Techniques of Material Characterization Prof. Shibayan Roy Material Science Center Indian Institute of Technology-Kharagpur

Module 01: Introduction to Microscopy and Optical Microscopy Lecture-01 Introduction to the Course and Basic principles of Image Formation

Welcome all, and today, I am starting an NPTEL online certification course and, the course title is techniques of materials characterization. My name is Professor Shibayan Roy; I am from material science center at IIT-Kharagpur. And today's module, the first module is introduction and, here we will be discussing about the basic general introduction to the course and certain basic principles of image formation that is what will be our course today.

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And we will be trying to cover certain concepts like, the general introduction to the instructor. First you should know me and what you can expect from this course. And then, we will discuss about the content of the course some suggested readings and the course will majorly covers the examples of microscopy. So, we will be seeing some microscopy images and we will discuss about them.

And then, we will go to a slightly different topic that is general description about microstructure because microscopy and microstructure these are very intently related to each other, so we will

see that. And finally, we will discuss about basic principles of image formation, this is what is the concepts we will be covering in today's course.

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So, a very brief introduction about myself. So, I did, as I said, I am an assistant professor in material science center at IIT-Kharagpur, I joined here in November 2015. And I did PhD from in materials engineering from IISc-Bangalore. Before that, and masters in materials and metallurgical engineering from IIT-Kanpur and B. Tech in ceramic technology from college of ceramic technology and the Calcutta university.

Broadly my research areas are mechanical properties of materials meso and micro-scale mechanical characterization, crystallographic texture and electron backscatter diffraction and additive and laser based manufacturing. Also, I have a keen interest on glass and glass ceramic materials. Here my homepage link is given if you want to know more about me, please visit here.

And many of the reading materials many of the slides many, many of the contents of this course are given here in this, so I request all of you to please go through this. If you have any further question you can always reach to me in this my email id here which is given here.

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So, first of all what it covers, what we can expect from this course. So, of course since as I said that will be mainly dealing with microscopy techniques. So, first thing you can expect is some basic principles of image formation which is what possibly we will discuss in this class and maybe in the next few classes. Some basic principles of image formation, basic attributes of any microscopy and so on, very generalized concepts.

Then we will move to specialized techniques, we will be first dealing with optical microscopes. There we will deal with image formation, we will deal with focusing, various modes like bright field dark field, polarized light microscopy, interference, contrast, transmission, fluorescent all of this different type of microscopes will be discussing in particular. And we will cover scanning electron microscopy.

They are of course will cover electron material interaction, image formation, various aberrations, astigmatism, various modes so on and so forth. If time permits, we will also try to discuss about little bit in compositional analysis EDS and WDS, crystallographic techniques like EBSD electron channeling contrast so on so forth. We will discuss of course transmission electron microscopy.

There also we will discuss about electron materials interaction, image formation, various modes bright field, dark field, high angle annular dark field so on. And then we will move to completely

a different characterization mode altogether and that will be diffraction based mode. And there we will first start with basic theories of diffraction. We will discuss about reciprocal lattice, the Ewald sphere.

Then we will move to electron diffraction and selected area diffraction patterns, little bit about use of TEM from all this perspective diffraction perspective. And we will move to final part, we will cover X-ray diffraction. And on the X-ray diffraction we will discuss about X-ray generation, X-ray scattering, X-ray intensity, X-ray diffraction pattern analysis and so on and so forth.

So, all of these various characterization techniques, they are used, as you can see here mostly there you can differentiate the primary differentiation that you can make from this is by the 2 factors. Number one that feature size that the probe and second is their probe size, which is again somehow related to the resolution that they can offer. We will discuss what is resolution and how you can determine and what makes resolution good or bad and so on in next few classes.

But for now, you can see that if you look at in the microscopy just simple microscopy and the three techniques that we are going to discuss here optical microscopy, scanning electron microscopy and transmission electron microscopy. These three techniques they are different in terms of the features that they are going to reveal or going to help you to identify. Optical microscopes, of course deals with coarser features, which are often in millimeter range, but yes, with some specialized techniques.

You can go down in feature size and you can reach all the way up to possibly micron level. And the probe size for this to get to obtain this you need to adjust couple of things you need to adjust probe size as well. So, you have to possibly go to a lower probe size when you are trying to get much finer details in the micron level. Possibly a better technique, if you are considering feature size in micron range is scanning electron microscopes.

