Fundamentals of optical and scanning electron microscopy
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Module – 02
Unit-5 Sample preparation and applications
Lecture – 09
Sample preparation techniques for optical microscopy
Polishing
Etching

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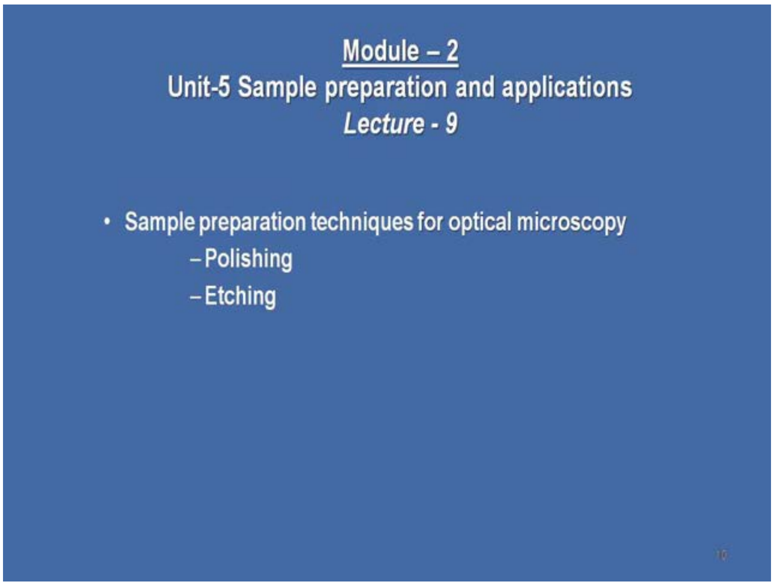

Hello everyone, welcome back to this material characterization course. In the last class, we just looked at the last variants of the optical microscopy and in this class I would like to give you a complete recipe of the material preparation for the optical metallography or microscopy analysis. It is kind of a complete 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 and then I will also give you some kind of guidelines for the ceramics as well as the polymeric materials.

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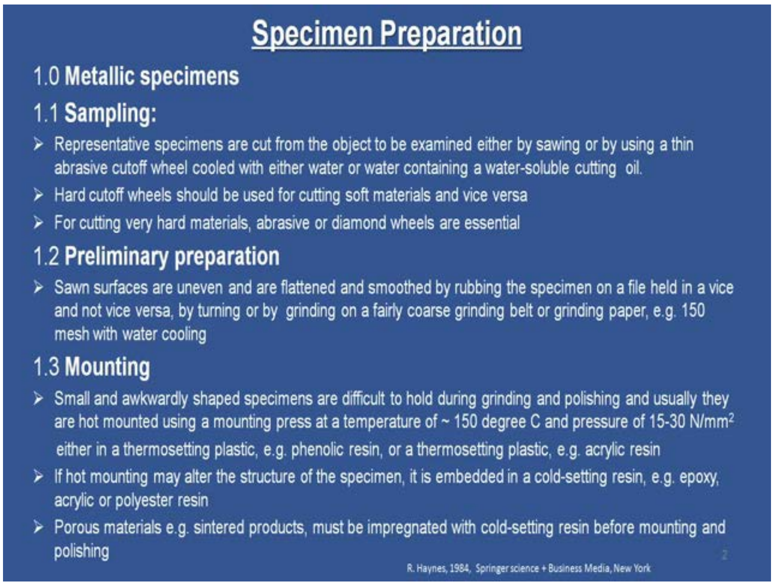

So, let us look at this the initial remarks. If we look at this, we will just go through the procedures for metallic specimens. The first step is sampling. This is very important, because it should be a representative of what we are talking about. So, the representative specimens are cut from the object to be examined either by sawing or by using a thin abrasive cutoff wheel cooled with either water or water containing water-soluble cutting oil. You see this is now completely is standardized you get a set of a 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 a water-soluble cutting oil. So, that why doing with adapting this coolant you would not make any structural changes in the material, 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 buy any standard cutoff wheel for this purpose, a supplier will give you by default what kind of materials we are going to cut based on that these cutoff wheels are supplied commercially. So, these are 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 are cutoff diamond wheels are used. I will also show this.

The first step is preliminary preparation after sampling. The sawn surfaces are uneven and are flattened and smoothed by rubbing the specimen on a file held in 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 preliminary in this manner.

