**Texture in Materials**
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**Module - 05**
**Principles of texture measurements by X-ray diffraction**
**Lecture - 22**
**Macrotexture and Microtexture Measurement**

Good afternoon everyone and today, we will start with module 5 that is Principles of texture measurements by X-ray diffraction. This lecture is lecture number 22 where we will try to understand Macrotexture and Micro-texture measurements.

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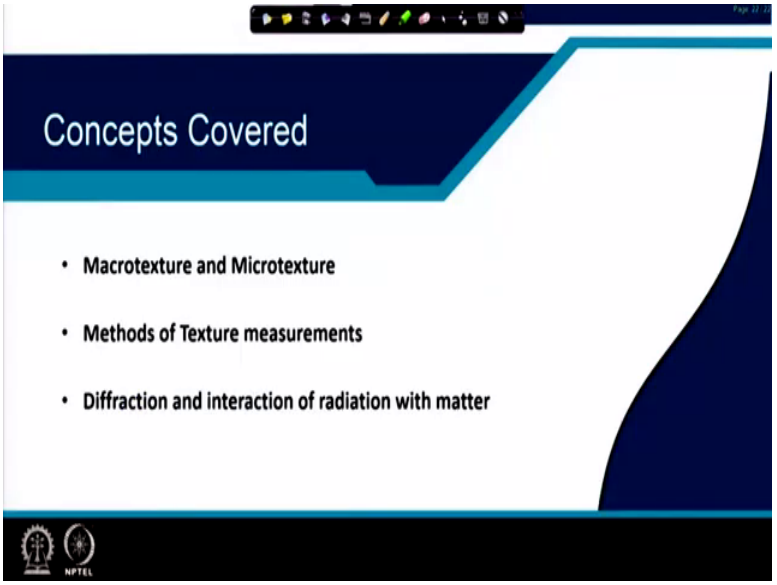


So, the concepts that will be covered in this lecture is micro-texture and macrotexture, method to of different methods of texture measurements, diffraction and interaction of radiation with the poly crystalline material which is matter.

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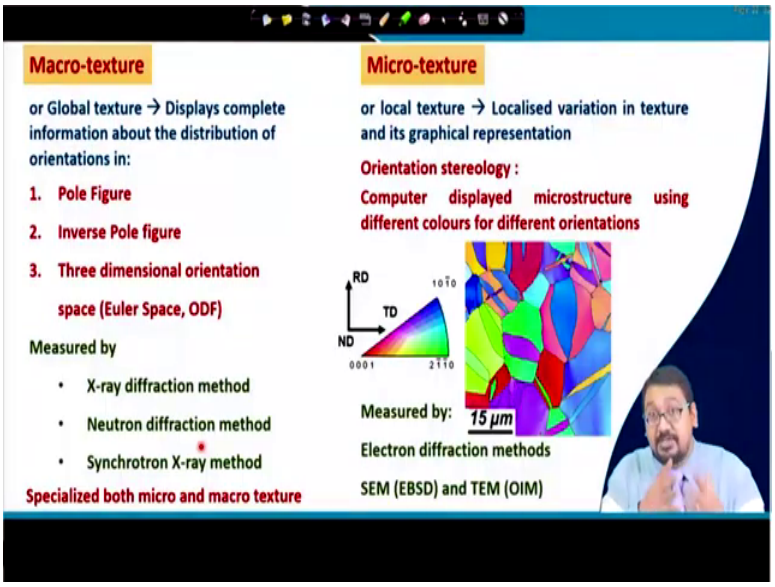


What is macrotexture? Macrotexture is usually called bulk texture or global texture and it displays a complete information about the distribution of orientation. It displays the complete distribution of a orientation in space for a particular poly crystalline material which has been processed by some method right say solidification or deformation or any other method.

The information that is obtained is usually represented in terms of pole figure or in terms of inverse pole figure and we have already seen that inverse pole figure is usually used for axial texture measurements. The three-dimensional orientation space that is Euler space consisting of Euler angles phi 1, phi, phi 2 is also utilized in form of Orientation Distribution Function that is ODF to show this texture or orientation information.

