Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy
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Week-11
Lecture-34

So, welcome to this class. So, just briefly in the last class, we talked about the resolution, we talked about the numerical aperture, the importance of Rayleigh's criteria, to determine the resolution of any microscope, and we also derived some basic equations related to the numerical aperture. and also how it depends upon the minimum distance which can be resolved by any lens or the microscope. So, what we got is x is equal to 0.61 lambda divided by NA here n

So, this is the minimum formula for the X to be resolved by a microscope, which is related to the microscope resolution. Now, what we will do is we will talk about two important aspects. One is the depth of field, and another one is the depth of focus. Okay, so first we will talk about the depth of field. So, the image is in focus when the object lies in the appropriate plane.

This is very important here: the appropriate plane. So, if the object lies above or below the plane or equivalent part, then what will happen is the equivalent part of the object will be out of focus. Out of focus, okay? So, the range of positions for the object for which what we call the depth of field is the range of positions, the range of positions for the object for which there is no change in the sharpness of the image.

This is known as the depth of field. So, here I am showing you a figure where you can see that this alpha is wrong here. So, alpha is only this much. So, here you can see that this particular plane is the optimum focal plane, and your beam is converging onto the sample surface, correct? Now, the height h.

This is the height, which is called the depth of field, and this height will be estimated by assuming that there is a diffraction effect that will limit the resolution. It will limit the

resolution of this particular value D1, correct? The sharpness of the image will not differ within these ranges. So, the depth of field, h, is the distance centered on the plane of optimum focus where the sharpness of the image does not change, okay? And if you see the simple trigonometry,

If you plot this particular triangle, then this is called h by 2, and this is called x by 2. This particular distance at this distance, so you can get that tan alpha is given by x by 2 divided by h by 2, which equals x by h. And if it is in a medium—air or some other oil medium—you have to include a refractive index, μ, fine. So, this is your tan alpha. Now, if you compare this particular formula with this, then you can write h equals 0.61λ divided by n or μ sin alpha into tan alpha, and this particular term is nothing but x, the minimum distance that can be resolved.

Okay, so here you can see that the depth of field. The depth of field, which is h, increases by decreasing the convergence angle, and this convergence angle is controlled by the objective aperture, okay. So, this is how the definition of depth of field, but the problem is if you lower the alpha, decreasing the convergence angle, it will have a direct effect on the resolution. So, it has to be an optimum value of alpha to be chosen for the optimum depth of field in any microscope.

So, instead of light, if we use electrons for imaging, then it actually improves both resolution because your lambda is very small and also the depth of field, okay? So, it means that if you use an electron source for imaging, you will have high resolution because of the lower lambda and the depth of field will be very good because your alpha will be minimum or lower, okay? So, this is called depth of field.

Now we will come to the depth of focus. Okay. Now, what is the depth of focus here? As I told you, depth of field is the range of positions for the object at a particular plane where the image is sharp. But what is the depth of focus?

Depth of focus is the range of positions of the image plane for a fixed position of the object. Okay, so it is a range of positions at which the image can be viewed without appearing out of focus. For a fixed position of the object, for a fixed position of the object. So, for example, you know that magnification is given by V by U, correct? Now,

if you differentiate this equation, Then what we will get is dv by du equals minus v squared by u squared, which equals minus m squared.

Okay, so dv equals minus m squared du. Now, what does dv mean? The effective shift in the image. So, dv corresponds to the shift in the image. Because V is the distance of the image from the lens.

So, dv corresponds to the effective shift in the image position relative to du. So, the change is the change in the position of the object. Okay. And this is directly related to the square of the magnification. Okay.

So, DU is the depth of field calculated by the above. And the equivalent depth of focus is dV. So, the depth of field can be calculated by this particular range of position for the object, and dV can be calculated by this formula, where it is usually bigger by a factor of M squared. So, it means that at higher magnification, the depth of focus is large, okay?

So, this is called the depth of focus, okay? So now, we have gone through just the basics related to the depth of field and depth of focus, and these are very important terms when you are using a microscope to see if your image is sharp enough, correct? Now, next is Next, we will talk about the aberrations. So, in any lens system, whether it is an electromagnetic lens system or an optical system, there must be aberrations related to the lens.

