Characterization of Construction Materials
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Lecture -38
Scanning Electron Microscopes - Parts and Functioning – Part 1

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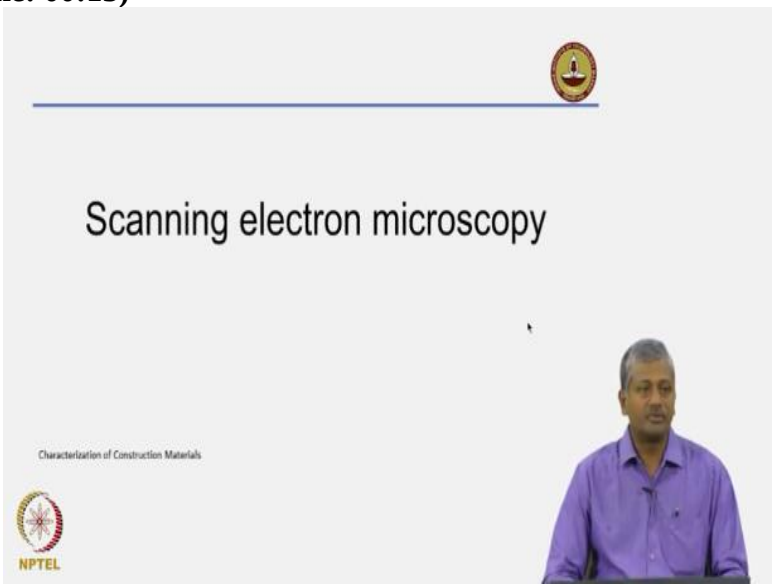

Hello everybody. So in the last couple of lectures we have been talking about the process of optical microscopy. We looked at different configurations of the microscope. In some cases, you have the reflected light imaging, and in other cases we have transmitted light imaging. The purpose of the imaging is obviously to look at the contrast between different phases present in the same material and we saw that the contrast was generally being generated by differential levels of absorption of the light that was passing through or different levels of reflectivity if it was in the case of reflected light microscopy, so reflectivity difference obviously comes from the densities of the different phases that are being imaged and denser phases tend to reflect more light.

Now we also discussed that the resolution of optical microscopy is getting limited because we are working in a band of wavelengths which are fairly large. To really cut down that wavelength, we need to actually start adopting other means of imaging. You already are familiar with X-ray imaging, so X-rays are able to penetrate very dense objects because of their high energies, and because of the extremely low wavelengths we can actually resolve atomic level structure also with X-rays. However, there was a lot of work done in the 1930s

to 1940s, where people looked at the power of using electrons for imaging and scanning electron microscopy is one such technique that came about, which used electrons for imaging the surface morphology of objects as well as to look at compositional contrast that may be arising out of the electron backscatter from the specimen.

So in this series of lectures we will take a look at the process of scanning electron microscopy, and how we can interpret the images successfully to obtain the right kind of understanding of the material characteristics at the micro scale. We already talked significantly about preparation for scanning electron microscopy. In most cases for building materials one of the common strategies, is to provide for a coating on the surface, because building materials are mostly insulating or non-conducting as a result of which when electrons are used for imaging, there may be a charge or build-up of the electrons on top of the surface, if the surface is non-conducting. So in order to make the surface conducting we deposit a layer of atoms of either carbon, or gold-palladium or chromium, to ensure that there is conductivity on the surface, and the electrons do not get charged.

You will see from the examples that we discussed, that is only one mode of imaging, the other mode obviously requires the sample to be impregnated by a low-viscous epoxy resin, and then polishing to a very fine level to ensure that you can actually get some good reflectivity or backscatter of the electrons from the sample surface. So we will take a look at both these kinds of operations of scanning electron microscopy.

