Characterization of Construction Materials
Prof. Manu Santhanam
Department of Civil Engineering
Indian Institute of Technology – Madras

Lecture - 37
Types of Optical Microscopy - Part 2

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Now the objective of imaging is to ensure that you are able to observe different phases clearly in an image. So, how does contrast and illumination come about in the case of microscopic evaluation? So, different degrees of absorption can produce differences in brightness. So when you are talking about transmission light microscopy, the light will get absorbed to different degrees by different phases. And light transmitted, obviously would depend on how dense the phases are, if the phase is very dense, it will absorb more light, if the phases light or porous, then it will permit more light to pass through.

Colour contrast happens when selective wavelengths are absorbed. So for example, the light is passing through the specimen, because of the kind of material it is, it may be absorbing higher or lower wavelengths or intermediate wavelengths depending upon the type of phase that that is there in your sample. So because of that you may get differences in the colour contrast.

Phase contrast happens when there is a shift in phase of light. Now, what do you mean by phase? When we plot the wave nature of light, incoming wave into the sample, and the wave that comes out of the sample may not be perfectly in sync. So that is basically the phase contrast.

And fluorescence contrast happens when incident light is absorbed and partially re-emitted at a different wavelength. So incident light gets absorbed by the sample. And then the sample itself emits a different colour of light or different wavelength of light and that is captured by your image. That is basically a fluorescence contrast. So, fluorescence microscopy is another set of techniques that can be employed for much better quality imaging, but we are not going to be talking about that in this case.

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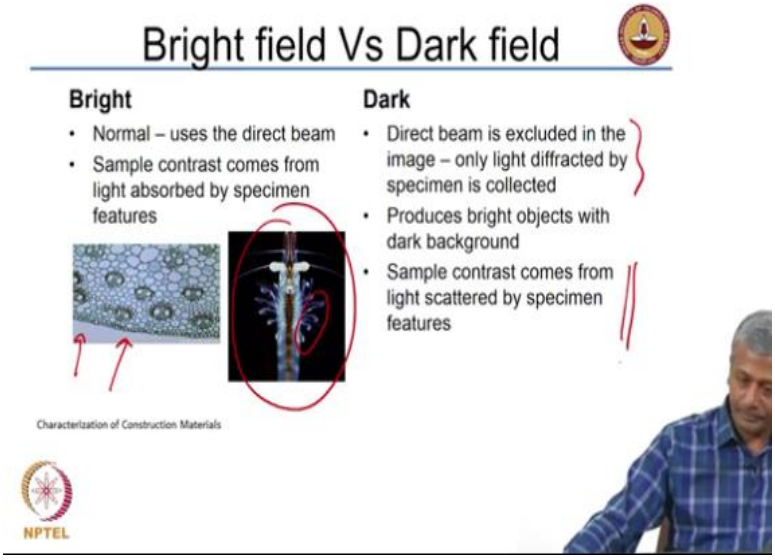

Now in biological assessment of samples and several times in transmitted light imaging, you can do a bright field or a dark field imaging. So what do you mean by bright and dark field? So when you have a specimen passing light through it, and collecting into the other side, you have the beam that is directly going through, you also have the beam that is getting diffracted from the sample. There are direct beams going and there are diffracted beams. So, what the bright field does is that it does the imaging by collecting the direct beam and the sample contrast comes from the light absorbed by the specimen features. So, different parts of the specimen will absorb different amounts of light and you get an image like this. That is again a biological sample and

you can see depending upon on the phase that is present in this sample you are getting different levels of contrast between the different phases.

Now in certain cases, you want to accentuate certain features of the object. So what you do is you go for the dark field imaging where the direct beam is excluded in the image and only light diffracted by the specimen is collected. That is called dark field imaging. In case of a dark field, you get an image like this (Image on right in slide). Look at how sharp the features of this insect are. It produces bright objects while the background becomes completely dark. In the case of a bright field imaging, the background is completely white. So if you have to image something like this, which has extremely fine features, these legs are extremely fine in this case, if you have to image that, you need to cut off the background completely. So because of that, what we do is we completely cut off the direct beam of light and only use the diffracted beam and the sample contrast obviously comes from how much scattering occurs from the different features of the sample. So, again you need to be clear about when you want to do one type of imaging over the other. So, for biological imaging, a lot of times this dark field imaging is preferred.

