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Lecture-9

Materials Characterization
Fundamentals of Optical microscopy

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Hello everyone! Welcome back to this material characterization course in the last class we just looked at the last variants of the optical microscopy and in this class I would like to give you a complete recipe of the material preparation for the optical micro metallography are microscopic analysis. it is kind of an a complete personal Person specific, about the quality of the sample preparation we have a set of guidelines with which we can go through most of the preparations which I am going to discuss in this class about metals and alloys.

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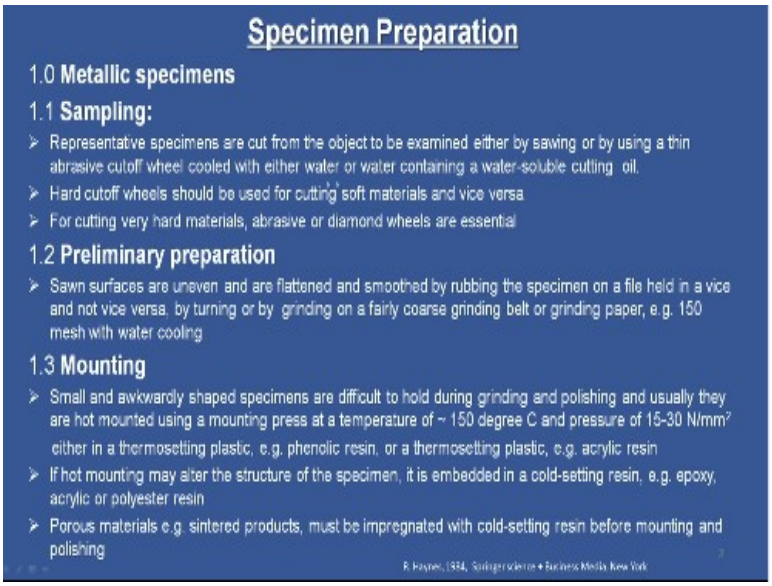

And then I will also give you some kind of guidelines for the ceramics as well as the polymeric materials. So let us look at this the initial remarks if you look at this we will just go through the procedures for metallic specimens : the first step is sampling. This is very important because it should be a representative of what we are talking; about so the representative specimens are cut from the object to be examined either by sawing or by using a thin abrasive cut off wheel cooled with either water or water containing water soluble cutting oil.

You see this is now completely standardized you get a set of recipe from the them the equipment supplier whatever you want to purchase in order to make the uniform sampling; that is if you buy a cutting machine you will also get along with the water-soluble cutting oil, so that by doing with adopting this coolant you won't make any structural changes in the material. You we all know that the heat produced during the cutting is being controlled by the cooling oil.

So the second important general guideline is hard cutoff wheels should be used for cutting soft material and vice versa; and this is also now standardized you, you by any standard cut off Phil for this purpose a supplier will give you by default what kind of materials we are going to cut

based on that these cut off fields are supplied commercially so these are all well established procedures.

You do not have to really bother about it. For cutting very hard materials abrasive and diamond wheels are essential. So we will see that what kind of abrasive and cut out diamond wheels are used I will also show this the first step is preliminary preparation after sampling ; thus all surfaces are uneven and are flattened and smooth and by rubbing the specimen on a file held on a vice and not vice versa.

By turning or by grinding on a fairly coarse grinding belt or grinding paper for example 150 mesh with water cooling; this is again a similar thing we have to be very careful about the cooling and the sample what we have taken from the object to be prepared preliminarily in this manner. Then we talked about mounting small and awkwardly shaped specimens are difficult to hold during grinding and polishing; and usually they are hot mounted using amounting press at a temperature of $1050^{O}C$ under pressure of fifteen to thirty newton per mm square either in a thermosetting plastic that is phenolic resin or thermosetting plastic.

Example acrylic resin. If hot mounting may alter the structure of the specimen it is embedded in a cold sitting resin for example epoxy acrylic or polyester resin. And porous materials for examples sintered products must be impregnated with cold setting resin before mounting and polishing. See most of the solid objects like metals and alloys you directly cut from the space I mean object or the material of your interest; but this mounting is mostly preferred for very irregular shaped specimens.

