

**Advanced Material Characterization by Atom Probe Tomography and
Electron Microscopy
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Week-11
Lecture-33**

So, welcome to this class. In the last classes, we discussed atom probe tomography. We discussed sample preparation, focused ion beam, the use of focused ions for sample preparation, and we also talked about reconstructions and how to use the atom probe to obtain chemical information. So, if you go back to the first class, I introduced this course as Advanced Microstructural Characterization by Atom Probe Tomography and Electron Microscopy.

So, from now onwards, all the lectures will focus on electron microscopy. So, in electron microscopy, what we will do is cover the basics of TEM-related topics and one more advanced technique, which is SEM-based, related to ECCI (electron channeling contrast imaging). We will also briefly cover SEM-based techniques like EBSD and TKD. The reason we need to introduce these techniques is that they will be used in correlation with TEM. I will

show you how these techniques can be used together with atom probe tomography to obtain structural information from macro to atomic scale. Then, how this structural information correlates with atom probe tomography, where you obtain chemical information. Okay, and all these electron microscopy techniques can be used to visualize or observe grain boundaries, grains, phases, precipitates, and defects. Okay, and in the introduction, I also mentioned that these are controlled by phase diagrams, solidification, and phase transformation, which overall determine the properties of any microstructure or alloy.

Okay, so this is the composition; this is the structure. And we have basically covered the reconstruction. So, I will show you in the last few classes, maybe two or three, how atom probe tomography can be used with electron microscopy to get correlative information on

structure and chemistry. Now, we will focus mostly on the electron microscope or the transmission electron microscope.

Fine. So, the content will be in the next 10-hour lectures or around the next 18 to 20 lectures. The content will be the basics. I will cover the basics of image formation, resolution, and aberrations. These are very important to understand image formation in the transmission electron microscope. Then we will briefly go through electron interaction with the sample surface and the signals we get in the microscope,

in a typical microscope, whether in transmission mode or scanning mode. Then we will talk a little about electron diffraction, the principles behind it, and reciprocal space. So, these things will be covered, including the basic construction of TEM and how electron diffraction is used for imaging sample features. Fine. And if time permits, we will also briefly go through chemical analysis in electron microscopes.

Okay. Anyhow, we have covered atom probe. Then there are other techniques, which I told you, based on ECCI, TKD, and EBSD. These three techniques I will cover briefly. Then I will show you some case studies.

These are based on different alloy systems. So, as a metallurgist—or as a materials engineer—we deal with different types of alloys, whether steels, superalloys, aluminum alloys, or semiconductors, okay? How all these techniques can be used to understand the microstructural behavior of these alloys, which controls the overall property of the alloy, fine? So, with this,

what we do is introduce the basics related to image formation. So, if you have any object, fine, you use an optical system. These are the basics related to any optical microscope. This optical system's main function is to magnify that particular object so that you create an image, okay, which is at a very high magnification. Okay, and to understand this magnification—the optical system and how it magnifies the object—there are several parameters or important concepts which need to be understood.

And which is very common, either you go to the optical, either you go to the electron microscope, either you go to the scanning mode or the TEM. These important concepts

will not change. Okay, so the basic concepts are magnification, resolution, depth of field, and aberrations. Okay, so these are the four common concepts which are important for most of the optical systems or the electron systems. Fine, so briefly, if you see that in SEM, in a scanning electron microscope, where you actually beam. Rasters on the sample surface, okay, and usually they will give you the information related to your morphology.

Shape of the sample, the sample features, fine. So this is a typical electron microscope where we can do it in a scanning mode. Okay, and in this SEM, usually we can perform electron backscatter diffraction, you can perform electron channeling contrast imaging. You can get the express X-ray spectroscopy, which is related to the energy dispersive spectroscopy, okay, which gives your chemical information at the local scale. We can also use the WDS, okay? So, we will also cover something about some basics related to WDS, which is Wavelength Dispersive Spectroscopy, okay?