Micro-texture: micro-texture on the other hand gives information about the local texture. Now, what is the difference between the local texture and a bulk texture? Local texture is localized variation in the orientation of the material, in the texture of the material and this texture representation can be given in terms of a graphical representation ok like the one shown in this coloured micrograph below. As we have shown this in previous lectures that these colour micrograph is not a random coloured micrograph, but it is a computer-generated microstructure, which gives the information of the orientation stereology. Now, this different colours of these different grains present in this microstructure shows different orientation and this is given by this triangular colour code. This is an example of a hexagonal close packed

material that is alpha titanium, which is a single phase and kind of a pure commercially pure titanium.

So, you can see that this material has a rolling direction vertical or transverse direction which is horizontal and in the plane of paper, it has a rolling plane that is perpendicular to the plane of a paper, it is ND normal direction. You can see that various grains have been represented by different colours and different colours represents the orientation of that particular grain in this microstructure. If it is red that means, this grain has an orientation 0 0 0 1. When it is green that means, this grain, this grain has an orientation of 1 1 2 bar 0 whereas, the blue grains have orientation near to 1 0 1 bar 0. So, this microstructure is a quantitative microstructure which is obtained by EDSD technique, and that when we talk about macrotexture, macrotexture as we know that it is measured mainly by X-ray diffraction or neutron diffraction methods, we will come to know in details in the later lectures that why this methods are used for macrotexture.

In case of micro-texture as I said electron diffraction methods like electron backscatter diffraction in a scanning electron microscope, that is SEM EBSD or Orientation Imaging Microscopy. In a transmission electron microscope that is OIM in TEM is used to obtain this kind of microstructure and this kind of microstructure is known as inverse pole figure map and is mapped by a triangular inverse pole figure showing different colour shades representing the orientation of the material. Now, if we look into another specialized technique, this technique is synchrotron X-ray diffraction method and this is a specialized technique, where the X-ray radiation is obtained from a cyclotron measurement. For example, in a utilizing a beam line so that a very low wavelength, parallel thin X-ray beams could be obtained and such X-ray beams have a very high penetration depth as well as very high special resolution. So, they become very specialized to obtain both micro-texture and macrotexture information depending upon the equipment that we are using.

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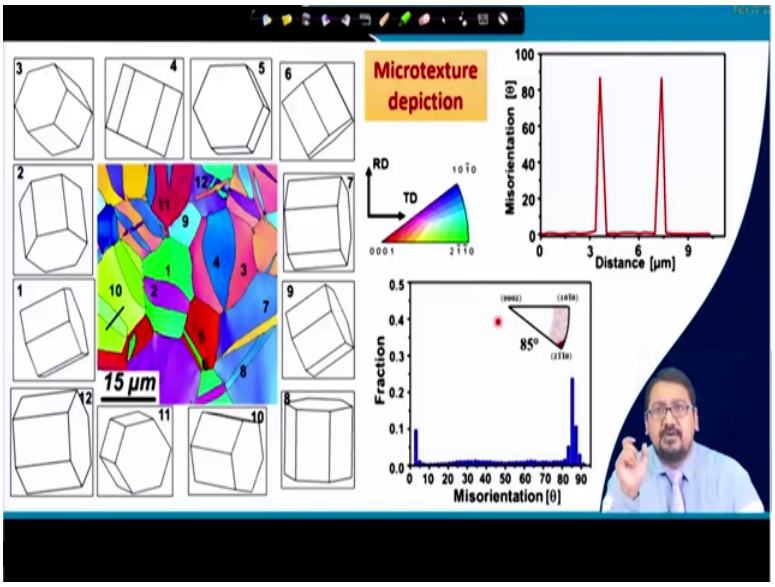


So, micro-texture depiction: Now the same inverse pole figure map that is the quantified orientation map microstructure obtained from a EBSD method using a field emission gun scanning electron microscope is shown here and this is the microstructure of pure titanium as I said. You can see that when we observe this microstructure, we can not only observe the orientation or the texture of this whole part of the sample, but we can observe the texture in even in individual grains. So, if you look at this green grain which is nomenclated as 1, what we can see is that the orientation of this grain is something like this with respect to the RD, ND and TD.

This orientation is represented in terms of unit cells and this unit cell, hexagonal unit cell can be obtained by using this EBSD method. Like that , we can observe the the this twin that has formed I know that it is a twin which has formed in the grain 1 and we have nomenclated it as 2 and you can see that the unit cell is sufficiently different from the grain wall.