And with suitable adjustment with this again maybe going through lower probe size you can go to something like even try and attempt to see features in the nano scale. Of course, the next technique we can think about is transmission electron microscope. Here, typically it is used for or features which are in the nanometer scales. And these days with modern microscopes with various fantastic advancements you can possibly even try to image in the atomic level.

This is roughly how the microscopy techniques are possibly different in themselves. Similar differences we can figure out between diffraction based techniques and so on. Here, what we try to show is again another feature or another distinguishing feature between different microscopy or different techniques altogether different characterization techniques altogether. That is the depth sensitivity, that means certain or certain characterization techniques are carry information only from the surface not from within the specimen.

But as many other techniques are available, which are possibly carrying information from deep within the specimen. So, certain characterizations are possibly giving you surface details more about the surface conditions, imaging whatever more information about the surface, whereas certain other are giving you information from the bulk of the specimens. Here also if you look at the microscopy techniques just now what we discussed.

If you look at something like, scanning electron microscope, here, versus the transmission electron microscope. Scanning electron microscopes, of course it is a very near surface technique, it gives you basically surface morphology of any material. Whereas, transmission electron microscopy will help you to get the details from deep within the specimen or it at a depth much more than the scanning electron microscope.

So, this way also you can possibly make a difference between different type of characterization techniques. And of course, all of this the purpose of showing you this is basically to tell you that which characterization technique to use and which characterization technique is suitable for a certain purpose. It entirely depends on what you want to get out of it, what feature you are going to look?

Whether you are looking to know or looking to get information from within the specimen or the surface itself is good enough for you to characterize. Based on that you can choose between

different type of characterization techniques even within one same type. Let us say just in imaging, if you consider depending on your need you can choose them. So, in this course, of course we will try to learn couple of basic characterization techniques.

But there are many, many variations available on these different techniques. And all of them will give you some different level of characterization, different type of information as per your needs. So, that is what is very important to understand. We will try to give you some overview and some basics about these characterization techniques. If you of course, want to know more about this characterization techniques or more about some variation of this characterization techniques you better should go through, all this suggested reading books.

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These books will help you to understand much better, I urge that you should go through them. If you want to know about microscopy in general, my suggestion will be to go through this book. This is a very simple one, and we will be following this book almost throughout this course, this is called electron microscopy and analysis and written by Goodhew and Humphrey, better to this as many versions editions, definitely better to use the latest one.

And this one is possibly the easiest one to follow, at least as per me, I have followed this and this is what I use for teaching this course similar course to my department as well. Of course, if you want to know more details about any of these techniques, please for light and optical microscopy

you should go through this book light and video microscopy. Scanning electron microscopy, again and compositional analysis techniques if you want to know please go through this book.

Scanning electron microscopy, and extra micro analysis by Goldstein, again, a fantastic book. Transmission electron microscopy, similarly there is a very nice textbook available, reference book available by Williams and Carter. And if you want to know about diffraction and if you want to know about X-ray diffraction in particular, there is no other better book available possibly according to me, in my opinion. Then this book called Cullity B.D Cullity, very basic book and very well written book published long back but still holds a position **ok**.

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So, let us start with microscopy. So, what is a microscope? Right, that is what we will start from the definition. A microscope, the book says, that Goodhew and Humphrey says that it is a system that transforms an object into an image **ok**. So, if you want to take if you see an three dimensional object, and if you want to make it permanent an impression and all, you take an image, which is generally two dimensional images, that is what microscopes.

But of course there are these days characterization techniques available which can give you three dimensional images as well. But mostly we will not go into that and we will stick with a general definition that. You basically take a three dimensional object, you see it, and you take an image,

you transform this to an image. If you go to Wikipedia, Wiki will tell you that microscope is an instrument which is used to see objects that are too small for the naked eye.

This is more like an very applied kind of a definition. And very much, I would say easier to understand, that is how we generally see a microscope. It is an instrument and it helps us to check out some things which are not really visible to us using our naked eye. And this is brings a different between these 2 definitions. If you go for this kind of a definition, the first one is system that transforms an object into an image.