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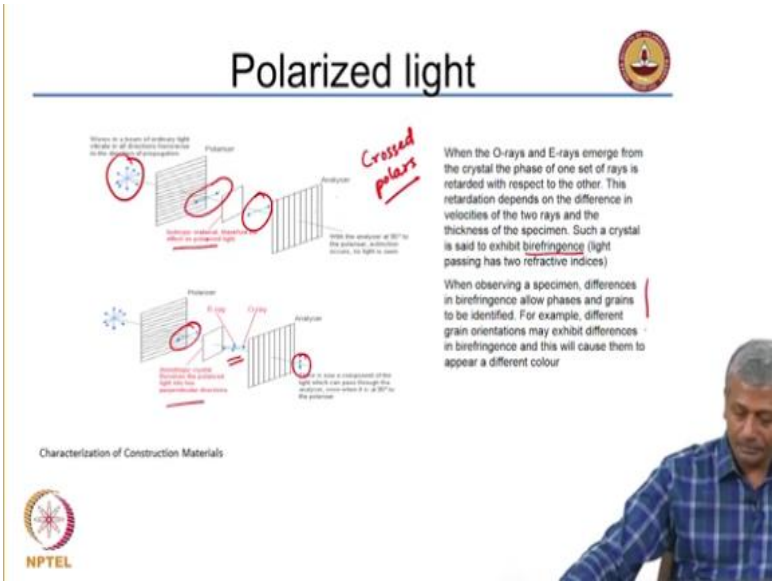

Once again, light has a dual nature - wave nature and a particle nature. Particles of light vibrate in all directions. A polarizer is a series of slits which cuts off the vibration in all other directions and has only one specific direction of vibration. When this light passes through an isotropic material, it does not have any effect on the polarized light, so it comes out just like that. And if

you have the analyzer, which is nothing but the polarizer, and you can keep it at different angles. So, in this case the analyzer is kept in a perpendicular direction. So, this is called crossed polars. So, here the polarizer and analyzer are kept perpendicular. So, what happens is this beam is coming and vibrating in this direction while the analyzer has slits in this direction (Refer figure on top in slide). So, this beam gets completely cut off now.

Now, what happens is if you have an anisotropic material which obviously does not have the same property in all directions. As a result of that, the incoming polarized beam is split into 2 beams or vibration in 2 directions. So, when this comes through the analyzer, one of the directions gets cut off and only one remains. So, when you do a crossed polar imaging in the case of an anisotropic material, you will be able to get some degree of contrast based upon how easily your beam is able to get resolved in 2 different directions. And that depends on a characteristic of the sample called by the birefringence. That means the light passing through such samples now has 2 refractive indices in 2 different directions. So what happens is when observing a specimen, differences in birefringence allow phases and grains to be identified. Without doing the crossed polars, you may not be able to produce the same level of contrast between phases that you will get when you have crossed polars.

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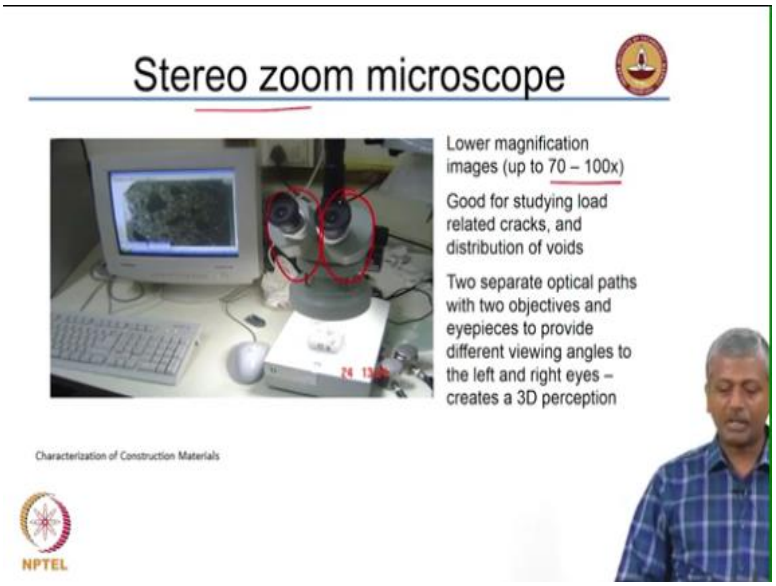