And these aberrations directly affect the resolution and the depth of field. Okay, so usually in microscopes, we assume that all the lens systems are perfect. Okay, it means that perfection means throughout the lens system, if you talk about this lens system, if a light ray falls on this particular lens system, then the focal point will be the same. at each position of the lens system towards one point. So, it creates a single image without any aberrations.

But lens aberrations cannot be avoided. So, there are three basic lens aberrations. One is chromatic aberration. The second one is spherical aberration. The third is astigmatism.

What is chromatic aberration? As the name suggests. It is, for example, if the light has a range of wavelengths with different colors, correct? So, if a range of wavelengths is

present in the light, okay, and your lambda has a certain range in the light, correct? And each lambda value—lambda 1, lambda 2, lambda 3—their focal point will be different,

after it passes through the lens system, okay? So, a range of wavelengths is present in the light and arises because a single lens causes light to deviate by an amount depending on its wavelength variation, okay? So, it means that the lens will have a different focal point corresponding to different lambda. This may be lambda 1, this may be lambda 2 focal point, and it is so—this is the chromatic aberration.

Now you will have a certain disk of confusion within this range of wavelengths, okay, corresponding wavelengths, the focal point of those wavelengths. So, these chromatic aberrations can be minimized by using different lenses with different refractive indices. So that it will eliminate the variation in the wavelength by using filters. Okay, so, in a transmission electron microscope, what we can do is use a monochromatic source to minimize the chromatic aberration. Now, the second aberration, a common type of aberration, is the spherical aberration.

Okay, so this is because of different path lengths of different rays. which are passing through the lens system. So, it means that if a ray passes through this location and if it passes through this particular location, then the focal point for this particular ray and the focal point of this particular ray will be different. Okay, so the portion of the lens farther from the optical axis

brings rays to a focus nearer to the lens than does the central portion of the lens. So, if a ray passes through, the focal point will be here. But if a ray is near the optical axis, the focal point will be somewhere here. Okay, so, this is the first position, second position. So, you will have a disk of least confusion, which is due to the spherical astigmatism.

Okay, so spherical aberration can be minimized by using apertures. Okay, so apertures mean you can use the aperture so that the rays which are away from the optic axis can be avoided to create the image. So you will come near to the optic axis. The third aberration is called astigmatism. Astigmatism is nothing but the object points off the optical axis.

The object point off the optical axis. Okay, so it means that the light rays are focused at different locations. At different locations if you take about the horizontal and vertical planes, it means that it means that the rays from this position will have a focal point here, okay, and the rays which are in this position which is This is the vertical plane; this is the horizontal plane.

The focal point will be somewhere else. This is called astigmatism. So here also, astigmatism can be minimized by using, usually in transmission electron microscopes, stigmator coils. Okay, so, to actually remove the astigmatism from the image formation or the aberration. Correct, so these are the basic types of three types of aberrations here.

Now, if you compare these aberrations based on the source you are using, So, if you compare these aberrations, based on either electrons as a source or light as a source, okay, so the light As we can see, the wavelength is between 400 to 700 nanometers, lambda. For the electrons, the wavelength is very, very low.

It is around 0.001 to 0.01 nanometer. Your lambda is very, very, very low in the case of electrons. And these usually used in the TEM and SEM as electron source. These are usually used in optical microscopes. So, the electrons they have a very small wavelength they usually scatter by gases very heavily as compared to light.

So, usually the optical paths whatever the electron wherever the electrons are used the The optical path is usually evacuated to a pressures, to a very low pressure of 10 raise to the power minus 10 pascals or 10 raise to the power minus 7 atmospheres, okay. So, and for the electrons, the lenses which are used in SCM or TM, those are called, those uses magnetic fields, okay. So, those are called electromagnetic lenses. So, here as I told you that this is in the vacuum the very low pressures.

So, here usually the mu refractive index is 1, related to 1, and in electrons, the alpha is very small. So here, what we can do is we can assume sin alpha is equal to tan alpha, okay? And therefore, if you see the theoretical resolution R1, we can put it as 0.61 lambda by alpha, okay. So if your lambda is very small, around 0.0037. Nanometer, which is typically achieved when you use a 100 kV voltage, the potential difference.