Now, you can see like that, we can map grain 3, 4, 5, 6 and we can show that all of these have different orientations of the unit cell with respect to the sample reference directions, important sample reference direction which in this case is RD, TD and ND. Now, therefore, each of these grains are being shown with respect to a different colour code with respect to this inverse pole figure colour code map.

Now, not only the information of the orientation of each grain is present in this scan, but it also contains the information of each pixel presents in the material. It can give the

information, if we draw a line in this in this grain number 10 and we can see that there is lenticular type of structure, which I am saying that these are kind of twins.

And see in case of titanium, extension twins usually develop, also contraction twins forms, but this is the case of the extension twin and this extension twin usually has a axis angle pair and that axis is usually 1 1 2 bar 0 and the angle is nearly 85 degrees. So, if we draw a point-to-point misorientation map and that at these two points, the misorientation reaches a peak which is near to 85 degrees, and we can also find out the length means the thickness of the twins.

So, that not only we can get the orientation of the material or orientation of each grain or each positions of the pixel, but also we can calculate the difference in orientation, the mismatch of orientation between two adjacent grains or in between if there is a twin present and we can show that ok. The angle, the grain boundary angle, the misorientation angle of the grain boundary is 85 degree in this case and this twin is nearly say 4 to 7 micrometre thick that means, 5 micrometre nearly roughly thick right. Now, not only this, as I said that the twin boundary is a axis angle pair right. So, we can obtain the misorientation angle distribution fraction for the entire microstructure, and that in this case, the fraction of certain misorientation angle is given in the y-axis and the degree of the misorientation angle is given in the x-axis.

Usually, in case of quantitative microscopies like electron backscatter diffraction techniques, the misorientation angles greater than equal to 15 degrees are considered to be high angle grain boundaries and that is they are considered to be grain boundaries right. Those angles which are lower than 15 degrees are known as low angle grain boundaries or low-angle boundaries and they corresponds to mainly geometrically necessary boundaries present in the material. Now, that when we look into this misorientation angle distribution fraction, we observe that there is a peak at around 85 degrees and as I have said that twins that usually form in titanium are extension twins, and they have a boundary, grain boundary or the twin boundary having an 85-degree misorientation about the 1 1 2 bar 0.

So, in order to see that why this kind of peak is observed in this material if we look into this material here, this microstructure here, we can see that here is one twin, there is another twin, there are few twins here, intersecting twins are present, this is one twin, here are another twins intersecting twins. In the whole microstructure, there are lot of twins present and

therefore, the boundaries of these twins could be observed more largely so, their fractions will be more.

So, if we look into this 85 degree boundary in terms of angular inverse pole figure an angular inverse pole figure like this one and we will see a high intensity at a certain axis right. This high intensity at a certain axis for this case comes at 1 1 2 bar 0 so, there is a lot of 1 1 2 bar 0 axis having 85 degree in angular and axis and angle relationship. Therefore, it is a kind of a special boundary usually called as a coincidence site lattice boundary, but we will come to that in the later section of the course.

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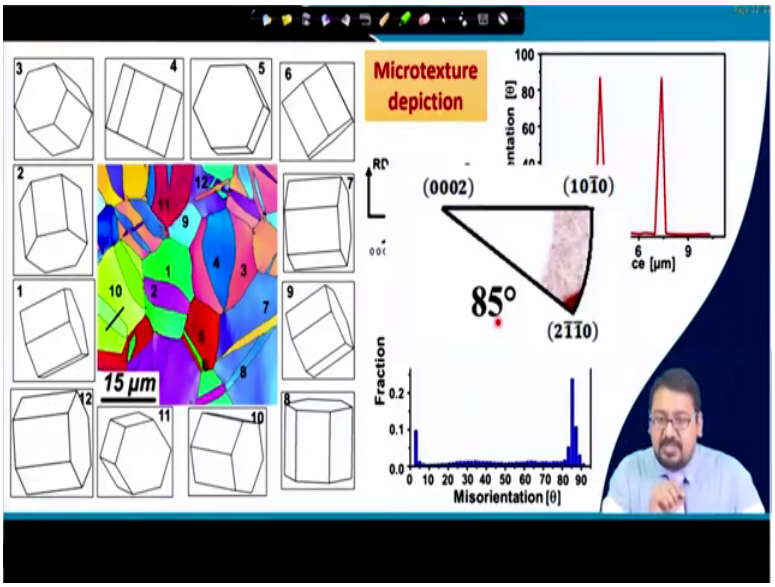


