Characterization of Construction Materials
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Lecture - 32
Optical and Scanning Microscopy- Introduction and Specimen Preparation – Part 1

Hello everyone, so today we will start on a new module on microscopy. In this chapter, we'll cover the basics of optical and scanning electron microscopy. Microscopy happens to be one of the most important techniques to be applied as far as characterization of materials is concerned. Whatever is the material, whether it is a construction material or manufacturing type of material, or deep understanding of material science is required, microscopy still remains the most central technique to the investigation of the characteristics of materials.

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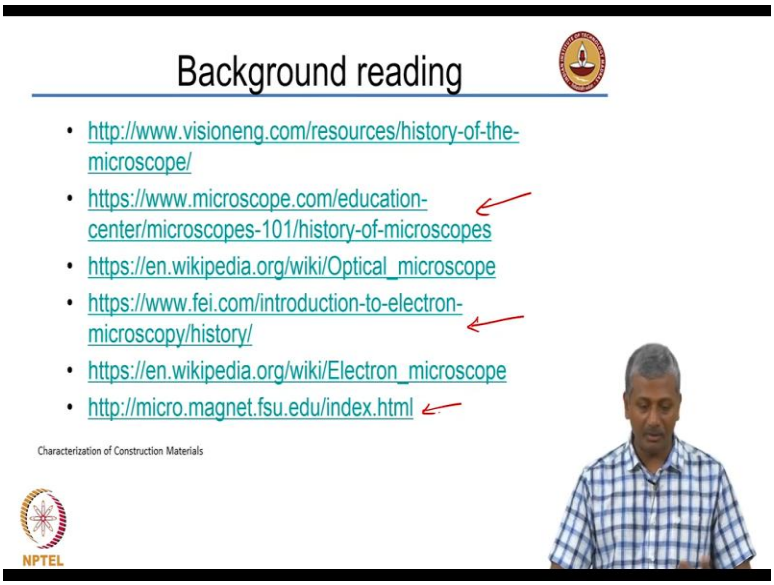

So, there is a lot of background reading that you can actually do in microscopy. Many of you may have already come across several articles with microscopy in it. You may have also performed microscopy yourself in school. If you have taken biology, there is all chance that you would have actually used the microscope, at least the light microscope where you would have observed small cells, or samples of blood or skin. They ask you to pick your own skin and bring the blood out, or maybe bite your nails or bite your skin and try to image that under the microscope. So that is a quite a common thing as far as microscopy is concerned in biology. So, amongst all scientific disciplines, probably the most commonly used investigative technique is

microscopy. And there is a lot about the history of microscopy, which I feel will not be really useful to go in detail as far as this course is concerned. But nevertheless, it is important that you understand what the background of this technique is. So there are several web pages that have been listed where you can get information on the history of development of microscopy, and how it has been applied over the years to the study of different types of materials.

So this particular reference is quite useful (Reference 4 in slide). It talks about electron microscopy and the history behind electron microscopy. And of course, even before that, there is a web page that deals with history of microscopes (Reference 2 in slide). So those 2 at least you should take a look at and see how things developed, how people actually understood the limitations of light microscopy and moved on to electron microscopy and so on. But in our case, what we will try to do is look at the principles of microscopy, how we can actually improve the kind of imaging characteristics that we get from samples, and then how to interpret these images.

As I had said in the initial lecture itself, one of the most important characteristics of characterization is to ensure that we understand how to interpret the result. Getting the result is something easy, because you have the technique to actually help you through it. But to get an interpretation of what you are looking at, and tying it to the overall story, at different scales, and that is basically the challenge as far as investigative characterization is concerned.

So I'd also like to bring your attention to this website (Reference 6 in slide) which is basically from the Florida State University. And this website has a lot of interesting information about different types of characterization techniques, not just microscopy. They have a lot of information here. And you can really learn a lot from what is actually presented on these web pages.