Then we talk about mounting. Small and awkwardly shaped specimens are difficult to hold during grinding and polishing and usually they are hot mounted using a mounting press at a temperature of 150 degree centigrade and a pressure of 15 to 30 Newton per mm square either in a thermo setting plastic that is phenolic resin, or a thermo setting plastic, for example, acrylic resin. If hot mounting may alter the structure of the specimen, it is embedded in a cold setting resin, for example, epoxy, acrylic or polyester resin and porous materials, for example, sintered products must be impregnated with cold setting resin before mounting and polishing.

See, most of the solid objects like a 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, where you cannot handle with your hand. Then this mounting techniques itself is followed and here itself as we just seen there are two types one is hot mount another is cold mount. I will just show you each of this, how it is working in a laboratory as well.

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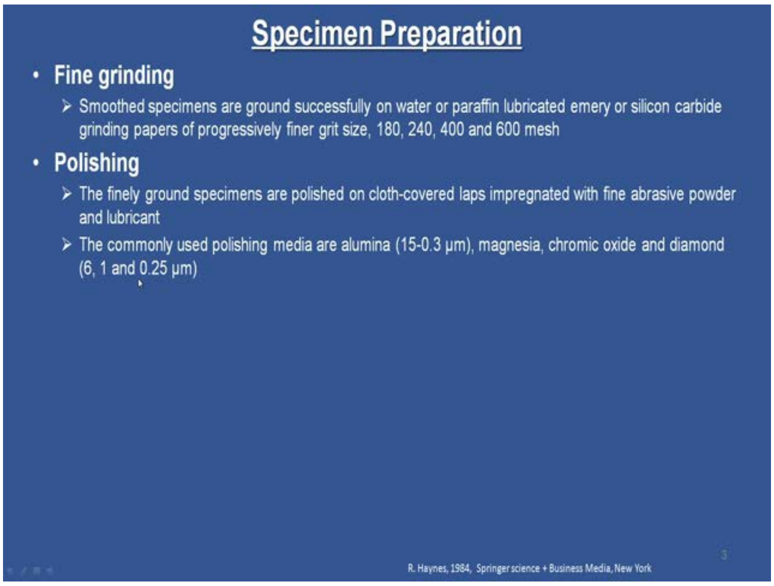

And after the mounting, we see the fine grinding. The smoothed specimens are ground successfully on water or paraffin lubricated emery or silicon carbide grinding papers of progressively finer grit size of 180, 240, 400 and 600 mesh. This is for making the surface more finer and finer and then fine polish. I will just demonstrate in the 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 magnesia, chromic, oxide and diamond, which is also very in the particle size from 6, 1 and 0.25 metal. These are all standard sizes, which are commercially available. And all these polishing media in general they are available in 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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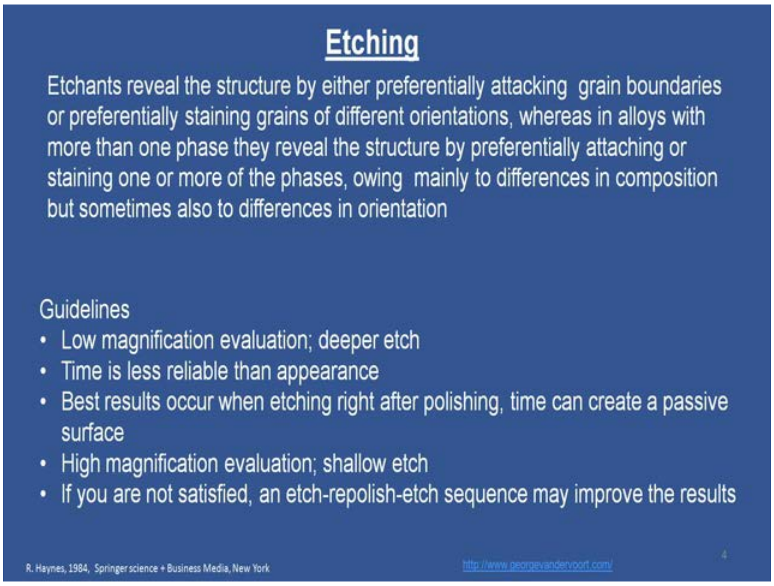

Now, comes to an important part of a metallography is called etching. So, first we will see what is this etching? Then we will go to the guidelines, and then how it works. Let me read out the initial remarks. Etchants reveals the structure by either preferentially attacking grain boundaries or preferentially staining grains of different orientations, whereas in alloys with more than one phase they reveal the structure by preferentially attaching or staining one or more of the phases, owing mainly to differences in composition, but sometimes also 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 or preferentially staining grains of different orientations. This also we will demonstrate to you, how it is being done in the laboratory.