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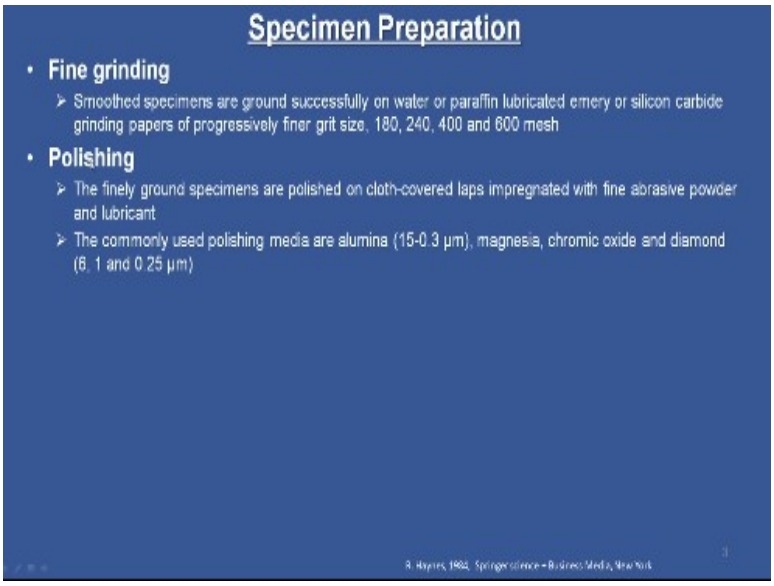

Where you cannot handle with your hand then this mounting technique itself is followed and here itself as he just seen there are two types one is hot bound another is cold malt and I will just show you each of this how it is working in a laboratory as well. And after the mounting we, we see the fine grinding. The smooth specimens are ground successfully on water or paraffin lubricated Emery or silicon carbide grinding papers of progressively finer grit size of 180, 240, 400 and 600 mesh this is for making the surface more finer and finer and then fine polish.

I will just demonstrate in the in the laboratory demonstration as well through videos what are these papers and how we are going to prepare the polished surface then come to the polishing the finely ground specimens are polished on a cloth covered laps impregnated with fine abrasive powder and lubricant the commonly used polishing media are alumina which typically ranges from 15 to 0.3 microns are magnesium chromic oxide.

And diamond which is also very in the particle size from 6,1 and .25 material these are all standard sizes which are commercially available and all this polishing media in general they are available in a variety of range of sizes according to the requirement of the user. So I will also show some live demonstration about how these things are used in the laboratory.

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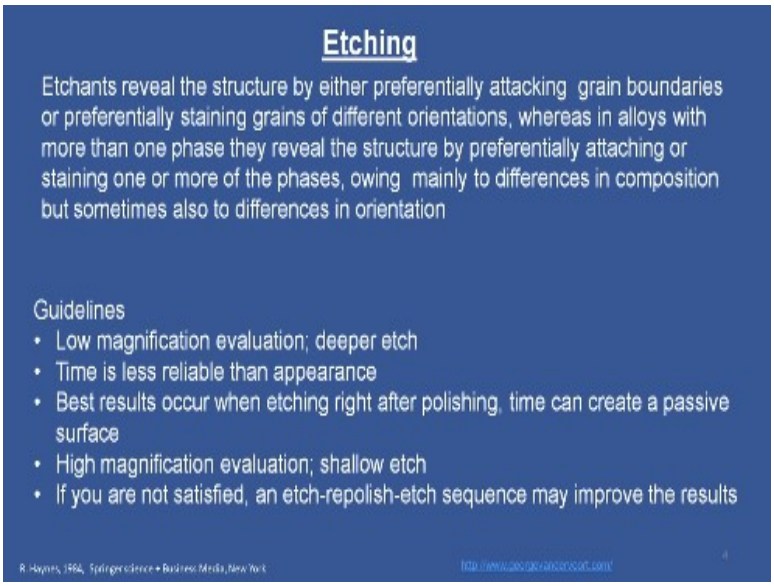

Now comes to an important part of my metallography is called etching. So first we will see what is this etching and then we will go to the guidelines and then how it works. Let me read out the initial remarks : etching reveals the structure by either preferentially attacking the grain boundaries are preferentially staining grains of different orientations where as in alloys with more than one phase they reveal the structure by preference attacking or staining one or more of the faces owing mainly to differences in composition.