If we look into this, we I have enlarged it, we can see that even the axis angle pairs could be identified using the micro-texture process. On the other hand, macrotexture does not give such in depth information rather macrotexture gives the information from the bulk sample and is a complete property of the sample, a representative information from the sample whereas, micro-texture though give depth in formation of the sample, the information is obtained from a very small area of the sample.

Therefore, may not be the complete representation or complete property of the sample. However, both this method together we can utilize to obtain information and for obtain information of any product to change processes in industry or to carry out research activity to understand more about different kinds of deformation behaviour or any other processes. That

happens in poly crystalline metals and alloys and are important for engineering application purposes, technological development purposes.

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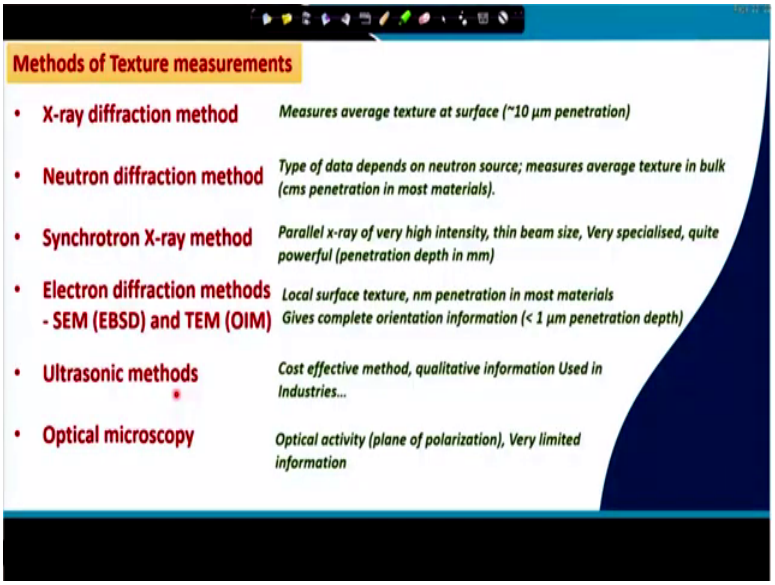


So, the methods of texture measurements. We found out that macrotexture measurements are done by X-ray diffraction methods and neutron diffraction method. X-ray diffraction method, measures average texture of the surface. Its penetration depth is low, but its penetration depth could be about 10 micrometre so, it can be considered utilizable for macrotexture measurement.

Neutron diffraction method, neutron radiations are very difficult to obtain. Usually, they are obtained in what you call nuclear reactor where neutrons are produced and then, they are utilized to in the beam line of the nuclear reactor to form, to obtain texture information when it is radiated on a surface so that the neutron diffraction could be obtained. Now, the neutrons actually penetration depth is very high and therefore, about centimetre. So, it can penetrate more than a centimetre inside the material, and we will talk about this in detail in the coming slides and in the next lecture, but the information obtained from the neutron diffraction is much more bulkier than the one which is obtained from the X-ray diffraction method.

Usually, the information obtained from the X-ray diffraction and the neutron diffraction are not that different, but in certain specialized case, when X-ray diffraction may not be able to give the information that is required or because of the heterogeneity in the texture present in the material, neutron diffraction may be utilized in some specialized cases. So, the third is the

synchrotron X-ray method. As I said that synchrotron is a specialized beam, it is an X-ray beam, and it is usually known as short wavelength X-ray beam.

Now, the intensity of the synchrotron is X-ray is very high, its wavelength lambda is lower than the wavelength lambda of X-ray, normal X-rays and X-rays are actually divergent beams, but as the intensity of the synchrotron X-rays increases, the divergence of that high-intensified short wavelength X-ray beam, synchrotron beam can be reduced, and parallel beams can be obtained. Under such situation, high-intensity very thin beams can be obtained and because of the reduction in the wavelength, its penetration depth increases to few millimetres and therefore, one can utilize this powerful specialized radiation in order to carry out both macrotexture and micro-texture.