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 surfaced 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 with difficult to be carried out to the perfection. So it is better, if you are interested, you practice it with lot of times spent on it, and carefully follow the guidelines.

There are general guidelines given by George Vandervoort, it is taken from George Vandervoort dot com. We can just go through for this etching. For low magnification evaluation, deep etch. What is deep etch we will see it in after sometime. 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, an etch-repolish-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 or information you are getting from the specimen, you have to re do this exercise. But then for the low magnification the guidelines is a deeper etch, that means, your etchings to be very strong, that means, you have to allow this etchants 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.

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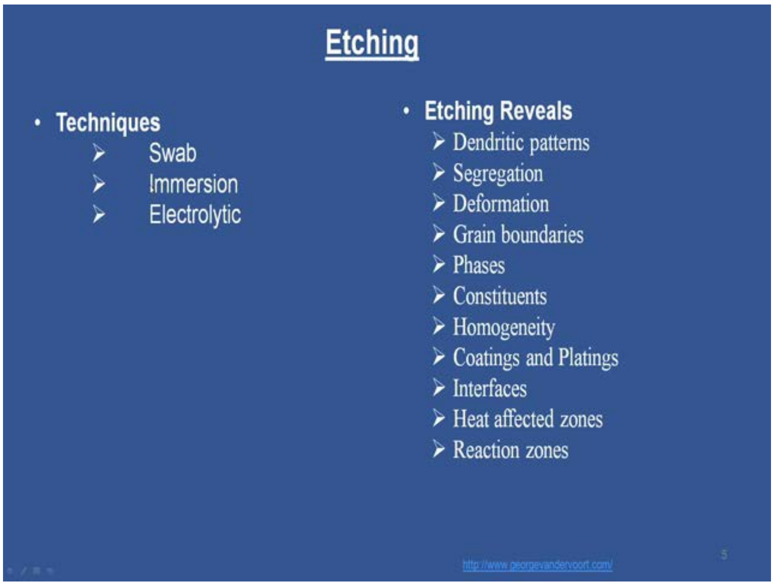

There are techniques etching has about three techniques generally, either you can swab, or immersion or electrolytic. So, these two are a manually done swab or immersion, we will demonstrate to you, how it is done electrolytic polishing is a separate technique. We will also talk about it in an appropriate time when it comes. So, what does this etching reveal? Etching reveals dendritic patterns, segregation, deformation, grain boundaries, phases, constituents, homogeneity, coatings and platings, interfaces, heat affected zones, reaction zones.

See, most of this the constituents are the information about the microstructure belong to metals and alloys and it need not be the same, but it 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 as perilites or a boundaries and amorphous and crystalline phases and so on. So, and also these etchants will also will act as a staining agents sometimes, you can just see the different constituents in the for example, polymeric material, it will show a different I mean staining contrast which you will be able to recognize and then study them.

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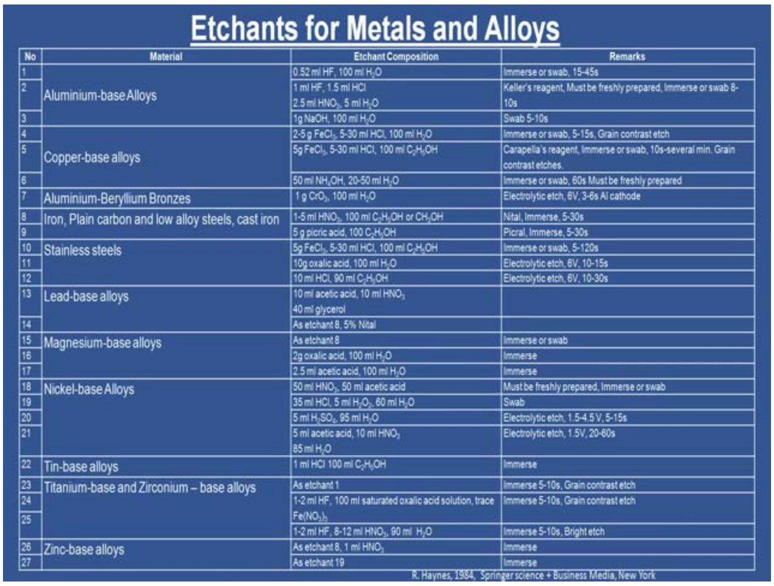