But sometimes so due to differences in orientation. So after polishing the specimen with all sort of Emery paper and then polishing media you need to do something called etching only, etching will reveal the structure by attacking preferentially attacking grain boundaries are preferentially staining grains of different orientations. This also we will demonstrate to you how it is being done.

in the laboratory. And it is very important and as I said all these procedures what I just talked about from the beginning to this point it varies from person to person it is like something like a cooking recipe each person will come out with different quality of the sample specimen surface to be examined under the microscope.

So it requires lot and lot of practice and then it is a kind of an expertise one develops to prepare this sample so though you may have a complete guideline, unless otherwise you do with your own hand in a laboratory these things are difficult to be carried out to the perfection so it is better if you are interested you practice it with lot of time spent on it and carefully follow the guidelines.

Okay there are general guidelines given by a George Vander bone we can just go through for this etching for low magnification evaluation deep etch what is deep etch we will see it in after some time, time is less reliable than appearance; best results occur when etching right after polishing time can create a passive surface.

For high magnification evaluation shallow etch if you are not satisfied and etch Polish etch sequence may improve the results. So as I just said you have to just look at the surface examine it whether you are satisfied with the kind of surface you are getting our information you are getting from the specimen you have to redo this exercise.

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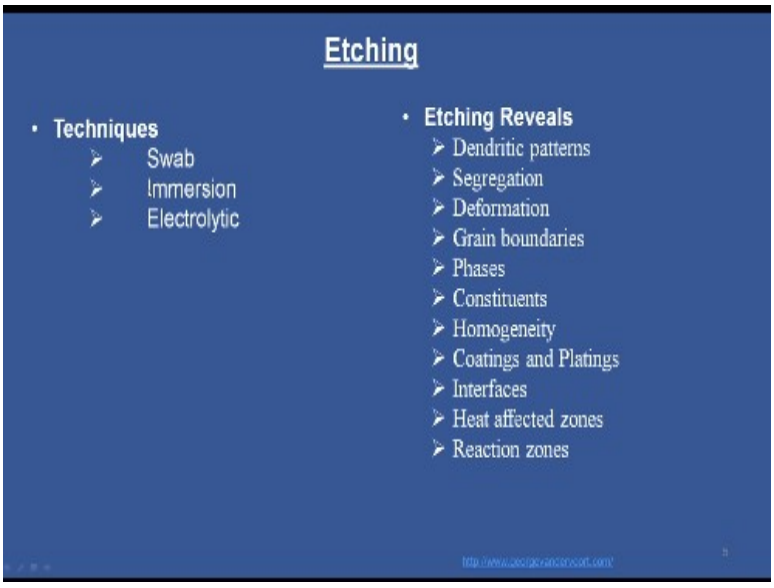

But then for the low magnification the guideline is a deeper etch that means you're etching to be very strong that means you have to allow this etch tends to be reacting with the surface bit longer than normally you do and similarly shallow etch means it should be sorry the time the etchant which spent on the specimen surface to be lower compared to the normal time.

you etch. And there are techniques etching has about three techniques generally either you can swap our immersion or electrolytic so these two are a manually done swab or immersion. We will demonstrate to you how it is done electrolytic polishing is a separate technique, we will also talk about it in an appropriate time when it comes so what does this etching revealed etching reveals dendrite patterns segregation deformation grain boundaries faces constituents homogeneity coatings and platings interfaces heat affected zone reaction zones.
So most of this the, the constituents or the, the information about the microstructure belong to metals and alloys and it need not be the same but depending upon the type of specimens whether it is a biological or a polymeric nature for example if you do etch the polymeric surface you may see a spare lights or boundaries and amorphous and crystalline phase and so on.