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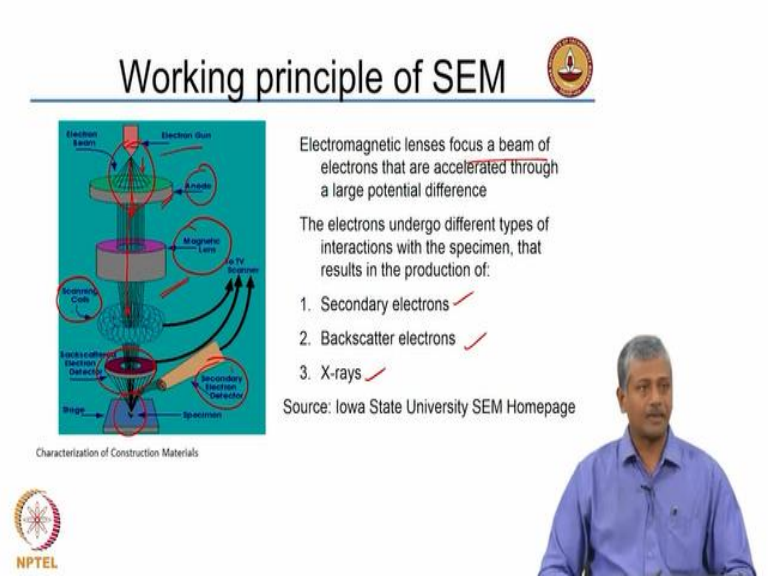

So, the simplest way to put forward is through the schematic diagram. As you can see here, there is a source of electrons, which is otherwise called the electron gun, from which fast moving electrons come rapidly down the central axis of the SEM column. Which is likened to your optic axis in the microscopes, in the microscopes the central beam of light that passes through lenses constitutes the optic axis. So in this case of course, all the electrons are made to move in a straight line or within that parallel beam of electrons to ensure that there is not too much straying of the electrons, out of this central axis. Now, how is that made possible? That is made possible by the use of magnetic lenses - by controlling the level of the magnetic field that you apply, you can actually control the path of the electrons and make them spiral down the optic axis.

After the magnetic lenses, you have the objective lens system - the objective lens system consists of various parts including the scanning coils. So please remember in the case of an electron microscopy, all the lenses will simply be electromagnetic lenses. We are not talking about any optical lenses here; we are only talking about electromagnetic lenses. So in this case, the control of the lens focus would be done by adjusting the magnetic field generated by these lenses. So, the objective lens system consists of what are known as scanning coils, and these scanning coils ensure that the electron beam follows a sort of a pattern on the specimen. So in other words, the specimen top is scanned by the electrons in a particular periodic interval. So you have a line scan, and then you come to the next line, and then you scan the next line and then next line and so on and so forth. This is similar to the scanning that was done previously in the old computer monitors - the cathode ray tube monitors. So the same idea applies here, so we are basically scanning line by line and then the interactions that are generated between the electrons which are bombarding the specimen and the specimen itself, lead to the generation of what we call as backscatter electrons, secondary electrons, and X-rays. And all these are then detected using suitable detectors which are placed at critical locations just around the specimen, and then they can actually capture all these electrons coming out.

So for example this is a secondary electron detector (shown in figure) that is placed at a very narrow angle, because those are low energy electrons escaping from the surface, and they do not need a very high energy to actually capture the electrons by the detector. On the other hand, the backscatter detector is almost placed right around the optic axis or electron axis, to ensure that the electrons that are undergoing elastic rebound with the sample are

getting perfectly captured, because the highest energy electrons will almost undergo an elastic rebound and come right up and at that location, you will be able to capture the electrons quite well. We'll look at the schematic and the actual type of detectors in just a few minutes.

So electromagnetic lenses are available in-between; there is a lens system that focuses a beam of electrons that are accelerated through a large potential difference and this is basically the potential difference between the electron gun which is the cathode, and another cap type system which is called the anode (labelled 'anode' in figure). So when you have a potential difference anode is a positively charged electrode. So electrons being negatively charged will move towards the anode and if you create a large enough potential difference you know that, that potential difference will result in kinetic energy, so it will increase the velocity of electrons which will cause them to move very fast through the anode down the optic axis, and then controlled by the magnetic lenses to ensure that all of them are on the central path, directly going to the top of the specimen.