Etchants for Metals and Alloys

No	Material	Etchant Composition	Remarks
1	Aluminium-base Alloys	0.52 ml HF, 100 ml H_2O	Immerse or swab, 15-45s
2		1 ml HF, 1.5 ml HCl 2.5 ml HNO_3, 5 ml H_2O	Keller's reagent, Must be freshly prepared, Immerse or swab 8-10s
3		1g NaOH, 100 ml H_2O	Swab 5-10s
4	Copper-base alloys	2-5 g $FeCl_3$, 5-30 ml HCl, 100 ml H_2O	Immerse or swab, 5-15s, Grain contrast etch
5		5g $FeCl_3$, 5-30 ml HCl, 100 ml C_2H_5OH	Carapella's reagent, Immerse or swab, 10s-several min. Grain contrast etches.
6		50 ml NH_4OH, 20-50 ml H_2O	Immerse or swab, 60s Must be freshly prepared
7	Aluminium-Beryllium Bronzes	1 g CrO_3, 100 ml H_2O	Electrolytic etch, 6V, 3-6s Al cathode
8	Iron, Plain carbon and low alloy steels, cast iron	1-5 ml HNO_3, 100 ml C_2H_5OH or CH_3OH	Nital, Immerse, 5-30s
9		5 g picric acid, 100 C_2H_5OH	Picral, Immerse, 5-30s
10	Stainless steels	5g $FeCl_3$, 5-30 ml HCl, 100 ml C_2H_5OH	Immerse or swab, 5-120s
11		10g oxalic acid, 100 ml H_2O	Electrolytic etch, 6V, 10-15s
12		10 ml HCl, 90 ml C_2H_5OH	Electrolytic etch, 6V, 10-30s
13	Lead-base alloys	10 ml acetic acid, 10 ml HNO_3 40 ml glycerol	
14		As etchant 8, 5% Nital	
15	Magnesium-base alloys	As etchant 8	Immerse or swab
16		2g oxalic acid, 100 ml H_2O	Immerse
17		2.5 ml acetic acid, 100 ml H_2O	Immerse
18	Nickel-base Alloys	50 ml HNO_3, 50 ml acetic acid	Must be freshly prepared, Immerse or swab
19		35 ml HCl, 5 ml H_2O_2, 60 ml H_2O	Swab
20		5 ml H_2SO_4, 95 ml H_2O	Electrolytic etch, 1.5-4.5 V, 5-15s
21		5 ml acetic acid, 10 ml HNO_3 85 ml H_2O	Electrolytic etch, 1.5V, 20-60s
22	Tin-base alloys	1 ml HCl 100 ml C_2H_5OH	Immerse
23	Titanium-base and Zirconium – base alloys	As etchant 1	Immerse 5-10s, Grain contrast etch
24		1-2 ml HF, 100 ml saturated oxalic acid solution, trace $Fe(NO_3)_3$	Immerse 5-10s, Grain contrast etch
25		1-2 ml HF, 8-12 ml HNO_3, 90 ml H_2O	Immerse 5-10s, Bright etch
26	Zinc-base alloys	As etchant 8, 1 ml HNO_3	Immerse
27		As etchant 19	Immerse

R. Haynes, 1984, Springer science + Business Media, New York

So, look at this table. You see there is 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 orients so on. So, we have the different alloys listed in this column, material column and you have this etchant composition listed again each of this material. Then you have the remarks which will tell you what kind of action you have to take care. So, 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, it must be freshly prepared immerse or swab or 8 to 10 seconds. So, these steps are quite crucial. So, whatever the standard timing which are being recommend for each of this etching action, that means, it will produce a normal grain contrast or whatever the constituent reveal normal contrast.