Now apart from the reflected and transmitted light microscopy, there is a special type of reflected light microscopy technique which is called the stereo zoom microscopy. And these are basically

low magnification, almost a simple extension of your camera but then with a slightly better ability up to 70 to 100 times can be magnified using the stereo zoom microscope. It is basically good to study macro defect features like cracks and voids in your sample. I will show you some examples later on.

So again, 2 separate optical paths with 2 objectives and eyepieces can provide different viewing angles to the left and right eyes. So what happens as a result of this is because your eye each eye is now going through a different set of eyepiece and objective lenses, you almost get a perception of a 3-dimensional image. Basically, this is how stereographic projection is done, which is responsible for capturing 3D films. In 3D films also, you have 2 different cameras at different angles capturing the same effect and the resultant effect on the eyes, it appears as if it is a 3D sort of a film, so that same thing happens in the stereo zoom microscopy, in which case both the optical paths are separate into the eye and gives a sort of a 3 dimensional feature to the specimen that you observe.

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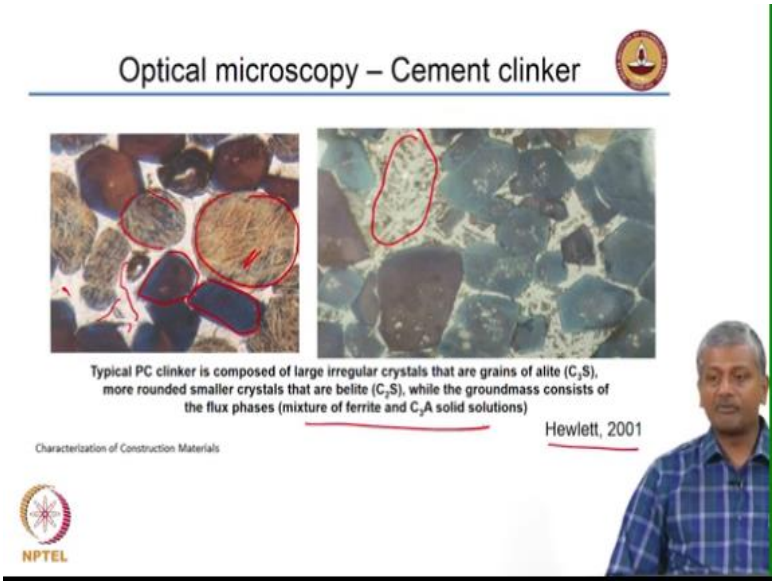

So, I will show you some examples of optical microscopy from construction materials. So, this is a very common image that you find in cement chemistry books. This one is actually from the chemistry of cement Lea's Chemistry of Cement, which has been rewritten by several authors, the editors PC Hewlett and this shows a typical Portland cement clinker. There are 2 different clinkers that are shown in this image. On the left you have a clinker that is composed of irregular

crystals. These irregular crystals are those of alite and the rounded crystals which show some striations on top, you can see the striations here, the rounded crystals with striations are belite crystals.

The material that is surrounding this, that is the white, and in the case of the image on the right, you can see streaks of white and brown, the material that surrounds these crystals is the ground mass. It is also crystalline, but the crystals are too small to be perfectly imaged. So, that groundmass is a mixture of ferrite and C_3A solid solutions.

So, this is reflected light microscopy. So, in terms of reflectivity, what phases would be more reflective? The denser phases will be more reflective. So, here the calcium aluminates, especially the calcium aluminoferrites - the white ones are all calcium aluminoferrites, those are more reflective. They are actually reflecting a lot more. This image has also been done after preparing this polished sample of the clinker and then etching it, because only with etching, you can be able to actually distinguish your grains in such a clear fashion.