So, the difference between the Express Spectroscopy and Wavelength Dispersive Spectroscopy is related to your resolution, energy resolution. Then, so this is a scanning electron microscope. So, in a transmission mode, in an electron microscope, You usually use a very thin specimen. So, and these thin specimens are electronically transparent.

Okay, so this is a typical instrument of TEM where the beam, a high-energy beam which is accelerated from a large potential, passes through the thin sample. Okay, and you can get the diffraction pattern. We will cover that. We can get the diffraction contrast to identify the phases, and also the electron beam can be used to image the atomic structure or the atomic columns. In a certain crystallographic direction of that particular phase, the material, or the alloy. Correct?

So, these atomic structures can reveal the internal defects, phase boundaries, okay, or the crystal structure. Okay, so these things can be done in transmission mode by using the transmission electron microscopy image. Now, if you see an image formation, There can be three types of image formation. The first one is projection.

Projection is nothing but how your object is projected, or you can actually mimic the projection of a shadow. Okay, so if you have an object, if you have a source, and if this source is a light source, If the light is projected on the object, at the back you can see the

image formation. So, this is a very basic example of a projection image. So, you must have seen the projectors in classrooms or during presentations.

Usually, they will project your screen onto the wall or onto the projector screen. So, this is the basic form of image formation. The second is an optical image. Optical image—what is the difference? They use conventional lens systems.

Fine? So, you can see that this is a lens here. This can be an optical lens, like a glass lens, or this can be an electromagnetic lens. Okay? This will come in later classes.

Okay? And based on the Depending on the position of the object, you can get different types of images. So, this is done by conventional lens systems—optical images. Then, another one is the scanning image.

This also uses a conventional lens system, but here the difference is that each point on the sample surface is scanned or presented serially with a high-frequency beam. Okay, so if you have a sample surface and if you have a beam, this particular beam is rastered in the x, y, and z direction. Okay? So the beam will raster at each point of the sample surface, and it will give you the image. By converting whatever interaction of the beam with the sample occurs, those interaction signals will be converted to your image formation. So, this is called a scanning image.

Scanning images are usually used in SEM and TEM. Optical images are mostly used in optical microscopes where there is no scanning of the beam. Okay. So, this is the basics of image formation. Now, here I am showing you a typical optical system.

So, here the ray diagram shows the concept of focal length and magnification. Based on the position of the object, this is your object. Okay? And here, U is the distance between the lens and the object, and this black dot represents the focal point of that particular lens. Based on the position of the object relative to the focal point, you can actually see three types of images under three different conditions.

Okay? So, in the first condition, this is your first condition where the object is placed between the f and 2f. So, assume this is the 2f position. If the object is placed between these two positions, the image is magnified, real, and inverted.

This is your image. Okay. So, there is a source of light here. That source of light, It hits this particular object.

Then you can see this ray diagram. It passes through the focal point and will form a real image, which is inverted and magnified. So, you will get a magnified, real, and inverted image. This is when your object is placed between F and $2F$. If the object is placed, as in the second case, in between the F . Okay.

So, this is your object. This is your object. Now, in this case, the image is erect if the object is placed within F , but it is virtual. It is not a real image. And it is magnified.

It is magnified, and it is not inverted, but it is virtual. Correct? If the object is placed well away from the $2F$ —so this is your $2F$ point—well away from the $2F$, then the image is demagnified. You can see that this is your object. And it gets demagnified.

It is a real image and inverted. So, the first condition and the third condition are used for magnification or for demagnification, okay, and based on the placement of the object with respect to the focal point of the lens. Now, if Here, one important principle we need to understand is the principle of reciprocity. Reciprocity, okay, and what is this principle?

It shows that the effect on the light rays does not depend on the direction in which the light is traveling. Okay, so this is the principle which will be very important to understand. Okay, so in conclusion, it can be summarized by a basic thin lens equation. Here, what is the thin lens equation? The f is the focal point of the lens, u is the object distance, and v is the image distance.

This is your focal point. Okay? And magnification is nothing but, as you can see, this particular triangle. This is one triangle and another triangle. Triangle 2.

