Fundamentals of optical and scanning electron microscopy
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Module – 03
Unit-6 Introduction to scanning electron microscopy
Lecture – 13
Interaction between electrons and sample
Imaging capabilities
Structural analysis
Elemental analysis

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Module – 3
Unit-6 Introduction to scanning electron microscopy
Lecture - 13

- Interaction between electrons and sample
- Imaging capabilities
- Structural analysis
- Elemental analysis

Hello everyone, welcome to this material characterization course. In the last two classes, we reviewed about the electromagnetic lenses and it is 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. Before I just start this lecture on Fundamentals of Scanning Electron Microscopy, I would like you to carefully go through what is shown on the slide.

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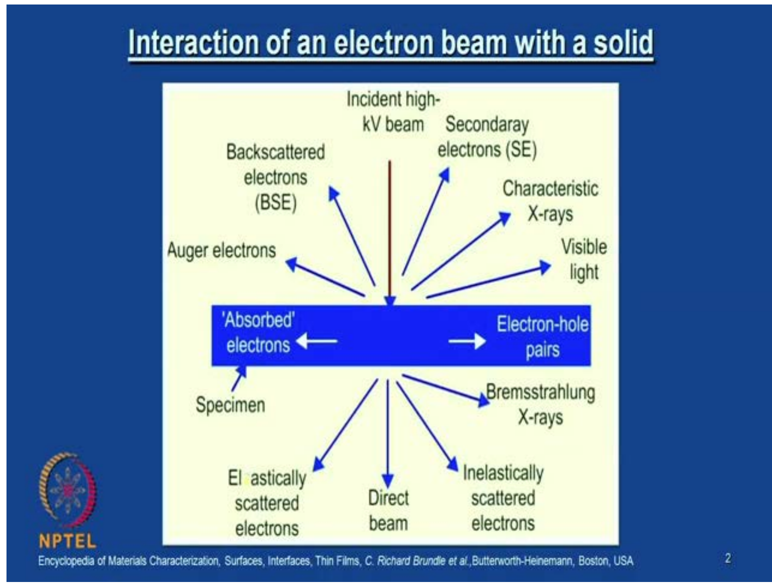

So, before we get into any of this electron optics or electron optics based instrumentation which is used for imaging of materials to reveal the micro structure details first we should know about the interaction of electron beam with the solid. It is a very gentle information which one should remember, I will tell you the importance of this the movement I finish this discussion here.

Look at this schematic, what is shown in the slide is you have a specimen and then this is an incident high energy electron beam which is falling on this sample. Then you get to see quite a bit of signals or which is coming out of this sample in all the directions. I would like you to carefully look at each one of them. So, what we are seeing is within this volume of the sample what we have written is an absorbed electrons, 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, then you have backscattered electrons, then you have elastically scattered electrons, then you have direct beam, and you have inelastically scattered electron, and then you have Bremsstrahlung x-rays.

By looking at this you just see that when high energy electron beam interacts with the specimen, it is always true that all this signals are generated. It is this the detecting

system which you employee 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 in optical microscope, a characteristic x rays can be used for n number of spectroscopic techniques to analyze the chemical details or chemistry of this specimen in a very, very high resolution in the materials which may contain very minute or trace elements.

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 this 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 the transmission electron microscopy. You have all this, other signal also being used in the transmitted electron microscopy for different applications. We will look at that in an appropriate time.

I just want you to look at all this signals which is coming out of the specimen. These can be broadly categorized into two segments; one is a forward scattering signals, all this are just direct beam, inelastically scattered electron, elastically scattered electrons, these are all forward scattering signals. Then you have backward scattering signals. Out of these two categories, the scanning electron microscopy uses only the backward scattering signals. 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 high energy electron beam interacts with the specimen all this 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 were 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 black board and then write few things on an introductory remark.

