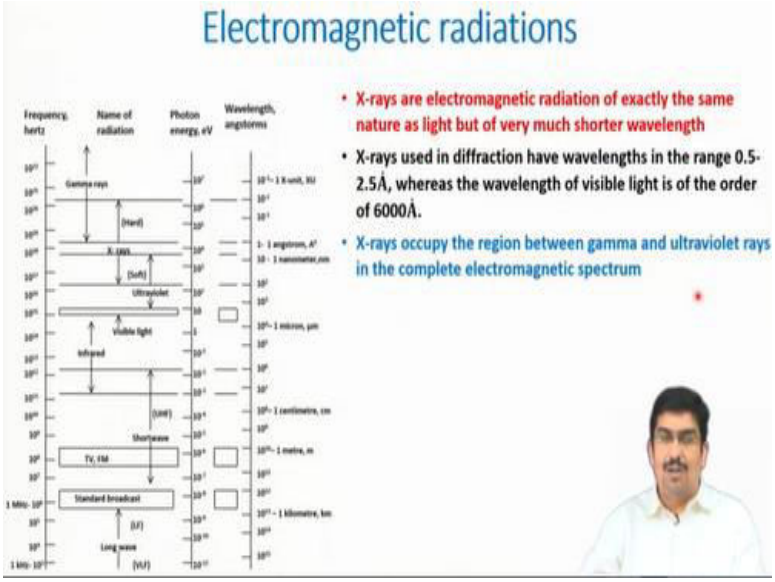

So, this is how you do it. We will stop it here and we will continue with this electromagnetic radiation and continuous spectrum part in next part. Thank you.