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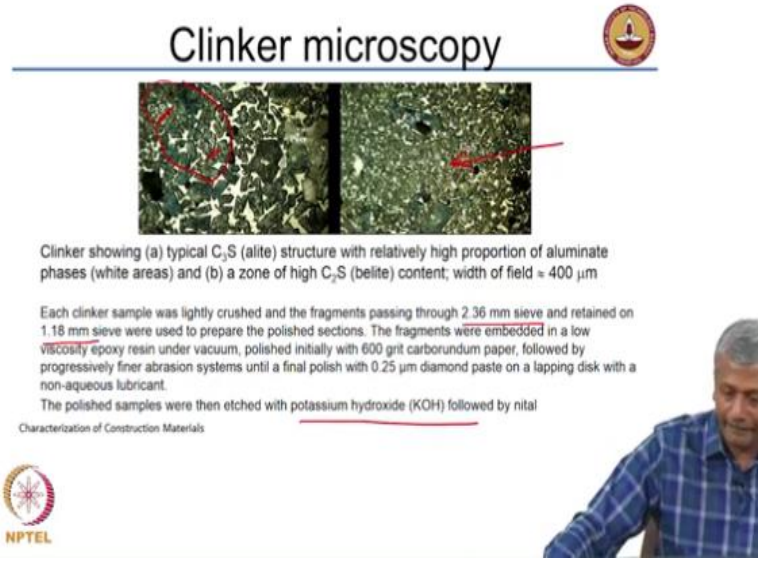

One more example here from one of our own studies, where we collected cement clinkers from different cement plants and prepared these flat surfaces subjected that to etching, where the etching is with potassium hydroxide followed by nital. So, in this case, we took material that was passing through 2.36 mm and retained on 1.18 mm sieve. So, these chunks of clinker were fairly

large about 1.18 to 2.36 millimeter in size and then we embedded them in epoxy, made a cut and then polished it. We got the right amount of polish to be able to distinguish the features - you can see C_2S, C_3S and the white ground mass in between.

One feature that was interesting for us in this study, just to tell you where the study actually has an application, we wanted to understand why does one cement lead to greater heat production as compared to the other. So one obvious reason is obviously the compositional contrast may be different, you may have more C_3S as compared to C_2S. But in these 2 clinkers, we actually saw that there was not too much difference in the C_3S and C_2S contents. But what we saw was, there were such large chunks of C_2S that were sitting all together. And it is very difficult for all these chunky or clustered C_2S to actually completely start hydrating. So there was a lot of heat difference because of the way that the phases were actually distributed. It is not just the amount of the phases that was important, but the morphology and the distribution of the phases also led to increase or decrease in the amount of heat that was created.

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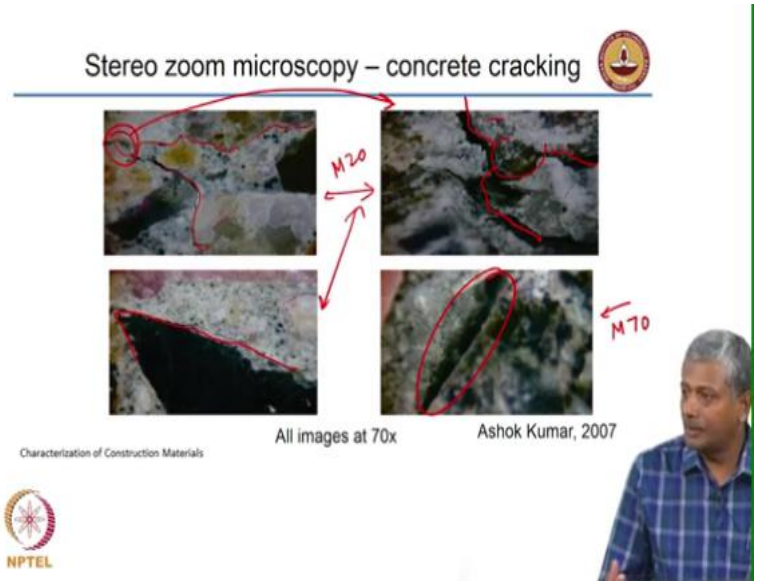

I had showed you earlier that we had prepared some concrete specimens, which were under load, and were encapsulated by the dental resin as an epoxy. So if this was the direction of loading, we slice these specimens in the perpendicular direction and polished it and observed that slice. So now please remember when you are loading something in compression is going to create more or less vertical cracking. If you avoid the friction between the top and bottom, between the platen

and the specimen, it will almost create a vertical crack. So what we are capturing by taking a slice like this is the path of those cracks, we are not actually getting the full length of the crack, but we are capturing the projection of those cracks in horizontal plane, so that is what is being seen here.