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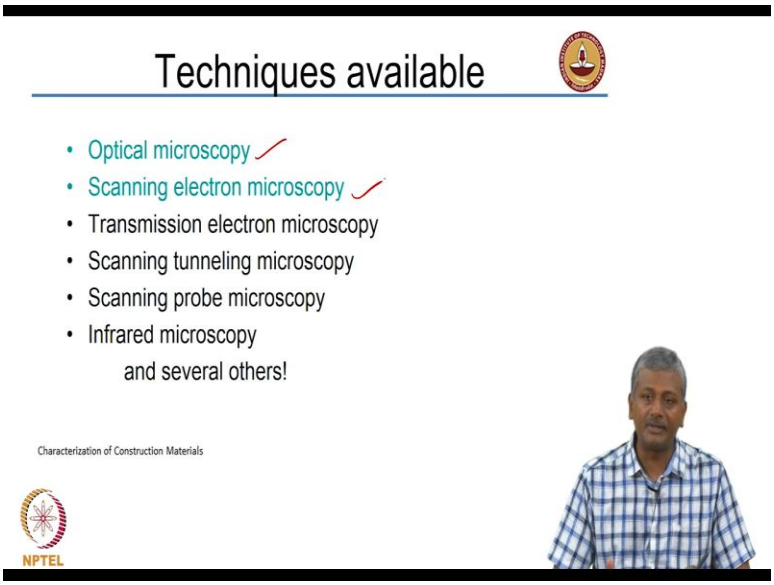

Moving on there are several different microscopy techniques available. You have optical microscopy, scanning electron microscopy, transmission electron microscopy and there are other smaller known techniques like scanning tunneling microscopy, scanning probe microscopy and infrared microscopy. Of course, I am only touching the tip of the iceberg. There are different types of microscopy techniques, that are either standalone techniques or derivatives of these primary techniques.

In our case, of course, we will concentrate our discussion on optical and scanning electron microscopy because these are most commonly utilized for the study of construction materials, particularly concrete. We will look a little bit also in to the basics of transmission electron microscopy, although we will not really cover that in much detail. I will bring that out primarily from the point of distinction between scanning electron microscopy and transmission electron microscopy.

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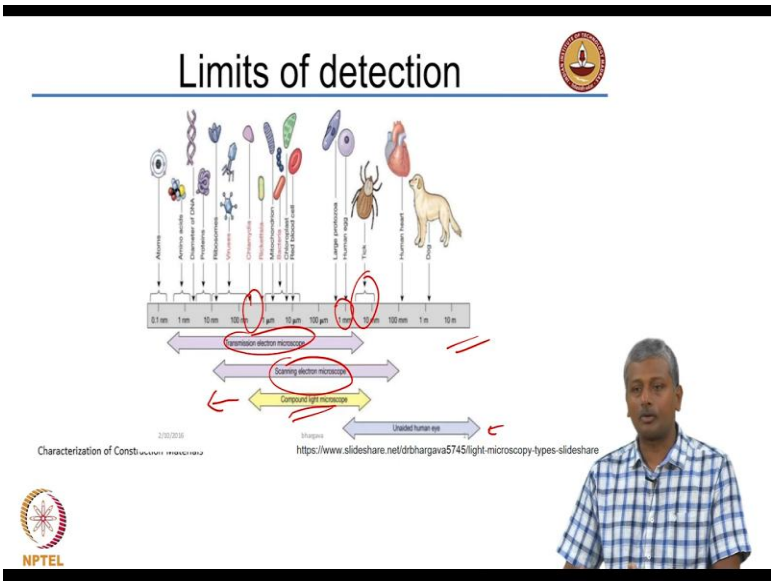

Now, you must understand that everything has a limit of detection; you cannot really use any technique to detect anything that you want. For example, you all know very well, from unaided human eye, the scales of detection probably range from less than one millimeter to, of course, we do not really have a upper scale as far as resolution is concerned, because the larger the object, the easier it is for us to see anything. But of course, then you have the issue of distance. There is a sight distance; you cannot really look beyond a certain distance. But you have a range of coverage possible with the unaided human eye.

But then if you want to go slightly further, you need to obviously go into the light microscope. Why do we call it the light microscope? Basically, it corresponds to light in the visible range. Visible range of light implying the range of wavelengths that constitutes visible light. That is part of the spectrum of light that we see. So, again, we will talk about that wavelength, when we look at the characteristics of the electromagnetic radiation. Now, compound light microscope, we are talking about several millimeters all the way down to about less than 1 micron, probably not much better than that. You probably would be able to see sometimes better than 1 µm, but not all the time.