Basically, this covers anything, any optical system and the best possible example that I can give you is our own human eye. So, our human eye is nothing but a system which transforms an object into an image in our eye. Of course this is the most complex one that has possibly ever been built, I do not know by whom. But yes, this is the most complex system, most complex optical based system that has ever been built.

And many of the concepts of microscopes that we will be discussing is basically builds around this the way our human eye works. So, we will have a very detailed discussion about the human eye before we begin optical microscope that time we will discuss more about this. But just understand that as per the definition goes this also can be classified as something like a microscope, principles are quite similar.

But of course if you go by the second one, you need an instrument. So, our human eye are sort of not falls in this. But again, if you see this very, very simple object, a magnifying glass that possibly all of you or all of us have played when we were kids, or many people are still using this, what it does? It just magnify sort of certain features or certain objects which are not very easily visible to our human eye, we just make it bigger for our ease.

So, even a magnifying glass can be considered a microscope from this definition, what is the taxonomy? That is the name meaning of the name, the word microscope came from 2 words, in Greek words micros which means small and skopein which means to look or to see. This tells us

actually, that any microscope will need three things three basic attributes, one is the source of illumination or signal.

So, certain things certain source needed, then source of a detection signal. So, this illumination signal will hit the object or maybe the object itself will produce something like an illumination. So, either way you need a detection signal, and then there will be some way of detection, that means the detection signal will be you have to work with it, you have to detect with it and form an image out of it.

This is what I am seeing in perspective of microscope, but this is very general description of any kind of characterization. You need a source of signal, you need a detection signal that means when the source signal interacts with the specimen with your object, it must detect, it must generate some kind of change in the signal or it must generate some signal on it is own. And finally, your job is just to detect the detection signal and get an idea about the object, go back and get an idea about the object.

That is how every characterization technique works; microscope is no exception to this general rule. So, if you look at now the microscopes, various types of microscopes from these three inherent attributes. So, based on the source of signal, we have difference between optical microscopes and electron microscopes. And if we use sometimes there are other kinds of sources like an ion.

If we use ion, that is an ion microscope, but generally we will restrict ourselves within the discretion of an optical and an electron microscope here. Based on the source of detection signal, now you have used the same source of illumination signal electrons, just the detection signal is different, you have scanning or transmission mode, versus EDS mode, energy dispersive spectroscopy which detects characteristic X-ray, we will learn about that.

If you just understand the way of detection, how the detection is working, there you can possibly make a difference between something like transmission electron microscope and AFM. These two are different source of illumination, source of signal is different, way of detection, source of

detection signal is different and way of detection is different also. But all of them even AFM atomic force microscope is called a microscope because it gives you an image which is not visible to you through your naked eye.

So, it is still a microscope in that sense, but it differs completely from a general microscope like TEM or optical microscope. Now if you look at the very basics of image forming principle, how the image is forming? What you will wonder to know that most complex of the microscopy techniques transmission electron microscope is very similar to the most simple of all the characterization techniques or all the microscopy techniques, optical microscope.

They work perfectly in the same kind of principle of image formation. Of course instrumentation is different between one because one uses light, source as an source of illumination, one uses electron as a source of illumination. So, instrumentation is very different but the image formation is very, very simple.



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So, now let us look at some of these images. Most of these images are from various periods of my research career. And I generally show these images before I begin my course to my students, every year I do that. And I ask them, what do you think about these images which characterization technique is used or what type of microscopy is used to get this kind of techniques, this kind of image?

So, every year I usually get students from various different departments like I have people coming with something like ceramic engineering undergrads, people coming at metallurgy department, people coming with mechanical engineering, civil engineering. Also one side I get people from chemistry, physics major and so on and so forth. Many of them have previous experience capturing images dealing with microscopes for the research also.

Some of them they could identify of course they could say that this is an optical image; this is an possibly scanning electron microscopy image. These 2 more or less they could identify, then when I ask them what about this, some people are smart enough they could identify that this is possibly an transmission electron microscope. So, when I asked them, what is the basis, why do you think this is optical one, this is an scanning electron microscopy image and this is an transmission electron microscopy image?

What happens is that at least 50% of them are confused, even it happens that many of them changes their answer, many of them says sir, this looks like maybe this is an optical, maybe this is not an optical, maybe this is scanning electron microscope and so on. Some people still stays and the kind of things they say for example in TEM they say sir, look at this the scale bar which is there tell you what is the size of this features basically.

