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Lecture-13
Materials Characterization
Fundamentals of Scanning Electron Microscopy

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Hello everyone. Welcome to this material characterization course. In the last two classes we reviewed about the electromagnetic lenses and its function, fabrication and some of the parameters which controls the electromagnetic lenses, how it is being used in the electron microscope. A kind of introduction with little more details we have gone through. Now from this class onwards, we will just start the scanning electron microscopy where all this electromagnetic lenses we have seen will be used.

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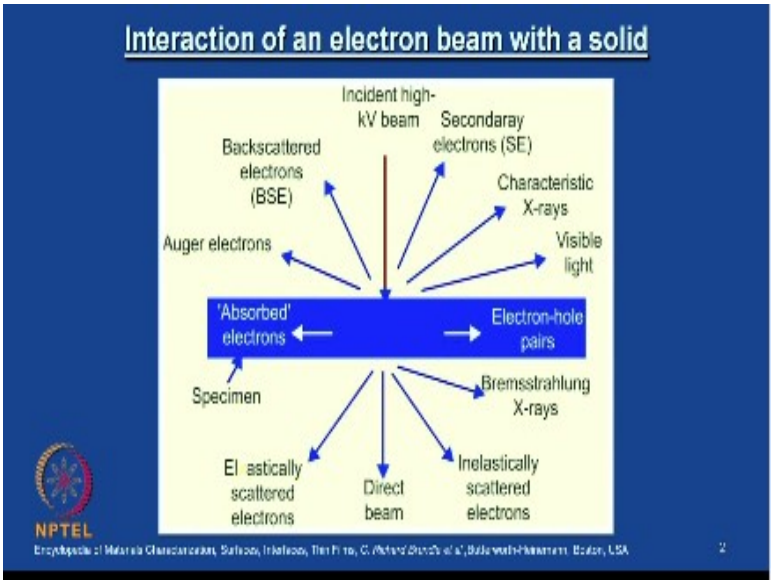

So, before I just start these lectures on fundamentals of scanning electron microscopy. I would like you to carefully go through what is shown on this slide. So, before we get into any of this electro electron optics or electron optics-based instrumentation which issued for imaging of materials to reveal the microstructure details, first we should know about the interaction of an electron beam with a solid. it is a very general information which one should remember.

I will tell you the importance of this the moment I finish this discussion here. So, look at this schematic. What is shown in this slide is, you have a specimen and then this is an incident high-energy electron beam which is falling on this sample and then you get to see quite a bit of signals are which is coming out of this sample in all the directions. So, I would like, like you to carefully look at each one of them so what we are seeing is within this volume of the sample what we are is an absorbed electrons.

Some that means some electrons are being absorbed by the specimen and some of them actually you get electron hole pairs generation and then you see a secondary electrons, characteristic x-rays, visible light and then you have backscattered electrons and then you have elastically scattered electrons and then you have a direct beam and you have inelastically scattered electron and then you have bremsstrahlung x-rays. So, by looking at this you just see that when a high-energy electron beam interact for the specimen, it is always true that all these signals are generated.

It is this, the detecting system which you employ to collect them and use them for imaging or analysis that characterizes the particular characterization equipment. For example, you just see that the visible light we used so far an optical microscope, a characteristic x-rays can be used for n number of spectroscopic techniques to analyze the chemical details or chemistry of the specimen in a very, very high resolution of in the materials which are I mean which may contain very minute or trace elements. So, in order to characterize them we may use this characteristic x-rays. We will look at that all the spectroscopic technique in a different lecture series but you just see here this is also one of the important signals which you get out of the electron beam specimen interaction.

And then this backscattered electrons and is secondary electrons are being used in SEM and then you see that auger electrons are used in auger electron spectroscopy and then a direct beam which comes from the specimen is used in transmission electron microscopy and you have all this other signal also be used in the transmitted electron microscopy for different applications. We will look at that in an appropriate time

So, I just want you to look at all these signals which is coming out of the specimen. These can be broadly categorized into two segments. One is a forward scattering signals. All these are just direct being inelastically scattered electron, elastically scattered electrons. These are all forward scattering signals and then you have a backward scattering signals. So, out of these two categories the, the scanning electron microscopy uses only the, the backward scattering signals. So, this is primary important information one should have before we get into the details.