If you see the symmetry of these two triangles, the ratio or the magnification the image or the object, the size of the image and the size of the object, is directly related to the distance between the center of the lens to the image, which is v , to the distance of the object from the lens center, which is u . So magnification can be given by v by u , and by using this thin lens equation you can actually get the magnification as f minus u minus f

or v minus f by f . Okay? So, if you measure the image distance and if you know the focal point of your lens, then you can easily calculate the magnification.

So, for large magnification, for large magnification, your v minus f must be small. u minus f must be small and positive. Okay? So, for large magnification, this particular u minus f should be small and also it should be positive.

Then only you will get large magnification. And this is achieved only by placing your object just above, just away from your focal distance, the focal point of the lens. Okay, so, this is your simple lens system for image formation. Now, we will go a little bit deeper into whatever you are getting—the inverted image in this case—this is your inverted image. You can use another lens system in the setup to make this inverted image a non-inverted image.

So, here I will show you some of the basic construction of this lens system. Now, you have an object here which is placed above the focal point Okay, which is at f_1 . Assume it is the focal point of the lens, the first lens. Okay?

And there is a second lens which is introduced after the inverted image of the object. Okay? Which has a focal point f_2 . Fine? So, this particular second lens

is called the projector lens, and this particular first lens is called the objective lens. Okay, so here we are introducing a new term which is the projector lens. The objective lens's function is to provide an inverted image at B. And the importance of the projector lens is that it gives you an upright image of the image formed at B. So, this is your first image. This is your projected image. So, this will give you the upright image of the first image which is formed from the objective lens.

Okay. So, the magnification at this position B It will be given by the earlier formula which is V_1 minus F_1 divided by F_1 . Correct? Now, this particular image's size is magnified again by using the projector lens.

So, it will give a further magnification of V_2 minus F_2 divided by F_2 . So, the total magnification is given by this particular equation, which is V_1 minus F_1 , V_2 minus F_2 divided by F_1 into F_2 . So, if you know the distance of the first image, V_1 , and the

distance of the upright image, which is V_2 , and if you know the focal points of the two lenses, then you can get the exact magnification of that particular original object. Sometimes, they also use a second projector lens.

Okay. So, from the basic optical system, we went to the next stage, where we are introducing a projector lens. Because of the introduction of the projector lens, the first lens, which is used, is termed as the objective lens. The objective lens is usually termed as the lens which forms the first image in any microscope. Okay, even in a transmission electron microscope, you can see that there is an objective lens.

And whatever image is formed, that has to be magnified and projected upright. So, that's what we call or use as a projector lens. Okay, now, in the next slide, what I will show is, till now, what we have assumed is that your object is self-illuminated. Your object is self-illuminated. It means that the object itself acts as a source.

It emits light. That's why we are able to form the image. That's why these light rays pass through these lenses. But it is not the case. So what is the assumption?

The object acts as a source where the rays start from the object and end at the screen where the final image is projected. So here, the illumination from the source is very important. That's why the next thing is one more attachment, which is called a condenser system. It means that before the objective lens, before the object or objective lens system, you will have a certain light source. And that light source—if your object is transparent—then illumination is behind the object.

If the object is opaque, illumination is from the front side. Okay, so there are two arrangements here. One is if your illumination is behind the object in this position—this is an object—if the object is transparent, then your illumination is in this direction. The rays will fall in this direction. But if your object is opaque, then usually what they use is a source of light, okay? A source of light in this case, by using the condenser system, and they will use a half-silvered mirror so that the illumination is from the front of that particular opaque object. Okay, so now we are introducing here a condenser system. So, as the name suggests, condenser

It condenses the light or the light beam. The main function of the condenser system is to condense the source, to condense the light. Correct? So, this is called the condenser lens system. So, transparent samples, as we know, most biological samples are transparent.