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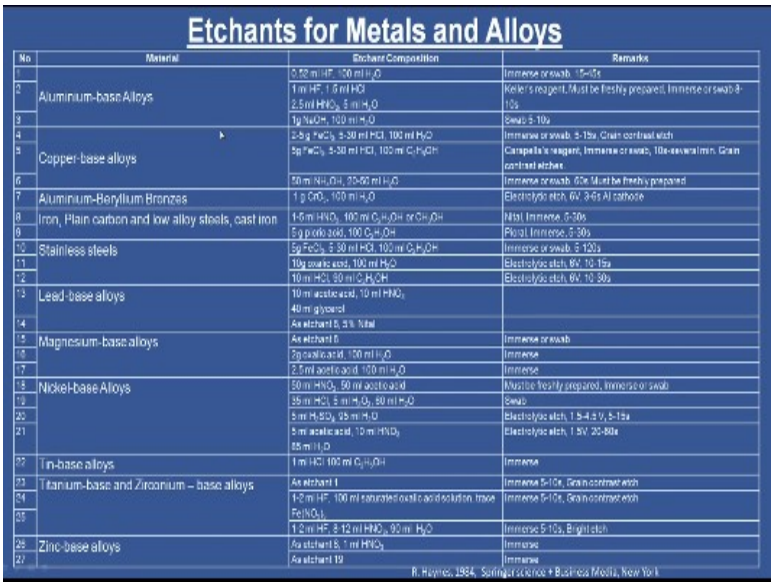

So and also these etch will also will act as sustaining agents sometimes the you can just see the different constituents in the for example polymeric material it will show a different staining I mean staining contrast which you will be able to recognize and then study them. So look at this table you see there is a huge table and it is very difficult to read it in one or two glances but this is what we have to keep in mind for metals and alloys you have variety of choices depending upon the requirement and the availability of your chemicals at you very and so on.

So we have the different alloys listed in this column material column and you have this etchant composition listed against each of this material and then you have the remarks which will tell you what, what kind of action you have to take care so for typically for aluminum base alloys you have the HF in water or HF and HCl and HNO_3 mixture this is typically called as Keller's reagent so must be freshly prepared immerse are swab for 8-10 seconds.

So these steps are quite crucial. So whatever the standard timing which are being recommended for each of this etching action; that means it will produce a normal grain contrastor whatever the constituent TV normal contrast when you talk about deeper etch or a shallow etch, these normal practices should be either exceeded our you should be reduced that time. So if you for example in this condition if you see 8-10 seconds are recommended if you keep it for 15 seconds to 20 seconds then it will become deeper etch or if you keep less than 8-10 seconds it will become shallow etch.

So this just to an example I am talking what is the deeper etch and what is shallow etch and then for example you have a copper-based aluminum beryllium bronzes; all of them will have a different, different etchants and you have ironed plain carbon and low alloy steels cast iron stainless steel lead based alloys magnesium based alloys nickel-based alloys tin based alloys titanium base and zirconium base alloys and zinc based alloys.

So you have a variety of Alloys and metals you, you see that different kind of etching compositions are recommended with a different I mean what kind of technique you use whether you immerse it or so a bit or electrolytic polish these details are also given in this table so I request each or each of one of you to go through this table whatever the material you are

interested and then see the correct modeling etching etchant composition and the, the remarks which is useful to prepare the metallographic specimens.

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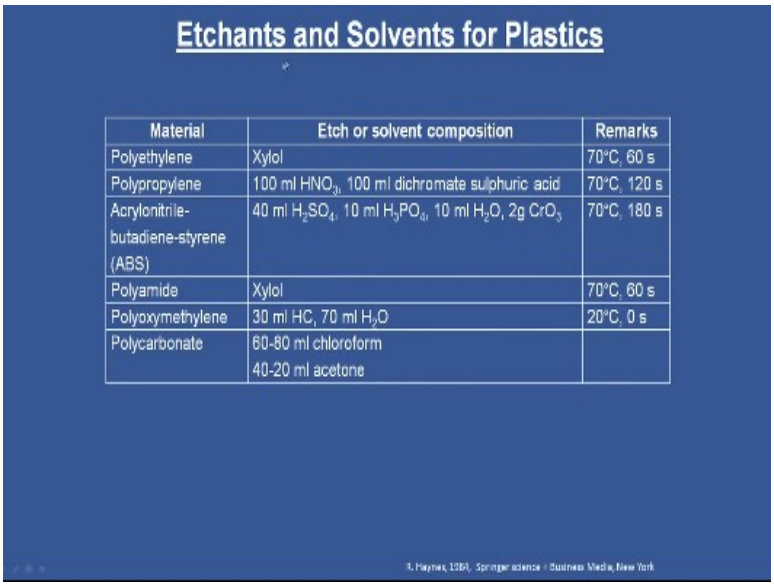