I will come to that—how to generate these electrons. Okay? Then your alpha, if it is around 0.1 radians, which is around 5 degrees. Then your R1 achieved is around 0.02 nanometers. This is the ideal resolution which usually you will get by using a lambda of this much value, 0.0037. But the problem with the TEM is you cannot achieve this ideal value.

As I told you before, there are certain aberrations which are related to chromatic aberrations. Okay, these are spherical aberrations and astigmatism. Astigmatism can be corrected fully by using stigmator coils. Chromatic aberrations—you can use monochromators. Okay, so your range of spread in the lambda or the energy spread will be minimum.

Spherical aberrations, as I told you, can be reduced by using the apertures. That is done by restricting the electrons to the path near the optical axis. Okay. So, usually the spherical aberration. So, you have an R1.

So, you will have a disk of confusion or a radius which is R2. The minimum distance is equal to CS alpha cubed. Here, CS is the spherical aberration coefficient, and alpha is a very small value. You have to select very small values by using apertures. But if you use smaller alpha values, you may affect the resolution.

So, there should be an optimum value of alpha chosen to balance the resolution. Okay, so your actual R is equal to R1 plus R2. Fine? So, this is just a mathematical equation where you can estimate the disk of confusion, the R2 value, as CS alpha cubed. So, by using an optimum aperture, you can usually resolve points 0.2 nanometers apart in TEM, okay.

This requires alphas to be very small, fine. Now, electron microscopes take advantage of depth of field, so then you can In the case of TEM, usually the depth of field is given by assuming it is given by alpha square. So you can see that here the alpha As you lower the alpha, R2 reduces, but as you lower the alpha, you can see that R1 increases.

So, you have to choose an optimum value of alpha where you can get the minimum spherical aberration with good resolution. And in TEM, the H is given by 0.61 lambda by alpha square for electron microscopes. Okay. And as alpha reduces, you can see that H

drastically increases. So you will have a very good depth of field in transmission electron microscopes.

Okay. And these electrons can be easily scanned on the sample. That leads to the ability to use it for SEM (scanning electron microscope) to create images or SEM and BSC imaging, or in TEM, we can use this for STEM imaging (scanning transmission electron microscopes), okay. So this is why we use electrons for imaging purposes. Fine. Now what we do is

The next part is how to generate an electron beam from the electron beam, correct? So First, we need to understand the electrons and their interaction with the specimens. Usually, in a typical atom, you can see that there are certain energy shells, which are K, L, and M. Okay, so these are very fundamental things that we just need to cover. There will be inner-shell electrons, the lowest-energy electrons near the nucleus, which are K and L. These are localized energies. There are outer electrons.

Highest-energy electrons with the lowest binding energies. So, these are called valence band, like the outermost occupied shells, and there is a conduction band where they share the outer electrons with a range of possible energies, and these are delocalised They are not localized; they are delocalized. Okay, so For an electron which is in the outer shell or the conduction band, a very small amount of energy is needed to detach it from the atoms or the outer electrons.

Correct? So, this principle is used to remove the electrons and to use these electrons for imaging purposes. As you know, in the nucleus, we have a positive charge surrounded by negative electrons that neutralize the charge. Fine. Localized electrons in the outer shells are shared depending on the type of bonding with the surrounding atoms. Now, we know that an electron can behave as a particle or as a wave.

And the electron has a charge of 1.6×10^{-19} coulombs and a very low mass of 9×10^{-31} kg. And these electrons, if they exist as particles, can be accelerated through a potential difference of V volts. Then its velocity V may approach the

speed of light. So, with this increase in velocity, the electron mass will increase according to this particular equation. $M = M_0 / \sqrt{(1 - (V/C)^2)}$.

If you consider it as a wave, it has a certain wavelength and momentum. And it is related to a famous relationship called the de Broglie relationship. And it is given by lambda which is given by h by p which is equals to h by mv. h is the Planck's constant. So the energy given to the electron

to the electron can be equated to the energy represent by the change in mass between these two equations. So, as I told you, as a wave, this is particular equation. So, energy given to the electron can be equated by relativistic change of the mass and by equating these two equations, you will get EV equal to M minus MEC square. C is the speed of light. E is the charge.