When you talk about deeper etch or a shallow etch, these normal practices should be either exceeded or it should reduce a time. So, if you for example, in this condition, if you see 8 to 10 seconds are recommend; if you keep it for 15 seconds, 20 seconds then it will become deeper etch; or if you keep less than 8 to 10 seconds, it will become shallow etch. So, just to an example I am talking what is deeper etch? And what is shallow etch? Then for example, you have a copper-base alloys, aluminum-beryllium bronzes all of them will have a different, different etchants.

You have iron plain carbon and low alloy steels, cast irons, stainless steel, led base alloys, magnesium base alloys, nickel base alloys, tin base alloys, titanium base, and zirconium base alloys and zinc base alloys. So, you have a variety of alloys in metals, you see that different kind of etchants compositions are recommend with a different I mean what kind of techniques you will use whether you immerse it, or swab it, or electrolytic polish these details are also given in this tables. So, I request each one of you to go through this table whatever the material you are interested, and then see the corresponding etching etchant composition and the remarks which is useful to prepare the metallographic specimens.

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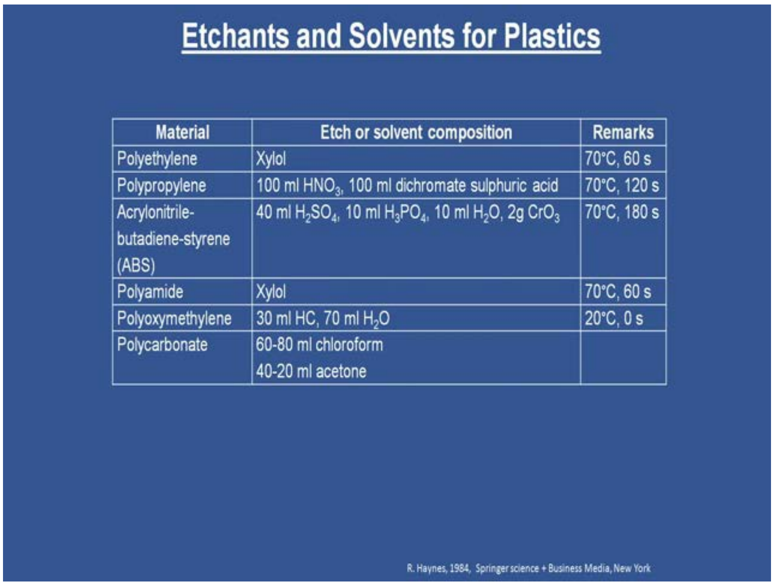

Material	Etch or solvent composition	Remarks
Polyethylene	Xylol	70°C, 60 s
Polypropylene	100 ml HNO_3, 100 ml dichromate sulphuric acid	70°C, 120 s
Acrylonitrile-butadiene-styrene (ABS)	40 ml H_2SO_4, 10 ml H_3PO_4, 10 ml H_2O, 2g CrO_3	70°C, 180 s
Polyamide	Xylol	70°C, 60 s
Polyoxymethylene	30 ml HC, 70 ml H_2O	20°C, 0 s
Polycarbonate	60-80 ml chloroform 40-20 ml acetone	

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, polyoxymethylene, polycarbonate for all this you have this a solvent composition is given here. Then you have in the remarks column you have the temperature at which these things to be carried out, and the time up to which this etchants to allowed etchants will be allowed to react with the specimen surface. And coming to the polymer, I only talking about etching here, but some of the sample preparation techniques which are meant exclusively for metals alloys may not suite here; for example, polymeric materials require something called microtone. I will talk about it in a due course.