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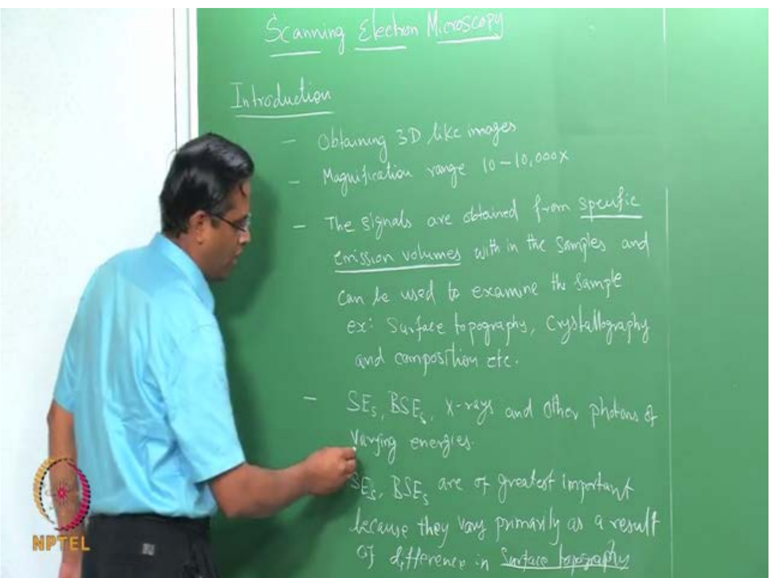

In an introduction to the Scanning Electron Microscopy, we should know what are its unique capabilities? Why do we opt for a scanning electron microscopy investigation in comparison to a light optical microscopy? The primary objective is to obtain the magnification with the high resolution. So, in very simple terms, 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 10 x to 10000 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, etcetera.

These unique characteristics be could not do with the light optical microscope. 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 polish the metallic specimen with the different kinds of emery sheets. 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 looked very polished and so on, then what we did it we also put that sample under the optical microscope then we could observe very closely spaced scratches. I would say this closely spaced an 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 had looking at the very high magnification 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. Any surface unevenness to the very micron to nanometer scale can be analyzed, and also the crystallography of the specimen and its chemistry can be analyzed with the scanning electron microscope primarily.

What are the parameters which enables this microscopic to do that? We will see that. 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 secondary electrons, backscattered electrons, characteristic x-rays and other photons of varying energies. See each radiation will have very specific energies which we will talk about.

The primary signals which is coming out of this SEM as I said it is a backscattered signal or backscattering signal back sorry I would say backward scattering signals it will be very clear, because there is another particular signal is named as backscattered electrons so you should not con confused with this because this is only coming the backward you know scattering. This is what meant, all this signals are backward scattering signals which are being primarily used in SEM. So, only these three signals are primarily used in an SEM, of course they are characters based on their energy that we will see in an appropriate time.

Out of all this backward scattered signals, only we talk about is, second electrons and backscattered electrons why, why are we talking only about this? Because they vary primarily as a 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. This is the core idea behind using this two and what are the other important things.

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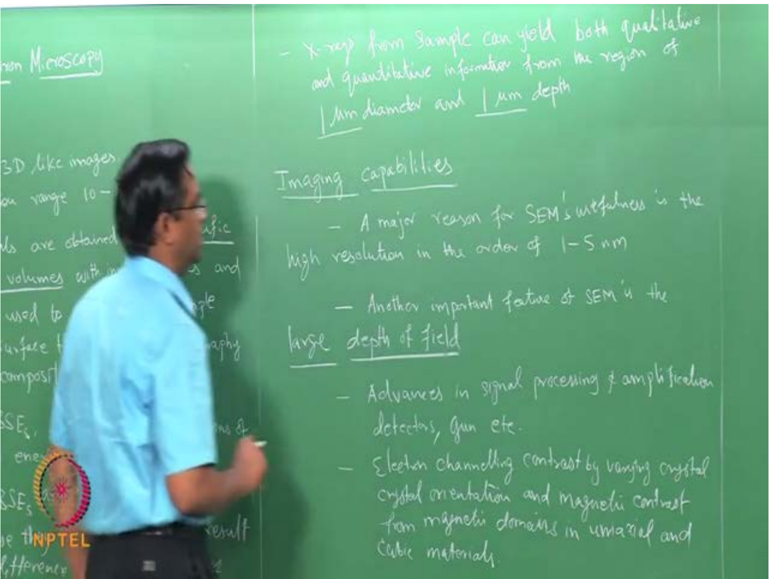

The other important information you get from SEM is, the x-rays that is characteristic x-rays come from a sample can yield both qualitative and quantitative information from the region of 1 micrometer diameter and 1 micrometer depth. This is a rough indication you get what is the region size from which you get this information which is in the order of 1 micrometer diameter and 1 micrometer depth from the surface. These are the information you get from this in general from SEM

We look at what are the Imaging capabilities. So, if you look at the imaging capabilities of this microscopy, the major reason for the SEM's usefulness is the high resolution in the order of 1 to 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 been exploited in electron microscope. In fact, 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 SEM's in the coming classes.