They says look at this, it is 2 nanometer and just now you told that transmission electron microscopes are good to give you features which are within nanometer size, fine, I agree with them. And then I said ok, look at this, so what do you think about this? They says sir, this is looks like an SEM microscope, I said why? They said sir, looks like very, very prominent features and sometimes very smart guy says that you could see like a depth perception.

You could see free kind of little bit a 3D kind of image here, I accept that, and I said why? Then what is the difference? They said sir, this looks like optical because here you are missing that 3D, you do not see that 3D kind of effect so easily plus many times people say that sir, this is a coloured image so that is why. But what we will learn next is that colour is like it just happens in

optical microscope, you could make a colour image even, this you can make a grayscale image or this you can make a coloured image artificially.

It is not nothing that but on an average yes, I accept sometimes their answer, because in optical images the colour comes naturally. Whereas this electron microscopy images, the colour is not so natural all the time, so I sometimes accept their answer. Then I ask look at this and tell me what exactly it is? They get confused because the scale bar tells the features are in the micron size and also this does not look like 3D perspective.

So, then I have to answer them that this is a transmission electron microscope. And there are certain features that this reveals which are only possible in transmission electron microscope. These are basically some feature called dislocations, which you can only see under a transmission electron microscope under certain particular condition, we will discuss maybe. Then I asked what is this? Here also a little bit if you look at very carefully, there is a little bit depth perception is there, very little but it is there not as prominent as this.

But there is a little depth perception is there plus it is a coloured image. So, then I asked them, what do you think about this? Most of the cases, my experience is they are not able to tell me exactly what this image is. And this I tell them, when I discuss about this technique, you will understand, I will again come back to this image, this is a special type of optical microscopy, a special contrast technique is used called interference contrast, differential interference contrast, DIC technique.

So, from all of these things, the bottom line is what I try to tell them is do not just look at this images and appreciate. Try to see different features from which you can tell exactly how this image is taken. If you understand the principle of image formation, possibly you can go backwards; you can just see this image and tell that these are the features from which I can identify this to be an SEM image, this to be a TEM image, this to be a TEM image and this to our optical images.

That is what exactly what I will tell to everyone who are listening to this course that in this course in this microscopy part, our job will be to identify this images based on their formation principles.

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So, that is exactly what I try to say here again the difference between scanning electron microscope and transmission electron microscope. Scanning electron microscope as I said gives you a little bit of 3D perception. And that 3D perception some sense is very similar to what we see with our naked eye, something, we also see the 3D image. That comes because of some other effect will not go into this.

Here basically scanning electron images gives us image some features or some morphology of the surface, which are very similar to what we regularly see with our own eyes. So, appearance wise, these images are very similar to the images that we see. Whereas, TEM transmission electron microscopes, images are slightly different, these are not something that we are used to see.

Basically this gives internal structure of materials, these are as I already discussed, here the information is carried from deep within the specimens. Both cases, several different type of images can be formed, because of different kind of formation mechanisms operating from them.

So, again as I said if you want me to understand this images, what they represent? The first thing we must understand is how these images are actually formed?

And this is very much true for transmission electron microscope because in transmission electron microscopes, I told you that this is an image which is representing dislocations. This I can tell because I know exactly how this image was captured, which condition? If I do not know this, the same kind of features or same kind of image can be produced just because of some other kind of mechanisms or even at times because of some artifacts in the characterization technique itself.

So, we need to know exactly how these images are formed in order to make out further information about these images.



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Now the point is what we are going to see out of these images? This part at least is not directly relevant or directly related to the characterization techniques. But this is about knowing what we are going to see? What is the thing that will or what exactly we are characterizing? Most often what we characterize via microscopes, of course we do not use the magnifying glass definitely, sometimes we do.

But what we use these microscopy techniques for is to characterize the microstructure of any material. This term may familiar seems familiar, if not please go through books and then you can

figure out that exactly what is this. So, microstructures has again, the definition says it is a very small scale structure of any material. And usually that kind of a structure is again not visible to our human eye and we need certain amount of magnification.

We need to zoom out what is magnification and all? We will go through; we need to zoom that is possibly more familiar word for many of you. So, we need to zoom in into that material or that structure to see it. So, it is very simple example is given from this reference here. That if you look at this, this is an engine block this is basically goes in an automobile engine. So, this is what you see in a macro scale that is what we call it.