But when you go on to scanning electron microscopy, you push the limits of detection, down to about a few nanometers. But then even scanning electron microscopy has its limitations with respect to nano-sized materials. So if you have to go really further down and image these kinds of materials, you will have to go to the transmission electron microscope which can go

further down in terms of your limits of detection. So, if you have to go to atomic scales or molecular scales, then you are talking about using transmission electron microscope.

In scanning electron microscopy, we will probably see a lot of different types of bacterialogical objects, or viruses and things like that which are of the micro sizes. In terms of your concrete or construction material scales, we are talking about how to make out different types of phases that are present within your cement paste for instance, as far as scanning electron microscopy is concerned. But if you have to go deeper into the structure of, let us say, C-S-H, which is a very high surface area material, and if you have to really look at how the spacing between the sheets is or if you want to find out what are the different compositions of C-S-H that are actually possible, you'll have to go much deeper than just scanning electron microscopy. But then we do not always need that in all the cases, only in a few studies, we actually need to go down to that scale.

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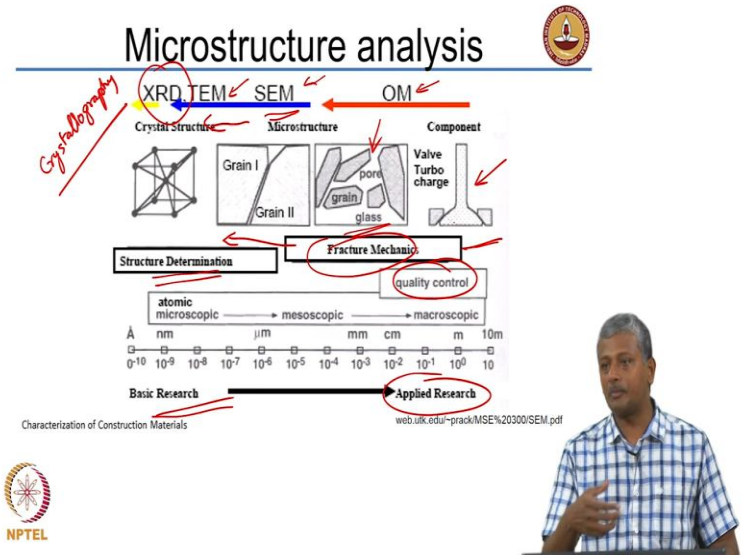

So, again depends on what you would like to do. We talked about this at the beginning itself, applying characterization techniques to the study of materials is an expensive proposition. You need to choose your techniques carefully, depending upon what exactly you want to find out. So, here, we have just looked at these primary techniques, optical microscopy, scanning electron microscopy and transmission electron microscopy, and of course, X-ray diffraction, which is the core characterization technique that you can apply to most materials.

What are we talking about when we look at these different types of techniques? For example, let us look at a component level, if you have a manufacturing component, of course, most characteristics or component are visually possible to observe you can see it with the unaided human eye. But if you have to look at for example, the makeup of the grains, how are these grains distributed in the microstructure of this component, probably optical microscopy may be good enough to look at the overall grain characteristics, look at the pores that are present between the grains and so on and so forth. But if you have to look specifically at the grain boundaries, how are the atoms distributed across the grain boundaries, what is the overall length of the grain boundary, what is the shape of the grain, on a much smaller scale, then you have to move to something like SEM. Now, when you go to TEM or X-ray diffraction, you are pushing the limits of what you can detect and you are going towards understanding the crystal structure, you are deciphering the crystal structure, or you are doing what is known as crystallography.

Now, it depends on what you want to do. For example, when you only talk about quality control, you are here at the visible range, or maximum, you can take probably a magnifying lens and see the components yourself. But if you look at optical microscopy, you can probably get the distribution of the grains in the microstructure, look at the pore sizes, any defects that are present, like cracks, for instance. And that could be in the realm of fracture mechanics. Fracture mechanics, we try to understand how materials fail, how can we actually understand the limits of these failures, and so on and so forth.

But of course, if you want to go towards something as complicated as structure determination, you need to go finer and finer. So, that is why we go to TEM or X-ray diffraction. So, again, this part on the right, where we are talking about length scales of the order of tenths of millimeters to about few meters, we are talking about applied research, this is not basic research.