The SEM's are becoming very popular because of the advances in the signal processing and amplification, like the kind of signals you receive second electrons, backscattered electrons or x-rays and when you have advanced processing signal processing and amplification detectors and then gun design, etcetera. So with that all this advances this SEM's can 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. These are all some of the highlights of the imaging capabilities of the SEM.

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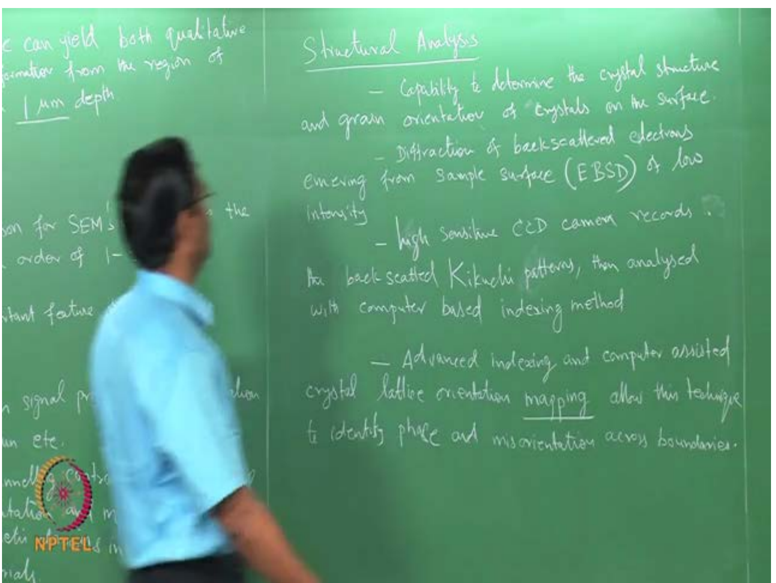

You will see that next is Structural analysis. If you look at what are the structural analyses one can do with this SEM, it has got a capability to determine the crystal structure and grain orientation of the crystals on the surface. Please understand you have to remember that it is all whatever information you obtain is only from the surface with very limited volume. You will just understand that in much more detail as we go into these lectures.

Then the diffraction of the backscattered electrons emerging from the sample surface, Electron Backscattered Diffraction, EBSD to the low intensity also enables this capability. Since, it is a low intensity we have very high sensitive CCD camera

recording, this is Charge Coupled Device camera records the backscattered so called Kikuchi pattern, which is nothing but this signal, and it is analyzed with the computer based indexing method. Then you have today, SEM's with advanced indexing and computer assisted crystal lattice orientation mapping is called EBSD maps, which allow this technique to identify the phase and the misorientation across the boundaries. So, this is also very powerful technique today and it is been applied everywhere in this itself separate research domain people can extensively use this and very powerful technique as for as SEM structural analysis concerned.

What else we can do with the SEM.? So far we have seen Imaging capabilities, Structural analysis and finally Elemental analysis.