The size range here is around millimeter, the size of this is within a millimeter, so all the small holes and everything you can very well see it with your naked eye, there is not no problem, no difficulty at all, fine. If you look out small portion of it, possibly you can still see certain few other features which are within millimeter range many a times. You can possibly see, you can try very hard and if you are having a good eyesight, possibly you can see this kind of a feature which are within millimeter range.

But better is if you try to use possibly a magnifying glass even better you use an optical microscope to see these features. Of course, if you want to now look what is inside this small different coloured features, you need a microscope most often you need optical microscope for this. And here you are seeing features which are within microns 10 to 500 microns features, you are seeing it and you need a microscope for this, to see this kind of features in this.

Now I want to look further deep into this and what I am seeing is some other kind of features, which are within nanometer scale. Of course as I said you possibly need an electron microscope, most often you need a transmission electron microscope. And if you want to look what is deep inside these materials or these features, I can reveal them and I can see some smaller features which are in the scale of 0.1 to 1 nanometer.

Almost in the atomic scale, definitely you need your transmission electron microscope to see them. The point is, this different scale structure is available, all different scales I have certain kinds of structures, distinguishing structure in this material. So, if I want to be happy, stay happy, I do not want to see all of any of this, I do not need a microscope. I can just simply stay with this, job is done.

But if I want to look at this kind of a structures, I need to see a microstructure under a microstructure; I need to have a microscope for this. Question is why do I need to see this? What is so special about them? That is because these structures at different scales, so these micro structures are different length scales; they control properties all the way up to here. So, the property of this component in the macro scale is actually governed by features which may be as small as this within the atomic scale that is why. That is why I need to know this micro structures and I need to have a microscope for doing this.

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This is very nicely shown with this again my own research, through that it is shown this relationship what we call a micro structure property relationship. Here this is something measurement of strength of a material. So, you see we have measured 2 different metals basically alloys, two different alloys aluminum based alloys. We have two different alloys and out of these two different alloys; the blue one is showing high strength at room temperature, very high strength at room temperature.

The green one does not show that much of a strength at room temperature, when we go to high temperature the story is completely reversed. Now, we see the green one shows much better strength compared to the blue one, why? Here what happens is the strength comes from the features which are in nanometer scale basically. So, the bulk strength of this material which is in the macro scale is controlled by these tiny features in the nanoscale that is why.

What happens here? Basically that this material if we take this green one which is a wonder material does not show much strength at room temperature but holds that string up to high temperature. What happens for them is? This material has these tiny features, small features which stay within that length scale, if you take it to high temperature. Whereas the other one, the green one, they have this small features at room temperature but those small features become much coarser much bigger.

So, they are not able to give you strength at high temperature. So, if I want to know this entire micro structure property relationship, I have to bring something in between which is the characterization.



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And that is exactly is shown somewhere over here I think is something called materials tetrahedron. That you have a material and characterization lies somewhere within the middle, we will come to that. But before that, let us discuss about couple of small other issues about microstructure and their relationship with the characterization techniques. There is something called representative volume element.

Just now I told you that ok, the properties are controlled by in this length scale, the properties are getting controlled fine, so I characterize this. Now what is the guarantee that this structure is there throughout the material? Other words, how much area of the material should I characterize

in order to know that what kind of a property I can expect? This is called a representative volume element problem.

That means, the microstructure which is characterized in the smaller length scale, whereas the properties which are measured at a larger length scale. How to do this something called length bridging or scale bridging? The properties are depending on the microstructure but the microstructure is characterized at a much smaller length scale than the properties. So, you have to make sure that the microscopy or microstructure that you are measuring through this microscopy really represent the entire material.

So, that is another very important point in this entire business of microstructure property relationship in the materials. And one simple example is given here that if you have this kind of a microstructure, and if you go and look some regions, you can possibly have this kind of a hexagonal arrangement. Whereas some regions you can possibly have a square kind of arrangement, and both of these can give you completely different properties.

So, if you characterize certain regions and find out this kind of arrangement, you cannot simply say that entire material is having this kind of arrangement. If you do that, possibly this correlation between microstructure property will be wrong. So, for that possibly you have to go and check some other parts and make sure that all other parts you have this, not this. If you have some regions, which is having this possibly have to do your characterization at a much larger area.