Basic research means we are in length scales of the order of Angstroms (Å) or nanometers. And that is only possible by a judicious combination of the techniques. You cannot just use one technique to get the entire range. You see that the range is quite vast. It goes from 10 meters to 1 Å, that is quite huge. You are basically coming 10 or 11 orders of magnitude. To actually investigate the material across all these orders of magnitude, you have to use all these techniques together. And that is one thing which we need to really comprehend is that no one technique can give you all the answers. It depends on what you are looking for. And you need to

choose your technique wisely, to ensure that you are able to get the best out of it for the particular problem that you are trying to solve.

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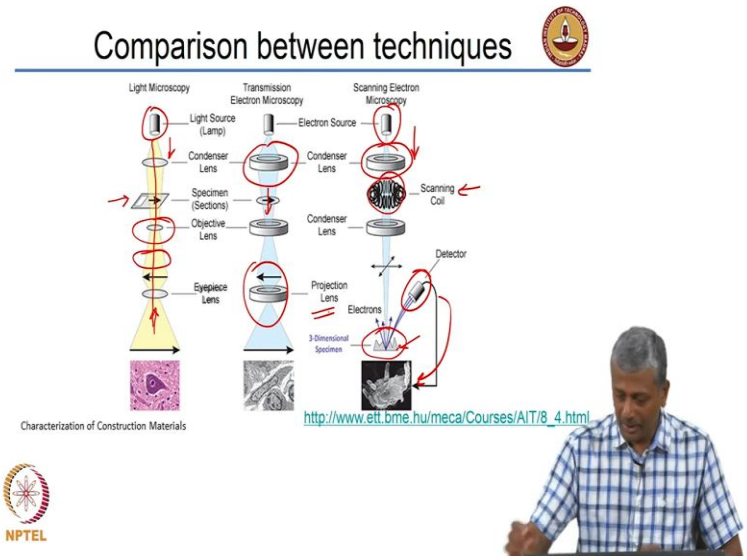

So with this background, let us first look at a comparison between the different techniques of microscopy that we are going to be talking about in this chapter. We will not talk about transmission electron microscopy much, but I wanted to bring that distinction up, so that you can get an idea about what is the difference between transmission electron and scanning electron microscopy.

So of course light microscopy is something all of you are familiar with. So here we have a light source, which is then sent through a system of lenses. What do the lenses do? They ensure that the light source moves in a collimated beam of light. So that is able to bring maximum light to the object that we are trying to investigate. Most of the times we deal with specimens that are transparent or translucent, which can pass the light through, and then we are able to see on the other side, what the structure of the specimen is, based on the amount of light that passes through the specimen. So, through the specimen, the light passes, and you also have an objective lens system, that is the lens which is closest to the object or specimen. You may have other intermediate lenses in between, but ultimately, you are viewing the image through what is known as the eyepiece lens. You have the eyepiece lens on the top through which the image is actually viewed. So you get contrast based upon the different types of light that are absorbed by the specimen, or the light that is transmitted by the specimen, depending upon how dense the phases

of the specimens are, so those are the contrast and we will of course, talk later about how you actually get contrast in light microscopy a little bit later.

In scanning electron microscopy, you do not use light anymore, you use electrons. The advantage there is that you have electrons which are of a much lower wavelength. So, when you reduce the wavelength, your ability to resolve features increases. We will look at that in more scientific detail a little bit later on. So here what you are doing is, you are basically having an electron source, which propels very fast moving electrons down the optic axis. We call it the optic axis, it is similar to your optic axis in your light microscope, where everything comes down in the centre, that is your optic axis. Similar to that in scanning electron microscopy, you also have the optic axis along which you are propelling very fast moving electrons. And since electrons obviously are charged, you can actually increase or decrease their speed by controlling the magnetic field in this optic axis. So magnetic fields are controlled by what are known as condenser lenses.

And then you have an object called the scanning coil, which takes the electron beam and scans it on the surface. Now many of you may be familiar with the older televisions that we have - the cathode ray tube based televisions, or the older monitor, CRT monitors, which we used for the computers. The newer generation probably has been used to LCD and TFT. But the older generation will be familiar with the cathode ray tube monitors. Even there, basically the entire screen is scanned. And because of the interactions of the photons, with the fluorescent elements on the screen, you get light of fluorescence that depicts the image on the surface of the screen. So the same thing happens in this case is that the electron beam is getting scanned on top of the specimen. So that is your specimen here, and the electron beam gets scanned on top of the specimen, the interactions that the electrons generate with the specimen are then detected suitably.