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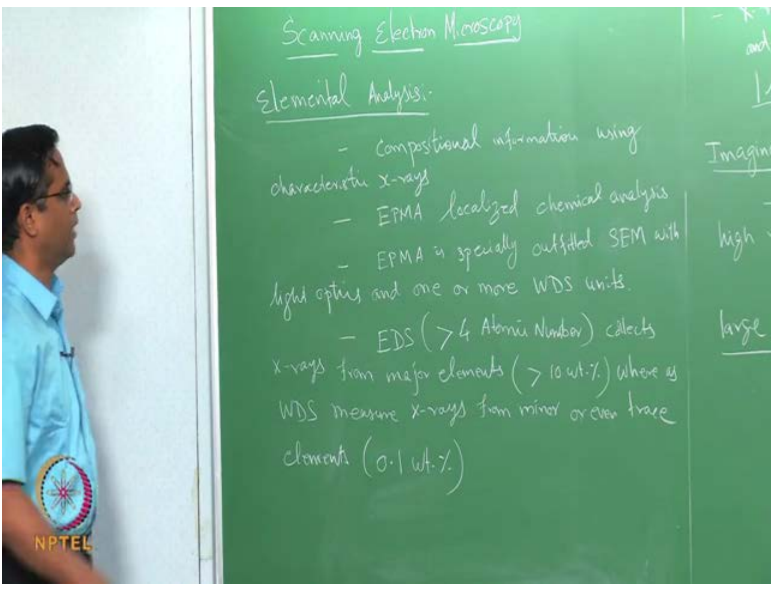

If you look at the elemental analysis capability of an SEM, you get the complete compositional information using characteristic x-rays. The tool generally referred as Electron Probe Micro Analysis, EPMA which can get the chemical composition from the very localized region and then provide complete chemical analysis. Then you have this EPMA is specially outfitted SEM with light optics and one or more WDS units, Wavelength Dispersive Spectrometer. We will see all this variants of the spectrometers as I mentioned which uses the characteristic x-rays which come out of the samples and then

do the chemical analysis we will look at them in a separate lecture serious. But then these are all the one of the primary attachment to the SEM's.

The another one is Energy Dispersive Spectrometer, which can detect the elements greater than 4 atomic number, correct 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 0.1 weight percent. So, this WDS is much more powerful compare to EDS. We will see why and so all this details in later, but these are all the basic details one should have about the when you look at the capabilities of scanning electron microscope. I have few more points to add in this segment.

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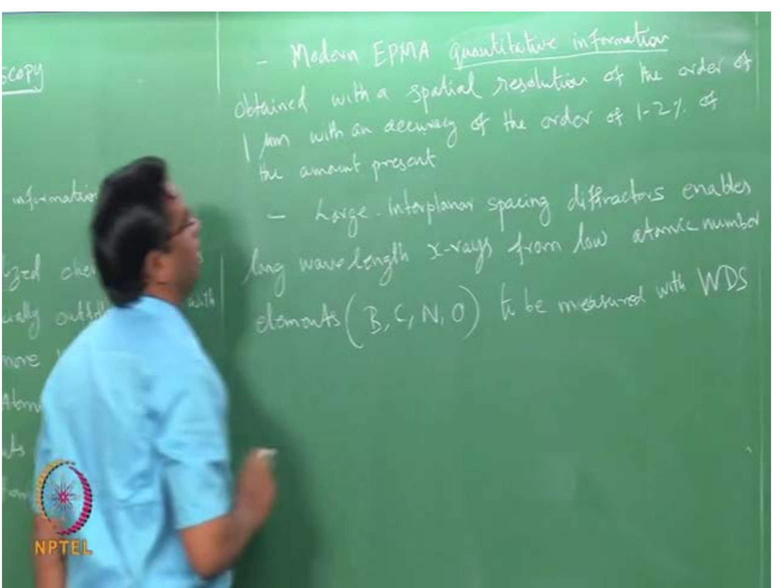

So, final point to the elemental analysis with a modern EPMA, you can get quantitative information from your specimen within a spatial resolution of the order of 1 micrometer with the accuracy of the order of 1 to 2 percent the amount present. And also this EPMA has a capability of analyzing the very low atomic number elements like Boron, Carbon, Oxygen because the WDS spectrometer uses a large interplanar spacing diffracts, a typically organic crystals which has got large interplanar spacing which enables long wavelength x-rays from the low atomic number elements. Since, this low atomic number elements has the characteristic x-rays of large wavelength or a long wavelength, so these

crystals enables the diffraction possible and then and they can be measured with the WDS.

These are all the basic capabilities of a Scanning Electron Microscope. I have just put them into three categories; one is imaging capabilities and structural analysis and then composition analysis or elemental analysis and SEM's are primarily used for only this purpose. Now, we will look at the some of the other introductory remarks.