The 2 images on top and bottom-left image are all belonging to concrete of grade M20. And the bottom-right image is for concrete of grade M70. Now we know from theory that in low grade concrete, there will be a lot more cracking, there will be a slow and steady failure. Whereas in high grade concrete, the cracking is sudden, the aggregates start cracking, in the case of low grade concrete the interface between the aggregate and paste will start cracking.

So, here you see, look at the extent of this crack, this crack actually starts somewhere here, it goes around the aggregate around this aggregate and so, on. The same thing happens, crack again follows the path, which is basically merging of the interfaces of different sorts of an aggregate. So, here again there is one more crack which is going around the aggregate. Again, this is actually a close up image of this part here which shows the branching. So, you have this crack which is branching out in different directions. So, because of the extensive crack branching in low strength or moderate strength concrete you get actually a quasi-ductile sort of a failure. You do not get a sudden failure. In the case of a high strength concrete, this crack is going through the aggregate in this case and that leads to a sudden failure of your sample. But there is no epoxy in it, because as I discussed earlier the epoxy was of high viscosity and could not impregnate the specimen well enough. So, epoxy also is a low density phase. So, this is a reflected light image. So, epoxy also will show up to be dark, just like the pores.

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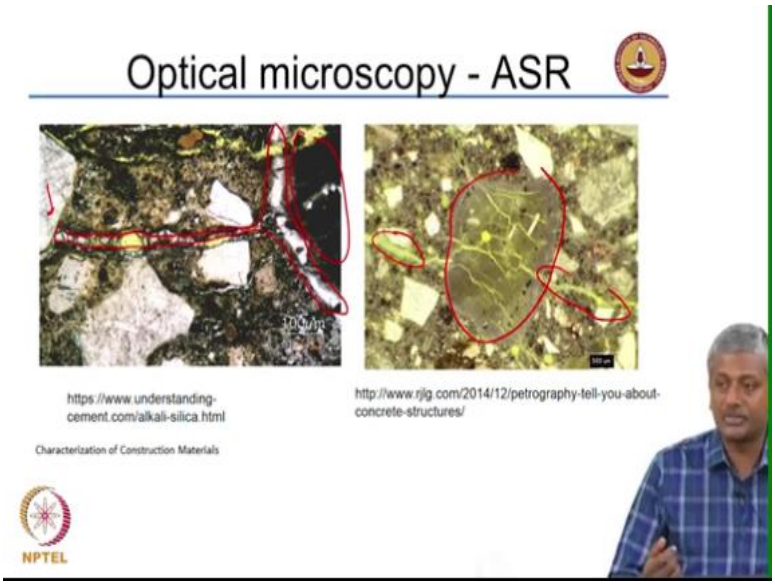

Now, this is an optical microscopy image of alkali-silica reactivity. So, alkali-silica reaction produces a reaction between the alkalies which can come from the cement primarily and from the other sources of materials inside the concrete and reactive siliceous aggregate and that leads to produce a production of alkali-silica gel which absorbs moisture and expands.

So, you see this entire deposit here of white (image on right) that is basically the alkali-silica gel, which is formed primarily from the aggregate. From the aggregate this gel is forming and then starting to expand and such expansions lead to cracking of your sample here (as shown in the Figure on the right). So, this is an aggregate which has got completely cracked because of the alkali-silica gel, which is expanding.

In this case, you can see the gel is almost yellow in colour. So, they actually did this with a fluorescent dye, they were able to produce this with a fluorescent dye. So, all the cracks appearing yellow is because the gel is present the cracks and they have added a yellow fluorescent dye which can be used for imaging quite easily. This is also an aggregate phase here.