The fourth method as I said is electron diffraction method, one can use SEM EBSD, or one can use TEM OIM. Now, SEM EBSD is much easier to utilize because preparation of sample of a TEM for orientation imaging microscopy is extremely difficult as compared to the EBSD microstructure preparation. Even though EBSD microstructure preparation is also considered sometimes difficult for some materials such as magnesium and titanium etcetera, in case of nano structure grains in many materials. These methods give local texture as said in the previous slide and it has a lower penetration depth, the penetration depth depends upon the atomic number of the material being irradiated, but on an average, it is said that the penetration depth is usually less than 1 micrometre. Therefore, it gives the complete information as I said in the last slides.

Apart from this, ultrasonic methods can be used to give qualitative information of the changes in the texture in particularly use in industries. See most of the time, ultrasonic methods are used to detect if there are any flaws or fractures in the material coming out of the production line, but if that if a certain texture is desirable, the ultrasonic intensity change that occurs because of that particular texture also changes if the orientation changes.

So, if a certain orientation or a texture is desirable, then the industry connects the final product or observes the final product under an ultrasonic method technology to see the changes in the intensity of the ultrasonic waves leading to see whether the material that has been produced is suitable for application or not. On the other hand, even using optical microscopy, one can get a slight understanding that whether the material is textured or not. Plane polarized light, if you use in an optical microscope, can deflect the light in such a way

that in a grain in several grains, it can produced different shades of colour indicating that the crystal structure on the plane is slightly different. Thus, indicating the change in the texture, but this is a very qualitative information and it only gives a very limited information about the texture of the material.

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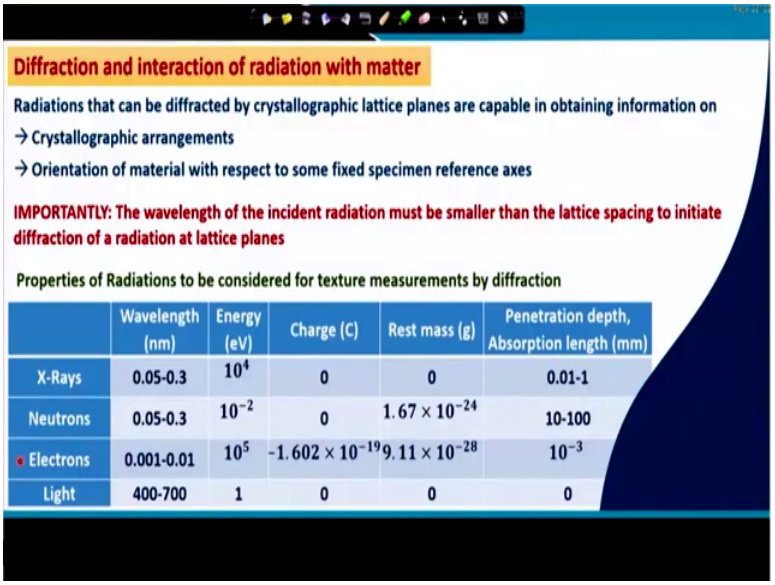


| | Wavelength (nm) | Energy (eV) | Charge (C) | Rest mass (g) | Penetration depth, Absorption length (mm) |
|---|---|---|---|---|---|
| X-Rays | 0.05-0.3 | $10^4$ | 0 | 0 | 0.01-1 |
| Neutrons | 0.05-0.3 | $10^{-2}$ | 0 | $1.67 \times 10^{-24}$ | 10-100 |
| Electrons | 0.001-0.01 | $10^5$ | $-1.602 \times 10^{-19}$ | $9.11 \times 10^{-28}$ | $10^{-3}$ |
| Light | 400-700 | 1 | 0 | 0 | 0 |

So, if we look more closely into the diffraction of these radiations which are electromagnetic radiations and their interaction with the matter, we can see that the radiations dive initially when it falls on the atom , it scatters in all the direction.