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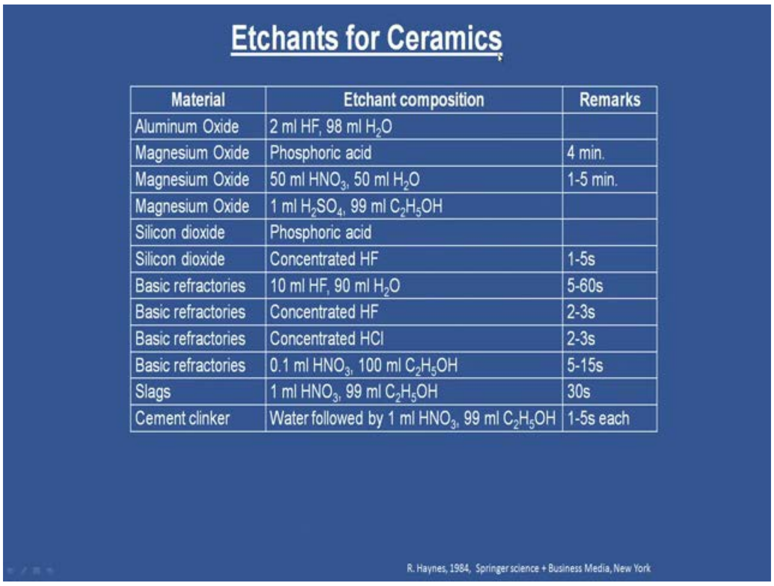

Then finally, the etchants for ceramics, you have the material column and etchant composition as well as remarks. You can see that aluminum oxide, magnesium oxide and then you have silicon dioxide, basic refractories, slags, and cement clinker. So, you have a typical etchants given for each one of this classification, and their time which is required to etch this surface is also given.

So, now what I will do is I will just take you to the lab where we will show you all this sample preparation. So, this is a typical a cold setting mount. I will just I talked about in the beginning of the slide; you have this cold setting compound and a cold setting liquid. As I just said they are all polymeric resign or a compound, and these are all commercially available a setting compound. So, they do not mark the actually chemistry of chemical composition of this on this bottle, but anyway they are all commercially available. So, as I just mentioned this cold setting is used for a irregular shape specimens or a specimens with you do not have enough holding thickness or height then you can use this.

So, this is a kind of a mount we one can use, this is a typical mount. You can also use a PVC pipe set cross sectional 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 whole mounted sample will come out of this mold so easily. So, this is typical grease which you can apply to the inside of the mold, and it should be uniform and then you see little bit of greases are kept on the table, so that that surface will also will become easy to remove when the molding is done.

Now, you keep your specimen, here we take this. First you prepare, let us prepare and then we will just pore this into that mold. You typically take some quantity of powder which depends upon the mold size, and you just mix this liquid - cold setting liquid and thoroughly stir it so that it homogeneously form 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 pore this mixture. So, then this mold is just allow to set for 30 minutes to 1 hour, and then we can be removed, and then sample can be, now sample is ready for the polishing.

So, now we will leave that. We will look at the other procedures then we will go back to this once it is set. So, this is the first grinding. It is an emery paper mounted on the rotating belt. And the sawn 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 sawn surface you can see, how it is being polished. 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 and these scratch marks under the microscope how it looks like later. So, once you make this kind of one directional, unidirectional scratch appear in this then you can rotate to ninety degree and then polish it to once again.

So, then once that belt grinding is done, we will talk and go to the fine grinding this what I just talked about 180, 240, and 300, and 600 and so on. So, we have to start with the coarse 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 coarser emery sheet that is 180 mesh kind 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 should be done with the increase in the size of the grid.

So, now, you see that scratch molds which we made out of the belt grinder should be perpendicular 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 yeah, 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 at 300 and then we will go to 800 and that is a finest polish one can get.

Now, it is clear that you do not see any you know deep grinding mark which we have generated at the previous emery paper as you progressively go to the finer emery sheets. This is the last sheet where 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 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. If it is a labor intensive process and after this what you should do is you have to take this to tape water and remove all this a derby which you corrected from this, all this. In fact, you will take cotton and then swab it like this, and make sure that none of the old debris or sticking onto this metal surface.

Now you see that after all this emery sheet it becomes finely polished and then you can dry it with an air blower or air drier. Make sure that each step you follow after grinding; now you will go for fine polishing. So far we have finished 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 disc polisher and what you are now seeing is disc polisher which has got a polishing cloth which is mounted on the labs. These are all commercially available a machines many suppliers are there. You can see that and these kinds of machines with come with lot of pre setting polishing recommendation. And if you are able to adapt those recipes, you can follow or you can follow your own style. You see that now the poly thing I mean it is a polishing cloth, and you can choose the rpm and the kind of you know settings, which you require for this sample, and then you start your polishing. Then you have this water coolant support is there which will be used while we are polishing these 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. So, here also we will use 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 slab. So, now we will put some powder on this polishing cloth, too much is not required. In fact, this itself an excessive edition, this much of polishing powder is not required, and you 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 this sample against this rotating slab, and this requires quite a bit of a practice.