H is Planck's constant, ma is the mass of the electron, but the mass of the electron changes when it approaches the speed of light. Correct. Now, with these equations—with all three of them—you can actually calculate the wavelength as a function of accelerating voltage. Okay. If you solve this particular equation, you will get this last equation, where lambda and voltage can determine the wavelength of the electron or— The wavelength of the electron can be calculated by the voltage or the accelerating voltage. Here are the values from a typical table, where you can see that with increasing voltage or potential—

or with increasing acceleration due to voltage—the wavelength also reduces significantly as the voltage increases. Correct. So, this is how we obtain different wavelengths, lambda, and you can see that these are in nanometers. These are actually very, very small distances, which relate to the inter-laminar spacings of any unit cell. So— These values, by using electrons with this wavelength, can actually resolve the inter-laminar or inter-atomic distances. That is why we choose this lambda with a very low value.

Fine. So, with this introduction, I will briefly go through the generation of a beam of electrons, which is done by two methods. One is the thermionic emission source, and another is the field emission source. We will come to the field emission later. Thermionic source. First, we will talk about the thermionic source.

This is the widespread use of thermionic emissions for the heating element. So it is exactly like a bulb. Correct? Here, what are the components? So there is a filament here.

Okay? There is a Wehnelt cap, which is called W. And here, there are anode plates. These are called anode plates. Anode plate. Okay.

Now, when usually across this filament, a small current is passed when there is no potential applied. Okay. But before, the filament current is high. So when you apply a voltage across the tip, and this is the anode plate.

Then what you are doing is extracting the electrons from this particular filament, and these are accelerated towards the column. So this is typically an electron plate. So, the anode plate for extraction is getting a positive potential. And these are applied as a negative potential. Okay.

Remember, in atom probe tomography, we have just introduced that time. Actually, you are applying a negative potential to the electrode. So that you will extract the ions from the sample. Correct. So, exactly a similar thing you can assume.

So, there is a tungsten filament which has a certain radius of curvature. And you are applying a very high potential for extraction. Correct. At the anode, so that it will extract the electrons from the tungsten filament. Now, if you are not applying any voltage, there will still be a small current passing towards the filament, and that particular current is called a dark current.

So, even though electrons are not extracted from the filament, there is a current passing through it, which is called dark current. It means that it flows before it emits light or electrons. Correct? So, if you see the electronic circuit design, this is your filament circuit. It is applied with a bias voltage, and this is your anode plate where you apply a positive potential. Due to

this field, electrons will accelerate. So here, you can see that the tungsten wire is a cathode, which is a filament, and it is heated by the passage of current to about 2800 Kelvin. So, you are not applying a voltage; you are just heating this filament by passing current through it. So, these are heated by the passage of current to about 2800 Kelvin

and are kept at a negative potential with respect to the anode, which is kept at a positive potential. And due to this positive potential, if you increase the potential voltage, these will extract these electrons across and accelerate towards the column, okay.

So, the filament controls the diameter of the area at the end of the filament, and tungsten acts like the Wehnelt cup. This particular Wehnelt cup, which is made of W, acts like a grid. So here, the parts of the emitted electrons cross at one point in space, and that diameter depends on the area of the filament emitting the electrons. This area is controlled by the potential between the filament and the grid RB. So you can see that these electrons are passed and concentrated to a particular spot. And this diameter depends upon the area of the filament emitting electrons.

And usually, this is the electron source. So the size of the electron source is very important for the resolution of a microscope. Here, you can see that this is your beam current. Beam current means the actual electrons, which are the current—electrons traveling after you apply the potential—and this is your filament current, okay. So the filament current increases initially.

As the filament current increases, you can see that this particular value is the dark current, as I told you. When you do not apply a voltage or a bias, then this current passes through the filament. Now, as you increase the filament current, you can see that your beam current increases significantly. But it reaches a saturation value, which is called Ic or Ie, the critical current or critical filament current. So there is not much change in the beam current.

It almost gets flattened. So it means that there is no additional change in the beam current. Okay? So it means that it has a direct consequence on the brightness. Brightness of the beam.