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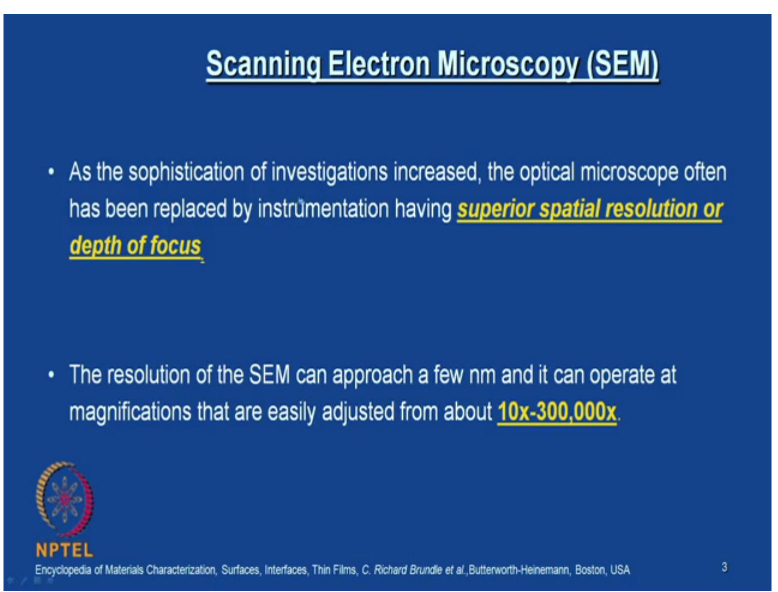

As the sophistication of the investigation increased, the optical microscope often has been replaced by instrumentation having superior spatial resolution or depth of focus. The resolution of the SEM can approach a few nanometers, as I mentioned and it can operate at magnifications that are easily adjusts from about 10x to 300,000x. Of course, this can be a subject of instrumentation we will talk about it in appropriate time.

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Scanning Electron Microscopy (SEM)

- The *depth* from which all this information comes varies from nanometers to micrometers.
- Likewise, the lateral resolution in these analytical modes also varies and is always poorer than the topological contrast mode.

NPTEL

Encyclopedia of Materials Characterization, Surfaces, Interfaces, Thin Films, C. Richard Brundle et al.,Butterworth-Heinemann, Boston, USA

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The depth from which all this information comes varies from nanometers to micrometers. This also a subjected to a specific instrumentation details we will look at them in appropriate time. Likewise, the lateral resolution in these analytical modes also varies and is always poorer than the topological contrast mode.

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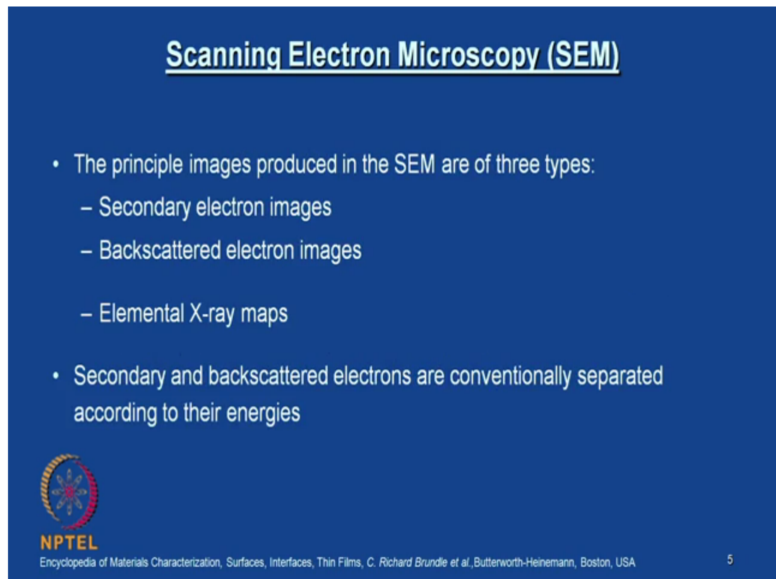

The principle images produced in the SEM are of three types namely; Secondary electron images, Backscattered electron images, and then you also have elemental x-ray maps. These are three primary images one can obtain in a normal SEM. Secondary and backscattered electrons are conventionally separated according to their energies.