So, all the other signals are not used in the scanning electron microscopy. We will see the details one by one but as an introduction you should know, in general when an electro I mean high, high energy electron beam interacts with the specimen all these signals are coming out and then the kind of detecting system which we use actually defines the characterization tool, whether it is a scanning electron microscopy or a transmission electron microscopy or any spectroscopy specific spectroscopy which where we look at the chemistry of the specimen.

So, this is primary important concept you have to understand before we get into the specific characterization tool. So, with this introduction I would like to start the scanning electron microscopy and let me just go to the blackboard and then right few things and an introductory remark.

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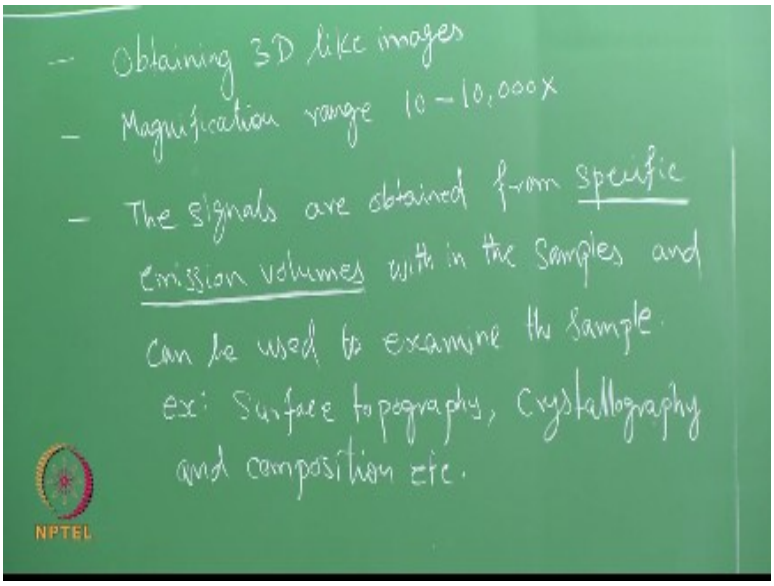

So, in an introduction to the scanning electron microscopy, we should know what are its unique capabilities. Why do we opt for in a scanning electron microscopy investigation in comparison to a light optical microscopy. The primary objective is to obtain the magnification with high resolution. So, in a very simple terms, you can you can obtain microscopic details 3D like images. We will see how this effect comes and what are the parameters which contribute to this phenomenon or any effect I would say. Magnification range of 10x to 10,000 x and more in fact it could be more also. Typically the signals are obtained from the specific emission volumes within the samples and can be used to examine the sample in terms of surface topography, crystallography and composition, etc. So, these unique characteristics we could not do with the light optical microscope and what is surface topography, the surface unevenness. So, you can just imagine what we have seen in a light optical system. If you recall we just polished the metallic specimen with the different kinds of emery sheets. Right !

So, the final Emery sheets which had very fine ceramic particles embedded in that sheet and then we just rub the sample against them and then that sample appeared almost like shiny and so on. With our naked eye the sample look very polished and so on. Then we what did it we also put

that sample under the optical microscope, then we could observe the very, very closely spaced scratches.

I would say this closely spaced and impression which was observed, like if you put the same sample under the SEM, you will see that there are hills and valleys because we are looking at at the very high magnification and rather a high resolution we are looking at it we are able to observe the small hills and valleys that is surface topology. So, this is one classical example you can just go and look back. This can be so, any surface unevenness to the very micron to nanometer scale can be analyzed.

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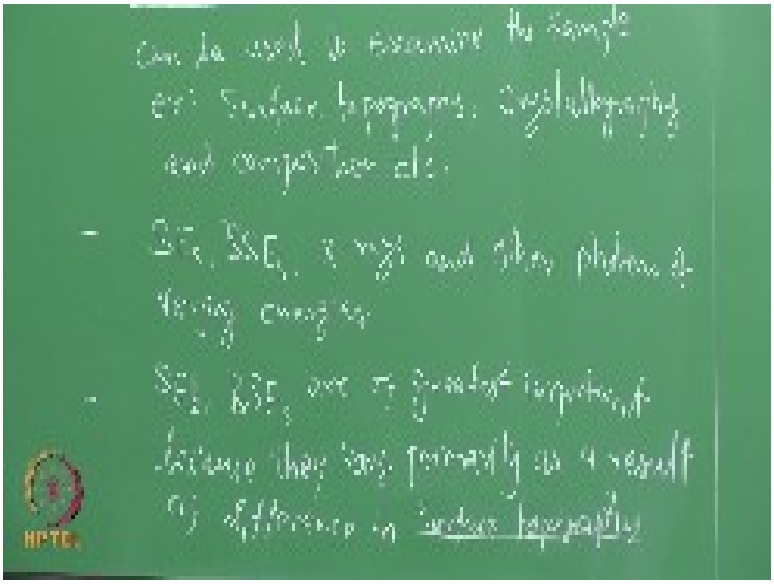