So your brightness will not change after a particular critical current IC. So, the B increases rapidly as your filament current increases, okay? Also, the brightness actually depends upon φ, which is called the work function, okay? So brightness depends upon your temperature and the work function. Higher brightness means you should get a higher

temperature, so you have to use a material or metal that has a very high melting point, which is usually tungsten, as we are using it. It has a melting point of 3653 Kelvin.

It is very high. And also, the work function should be low enough. It is usually around 4.5 electron volts. It should be low. So that your electrons—the probability of the higher density of electrons—can be removed very easily.

So your brightness will be very high. So for a typical thermionic emission with a tungsten wire, the brightness is around 10^9 A·m^{-2}·sr^{-1}. Fine? So this is related to your brightness. So here, I am showing you a typical image of an electron gun.

That electron gun consists of a—this is—it goes to an HD cable. You can see there are two points here, and in these two points, there is a connected filament. Okay, so this is your filament. This is your Wehnelt cup, and this is your anode plate where you are applying a positive potential, and it is negatively biased. Correct?

So there are three major parts of the thermionic gun from top to bottom. You can see that there is a cathode. This is your cathode; there is a Wehnelt cylinder, and there is an anode, which is shown separated here. So, the Wehnelt screws into the cathode. So, you can attach this particular Wehnelt cup or Wehnelt cylinder to the cathode, and both are attached to the high-voltage cable, which also contains power

supplies for heating the cathode and biasing the Wehnelt. The anode sits just below the vignette and the whole assembly sits on the top of the column of the lens. So if you have seen any microscope, there is a column, there is a panel, and there is a screen here. Correct? And usually the gun or the electron gun sits at the top region.

Okay, and this is connected to your HT cable. So here you can see an electric circuit where you can see that the filament, there is a bias resistor, there is a Wehnelt cylinder and there is a crossover which is controlled by the positive potential applied at the anode, okay? And here there is a typical image of a filament. With the two tips which are connected to a thin tungsten wire. As I told you, here we use tungsten as the source.

Thermionic. So there is another source which is called LaB6. The advantage of LaB6 is the work function is very low. It is lower than 3 electron volts. So you will get 10 times brighter than by using tungsten.

Okay, so the LaB6 filament can also be used as a source for the electrons. Now, here there is a very good example of the effect of bias voltage on the acceleration of electrons towards the column. Okay, so you can see that this is your filament, this is your filament, and this is the Wehnelt grid. You can assume this is the Wehnelt grid, and this is your anode. Now, if there is no bias, if there is no bias, then what will happen?

There will be no deflection of electrons which are coming from the needle tip or the tungsten tip. So, these electrons will flow in this direction. So, it has a long spread between these electrons. Here, you will get the maximum current, but the beam will not be focused enough. So, you cannot use this spread electron beam for your imaging purpose.

However, if you put an optimum bias, you will get an intermediate current, but you will get a very fine spot. By applying this voltage, these electrons will actually be deflected back to a certain point. This is what we call the gun crossover. Okay. But if you apply a very high bias, the electrons will not be able to reach the anode.

So, they will be deflected back. So, you will not get any current, any beam current for imaging purposes. So, here you can see the variation of the bias voltage versus the emission current, and you can see that in a certain region of the bias voltage, you will get the maximum brightness. And maximum brightness. So, your aim is to get not the maximum current or the minimum current.

Your aim should be to use the maximum brightness at a certain intermediate current so that you can use these electron beams, focused electron beams, for imaging purposes. Okay, so usually these electron beams, these electrons which are flowing from the filament, are focused by using these biasing Wehnelt grids. Okay, as I told you in thermionic emissions, this is the same; it is related to the brightness. So, the brightness definition is the beam current density per unit solid angle. So, this particular solid angle is nothing but a steradian, which is a unit of solid angle.

What is a steradian? It is the solid angle which encloses a surface on a sphere equal to the square of its radius. So, this is a square. For thermionic emissions, the brightness can be related to your voltage, the temperature, and the work function.

As I told you, the work function and the temperature are both very important for high brightness. And these two depend upon your filament material. Okay. So, in layman's terms, we can say that brightness is a measure of how many electrons per second can be directed at a given area of the specimen. Okay, so this is generally related to brightness.