Now, when it scatters in all the direction, it is interacting, we are considering only one atom, but when there is a periodic arrangement of atom in space, the scattering of this these electromagnetic radiation from one atom in all the direction. The intensity of the scattering may not come out fully from all the direction, but it may come up from certain direction and these direction is following the Braggs law and it is following the structure factor relationship because of the periodic arrangements of atom in that particular poly crystalline material.

So, diffraction techniques are reinforced scattering, we have already talked about this in much earlier in this lecture series. Using these method, we can obtain crystallographic arrangement and most importantly for this course, we can obtain the orientation of the material with respect to any fixed sample, important sample reference directions or reference axes. So, that in order for the material sorry in order for the radiation to get diffracted via a

periodic arrangement of atoms in a poly crystalline material, the lattice spacing of these atoms, or the basis will have to be slightly larger than the wavelength of the incident radiation right. So, that is how if the wavelength of the incident radiation is much smaller than the lattice spacing, then only the diffraction will initiate in this lattice planes.

So, let us see the properties of radiations to be considered for texture measurements by diffraction. Here, we have shown the wavelength of if the X-ray is in a range of 0.05 to 0.3 nanometre, its energy in electron volts is 10 to the power 4, it has no charge, no rest mass and it has a penetration depth or absorption length of 0.01 to 1 millimetre. Now, why I have written 1 millimetre? Because I have considered short wavelengths and synchrotron wavelengths; synchrotron X-ray radiation with a lower wavelength. So, that interaction of X-rays with the matter, decides the penetration depth and the absorption. Neutrons have the same wavelength, its energy is also less, its charge is 0, it has a higher rest mass, X-ray does not have any rest mass, but it has 1.67 into 10 to the power minus 24 grams rest mass per neutron and so, its penetration depth is much higher about 10 millimetre to 100 millimetre or sometimes more for some materials.

Now, why is this we will come to this in a later stage, but still, that neutron diffraction it has no charge, and it interacts only with the nucleus of the material and therefore, it has a lot of space to go and have a large penetration depth without any absorption, just by logic .

So, let us go to the electrons. Now, electrons have a very low wavelength and it has energy much higher even than that of the X-ray. It has charge, a negative charge and it has a rest mass though lower than the neutron, but because of its interaction with different shell electrons and also, the nucleus of the material its penetration depth is very low 10 to the power minus 3. Here, we have also given the the wavelength of light, it is of the order of 400 to 700 nanometres which is much higher than the lattice spacing of any periodic lattice arrangement of any metals or atoms. Therefore, even though it does not have, it could not penetrate the atom; it cannot give any diffraction from any poly crystalline material. So, light because of the higher wavelength larger than the lattice spacing could not give any diffraction.

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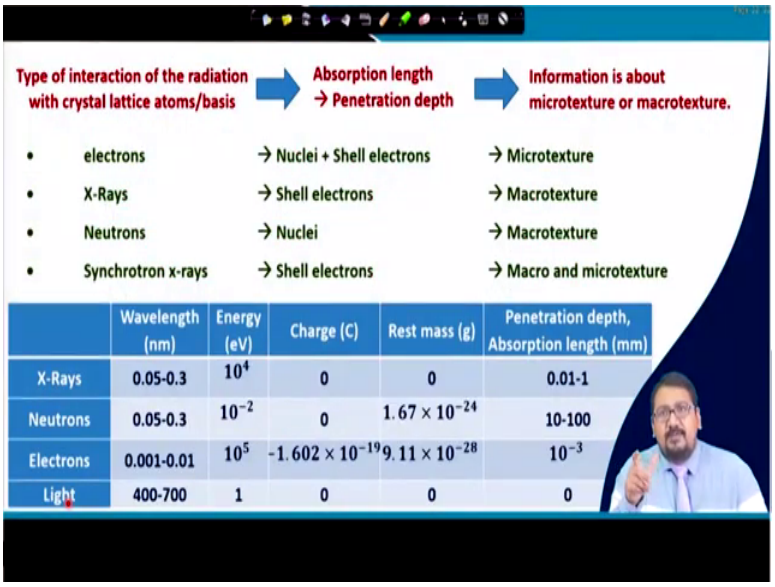


If we look deeply into this these radiations, which the absorption length and penetration depth of this radiation, these radiations depends upon the way these radiations interact with the matter. Therefore, electrons, it interacts with nuclei and the shell electrons and so, its penetration depth is very low and therefore, it gives only micro-texture information.