That is what is called representative volume element. So, when you do characterization of microstructure, this one assumption is inherent that your characterization really represent the area that you are choosing the kind of characterization technique you choosing is really representing your microstructure. Because ultimately that microstructure is related to the properties, so this is another very important consideration when you choose your characterization technique.

So, we can go to another important aspect about microstructure. That is the microstructure is generally an image as you can see. For example when all this micro structure that I have shown you, you are seeing primarily that is an image exactly an image. The problem with an image is that it is kind of a qualitative information and image is a qualitative, all it can say you is how does this feature looks like, whether this feature is spherical, whether this feature is rectangular, whether this feature has a square shape what exactly it is.

So, it is very much a qualitative, it will not be able to give you quantitative information. And when you ask for a quantitative information you need to know this scale bar. For example, here in this image, you have this scale bar basically the scale bar tells you the features, what is their size. So, you are transforming one stage you are going one stage ahead and you are sort of transforming this image which is a qualitative information to a quantitative information but that is very general concept.

Problem is, what is the problem in this? Why we are not so happy with qualitative information? That is because most of the properties that we have is a quantitative information, properties like I told you strength. So, strength has a value, what value it is? If I go for some other kinds of properties, I will get other kinds of value. So, these properties are always more or less always, not always I should say.

So, most often properties are quantitative information. So, if you are trying to link the microstructure to the property that is what is a problem. One is giving you mostly a qualitative information, whereas the other one is giving you a quantitative information. So, this link is not so easy to happen. That is why if you can convert this micro structure and that information to quantitative information, it will be very easy to predict the properties or it this length bridging scale bridging will be much easier.

So, this is what the recent trends of characterization techniques that you can go for something known as quantitative microstructure. So, here it is much more clearer, microstructure if you make it quantitative, processing by which you get this microstructure that also is a quantitative.

Mostly you know exactly what temperature? What strain rate and other things that you use, what kind of a deposition rate we use and so on. So, it is also a quantitative by itself.

Property of course is a quantitative, performance the final performance of the material again a quantitative property, except all of this only the microstructure usually used to be qualitative information. But modern trend is you go for characterization techniques, which gives you more than only qualitative information, some quantitative information. So, this is an example of one such characterization, which we if time permits, we will try to discuss very quickly called electron backscatter diffraction.

That gives you a quantitative information of the microstructure. And then you can use it for predicting your properties very easily and do this length scale bridging.

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So, with this, we will be moving to generalized concept of imaging. So, please keep this information about microstructure with you. In future we will be possibly needing it, and we will be recalling this microstructure description about microstructure time and again. But let us discuss now with generalized concept of imaging. The first thing to understand is that the concept that way images are form.

These are basically very much general for any kind of microscopy that you use, whether you are using a light microscope or whether you are using an electron microscope. The way images are formed is quite, similar, this I think, I have already told you. So, there are three different ways that an image can be generated. So microscopy, the purpose is to generate an image and there are only three ways that you can generate an image.

The first one is called projection image, which is the most common example and the most commonest example we will go to that is basically shadow formation. If you take your shadow you have something like an source, you are the object and you are seeing your shadow on the ground which works like an imaging plane. So, this is called a projection image or at times it is also called parallel images.

Then of course, you can form you can do the same kind of things with the lens system, you can use some kind of lens best example is something like optical images or optical systems. As I have already said our human eye is the best example of this optical system lens based system. At times both of these projection image and lens based images most often they are parallel image, direct images, they are formed all parts of it are generally forms simultaneously, both the projection image and the optical images.

There is another way of forming any image that is called a scanning image. And in scanning image what happens is different points are presented serial in that images, what we generally know as digital image in terms of the digital image, what we know as something like pixels. So, pixel by pixel generation of an image that is another way of forming any kind of images. Best known example is possibly any kind of television picture images or any digital display images.

They are basically formed pixel by pixel but at such a fast rate that we cannot distinguish them this image formation mechanism pixel by pixel we cannot distinguish them with our human eye. (**Refer Slide Time: 43:28**)

So, now we will be discussing about ways of image formation and this we will be doing it in the next lecture. And there we will be discussing how many different ways are there to form images. And we will stop here for today and we will do it for the next time, Thank you.