Similarly if you look at the etchant and solvents for the plastics; you have polyethylene polypropylene; acrylonitrile butadiene styrene abs and then you have polyamide polyoxy methylene polycarbonate; for all this you have this solvent composition is given here; and then you have in the remarks column you have the temperature at which these things to be carried out under time up to which this etchants to allowed ; etchants will be allowed to back to the specimen surface and coming to the polymer I am only talking about etching here but some of the sample preparation techniques which are meant exclusively for metals alloys may not suit here for example polymeric materials require something called microtome I will talk about it in a new course.

And then finally the etchings for ceramics you have the material column and it should composition as well as remarks you can see that alone aluminum oxide magnesium ask oxide and then you have silicon dioxide basic re-factories slacks and cement clinker so you have a

typical it change given for each one of this classification and, and the time which is required to etch

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this surface is also given. So now what I do is I just take you to the I will just take you to the lab where we will show you all this sample preparation in so this is a typical cold setting mount I just I talked about in the beginning of the slide you have this cold setting compound and a cold setting liquid the day as I just said they are all polymeric receiving our compound and these are all commercially available setting compacts.

So they do not mark the actual chemistry of chemical composition of this on this bottle but anyway they are all commercially available. So I say just mention this cold setting is used for a irregular shape specimens are specimens with you do not have enough holding thickness or height then you can use this. So this is a kind of amount we one can use this is a typical mount you can also use a PVC pipe set cross section also and here we will just demonstrate how this mounting is done through this take this mold first and then you try to apply grease inside so that the once the mounting is done the, the whole mount mounted sample will come out of this mold so easily.

So this is a typical grease which you can apply to the inside of the mold and it should be uniform and then you see a little bit of greases are kept on the table so that, that surfaces also will become easy to remove when the molding is done. So now you keep your specimen here we take this first you prepare let us prepare and, and then we will just pour this into that mold. You typically take some quantity of powder which depends upon the mold size, and you just mix this liquid core setting liquid and thoroughly stir it.

So that it molding from the paste. So a continuous stirring is required in order to make it uniform paste and then you can keep the sample inside the mold and then pour this mixture. So then this mold is just allowed to set for 30 minutes to an hour and then we can be removed and then sample can be now sample is ready for the further polishing.

So now we will leave that we will look at the other procedure then we will go back to this once it is set. So this is the first grinding : it is a memory paper mounted on the a rotating belt and, and thus on surfaces which I talked about in the beginning of the lecture is being ground on this emery, this is about 150 mesh emery. So you first prepare the, the sawn surface you can see how it is being polished.

Yeah this is how you hold the specimen and you have to be very careful that you know by holding it, it requires practice otherwise your sample will fly away and also you should make sure that you should not hurt your fingers; it requires practice, so you can see that the scratch marks are falling in one direction we will also look at until this the scratch marks under the

microscope how it look like later so once you make this kind of one-directional unidirectional scratches appear in this then you can rotate to 90^{O} and then polish it once again.

So then once that weld grinding is done we will talk we go to the fine grinding this is what I just talked about 180 240 and 300 and 600 and so on. So we have to start with the course Emery paper that is about 180 mesh. So these are all the emery sheets kept in this increasing order of grid size and you can just start from the, the coarser Emery shape that is 180 mesh kind of, of a paper.

So now you will see the same sample will be brought here and then you can see that how it is being polishing is done with the increase in the size of the grid. So now you see that scratch marks which we made out of the belt grinder should be perpendicular to this polishing direction; so you have to rub it like this until all the scratches we have generated in the previous polishing technique.