Electrons can be deflected using condenser coils and thereby thin beam of electron can produce and therefore, it can give in depth information in that micro-texture regime. X-rays on the other hand interacts with shell electrons, it does not have any charge, it does not have any mass so, it can go deeper, but still because it is interacting with the shell electron, its absorption is high and therefore, it could go up to few micrometer level only and therefore, it can give macrotexture information.

Neutrons interact with nuclei, and it can penetrate deeper, it has a lower, very low absorption coefficient and we usually call it linear absorption coefficient or mass absorption coefficient and therefore, it gives information of the macrotexture, deeper information of the macrotexture because it can penetrate up to few centimetres. Synchrotron x-ray on the other hand interacts same way as the normal X-ray with the shell electrons, but it has a lower wavelength and this leads to a higher penetration depth, a higher penetration depth than few tens of micrometer to millimetre level and therefore, can give a better information of the macrotexture. On the other hand, it can be produced as if a thin beam as I said earlier therefore, can be used for micro-texture either.

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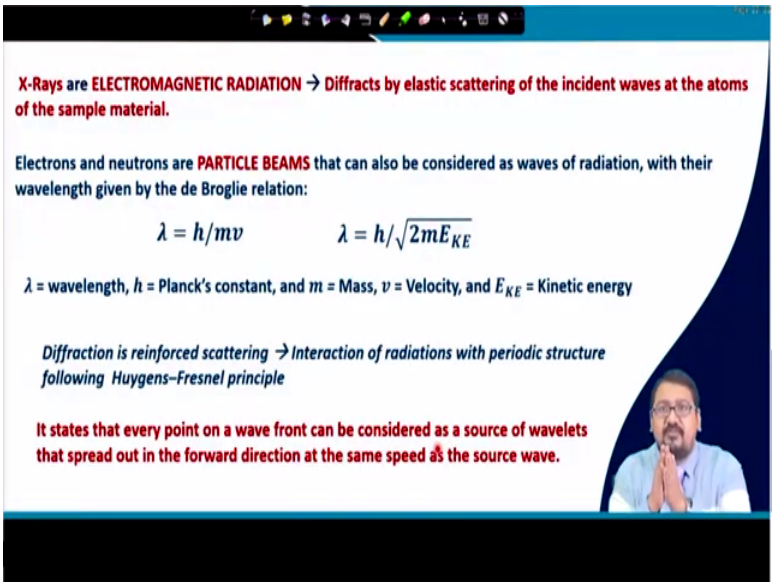


So, X-rays are electromagnetic radiations which diffracts mainly by elastic scattering of the incident waves at the atom in case of any sample material. But if we look into the electron beam or the neutron beam, they are particle which are travelling at a higher speed so that it becomes a particle beam and therefore, these particles exhibit certain wavelength and they look like a wave of radiation and therefore, they exhibit certain wavelength.

If we look into the de Broglie's relationship, which is given by lambda equal to h by mv; where h is the Planck's constant, lambda is the wavelength, m is the mass, v is the velocity and if E is the kinetic energy of the material that is equal to half mv square. Then the lambda can be calculated to be equal to h that is Planck's constant divided by root over 2 times mass into kinetic energy. So, if you calculate, we can obtain the lambda from this and that thus the electron and the neutron behaves like a wave and so, even though it is a particle using the de Broglie's relationship. As diffraction is reinforced scattering, and it produces because of the presence of the periodic structure of the material, which is what in which it is interacting.

It follows the Huygens-Fresnel principles, which states that every point on the wave front can be considered as a source of wavelength. Each point of the new waveform front is; considered as a source of wavelets that spread out in the forward direction, it is spread out in the forward direction at the same speed as the initial source wave that is all.