So it is not like your light is or electrons are passing through the specimen, they are getting reflected, or they have some other interactions with the specimen that generates some sort of detectable signals, which are then picked out by the detector, and then shown on the screen. We will talk about what exactly these different types of interactions are.

But when you move to transmission electron microscopy, you cannot work with specimens that are of this type, you need to actually grind them into an almost flat or extremely

thin material which can transmit the electrons through. So we are talking about extremely thin materials or thin sections through which the electrons can get transmitted. So here, what you do is you have the electron source, you have condenser lenses to control the speed and the trajectory of the electrons. And these electrons go right through the specimen, just like in a light microscopy system, and then they are imaged through the eyepiece. So in this case, this eyepiece lens is also called the projection lens.

Now, the distinction is primarily between transmission electron microscopy and scanning electron microscopy. In the scanning electron microscopy system, you are scanning the beam of electrons on the surface of the specimen and looking for interactions that are generated out of it. In TEM, the electrons go right through the specimen and are looked at on the other side.

Now, all of you are also familiar with one more similar such system called X-Ray imaging. What do you do in an X-ray imaging system? You have the object, you send X rays and the X rays go through the object and are imaged on the other side. How does the contrast come about? Based on the absorption of the X rays by the different phases that are present. So when you, for example, take an X-ray of your body, you get an image which shows the bones and the flesh does not really appear very clear. That is actually a negative image because your bones are a lot more denser than the flesh. So they tend to absorb more X-rays. So in reality, the image should be the bones looking dark in the flesh looking light. But what we do is a negative of the image, because what we want to use the X-rays for, is to look at defects and dislocations on the bones. So that is also another type of transmission technique where X-rays go through the material. And that happens to be also a very popular technique, as far as material characterization is concerned, but not so often used, because very often the strength of the X-rays that you need for imaging objects, makes them very hazardous, because you need a very high intensity X-ray to image objects and look at the defects inside. In medical imaging, the X-rays are not very strong, and people are not exposed to those extremes for a long period of time, which is why they are safe to use. So with this distinction, I hope that you understand what the limitations of the methods are and when to apply these methods, let us move on from here.

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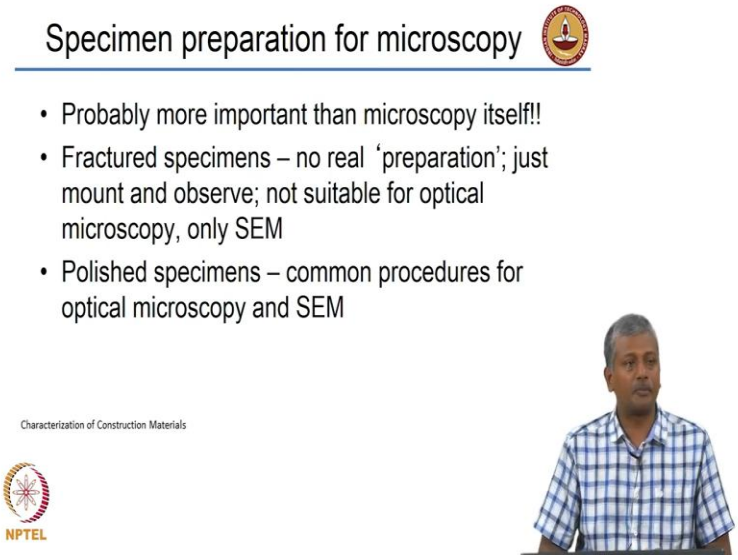

Let us first talk about the most important subject of specimen preparation for microscopy. Now, very often, the preparation itself is more important than the application of the technique. How you prepare the specimen can make the interpretation either very easy for you or extremely difficult. So, preparation of the specimen is key as far as getting good microscopic images are concerned.

Now, when we want to look at fractured specimens from materials, so, in such cases, for example, when we do a uniaxial tension test on the steel, we pull apart the steel rod, and then you know that there is a specific shape to the failure that you get, because of the ductility of steel, you get a cup and cone type failure. Now, if you want to observe this cone or cup under the microscope, all you need to do is just stick it under the scanning electron microscope, and then take a look at the features of the grain boundaries and so on.