So you have to do this, this is kind of laborious and boring; you have to bear with this if you want a good surface. So now we can just go to the now you can see that the scratches are just getting away and you start seeing the new scratches in the perpendicular direction to what we have made in the previous grinding you see that the old scratches are going very slowly.

And now we are coming to the next emery paper so like that we can just go one by one. You see that now the surface is becoming much fine and close to uniformly polished region; now we are with 300 and then we will go to 800; and that is the finest polish one can get. So now it is clear that you do not see any you know deep grinding mark which we have generated in the previous emery paper as you progressively go to the finer emery sheets. This is the last sheet there is 800 mesh and after this you see that surface will become almost fine polished condition.

And unless you make sure that when you jump, jump to one paper to the other unless you make sure that the previously generated scratches are removed you are not going to get the a good polished surface : this you have to keep in mind and if it is a labor-intensive process and after this what you should do is you have to take this to a tap water and remove all this debris which

you corrected from the all these; in fact you take a cotton and then swab it like this and make sure that none of the old debris are sticking onto this metal surface.

So now you see that after all this Emery sheet it becomes finely polished and then you can dry it with them air blower or a dryer. Make sure that if each step you, you follow after grinding now you will go for fine polishing so far we have finished a fine grinding now we will go to fine polishing you make sure that the surface is dry and clean from the dust and any other particles.

So now we are now looking at that a disc polisher and what you are now seeing is disc polisher which, which has got a polishing cloth which is mounted on the labs, these are all commercially available machines many suppliers are there; you can see that and these kind of machines with come with a lot of presetting polishing recommendation, and if you are able to adopt those recipes you can follow or you can follow everyone style you see that now the quality thing I mean it is a polishing cloth.

And you can choose the RPM and the kind of settings which you require for this sample and then you start your polishing and then you have this water coolant support is there, which, will be used while we are polishing this sample. So like I said in the beginning of the class for this particular sample we will use alumina which is available in the wide range of particle size, and so here also we will use the alumina with some specific particle size; they are also commercially available.

Then we will show how to do the polishing in the with the polishing. So now we put some powder on this polishing cloth too much is not required in fact this itself an excessive addition this much of polishing powder is not required but you can, can put little less and then let it become uniform on the cloth and then you can also drip the water very slowly and then you have to be very, very careful in holding the sample against this rotating lap and this requires quite a bit of a practice it is not just holding the sample.

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You have to apply some uniform pressure and if you if you are if you are not applying the uniform pressure then the polishing will be one-sided and it will not be throughout the sample you can carefully see that the person who is doing this is rotating the sample while holding this that is just to in order to make sure the uniform polishing all over the surface this rotation is essential and if you do not hold it carefully it will just fly off from this rotating cloth.

You have to be doubly careful about this so you do this for about five minutes you see this is what will happen if you do not hold it properly and you have to be very careful about holding the specimen and then rotate it this requires lot of practice; this is why we intentionally did it to show what kind of mishandling will happen so you have to be extra cautious so once this pulsing is done what you should do is you have again wash it with the running water with the cotton swab it nicely and make sure that all the scratches are removed in fact this is not final polish.

But before that you would like to show the next polisher is diamond this is a is again commercially available, all will get it in all the metallurgical or materials metallography suppliers; and again it has got a different cloth you cannot use the same cloth what we have used for alumina powder you see that the kind of a gun a diamond paste is squeezed out and it is about quarter micron and you have to take it within a very small quantity even this is little higher in my opinion so right and then you can just wipe it on the cloth uniformly and then you have how to just apply some kind of a lubricant which is supplied along with this diamond powder.

So little bit of lubricant and then again you start polishing it. Here again you can choose either a preset recipe are you can choose your own recipe depending upon the RPM and time and so on. So hold this gently again that did be very careful while holding and rotating it otherwise the sample will fly off, and it can cause some accidental also. In between you can apply a little bit of lubricant if you if it required; and you see that again you wash it with running water with a cotton swab and make sure that all the debris is everything is removed from the sides as well.

