Characterization of Construction Materials
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Lecture - 33
Optical and Scanning Microscopy- Introduction and Specimen Preparation – Part 2

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Slicing paste samples

First specimen (disk) is typically discarded

Scrivener et al. 2015

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Now in some cases for example, when we have prepared paste specimens, just like you do for X-ray diffraction, you must have learned in X-ray diffraction that you do not always need to powder the specimen, you can actually use slices of the specimen, of the cement paste and directly image the slices. So as soon as you cut the slice you can put that under the X-ray diffraction instrument and image it or rather collect the X-ray patterns from it.

Similarly in microscopy also, you can actually directly take cement paste slices and simply cut it off and use it for microscopy. Only thing is, again, you need to embed this in epoxy and then do the grinding. You do not need to break off chunks of this material. When you are dealing with concrete or mortar, you can either slice it, or better still, to have a more representative sample collected, what typically is done is, when you actually crush the concrete cubes for compression, you take a small chunk of mortar from inside that concrete that represents the entire concrete sample. And then you use that impregnate that in epoxy and prepare your specimen for that. So, you can choose specimens or samples of materials from different types of objects and different types of tests.

So, you can actually prepare these paste cylindrical specimens, just for the purpose of microscopy and simply cut them from time to time. A very important question I want to ask you, what is the difference between a specimen and a sample? I am mixing up both, but obviously, there is a difference. Why would we have 2 different words, sometimes when we refer to the same material? When do we call something a sample and when do we call something a specimen? A specimen has a specific dimension.

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Preparation of polished specimens of porous building materials

Saw cutting

Epoxy impregnation

Polishing

Polished specimens

Thin section

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So in the previous example, when we had these small chunks of mortar or concrete or paste, which were taken out from other tests to prepare for microscopy, those were ‘samples’. But after we did all this epoxy impregnation, we got these ‘specimens’. So we have prepared a specimen from the sample. So please understand that distinction very often in technical writing, I see that people make this mistake and they do not distinguish sample from specimen. Specimen always has a distinct dimension. Sample could be anything. A sample could be anything like a powder, for instance, a powder is a sample. So when you prepare a powder dispersed on a glass plate for X-ray diffraction, it is only a sample of the material. But when you take this powder and you actually use those sample holders for XRD, because that is a disc-type specimen that you actually get, so that becomes a specimen. But it is still a powder sample which is forming that specimen.

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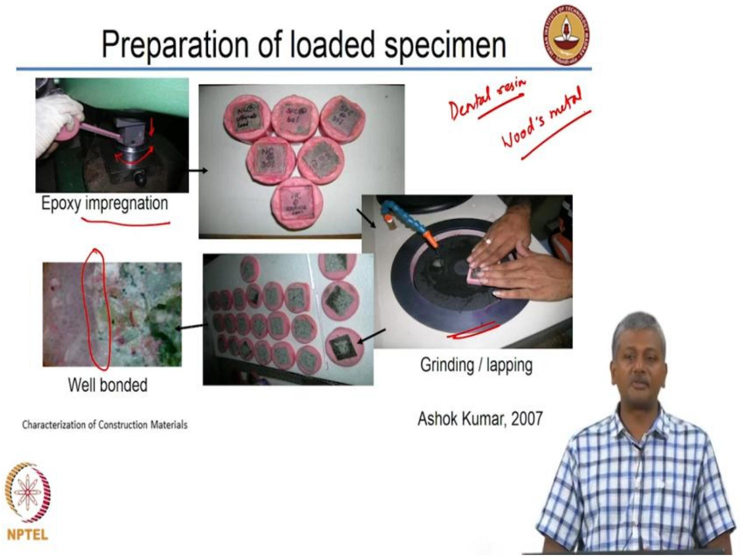

Preparation of loaded specimen

Dental resin

Wood's metal

Epoxy impregnation

Well bonded

Grinding / lapping

Ashok Kumar, 2007

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NPTEL

Now, in some cases, people have also tried to investigate how the structure of the material behaves or changes when loading is applied to the material. So, in this case, we were trying to investigate concrete cubes that were loaded in uniaxial compression, and we wanted to preserve the cracked state of the cube at different stages of loading, the idea was to look at how cracking progresses inside concrete with different stages of loading.