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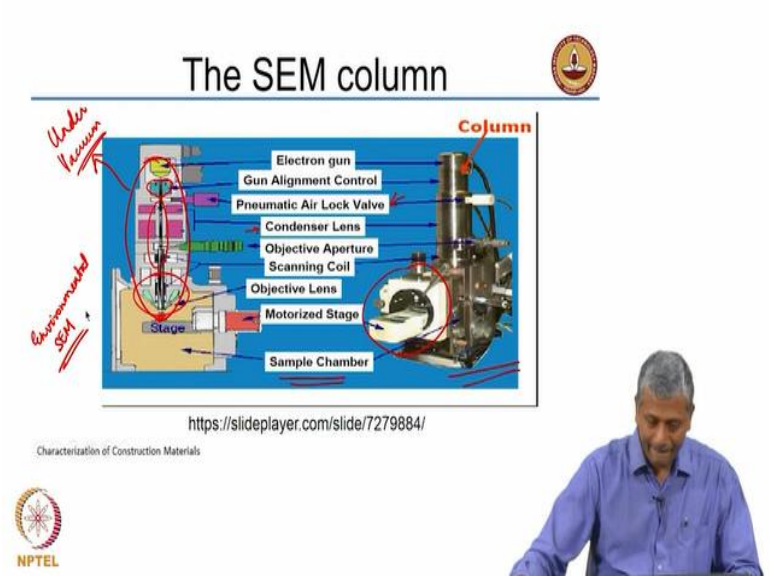

So let us look at the SEM column itself as to how the instrument is actually arranged. So this is a picture of a typical scanning electron microscopy system, so we have a column and then you have a sample chamber. So we have an SEM column, which is composed of course of the electron gun. You have the alignment control which ensures that the electrons from the gun are going right down. There is an airlock valve provided at that location to ensure that you can actually have a different system of evacuation, when the electron beam enters this

objective lens part. So objective lens part consists of condenser lenses which ensure that the magnetic fields are adjusted in a way to keep the electrons down the optic axis, and then you have the objective aperture and scanning coil system which ensures that you get the scanning of the electron beam on the surface; the aperture basically controls the size of the electron beam, just like in the light microscopy where aperture controls the amount of light getting on to the specimen, here it simply controls the size of the electron beam.

So just from first principles if you have a small aperture what will happen to your resolution? It will improve. The smaller the width of the electron beam, the greater the detail that you will see in very short distances. So resolution improvement can be done by reducing the aperture. But if you reduce the aperture, intensity of the electron beam will get reduced. So you have to ensure that you are choosing the right sort of settings for your imaging system, we will come back to that discussion later.

Beyond the scanning coils you have the objective lens system again. It is nothing but an electromagnetic lens which ensures that, by controlling the magnetic field of those lenses you are able to focus the electron beam right on the specimen. The electron beam should converge right on top of the specimen. So that is basically your focus in that case.

The sample chamber is usually set apart from this SEM column, why is that? Now you know that electrons have to move down this optic axis in a nearly centralized arrangement, so they need to come right down the centre of the optic axis. Now, if there is any gas present in this system, the electron beams may tend to stray from the centralized location and further what might also happen is that the electrons may lose their energy in trying to ionize the gasses, because they will have bombardments with the atoms of the gas, and that will slow them down. We do not want that to happen, so we want to control this SEM system under a very high vacuum. So this SEM column, you need to have a very high vacuum to ensure that electron beam does not stray, does not ionize any gases that may be present. So you need to have a very high vacuum level in the electron beam chamber, that means the SEM column.

But once the electron beams comes on to the specimen, there are certain cases in which we want to control the pressure inside the sample chamber because not all samples can be imaged under high vacuum conditions. For high vacuum to be possible, the samples that you image have to be completely dry, completely free of moisture. But in certain cases, for

example, when you are evaluating biological samples, which are wet, which will have water inside, or sometimes if you want to image, fresh cement paste, evolving properties of cement paste, in such cases, you'll have to control the pressure in the sample chamber because you cannot obviously have high vacuum when you have moisture present in your system, because you are going to drive out the moisture that is going to clog all your exits and so on, because of which you'll have complete breakdown of your microscope.

So we need to ensure that the sample chamber is capable of varying degrees of pressures, not necessarily vacuum. In most conventional microscopes, you will not have different vacuums, that means that you will have to operate the sample chamber also at very low pressure conditions or high vacuum conditions.

But in other cases when you are working with biological samples and when you want to do imaging of wet substances, you need to enable, what is known as the environmental mode. In Environmental mode you do not have a vacuum in the sample chamber. So you are able to image the sample with respect to its original state. You do not need to actually alter the state to make the sample completely dry.