So, you use the light source behind the object, and most metallic samples or bulk samples are opaque objects. So, you will use the light source in front of the object to lens. Okay. So, what is the condenser system? As I told you, it is necessary—the condenser system—it is necessary to collect the light, to collect the light

which is diverging from the source. You can see, if there is a light source, a point source, you can see that the light is diverging. Okay? And to make these divergent light rays converge, or to converge these light rays, you use the condenser lens system. And this condenser system has two main functions.

One is the object. It appears brighter. Contrast will improve. The second is it is used to control the angle at which the illumination arrives at the specimen. Okay, so this is the function of the condenser lens system. Similarly, in the transmission electron microscope, you have a condenser lens system, an objective lens system, and a projector lens system. These three systems are common to any microscope.

Whether it is optical, scanning electron, or transmission electron microscope. Okay, and this condenser lens system in TEM is usually used to converge the beam, which is very important in TEM. So, convergent beam diffraction patterns are taken. We will come to that at a later stage.

But remember, for any image formation in a basic electron microscope or an optical microscope, these three main systems are very much needed. Condenser system, objective system, and the projector system. So, you can see that at each lens system, whether it is an objective lens system or a condenser lens system, you can see these dark areas, which are called apertures. These are called variable apertures at the condenser lens. These apertures are very important to control the area of the specimen which is illuminated and the angular spread.

Okay, so these apertures are used to control the area of the specimen. So, if you see in the optical system, recently we talked about magnification, and magnification is a function of the final image distance from the lens, the object distance, and also the focal point. So, in light microscopes, usually as in this equation, the variation of V_1 and V_2 is very difficult in optical microscopes. But F_1 and F_2 are feasible.

That's why in light microscopes, usually you will change the lens when you want higher or lower magnification. Because changing the distance of the image, V_2 or V_1 , or U_1 is very difficult in the optical system, in the optical image system. So, you can vary the focal point, F_1 and F_2 , by changing your objective lens to one with a different focal point. But in electron microscopes, in electron microscopes, you do not have to change the lens; this problem will not be there.

So, this parameter can be adjusted. So, if you compare any lens system magnification, you can see that in light microscopes, the resolution is around 200 nanometers because it uses light as the source, and the human eye can detect objects up to 0.2 mm in size. Okay. So,

Here comes another important point: you cannot magnify the smallest details, which can be resolved up to 200 nanometers, to a large size up to 0.2 mm. So, any magnification larger than 1000x makes the details appear larger and larger. So, you can magnify infinitely. You can magnify it.

The thing is, if you, even though if you are magnifying, that it, the finer details, which is λ , cannot be resolved. Okay? So, there comes the resolution. Even though you can magnify higher and higher, but if your final detail, it is not necessary that the two points or the final details can be resolved by magnifying. Okay?

So, then as I told you, it comes the resolution. Okay? So, what is resolution? To understand the resolution, first is you need to understand the numerical aperture. So, what is numerical aperture?

In any lens system in an optical lens system if you have a lens it has a certain numerical aperture. What is numerical aperture? It is the measure of its ability to gather light and

resolve finer details at a fixed object distance at a fixed object distance so now if you have a lens fine now you have a object here this is your object And this is your light beam.

Okay? This is your lens system. And this is fixed here, which we term as D . Okay? And this is your beam. The whole angle is θ .

Okay? And the half angle is α , okay? And the distance between the lens and the object—this is your object—is small d , okay? So, the angle α is the half angle of the angular aperture. So, this is your angular aperture, θ , okay?

So, this is your angular aperture. This has to be converted to the numerical aperture. So, usually the NA (numerical aperture) is given by $N \sin \alpha$. Here, N corresponds to the refractive index of the imaging medium. Okay.

And usually, it varies from 1 to 1.51. 1 is for air. 1.51 is for oils. This is your definition of numerical aperture, and any lens system will come with this numerical aperture. Fine. So, as per the formula for numerical aperture, this will be maximum when your α is equal to 90 degrees because the highest value of $\sin \alpha$ is at 90 degrees.

So, $\sin \alpha$ is equal to 1. Okay. So, based on this, what we can do is if you have an objective, this angular aperture—fine—so based on the distance between the object and the lens system, you can actually see that different numerical apertures can be achieved. So, for example, if you have an object here, which is at the same place—now this is your, okay? So, the beam—you can see that at each condition, your angular aperture increases, correct?