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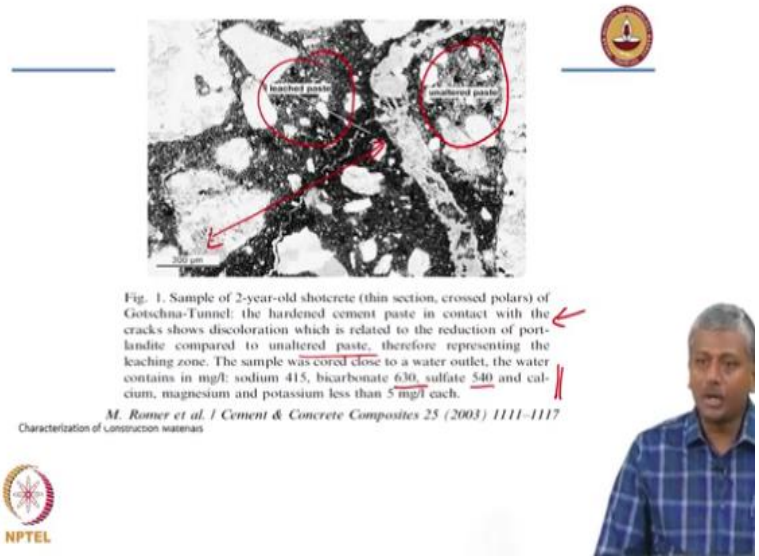

Now, this is an image of shotcrete from a tunnel which was subjected to sulphate attack, this tunnel is in Switzerland. And what we see here is that, of course, the image is not very clear, but you can see that this concrete lining from the tunnel has different zones. On the right, you have an unaltered paste zone and on the left you have a leached paste zone. Now, of course, how do you make out the difference - the difference is primarily because of the difference in darkness of the altered paste zone, you can see the leached paste.

So, hardened cement paste in contact with cracks shows discoloration, which is related to the reduction in portlandite, which is calcium hydroxide. So sulphates enter, they will react first with calcium hydroxide and consume the calcium hydroxide phases. And the unaltered paste on the right side, so this (left side) basically is the leaching zone. So all the zone where the water with sulphates is entering the concrete is leading to a depletion of the mineralogical phases from the cement. And of course, please remember this is not really a concentrated sulphate solution, this has bicarbonate and sulphates, which are not too large. This is in milligrams per liter or parts per million. This is not a very concentrated solution, but even water with such low levels of sulphates and bicarbonates can slowly tend to leach out your phases from the cementitious matrix.

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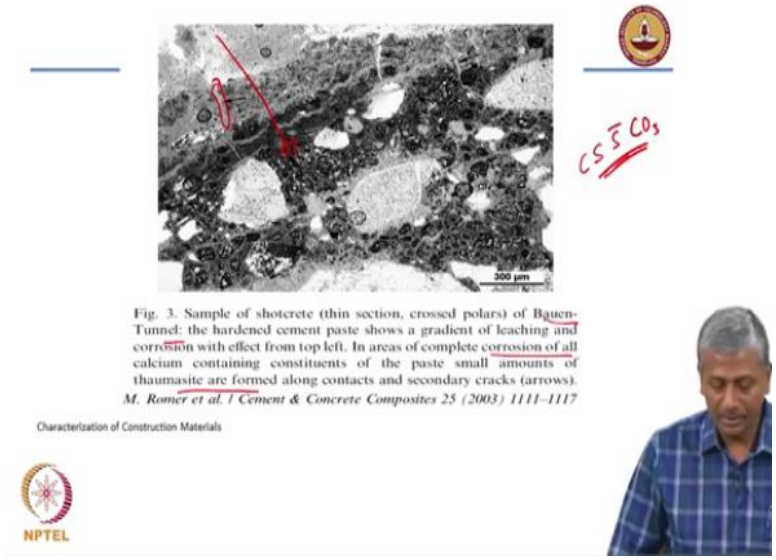

Again, this is from another tunnel in the same location, in Switzerland. The hardened cement paste shows a gradient of leaching and corrosion with effect from top left, you can see how the grey level changes from the top. The light grey regions are where you do not really see much alteration but the dark grey regions are where you are seeing a lot of alterations. So in areas of complete corrosion of all calcium containing constituents, small amounts of thaumasite are formed along the contacts and secondary cracks. So, thaumasite is a phase basically there is forming in such locations here (shown in figure). So, thaumasite is basically a calcium silicate sulphate carbonate ($CS\bar{S}CO_3$). It happens when sulphate attack happens in the presence of carbonate species. So, you get this sort of a thaumasite formation in this case.