So, here, what we did was, as the specimen was being loaded, we had a sleeve which was put around the specimen. And at the particular load, let us say 30% of the ultimate load or 60% of the ultimate load, we stopped the instrument. We did not unload because if you unload what happens in concrete some of the cracks may close, depending upon the state of deterioration in the concrete. So, we wanted to preserve the structure during cracking. So, at that particular load level, we had the sleeve around the concrete specimen. And then we simply poured this epoxy, which cures very fast and in this case we chose this dental resin. If you get a tooth replaced in your jaw, then very often the dentist will use this dental resin to ensure that it sets in place very fast. So it takes only about 5 to 10 minutes to set. Typical epoxies take about few hours to cure, but dental resins set very fast. The only problem with dental resin is that it is a high viscosity material. Now, why is that a problem? Why is high viscosity a problem? High viscosity means it is not going to penetrate into the pores very well, so in this case, our aim was to primarily preserve the structure. But we could have done better with a material which is of much lower viscosity. But the problem was that if you choose that kind of a material it takes longer to cure. And we did not want to actually wait too long because the loading was still on the specimen. So, in this case, we simply impregnated or rather we encapsulated the specimen in the epoxy. So, impregnation is not the term here, we should talk about encapsulation of the specimen and then these were the specimens that were obtained from the epoxy encapsulation.

And then these were sliced and then polished. So, we did grinding and lapping to ensure that we are getting some degree of polish. And when observed under the optical microscope, you see that the bonding between the epoxy and the concrete is quite good. There is no gap, that means, it seems to have encapsulated the concrete pretty well. Whether it kept the cracked structure preserved or not is debatable, but at least it ensured that complete unloading was not possible while the epoxy encapsulation was on. So because the epoxy gets hardened, when the load is still on, you do not expect the unloading to happen. If the epoxy is left to cure for too long, and then you unload, it is possible that some cracks may close. So we will come back to how this was actually analyzed a little bit later. But this is how we actually prepare the specimen under load.

So we are talking about polymers and polymers do tend to creep significantly and the concrete also would be creeping at the time of maintaining the load. But we are only talking about 5 to 10 minutes. So because of that creep, there may be incremental changes in the structure still, you may have increased strains, but then it is only for 5 to 10 minutes which is why we need this resin to actually set very quickly.

There are other techniques also people have actually used, something called ‘Wood’s metal’. If you do a search for this in research papers, you will see that people have actually investigated same kind of experiment with Wood’s metal, which has a much lower viscosity and it is able to penetrate the material to a much better degree, and hold the specimen intact and prevent it from failing.

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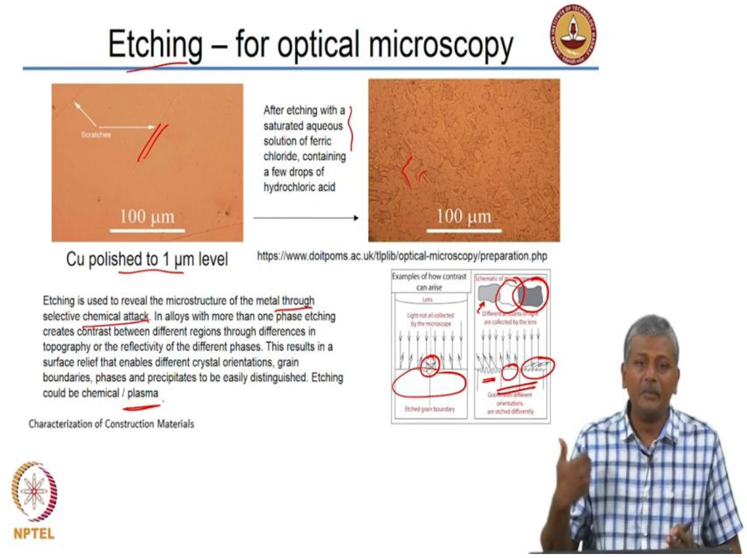

Etching – for optical microscopy

After etching with a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid

Cu polished to 1 µm level

https://www.doitpoms.ac.uk/tlplib/optical-microscopy/preparation.php

Etching is used to reveal the microstructure of the metal through selective chemical attack. In alloys with more than one phase etching creates contrast between different regions through differences in topography or the reflectivity of the different phases. This results in a surface relief that enables different crystal orientations, grain boundaries, phases and precipitates to be easily distinguished. Etching could be chemical / plasma

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In some cases, when you do optical microscopy, you may not get sufficient contrast by just the process of polishing and preparation of the sample. You may need to do something beyond polishing to ensure that you are able to reveal the contrast that exists between the grains of the material and this process of revealing the contrast by doing chemical attack is known as etching.