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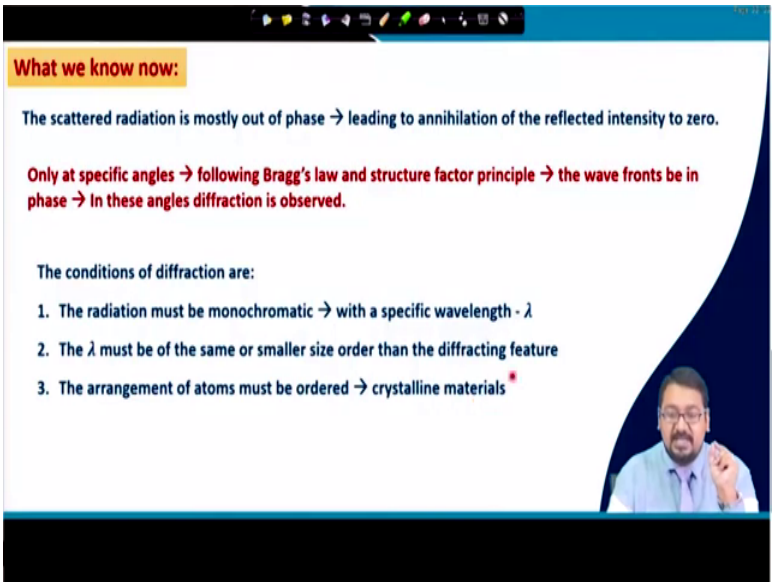


So, what we came to know from this lecture? We came to know that most of the scattered radiation, its scattered in all the direction therefore, they are in out of phase. So, this leads to mainly annihilation of the reflected intensity to mostly zero. So, only at specific angles which is following the Bragg's law and the structure factor principle, the wave fronts are actually in phase, and this are the angles at which diffractions are observed.

So, what are the conditions of diffraction? To summarize, it is that radiation has to be monochromatic, it cannot be polychromatic, and it should have a specific wavelength that means. Secondly, the lambda must be of the same or a smaller size order than the periodic lattice arrangement of that particular material in question and 3rd and the most important thing is the arrangement of the atom has to be in order right, it has to be a crystalline material.

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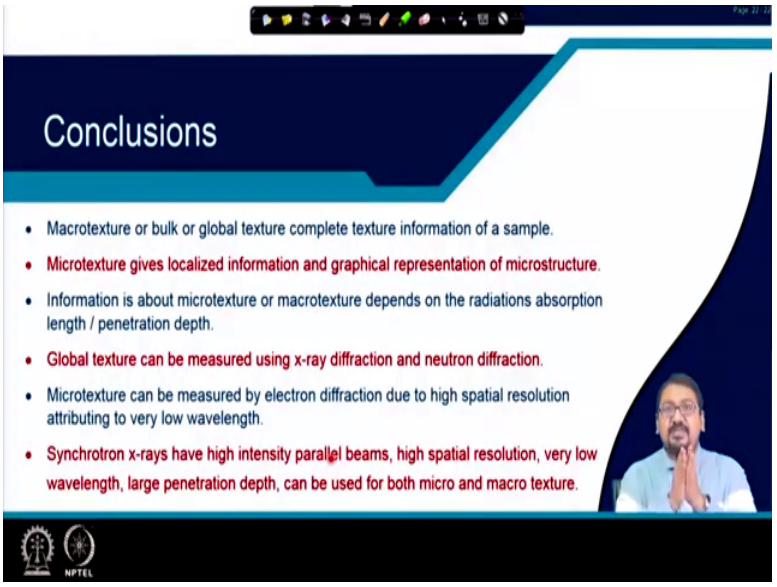


So, It can be concluded that macrotexture or bulk texture or global texture gives the complete information of the texture of the sample that means, it can give the property of that sample ok. Micro-texture gives the localized information of the texture, and it can be shown in terms of a graphical representation like the inverse pole figure map that we discussed in the initial slides and this give the information of the micro-texture, it may not be the texture representation for the whole sample.

The information is about micro-texture or macrotexture, it depends upon the radiations absorption length ok which is mass absorption coefficient, we will talk about this in the next lecture, and it decides the penetration depth. Global texture can be measured by x-ray diffraction and neutron diffraction. Micro-texture can be measured by electron diffraction because it has high spatial resolution, and it has very low wavelength.

Synchrotron x-rays have high intensities and therefore, can be converted into parallel beams, it will then have very high spatial resolution, it will have a lower wavelength as compared to the x-ray therefore, and it will have a lower absorption coefficient and therefore, a higher penetration depth and can be utilized for micro and macrotexture measurements both.

Thank you very much.