And also the crystallography of the specimen and that under its chemistry can be analyzed with the scanning electron microscope primarily. So, what what, what are the parameters which enables this microscopy to do that. We will see that. So, what are the typical signals we are going to get from the SEM, secondary electrons, backscattered electrons, x-rays and other photons of varying energies.

Just you look at that slide again what I have just shown here. This is second electrons, backscattered electrons, characteristic X-rays and other photons of varying energies. See, each radiation will have very specifi energies which we will talk about. So, the primary signals which is coming out of this SEM as I said it is a backscattered signal or back scattering signal sorry. I would say back towards scattering signals. It will be very clear because there is another particular signal is named as backscattered electrons. So you should not confuse with this because this is only coming the backward you know scattering. This is what I meant all these signals are backward scattering signals which are being primarily used in may SEM. So, only these three signals are primarily used in an SEM of course they, they are characterized based on their energy that we will see in an appropriate time.

And out of all this backward scattering signals, only we talk about second electrons and backscattered electrons, why ? Why are we talking only about this, because they vary primarily as the result of difference in the surface topography. The amount of secondary and backscattered electrons which is coming out of the specimen surface is primarily depending on the surface topology.

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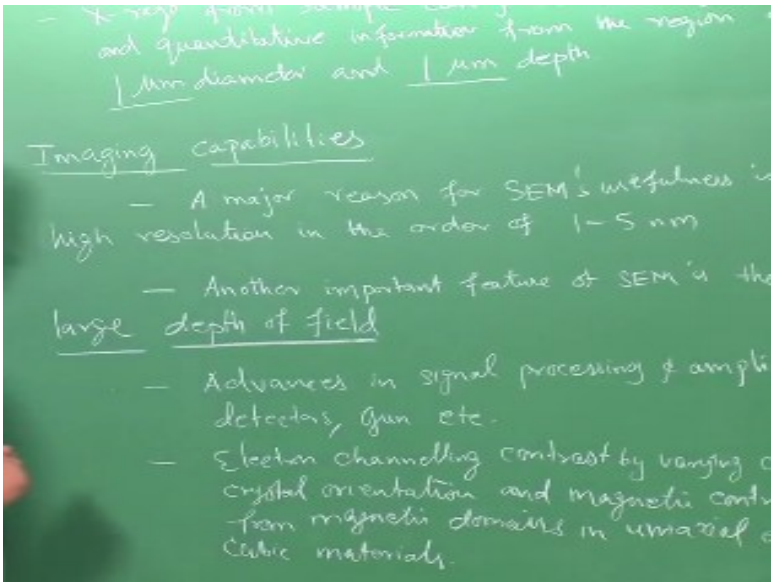

This is the, the core idea behind using this tool and what are the other important things ? So, the other important information you get from the a SEM is, the x-rays that is characteristic x-rays come from the sample can yield both qualitative and quantitative information from the region of 1 micrometer diameter and 1 micrometer depth. This is a rough indication. You get what is the region size from which you get this information which is of the order of 1 micrometer diameter and 1 micrometer depth from the surface.

So, these are the information you get from this in general from SEM and we look at the what are the imaging capabilities. So, if you look at the imaging capabilities of this microscope, the major reason for the SEM usefulness is the high resolution in the order of 1to 5 nanometers and another important feature of SEM is the large depth of field. We have already discussed in the fundamentals of the optics. We have seen what is depth of field, how it is being exploited in electron microscope.

In fact the what we have just stated in the beginning 3d like images it is partly because of this effect. You have high or very large depth of field in an SEM. We will also see it using a ray diagram how it enables this effect when we discuss the other functions of SEMs in the coming classes and the SEMs are becoming very popular because of the advances in the signal processing and amplification like the kind of signals you receive secondary electrons, backscattered electrons or x-rays and then you have advanced processing signal processing and amplification detectors and then gun design etc.