So, etching is basically the revealing of the microstructure of the material through selective chemical attack. Now for instance, if your material, like even a metal for instance, this is actually the image of a copper polished to 1 µm level. And this is the image of the copper as taken directly under the optical microscope; you see some scratches here from the polishing. So, those scratches are from polishing. Those are not really grain boundaries or defects in the material, those are simply scratches that have been induced by polishing.

Now, the same material, after you etch it with a saturated aqueous solution of ferric chloride containing some hydrochloric acid, let's see what happens to the same material. What has happened here is, the chemical seems to have attacked some locations much more than the others, thus producing these crevices and pits and all that. Those crevices and pits happen to be the boundaries between the different grains of copper that are present on the surface. So, etching has revealed the features of this material significantly.

So just to give you another example of how contrast can arise by etching: if you are talking about a material which has a grain boundary like this, and if you do not have a proper distinction at the grain boundary, a lot of this light that gets reflected from the surface may not actually reach the lens. When you do etching, what you are doing is, maximizing the amount of light that reaches the lens from different types of grains or from different grain boundaries.

So here for example, there are certain cases where the light may get completely scattered and not reach the lens, and those phases will turn out to be dark or black. In locations where you do not have any defects on the surface, where you have almost a planar surface reflecting this light, it goes straight up and your image will look very bright. In the intermediate locations, where the light is getting scattered only by small angles, you may not get the brightest of images but you will get an intermediate gray level. Now because of this, obviously you can have a much better contrast in your surface as opposed to how you will have without any etching that is being performed on the specimen. So, etching can be done either by chemicals or etching can also be done through plasma; basically in plasma, we have a very high, sort of an ionic cloud which comes and mills the specimen or sort of attacks specific phases on the surface of the specimen.

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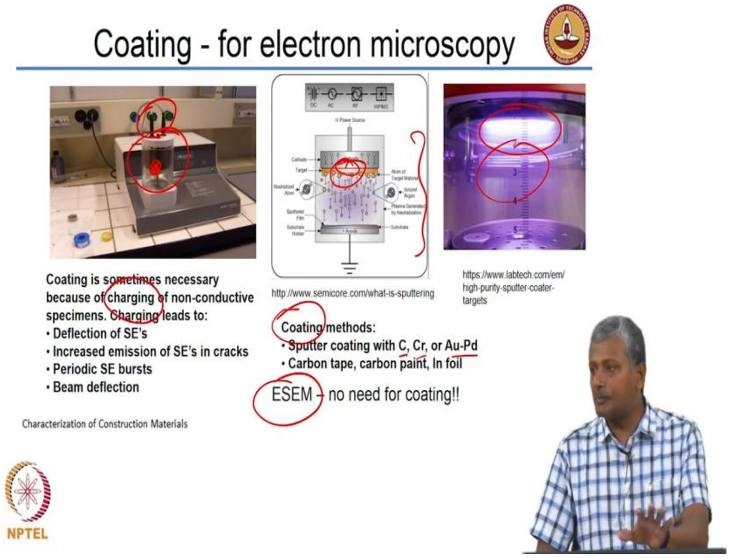

Coating - for electron microscopy

Coating is sometimes necessary because of charging of non-conductive specimens. Charging leads to:
- Deflection of SE's
- Increased emission of SE's in cracks
- Periodic SE bursts
- Beam deflection

http://www.semicore.com/what-is-sputtering

https://www.labtech.com/em/high-purity-sputter-coater-targets

Coating methods:
- Sputter coating with C, Cr, or Au-Pd
- Carbon tape, carbon paint, In foil

ESEM → no need for coating!!

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And another step, in some cases is that of coating. Now when we're dealing with metals like steel or aluminium or copper, imaging these in electron microscopy is quite easy because these electrons can easily interact with the sample, get reflected or get absorbed or get scattered and so on and so forth. And you do not really have a buildup of the electrons on the surface because the surface is conductive. So the electrons fall on metal, they will get conducted and simply go down the metallic parts of your scanning electron microscope. If you have a non-conductive material like a biological material or a ceramic or a construction material, most construction materials are not conductive, so when the electron beam falls on the surface, because it cannot conduct this electron beam anymore, you can have a buildup of the electrons on the surface.