So, this is your angular aperture. And this is your object. Now with this, you can see that if the n changes or if the α changes from 7 degrees to 60 degrees. So this might be from 7 degrees to 60 degrees because it is a half angle, correct? This is your θ , sorry.

This is your θ . This is your α . This is your α , okay? So, as α changes from 7 to 60 degrees, your numerical aperture changes from, assuming it is in air, 0.12 to 0.87, okay? So, it means that, from this formula, if n is high, your NA will also be high.

If the refractive index of the medium is high, then NA will also be high. Okay. So, the importance of numerical aperture will be covered in just another slide. So, before going to the actual effect of the parameters on the numerical aperture, we also talked about the resolution, which is actually given by Rayleigh's criterion. Okay.

So, what is Rayleigh's criterion? Rayleigh's criterion is nothing but the closest spacing between two points which can be clearly seen through the microscope as separate entities. That is called Rayleigh's criterion. So, this is not the same as the smallest point which can be seen by magnifying it. So this is called your resolution limit.

This is called your resolution limit. Now, to understand this resolution limit, we need to go through a little bit and explain about the diffraction limit. So, even if you have a microscope, all the lenses will not be perfect and will introduce distortions in the image. So, as your light travels through space, there will be bending around the obstacles, interfering constructively and destructively. Especially these light rays, if they move around pinholes for light, then the parallel beam of light, which is a spot, transforms into a series of cones that are seen as circles known as Airy rings.

So if you have a pinhole and if your light passes through this particular pinhole, you will have concentric rings in two dimensions. And if you see perpendicular to this screen, you will see that these are the concentric rings. These are called Airy rings. And the diameter of the central ring, this particular diameter of the central spot, is inversely proportional to the diameter of the aperture or the opening of the pinhole from which the diffraction is occurring.

Therefore, the smaller the aperture is. This is a big aperture; this is a smaller aperture. Therefore, the smaller the aperture is, the larger the central spot will be. The larger the central spot will be. So, diffraction can occur from every small point in the object and can suffer diffraction.

Okay, so the small points in an object can also become Airy discs in the image. This particular Airy disc—the intensity will be maximum at the center. And there is a disc which comes after a particular distance; it increases, then decreases again. This is directly related to the constructive and destructive interference of the diffracted beam or the light.

So, for a circular aperture, if you have a circular aperture of diameter D , the first minimum

in the diffraction pattern occurs at θ equal to $1.22 \lambda / d$. d is the aperture size. Okay? Here, λ —we can take it as light, which is equal to the wavelength of the light—and this relationship has come from a Bessel's function. We will not go into detail about the Bessel's function. So, Bessel's function is nothing but the light intensity.

Okay? Light intensity $I(\theta)$ is a function of $I_0 [2J_1(ka \sin\theta)/(ka \sin\theta)]^2$. Okay, so it means that $J_1(x)$ is given by the Bessel function of x . In this Bessel function, the first minimum occurs at $\theta = 1.22\lambda/d$. Now we will understand the minimum. So, if you have an aperture or a pinhole, and if light passes through the pinhole, what will happen is, if you project it on the screen, you will have a large central peak of intensity,

there will be a minimum, then an increase, then another minimum, then another increase. Okay? Now, the first minimum which appears—this is your central point, 0—the first minimum occurs at a distance of $1.22\lambda/d$. d is the diameter of that particular aperture, and this first minimum is related to θ , the first minimum appearing in the function. Okay.

Now, we will introduce Rayleigh's criterion here. Now, what Rayleigh's criterion tells us is that if there are two objects, O_2 and O_1 , and they are self-illuminating objects, and if they are passed through a pinhole, okay, and if you project the intensity on a projector, these two objects can be resolved only when the minimum of one object is at the maximum point of the other object's intensity. It means that if the maximum intensity point of one object merges with the minimum intensity of the second object. And this is the minimum distance that can be resolved by Rayleigh's criterion.