It is not just holding the sample you have to apply some uniform pressure. 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 in order to make sure the uniform polishing all over the surface this rotation is essential. 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 5 minutes, you see this is what we happen if you do not hold it properly, and you have to be very careful about holding this specimen, and then rotate it this requires lot of practice, this is why we intensely did it to show what kind of mishandling will happen. So, you have to be extra cautious.

So, once this polishing is done what you should do is you have again washed it with the running water with cotton, swap it nicely. And make sure that all this scratches are removed, in fact, this is not final polish, but before that we would like to show the next polisher is diamond. This is again a commercially available or 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 with very small quantity even this is little higher in my opinion, so that and then you can just wipe it on the cloth uniformly, and then you have to just apply some kind of lubricant which is supplied along with this diamond powder. So, little bit of lubricant and then again you start polishing it.

Here again we can choose either a preset recipe or you can choose your own recipe depending upon the rpm and time and so on. So, hold this gently, again you have to be very careful while holding and rotating it; otherwise, the sample will fly off, and it can cause some accident also. In between you can apply little bit of a lubricant, if it required. And you see that again you wash it with the running water with cotton swab, and make sure that all the debris everything is removed from the sides as well as the surface on the specimen. Now you see that the sample is almost getting close to a mirror polish. So, again, it is now dried with a blower, you see that it is become a mirror, mirror surface.

So, now we will go to etching. Since it is steel specimen or iron based alloy use a nitol or picral. I will just for typical etching solutions are kept in this laboratory like this. And you also have different other etchants like I listed in the table. It is depending upon the kind of the requirement; each laboratory will have its list of etchants. Since ours is a metallurgical and materials department, you have mostly etchants belong to metals are kept in this or it will shown.

Now, we will see how this etching action is done. So, you just keep the running water just slow speed, and take the polished surface, you have the two options either you swab it or immerse it. Both the technique will work in this case. What we is going to demonstrate or going to see is that going to swab. So, take cotton and make the cotton completely wet with this etchant that is nitol and cotton should be sufficiently wet with the nitol solution or etchant and you swab it on the polished surface very close to the running water yeah, see, it 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 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 or less dark then also it is a not a perfect etch that also comes with a 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. 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 there; after 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 as come out of the table surface, because we had already had applied the grease. And you see that the specimen is nicely mounted with the cold setting compound. Now, it has been slide out from the mold with is 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 very small sample is 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 is how the cold mount is useful, if your material is sensitive to heat; otherwise we can do with the hot mounting, and then you can take it to microscope for viewing.

So, this we have already seen it in a metallurgical microscope, how it is being looked at it. Now, I will just show typically how these scratches of various papers, which we are gone through, will look like then to the microscope. So, you see that a belt grinder, the 150 mesh make a kind of deep scratches on your sample, give you an idea. Then I will also show you some of the subsequent scratches, which the finer grid papers make, this is the deeper scratch marks which your belt grinder makes. And once you go to the fine emery's, this complete is straight line marks which are made by the abrasives will be eliminated slowly as you progress with the grid.

Let me go to the next paper. Let us see what kind of, so this is a next paper, you see that how the belt mark is being removed, and the new scratch marks are appearing perpendicular to the first emery paper that is about 180 mesh typically you can see that some of the marks are left which from the belt grinding. And like that you have to make sure all this deep scratch marks are removed, and then you have new scratches, which is coming parallel or perpendicular to the old scratches or lines.

It just to give you an idea what kind of a surfaces you will see under 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 emery 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 about 800 mesh, this is the final 800 mesh paper. You see that fine scratches, which are very close nature and this surfaces is taken to your alumina polishing on the lab rotating labs. So, you have all the scratches are in the same direction and this is how after final polishing your sample will look like, and this is how your micro structure 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 an optical microscopy and as I just mentioned, I have not included the sample preparation techniques for polymeric samples and also electrolytic etching. I will take these two techniques when I talk about electron microscopy and its sample preparation where there 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 micro structure under the microscope. We will see in the next class, some of the problems and numerical examples in the tutorial class.

Thank you.