So, without all this advances this SEMs and also do imaging something like using electron channeling contrast by varying the crystal orientation and also magnetic contrast from the magnetic domains in the uniaxial and the cubic materials. So, these are all some of the, the highlights of the imaging capabilities of the SEM.

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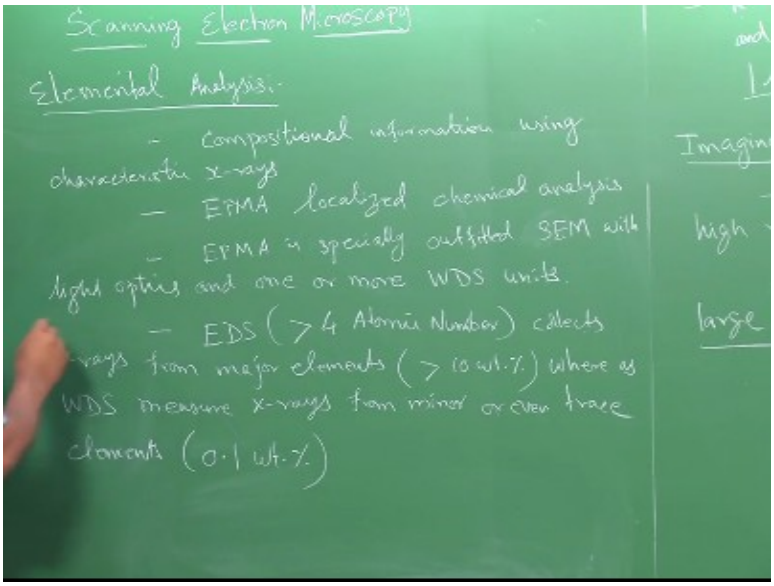

You will see that next is structural analysis. Okay. If you look at the what are the structural analysis one can do with this SEM, it has got a capability to determine the crystal structure and grain orientation of the crystals on the surface. Please understand you have to remember that it is all whatever the information you obtain is only from the surface with very limited volume you will just understand that in much more detail as we go into this lectures and then the diffraction of the backscattered electrons emerging from the sample surface. Electron backscatter diffraction (EBSD) with the low intensity also enables this capability and since it is a low intensity we have very high sensitive CCD camera recording. This is charge coupled device camera records the backscattered a so called Kikuchi pattern which is nothing but this signal and it is analysed with the computer-based indexing method.

And then you have today SEMs with advanced indexing and computer-assisted crystal lattice orientation mapping, that is called EBSD maps, which allow this technique to identify the face and the Miss orientation across the boundaries. So, this is also very powerful technique today and, and it has been applied everywhere. This itself separate is a research domain. People can extensively use this and very powerful technique as far as SEM structural analysis concern. And what else we can do with the SEM so, so far we have seen imaging capabilities, structural

analysis and finally elemental analysis. So, if you look at the elemental analysis capability of an SEM, you can get the complete compositional information using characteristic x-rays. The tool generally referred as Electron Probe Micro Analysis (EPMA) which can get the chemical composition from the very localized region and then provide a complete chemical analysis and then you have this EPMA is specially outfitted SEM with light optics and one or more or WDS units (Wavelength Dispersive Spectrometer). We will see all this variance of the spectrometers as I mentioned which uses the characteristic x-rays which comes out of the sample and then do the chemical analysis. We will look at them in a separate lecture series but then these are all the attachment one of the primary attachment to the SEM.

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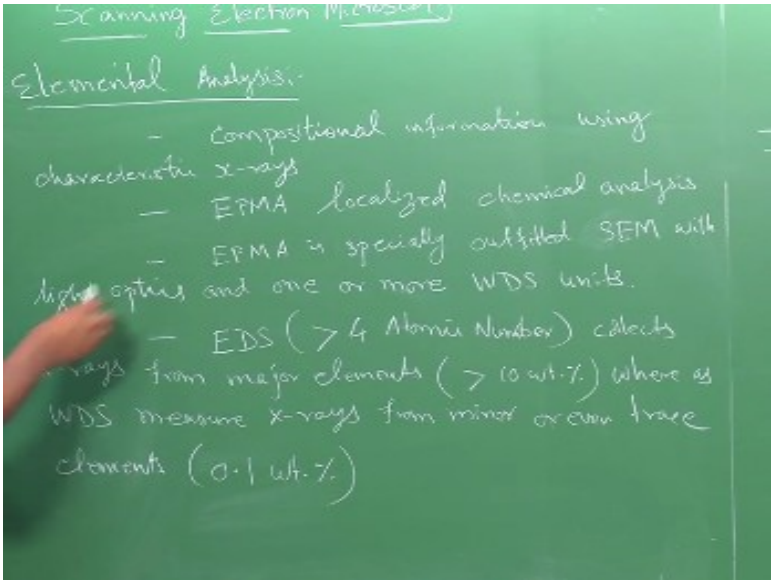