So, here you can see that this case, the first case is resolved. The second case is the Rayleigh limit. And you can see that in the third case, these two intensities are not resolved. So, this is not resolved as per the Rayleigh's criteria. So, remember the first minimum of the diffraction pattern matches with the maximum part of the other intensity or other object intensity and

the first minimum occurs at θ equal to $1.22 \lambda / d$ which is given by this formula from 0 which is derived from the Bessel's function. So, two points are just resolvable if they are separated by an angle. This is called θ , θ minimum, which is equal to $1.22 \lambda / d$. Here, λ is the wavelength, which shows that it is a function of wavelength which the light is used and also the d , okay, so diameter of the aperture. So, the microscope resolution.

The microscope resolution can be defined as the ability of the lens to produce sharp images of two closely spaced point objects. Okay? So, if you have two objects. Okay?

And this is your lens system. If you do it, this is your θ , this is your x , this is your distance between the lenses. So, these two objects, the smallest distance x by which the objects can be separated is the resolution. So, according to Rayleigh, θ equals, from the Bessel function, it is around $1.22 \lambda / d$

and from this geometry, which equals x / d . d is the distance between the two objects, correct? With the small angle approximation, usually we can say $\tan \theta$ equals $\sin \theta$, which equals θ . Therefore, the resolving power x is given by $1.22 \lambda / D$. This is your resolving power of any microscope. Now, just now we covered the numerical aperture. Right?

Just now we covered the numerical aperture. Right? So, based on the numerical aperture, the formula which is given by NA equals $n \sin \alpha$, correct? Now, by using this formula, we can connect the resolving power with the numerical aperture, okay? So, θ equals x / d , which is by the geometry, you can also call it as D / d ,

for small angles, okay? So, as the $\sin \theta$ is equals to d / d . Now, for any microscope, okay? So, here these are the two. So, this is your x is the resolving power and this is your microscope objective. and this is your θ , which is an angular aperture, and this is α , is the half angle of that angular aperture.

Now, this is your object. The distance of the is d , and the small d is the distance of the object from the lens. So, from this geometry, what we get a formula is x is equals to $1.22 \lambda d / d$. This is your θ . So, now by using these two geometries, you can see

that the theta is equal to x by d which is equals to if you compare these two geometries, it is equals to d by d .

We know that then $\sin \theta$ by 2, θ by 2 we know that θ by 2 is α is equals to if you assume that for small approximation $\sin \theta$ is equals to θ equals to d by d . So, this can be written as θ by 2 which is equals to x by $2d$ given by d by $2d$. Hence, $\sin \alpha$ can be given by d divided by $2d$. So, here $2 \sin \alpha$ equals d divided by d . Now, we know that NA is given by $N \sin \alpha$. And we have x equals 1.22λ divided by d . So, we can write x as 1.22λ divided by $2 \sin \alpha$.

And with this, if you substitute NA into this formula, then you will get x equal to 0.61λ divided by NA multiplied by N . Here, NA is the numerical aperture. And x is the resolving power of the microscope. Okay? So, it means that the larger the NA, x will be smaller.

So, it means that the resolving power will be higher. Here, λ is the wavelength, and n or μ (you can call this particular n) is the refractive index. So, you can see that by decreasing λ , your best resolution is achieved when you decrease λ and increase the refractive index or α . Okay?

Best resolution means x should be smaller. Okay? So, for your light, your λ is: green light is around 400 nanometers. If you do the μ in oil immersion, μ will be higher, and $\sin \alpha$ should be towards 1. It means α should be higher.

Okay? So, then you will get the maximum best resolution based on the λ , NA, and the refractive index μ . Okay? So, I hope that now you have understood the basic concept related to resolution. Okay.

And this particular concept is the same for all microscopes, whether it is an optical microscope or an electron microscope, okay. So, with this, I will end this class now, and we will talk more about the depth of field and depth of focus and how important those terms are for imaging purposes, okay. So, we will meet in the next class. Thank you.