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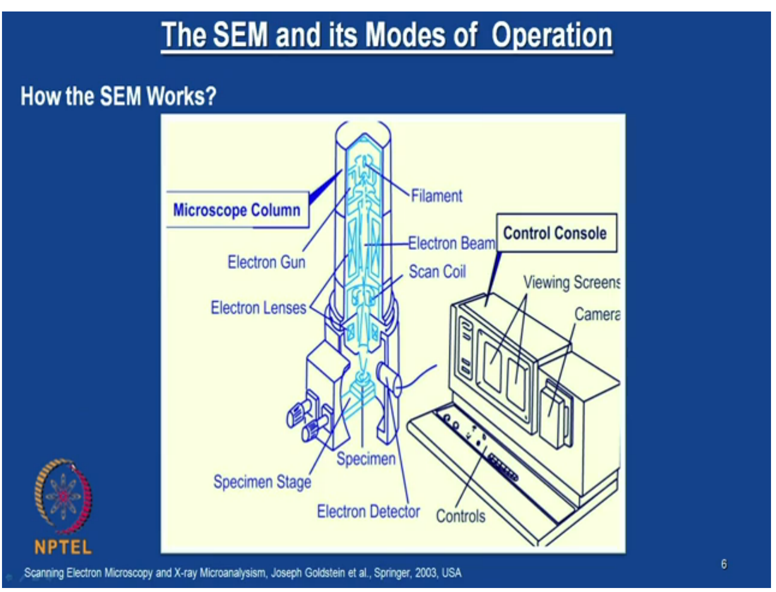

We will now see how this will have some schematics to show how this SEM works. You have the two separate entities; one is Microscope Column on the top to bottom, the other is a Control Console. You have the Electron Gun, which comes from the top of the microscopic column and then you have a further all the Electron Lenses and a Scan Coil and the beam reaches all the way up to this Specimen chamber, which is maintained at the with the vacuum of a 10 to the power minus 4 pascal, which is in the order of one billionth of the atmospheric pressure for your reference. On the right hand side you have the CRT screens, viewing screens, and then camera where all this scanned images are being used.

This is primary classification of this equipment, microscopic column and a control console.

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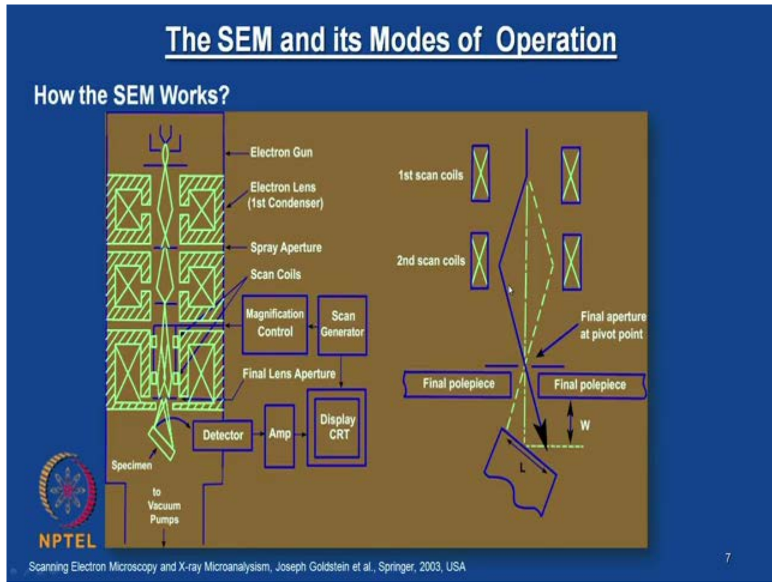

Then you look at this next schematic, and you have this complete schematic of the cross section of the Scanning Electron Microscope. What you see is then electron gun which generates the electrons and accelerates to; typically from 0.1 to 30 kilo electron volts, and then it is being passed through electron lenses and also it can scan coils. So, these electron lenses what they do is, the probe diameter which is being produced by this tungsten hairpin, typically it is not sharp enough to reabsorb the structure. These electron lenses demagnified to very sharp spots or a sharp probe, and then they are being rusted on to this coil to this specimen. 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 this Scanning Electron Microscopy in next class as well.

Thank you.