The stage on which the sample sits or specimen sits, is a stage which can be moved in the X and Y-directions as well as in the Z-direction, and in some cases you also get stages that can be rotated, because sometimes rotating can give you very distinct features of certain crystalline species. So you can have a stage which has only three axes of movement or sometimes it may also be equipped with rotation capabilities. So the rotation capability actually is there in the high-end microscopes. You will have to actually order that specially to get the sample stage which can actually be rotated in addition to being moved the X, Y, and Z directions. We will take a look at each one of these components separately.

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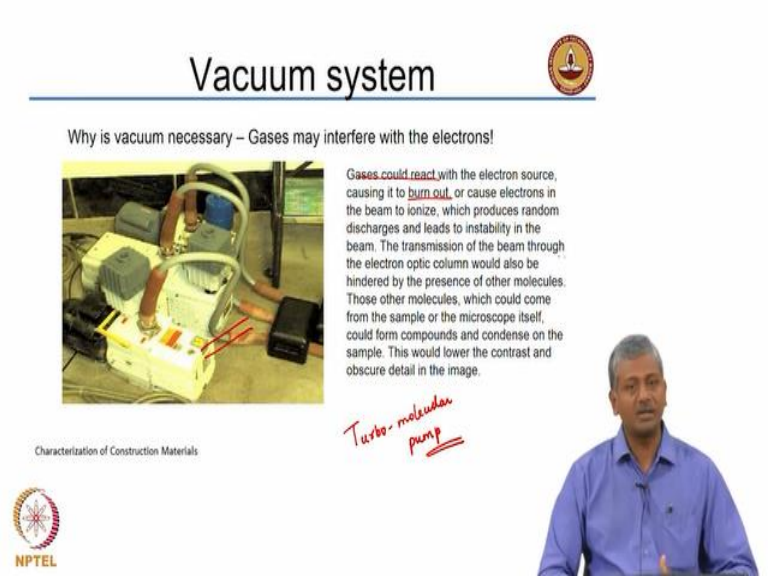

So first of all we need to understand that we need to have a good vacuum system in place. So generally the vacuum system consists of a regular vacuum pump, typically an oil-based vacuum pump and there is also another system called a turbo-molecular pump, which is present within the SEM column. Now depending upon the amount of reduction in pressure or extent of vacuum that you need, at certain points, this oil-based system will stop operating, and the turbo-molecular system will start operating this. And when the turbo-molecular system starts operating it will then be able to evacuate the chamber to very low pressures or very high vacuums. And again, we need vacuum because the gases present in the SEM column can react with the electron source, causing the electron source to be losing its energy, basically burning out because it is spending all its energy in trying to ionize the atoms and then the beam becomes unstable because the ionizing atoms will discharge. The transmission of the beam through the electron optic column will also be hindered by the presence of these gaseous molecules in the air. So we need to completely evacuate, which makes it very essential imperative to have a good quality vacuum system installed. In many microscopes, one of the common reasons which makes the vacuum system ineffective is that we sometimes do not control very well the sample preparation. If there is any moisture, if there are any loose powders in your sample, which can fly off, that can also affect your vacuum system, because they clog the evacuation exits and so on and so forth. So because of that, you need to ensure that you do not have any loose powders in your sample, because in cementitious materials sometimes we have to work with loose powders but we need to ensure that they are prepared sufficiently well, so that they will not be sucked out when the chamber gets evacuated. Other thing is of course you do not need, you should not use wet samples. If your SEM does not have an environmental mode or a high pressure mode.

So for powdered samples typically what is done, one aspect is to take a double-sided tape, stick it on a glass slide. Take a double-sided tape, spray the powder on it, and then rub the powder onto the tape so that it sticks properly to the tape. So mostly what will happen is in such cases when you actually evacuate the chamber, the powder will be sufficiently well stuck to the tape so that it does not come off.

The other option is to take the powder and immerse it in epoxy and then you prepare a flat sample, polish it, and then observe under the microscope. So for powders that is the best way to do it. Because if any loose powder is present on your tape, which you have not carefully removed, then this will start getting into your evacuation points, and then that will cause your vacuum system to start collapsing. And secondly, it will also lead to contamination of the chamber, which you do not want it to happen. So, with super-fine materials like silica fume, the better method would be to embed in epoxy. Just take a cylindrical container of low-viscous epoxy and put your powder into it. So it is encapsulated properly, then you slice, prepare the sample, and prepare the specimen surface by polishing it and then imaging.