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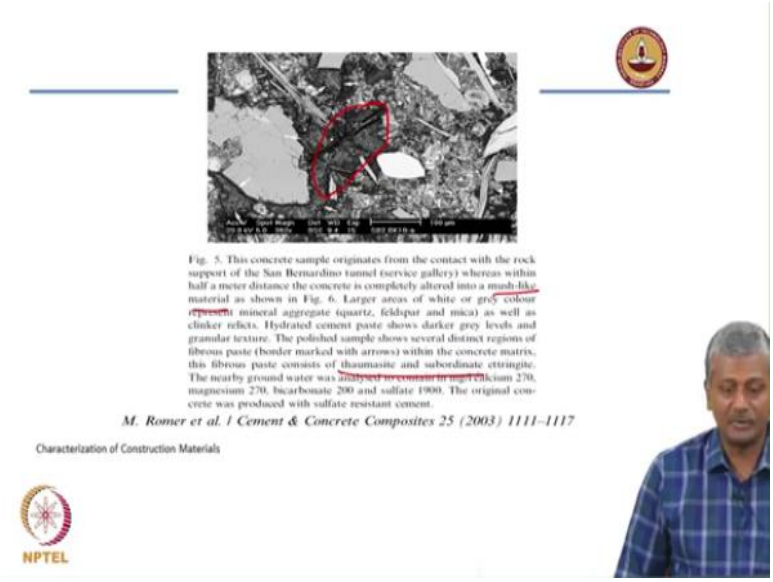

Again from the same series of specimens, but in this case of course, you see a much clearer image. So, again you see here, this entire area is converted into a mush-like material and this mush-like material was later found to be thaumasite and ettringite that was there in the sample

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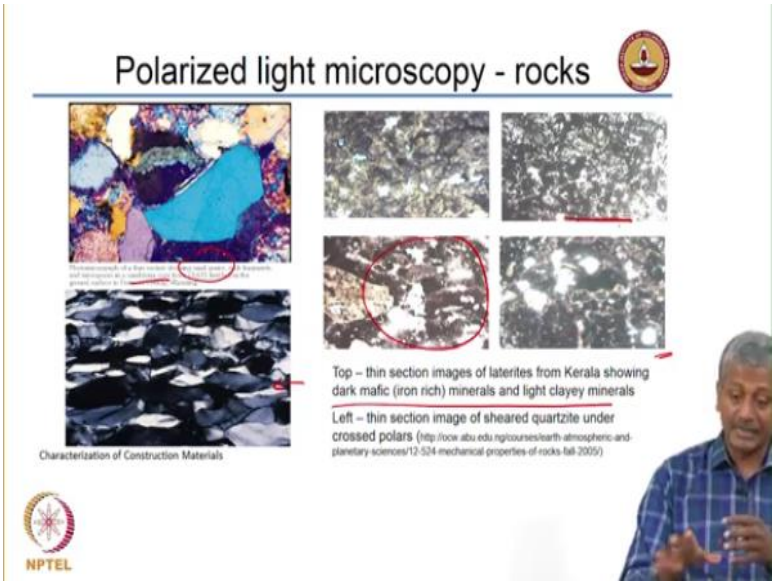

Now, very often polarized light microscopy is used successfully for rocks and here you can see why it remains to be such a useful technique. So, on the top left you have a thin section showing sand grains and rock fragments in a sandstone core. So, they took this sandstone core from 13,000 feet below the ground surface. So, you can imagine 30,000 feet below the ground surface the pressure would be significantly high that could have fused all the ingredients together.

And you can see the different phases very clearly. This is polarized light microscopy, where the light is coming through the sample, which is a thin section. So because of the differences in the extent of absorption of light and also the preferential absorption of certain lights, we obtain different colour contrast because other wavelengths of light are actually transmitted through.