This one, the another one is energy dispersive spectrometer, which can detect the elements greater than four atomic number, collect the characteristic x-rays from the major elements approximately you should have about 10 weight percent whereas the WDS measures x-rays from the minor or even a trace elements of point one eight percent. So, WDS is much more powerful compared to EDS. We will see why and so all these details say later.

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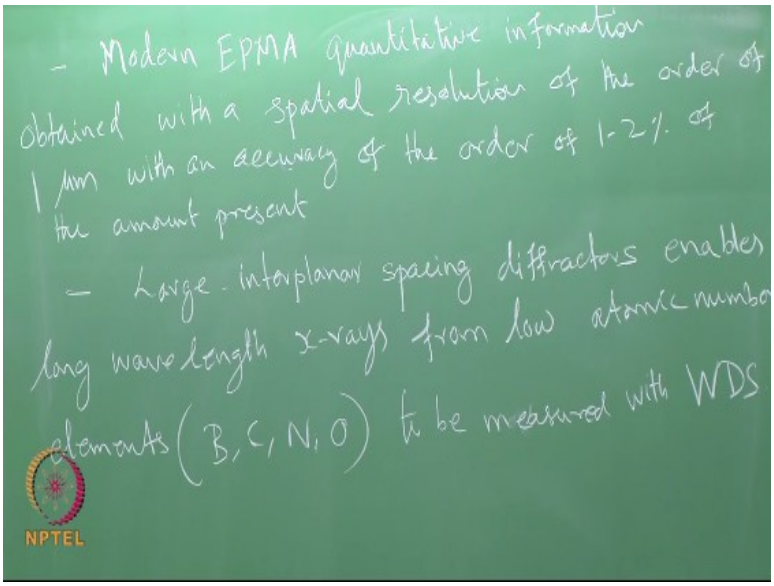

But these are all the basic details one should have about the when you look at the capabilities of a scanning electron microscope. I have few more points to add in this segment. So, the, the final point to the elemental analysis with a modern EPMA, you can get a quantitative information from your specimen within a spatial resolution of the order of 1 micrometer with the accuracy of the order of 1-2% the amount present and also this EPMA has a capability of analyzing the very low atomic number elements like boron, carbon, oxygen.

Because the WDS spectrometer uses a large interplanar spacing diffractors typically organic crystals which has got large interplanar spacing, which enables long wave length x-rays from the low atomic number elements. Since this low atomic number elements as the characteristic x-rays of large wavelength or a long wave length. So, these crystals enables the diffraction possible and then and they can be measured with the WDS. So, these are all the, the basic capabilities of a scanning electron microscope. So, I have just put them into three categories, one is imaging capabilities and structural analysis and then composition analysis or elemental analysis and SEMs are primarily used for only this purpose. Now we will look at the some of the other introductory remarks.

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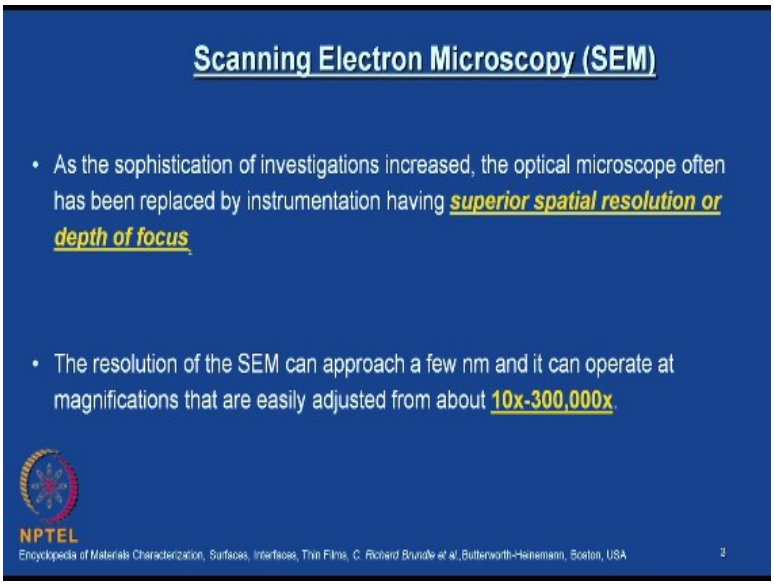