So, what will happen as a result of that is the resultant image that you see will be completely white, because electrons are getting charged on top of the specimen. So we basically call this issue as ‘charging’. Charging is the accumulation of the electrons on top of the specimen which is non-conductive. And charging can lead to all kinds of problems in imaging like deflection of your secondary electrons, beam deflection and so on and so forth. We will discuss this later when we actually look at what these secondary electrons are.

So, to avoid this, what we need to do is at least make the surface of this material conductive. So, if you are dealing with asphalt concrete or cement concrete and you want to image it in the scanning electron microscope, you need to at least make the surface conductive.

This problem is not there with light obviously, because any material would reflect light or any material depending on how translucent or opaque it is, will transmit some light and reflect some light. But in the case of electrons, only the conductive materials will be able to take the electrons through a certain path, the non-conductive material will lead to a charging or collection of the electrons on the top. So, to avoid that, we need to make the surface conductive and this is done by a coating. So you can do coating with several different techniques. All you need to do is coat your non-conductive material with a film of conductive material. But of course, this film should not be something that covers the surface, it should be something that simply makes the surface conductive. That means, we are only depositing an atomic or an ionic layer of this material on the surface.

So, common conductive materials that we typically use are carbon, chromium or mixture of gold and palladium (Au-Pd), you can see in this picture, you have the cloud of Au-Pd which has been made to ionize with the help of very high potential difference. So, again the whole process is actually depicted in this case. Typically, what happens is when you put your specimen inside this chamber, you then evacuate the chamber. You have a very high vacuum present inside. And under this high vacuum, you apply a very high potential difference between these 2 electrodes, which causes the target metal (labeled in the Figure in slide), like carbon, chromium or Gold-Palladium (Au-Pd) to start releasing the ionic species from this material as a cloud and this cloud goes and starts depositing on top of the specimen.

So, actually when you observe a specimen of scanning electron microscopy, which has been coated, you can distinctly see some shine or color on the surface depending upon the type of material that is coated. If you look at a carbon-coated specimen, you will see that the surface becomes slightly dark. If you see a Gold-Palladium-coated specimen, there will be a pinkish or violetish tinge on the surface of the specimen. That is essentially just that a layer of atoms of these materials has been deposited on top and these atoms have been made to get ejected from the specimen or from the target metal, that is either carbon or gold palladium, by applying a very high potential difference. So that is called Sputtering; Sputter coating which ensures that you are able to cover the surface of your specimen which is non-conductive with a layer of these conductive atoms.

With this, you are able to now take the charged electrons which come to the surface and transmit them around the specimen down the other parts of the microscope and the electrons do not get charged on the surface, all you get are the interactions that come out from the surface which we are trying to image in scanning electron microscopy.

Now in certain cases, like in Environmental Scanning Electron Microscopy (ESEM), we do not need coating. Now, what is ESEM? It is SEM that is done at normal atmospheric pressures or slightly lower than normal atmospheric pressures. In most cases, scanning electron microscopy, we have to obtain a very high vacuum inside, to ensure that our electron beam goes straight down and interacts with the specimen. But in some cases in Environmental SEM, ESEMs, you may actually have the electrons directly interacting with the specimen without the need for a very high vacuum to be maintained. In such cases, you may not need coating. So, in several cases of biological samples when you want to actually study the structure intact of these biological components, you can actually go with Environmental SEM because many of your biological samples also have some liquid in them. Whenever we were dealing with high vacuum, we cannot use a liquid material because when you try to evacuate, all the liquid will vaporize and then you will have a problem with clogging of all your detectors and all the exits within the microscope. So, because of that for biological samples which are wet, you can apply Environmental Scanning Electron Microscopy, in such cases you do not need to do coating. You cannot actually do coating also for some objects which are wet. Even here, as I said in this chamber in which coating is getting done, you are applying a very high vacuum. So, you cannot have objects which are wet, you need to ensure that objects are completely dry.

Now, drying of the objects is something that you may have learned previously. When we talked about cementitious materials, Dr.Piyush would have mentioned to you that to prepare cementitious materials for most of the techniques, we need to stop the hydration or remove the moisture that is present inside the sample. This can be done in several ways, you can either oven dry the sample or you can do a freeze-drying of the sample or you can do a solvent exchange in which case all the water comes out and the solvent volatilizes. So, only dry samples are used for testing as far as microscopy is concerned, unless you go for Environmental Scanning Electron Microscopy where you can even use wet samples. So we will stop with that and move on to optical microscopy in the next class. Thank you.