Now, of course, you cannot do the same with optical microscopy because you have to remember that light microscopy depends on objects that are extremely flat. Now we will talk about the reasons as to why that happens a little bit later on. But for most cases, when you want to observe fractured specimens, under scanning electron microscopy, you do not need major specimen preparation techniques, all you need to do is fracture the specimen and directly stick it into the microscope.

Now, as far as optical microscopy is concerned, and certain types of scanning electron microscopy, like those that depend on rebound of the electrons that hit the surface, in those cases, we need to polish the specimens. The specimens have to be perfectly flatly polished to ensure that these reflections can be made possible. Now in light microscopy, unless your specimen is transparent or translucent, in which case the light will go directly through, in other cases, you will be relying on the reflection of the light from the surface in such cases, you need to ensure that the surface is perfectly flat. So, you get a perfectly clear reflection from the phases that are present on the surface. So, polished specimens you commonly use for optical microscopy and for backscattered scanning electron microscopy.

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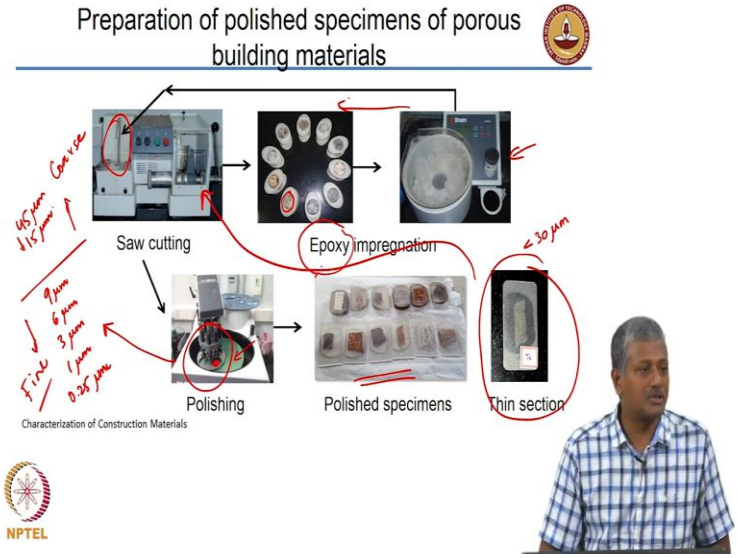

So, how is this polishing done? So, this is the technique of polishing, first you start with saw cutting. So you have here a saw, diamond-tip saw typically because most construction materials can be easily cut, including steel with a diamond-tip saw. So the saw basically ensures that you are able to cut down your specimens to a small size, ofcourse, you cannot use very large size specimens in your microscope because no microscope is actually geared to handle extremely large specimens. So, now you have this saw cutting that is done to reduce the size of your sample into something that is more manageable in a microscope.

So, to prepare the specimen for polishing, you have to first impregnate that with epoxy. Now why do you think they impregnate the sample with epoxy? So samples are not perfect

solids, unlike metals, metals are mostly perfect solids, in those cases you do not really need to impregnate, you can directly image by polishing those specimens directly. But if you have to image construction materials, you need to impregnate it with epoxy because construction materials are porous and if you do not impregnate it well, the act of polishing may destroy your structure. So, the reason for impregnation is that the epoxy will get into the pores and into the cracks and voids and preserve the structure while it is being polished. So, epoxy impregnation is an important part of preparing a polished specimen. So, of course, this epoxy is impregnated under vacuum which causes this epoxy to boil up and then enter the pores of the construction material.

And then after that, you again send it back to actually make a finer saw cut from there. After you do the epoxy impregnation, you can make a finer saw cut, because what will happen is when you take your specimen and impregnate with epoxy, even the surface of the specimen will get covered with epoxy. Now, if you take that specimen to image, you will not see anything but epoxy, so, you will have to actually do another finer saw cutting to expose the surface of the specimen. You can avoid that step and directly go to polishing, but that will take you longer to polish the epoxy out. The epoxy needs to be polished out or you can simply cut it off and then go for polishing. So, you can actually avoid the second saw cutting technique and directly go to polishing also.