As the surface of the specimen now you see that sample is almost getting close to a mirror polish so again you yeah it is now dried with an blower you see that it has become a mirror mirror surface so now we will go to etching; so since it's a steel specimen our iron-based alloyed we use a nital our picral eland just for a typical etching solutions are kept in this laboratory like this and you also have different other agents like I listed in the table they are depending upon the kind of requirement each laboratory will have its list of agents.

Since ours is a metallurgical and materials department you have mostly etchings belong to metals are kept in this art is shown now we will see how this etching action is done, so you just keep the running water the slow speed and take the a polished surface. You have the two options either you swab it are immerse it both of both the technique will work in this case what we is going to demonstrate are going to see is that going to swab so take a cotton and make the cotton completely wet with, this etchant that is nital.

And you the cotton should be sufficiently wet with the nital solution or etchant and then we will show a bit on the polished surface very close to the running water yeah see is nicely swabbing on this so you can just notice one thing very importantly the surface of the metal become slightly dark; you can see that once you recognize this the color change, you can you can be rest assured that your surface is nicely etched, and this color also you have to keep in mind whether it is shallow etch or a deeper etch if it becomes too dark then that is an indication of a deeper etch.

And if it is become too dull are less dark then also it is a not a perfect etch that also comes with the practice so once you are satisfied with the etching time and the surface color, then you thoroughly wash it with the running water, like this and then again you wash it with the distilled water and alcohol to make sure that no dirt or anything is sitting on the surface; so after this your sample is you just make it dry and you have the surface ready for the examination.

The final blowing is thereafter this the specimen is now ready for the Metallographic examination. Now we will go back and take look at the cold setting what we have kept in the beginning of this exercise, you see now the mount is easily has come out of the table surface

because we had already applied the grease and you see that the specimen is nicely mounted with the cold setting compound.

And now it has been slide out from the mold with ease because of the application of the grease. So now you can easily hold this and then polish this sample as required here we have just shown a bigger sample typically an intricate or irregular shape of a sample will be mounted like this and then it is always easy to hold; you see that a very small sample has been mounted in fact this is a ceramic sample which is being mounted again on a cold setting compound you see now it is very easy to hold this and then go ahead with the polishing procedures. So that I show the the cold mount is useful.

If it is if the if your material is sensitive to heat; otherwise you can do with a hot molding and then you can take into microscope for viewing. So this we have already seen it in a metallurgical microscope how it is being looked at it. Now I just show typically how the scratches of various papers which I we have gone through will look like under the microscope.

So you see that how you are rebelled grinder the 150 mesh make a kind of a deep scratches on your sample; give you an idea; then I will also show you some of the, the subsequent scratches which the finer grit paper make yeah this is the deeper scratch marks which your velcro intermix and once you go to that fine emeries this completes straight line marks which are made by the abrasives will be eliminated slowly as you progress with the let me go to the next paper let us see what kind ; so this is the next paper you see that how the belt mark is being removed and the, the new scratch marks are appearing perpendicular to the, the first emery paper.

That is about 180 mesh typically you can see that some of the marks are left which from the belt crying and like that you have to make sure that all this deep scratch marks are removed and then you have a new scratches which is coming parallel or perpendicular to the old scratches or lines just to give you an idea what kind of surfaces you will see under a microscope because you may see some straight lines with your naked eye but it is very much appreciable under the microscope you see that how deeply the scratches are being made by the my sheets and you can see that

further how it is being removed with the 400 or 600 and I would like to see the last one but 800 mesh.

Yeah this is the final 800 mesh paper you see that fine scratches which are very close nature and this surface is taken to your alumina polishing on the lap rotating labs. So you have all the scratches are in the same direction and it is how your final after final polishing your sample will look like and this is how you are microstructure after polishing you will see; so now I believe that you have some fair bit of an idea how the samples are being prepared for the optical microscopy. And as I just mentioned I have not included the sample preparation techniques for polymer examples and also electrolytic etching.

I will take these two techniques in a when I talk about electron microscopy and it is sample preparation where they are those techniques are also being used so I will combine this. So as far as the optical microscopy variants are concerned especially for metals and alloys and ceramics I think you have some better idea about how to prepare the samples and how to look at the microstructure under the microscope. We will see in the next class some of the problems and numerical examples in the tutorial class thank you.

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