Now, another, as I told you, there are two types of sources. One is thermionic. Another is field emission. What is field emission here? So, here I can show you that.

So, this is just an example of thermionic. So, this is your tungsten filament. LaB6. This is nothing but a... Crystal LaB6 single crystal where your work function is much lower.

Around 3 electron volts, your brightness is 10 times higher. Correct? This is the difference between thermionic emissions. Now, there is another source which is called a field emission source. In the field emission source, they also use a very sharp tungsten tip.

So the radius of the tip at the apex will be less than 50 or 200 nanometers. A sharp tungsten tip is used and usually the electrons are accelerated by generation of electric field. Remember in the second class when we started the introduction of the atom probe, I told about the two different aspects. So, Professor Mueller during his investigation first he was working about the electrons, how to extract electrons by applying a positive potential to the anode and negative potential to the cathode due to which he generates a very high amount of electric field which will extract these electrons.

And as I told you during in parallelly in Ernst Ruschka he was also using these electrons for the designing microscope. So due to the de Broglie due to the Heisenberg and uncertainty principle these electrons and they have a very low mass. So the uncertainty of electron. to capture to a particular position is very difficult that is why the resolution was for imaging the resolution was very less than it is almost not less than 2 nanometers. That

is why the Professor Muller went to reverse the potential with the negative and positive so that you can use the ions for the field of operation.

So that is a different thing. Now here exactly in a similar way if you apply a negative potential and a positive potential then you will generate a very high amount of electric field and due to this electric field so there will be a these electrons will be tunneled or quantum tunneled. So, we described about this process during atom probe also but similarly here these electrons are removed from the specimen or the filament. So, as I told you here, the field emission source—the metal or the source, usually tungsten—the metal surface is subjected to an extremely high electric field.

which is greater than 10 raised to the power of 9 volts per meter, and due to this very high electric field, the probability for an electron to leave the surface without needing to be given the amount of energy for the work function, it can tunnel very easily due to the presence of a very high electric field. So here, the temperature is not playing an important role. Here, the important role is played by the electric field. Okay, so here, usually, a greater number of electrons can be drawn from the same area of tungsten.

So, your B increases up to 10 raised to the power of 13, which is much larger than thermionic sources, which is here around 10 raised to the power of 9. Correct? So, it is almost 4 orders of magnitude larger in brightness by using this field emission source. And this field emission source is nothing but the tunneling of electrons from the metal surface by using a high electric field near the metal surface. Okay? So, this is a typical equation where you can see that the field emission current depends strongly on the applied field, and Ef is the Fermi energy.

Fermi energy, which is 5 electron volts for tungsten at room temperature. So, this is the density of electrons that can be removed in the field emission source. So, this is a typical cross-section of an electrical circuit where you can see this is a field emission electron gun, and it is nothing but a tip. Okay, and this tip is made very sharp in order to get a very high field strength with low voltages. Okay, the field-emitting tip has a very strong curvature, meaning the needle specimen, the needle is around less than 50 nanometers.

And this is done by etching a single crystal tungsten wire to a needle point. And it is less than 10 nanometers or it is up to 50 nanometers. Okay. The grid, similarly the grid will be there. There is an anode.

There is a crossover. The grid provides the field that extracts the electrons. Okay. by tunneling mechanism due to the generation of large amount of high degree of electric field near to the tip surface. So, there is a very small tip radius gives a very high field strength which is 10 is to the power 9 to 10 is to the power 10.

Anode is positively charged as I told you so that it can extract the electrons and its function is to accelerate the electrons to the potential. With this grid also you can generate a crossover which is called a GERN crossover and your electron the energy spread in this is very minimal as compared to the thermionic sources. So thermionic sources are they are heated up by passing a current. And there is an application of high voltage so that the electrons will come out at high temperatures. So, usually you use a very high temperature source, high melting point source with a lower work function.

But in field emission, you use the tunneling process by applying a high electric field. Okay, so with this, I will end this class now, and I hope that you got a feeling for how electrons are extracted from a metal surface. Usually, tungsten is used, and in subsequent classes, we will cover more topics related to electron microscopy. Thank you.