As the sophistication of the investigations increased the optical microscope often has been depressed by instrumentation having superior spatial resolution or depth of focus so the resolution of SEM can approach a few nanometer as I mentioned and it can operate at magnification that are easily adjust from about 10x to 300,000 X of course this can be a subject of instrumentation.

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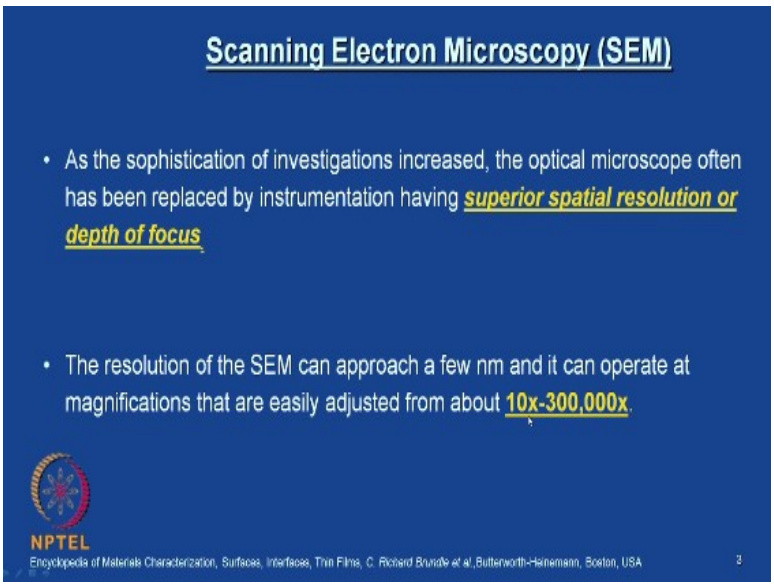

We will talk about it in an appropriate time. The depth from which all this information comes varies from nanometers to micrometers is also a subject to specific instrumentation details we will look at them in appropriate time. Likewise the lateral resolution in this analytical modes also varies and is always poorer than the topological contrast mode. So, the principle image is produced in the SEM are of three types, namely secondary electron images, backscattered electron images and then you also have elemental x-ray maps, and these are the three primary images one can obtain in a normal SEM secondary and backscattered electrons are conventionally separated according to their energies.

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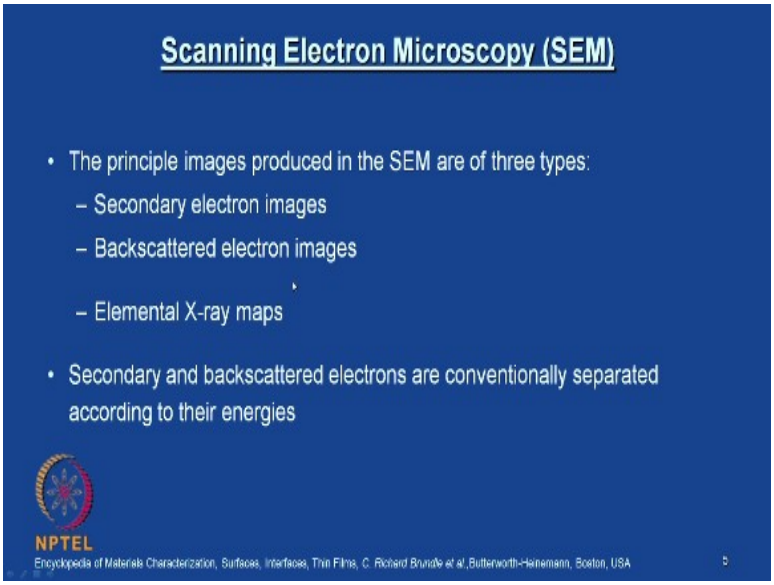