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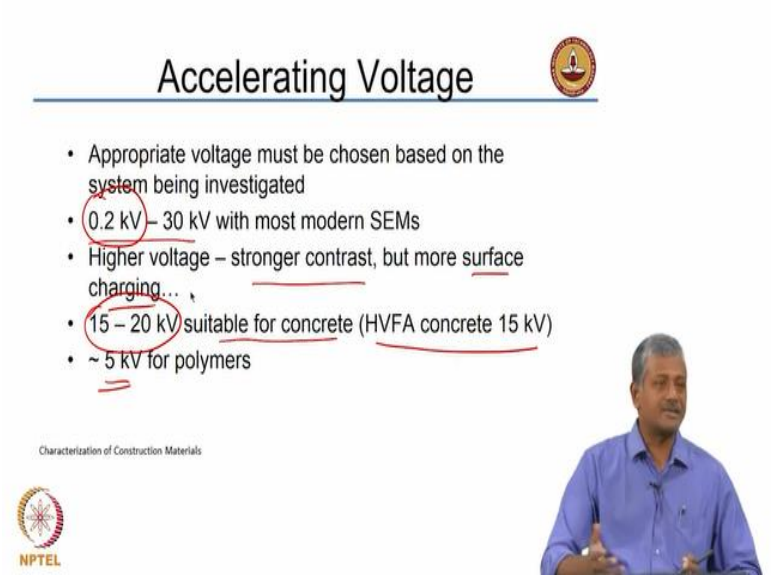

The electron beam speed is controlled by the potential difference that is applied between the electron gun and the anode. So that controls the electron speed. The movement of the stage up or down only controls the working distance, that is the distance between the objective lens and the sample.

The accelerating voltage is basically the potential difference, which is applied between the electron gun and the anode. Now, to image different types of materials, we need different accelerating voltages, if we are trying to image something which is soft, we do not need extremely high accelerating voltages, because the higher the accelerating voltage, the greater will be the depth of penetration of the electron beam into the sample. So appropriate voltage has to be chosen based on the system being investigated and generally most SEMs have a range between 200 Volts (0.2 kV) and 30 kiloVolts, which is a very large operating range. That means they are capable of imaging different types of systems. When you are looking at biological systems, you are operating at very low voltages because most biological systems we have single-cell systems or very small cellular systems that you are trying to image with the scanning electron microscope, if you have very high energy electrons, they may tend to actually damage your entire sample itself, because of the interactions. So you want to reduce the voltage and at which these electrons are going down.

But when you are moving to dense systems like ceramics, like concrete, you have to adopt higher accelerating voltages, because what will happen if you go with very low voltages is that the beam penetration into the sample will be very limited and because of that you are not going to get a good representative understanding of what that sample is made up of. So if you want good beam penetration, you can operate at high operating voltages. So with higher voltage you get stronger contrast, but you are also liable to get more surface charging, because these electron beams are going very fast down the optic axis and collecting on top of the sample, so we need to ensure that when you are operating at very high voltages, you need to conduct the system properly. Otherwise what will happen is electrons will start building up on the surface.

Now, you do not always need extremely high voltages when you work with concrete-based systems. It depends on what type of imaging you are performing. If you are just doing a morphological imaging on the surface, the accelerating voltage does not have to be very high, because the beams are basically dislodging the secondary electrons present on the surface. But if you are trying to understand the compositional assessment of the phases that are present in your material, then you want the electron beam to at least penetrate some distance, so that you get some representative image of what you are actually observing. So for that you need to have a higher accelerating voltage.

So for most concrete applications, a voltage in the range of 15 to 20 kV is suitable. For softer concretes you may want to have lower voltages, but for most dense concretes, you will have to have higher voltages. And for polymers, you adopt lower voltages because, obviously, the polymers are much softer, and have lower elastic moduli as compared to concrete. High volume fly ash concrete, just an example is provided for a softer system, because high volume fly ash concrete, because it gains strength much slower, and probably never reaches the strength potential of 100% Portland cement concrete. You may actually want to reduce the amount of accelerating voltage for such softer systems.