Now, this is the case where thin section image is taken for sheared quartzite under cross polars (Left image). So, you can again see when you shear quartzite mineral and you observe under polarized light microscopy with a cross polar, you are able to make out the grain features after the fracture that has happened.

And this is an example from our own study here at IIT Madras (images on top), where we were trying to understand the nature of laterites from different quarries. So, here these are thin section images of laterites from Kerala showing the type of mineral phases inside laterite. So, we have dark iron-rich minerals and light clayey minerals. Of course, this is light that is passing through. So, the light lighter phases will let more of the light pass through; darker phases will be absorbing more light. So, you can see here, these are quarries that are located within a distance of 20 to 30 kilometers from each other, but look at the differences of the mineral composition that you get. In this picture (bottom-right) for example, you get a lot of dark phases and less of white phases. In this one (top bottom-left) you have an irregular distribution of white phases. So, here you have more of the white phase (top-right) evenly distributed across the matrix.

Now, when you look at the material characteristics on the macro scale, you see that the strength was highest in those cases where the clayey fractions were the least and where the dark iron-rich fractions were more. So laterite is a material which basically continues to evolve as more and more leaching happens of the silica and the alumina and enrichment of the iron takes place over time. And because of that, induration or in-place gain in strength of the laterite can happen and that leads to differences in the performance of the laterite with respect to mechanical characteristics, and that can be actually looked at very clearly under the microscope to get an estimate of what different phases are actually forming here.

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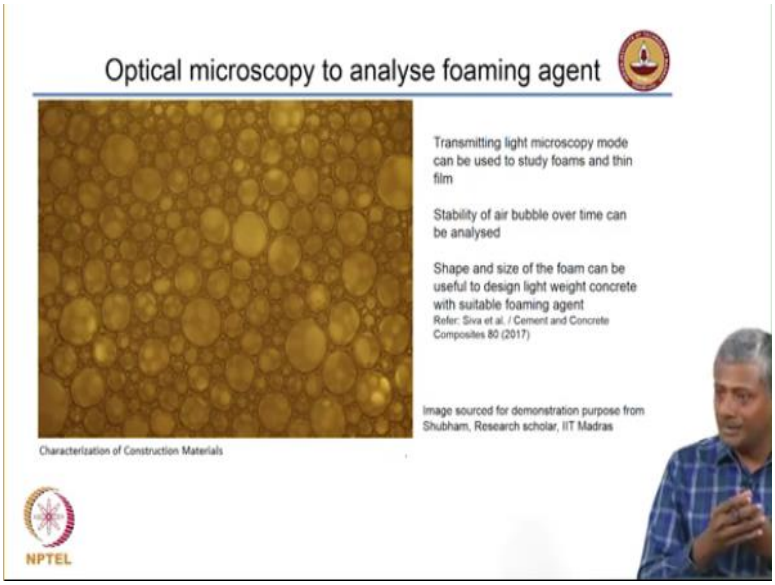

So, again one more picture of optical microscopy to analyze a foaming agent. So, here foam is actually used to produce foamed concrete and foam is generated from sodium lauryl sulphate or natural derivatives of extracts of trees and so on. So, transmitted light microscopy image is used here to study foams and thin film. So, on a glass slide this entire foam is actually spread out and then imaged under transmitted light.

So, what happens is, with time some of these bubbles will start to disappear, i.e., collapse. But what we want as far as foamed concrete is concerned is, when we mix these bubbles along with the cement paste and cement mortar, the bubbles should be retained to ensure that the foam forms a complete porous structure inside the sample. But foam stability can sometimes be questionable if you do not use the ingredient for foaming and sometimes when the right conditions do not exist, which are generated by a foam generator. So, the stability of the air bubble over time can be analyzed using this sort of an experiment. So here this is a study which was conducted in our own lab, where shape and size of the foams was used to design lightweight concrete with a suitable foaming agent.

So, we have looked at different sorts of examples from optical microscopy. One thing that was common is that we could only go to a certain extent as far as resolution was concerned, because of the limits of the visible light in terms of its wavelength. So, this will lead us in the

next lecture into scanning electron microscopy where we can actually get much better resolutions and use it for a wide variety of studies. Thank you