So polishing involves grinding it on a plate with some sort of an abrasive, the simplest abrasives are obviously carborundum or even sandpaper for an instance. So if you want to do coarse polishing, those are good enough. But if you want to do finer and finer polishing, you need to reduce the sizes of the grains that are abrading your system progressively, typical material that is used for polishing is diamond paste, we use a diamond paste or a diamond spray, which is sprayed onto this circular table that is rotating at a certain speed. And then on top of that you have this specimen holder, that holds your specimen in place here and grinds it against this table which has the diamond spray on it. Of course you cannot have solid diamond spray you need to have a lubricant in which this diamond spray disperse because without the lubricant, you will produce too much heat that will again damage your specimen. You do not want to generate that heat.

In this case we do not use IPA because IPA is volatile, it will just get off immediately you need something that stays. So lubricating oils and greases are typically used in this case, or you can use ethanol which goes much slower than IPA. But there are special lubricants available for this purpose, which can help in keeping the material in a liquid state. So, we want the spray to be dispersed in this lubricant and then interact and abrade the surface of the porous building material. If the spray is too dry, it will generate too much heat. So, after this polishing, you get these polished specimens, which are then ready for imaging.

Now, of course, you may want to go a step further to produce a translucent specimen, like for example, if you are doing a through transmission microscopy, then the specimen has to be really thin. So, for that what you need to do is to prepare the thin section, you need to go back to this instrument and do a very fine milling of the surface. So, continuously you will be milling the surface to bring down the thickness. The thin section, as the name implies, has a thickness of less than 30 μm. So, we are talking about grinding the specimen down to about 30 μm in size or less because otherwise, light will simply not be able to transmit through these objects. So, for thin section microscopy, you need to do a progressive milling to ensure that you get this thickness.

One thing I want to tell you is this preparation technique, for one single specimen can be as long as 15 to 20 hours. So it is not something simple, because the epoxy impregnation and the curing or setting of the epoxy takes itself about a few hours and after that you need to do the fine saw cutting you need to do the polishing. Polishing has to be done with successive diamond spray sizes. Typical sizes I will just tell you, in the case of polishing of cementitious specimens, you typically start off with diamond sprays of sizes, 9 μm, then you go with 6 μm, then 3 μm, 1 μm and if you need much finer polishing, you can go to about 0.25 μm. So, 5 different diamond spray sizes are used. And of course, you have to realize that each time a new diamond spray is put onto this rotating table, you need to first clean out the table and then put it because otherwise the old abrasives will still be on the table and you will not really get the fine polish that you want. So, this is the fine polishing and above this is coarse polishing, where we are talking about 45 μm up to 15 μm. So, depending upon your experience with how this polishing goes, you can then alter your technique to ensure that you minimize the number of steps required to produce a given sample.

Now, of course, in terms of concrete if you are comparing paste with mortar with concrete, the time taken for the preparation of these specimens will be quite different. Which specimen do you think will take less time. The paste may take less time, but the only problem is paste is a very soft material, so we need to handle it carefully. Because, if you continue to polish it too much, it will keep on getting abraded. When you move to mortar and concrete, you have 2 dissimilar phases in it, one is the paste phase, one is the aggregate phase, the aggregate phase is hard, and paste phase is soft. So, some level of grinding may be enough for the aggregate phase. But then you need to ensure that the paste also gets ground to the same level of fineness.

So, altogether put, this technique of polishing is not really a science, it is an art. So you need to actually practice it a lot to try and master it. And I can tell you with 100% confidence that you will not be able to master it. Out of 100 samples that you prepare, you will probably get about 20 which are working well, and the remaining 80 will be wasted. But then that is the price you pay for doing this kind of microscopy technique. Of course, I am not saying that for everybody, this is the case. I am talking about the average research investigator. If you talk about people in material science, they probably have much more input as to how to prepare these samples in a much better fashion, but even in cement chemistry, there are people who can actually do a much better job. But again, those are the ones who have perfected the art. So it is not possible for everybody to do that all the time. I have lost countless samples doing these kinds of investigations. And it takes a long, long time to actually do this kind of preparation. So that is why, once again, I come back to the original scenario. Please do sophisticated techniques only if you think that they are going to give you some sort of an answer into the question that you are looking at. If you can get an alternative method of answering that question, it is all the more better.