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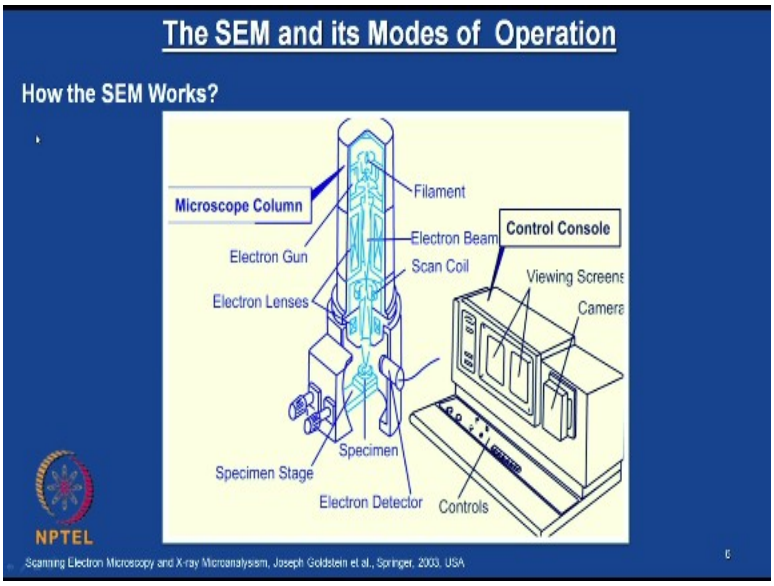

So we will now see how this will have some schematics to show how this SEM works. So, you have the two separate entities one is a microscope column, from the top to bottom, the other is a control console. So, you have the electron gun which comes from the top of the equipment column, microscopic column and then you have further all the electron lenses and as can coil and the beam reaches all the way up to this chamber specimen chamber which is maintained at the with the vacuum of 10 to the power minus 4 pascal, which is in the order of1 billionth of the atmospheric pressure for the for your reference. And you have on the right hand side you have the CRT screens screens and then camera where all this scanned images are being used. So, this is a primary classification of this equipment microscopic column and a control console and then you look at this next schematic and you have this a complete schematic of the cross section of the scanning electron microscope and what you see is an electron gun which generates the electrons and accelerates to typically from point 1 to 30 kilo electron volts and then this being passed through electron lenses also and also a scan coils. So, these electron lenses what they do is the, the probe diameter which is being produced by this the tungsten hairpin typically, it is not sharp enough to resolve the structures and this electron lenses dig magnified two very sharp spots or a sharp probe and then they are being rested on to this coil to the this specimen.

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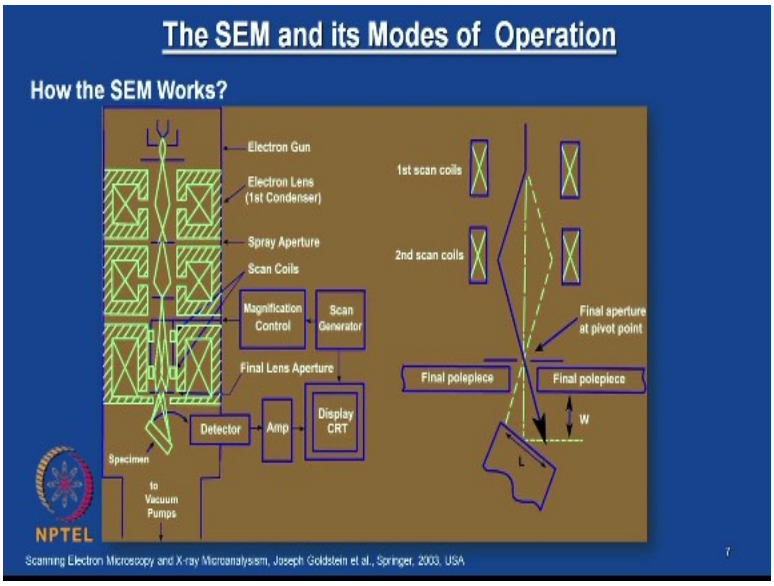

And then you have this detector the signals which comes out of this specimen which is kept under the vacuum and then it amplifies and it goes to the display. We will continue the discussion of this general function of this scanning electron microscopy in next class as well. Thank you.

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