Materials Science and Engineering
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Lecture – 08
Metallography

Hello friends, in the previous lecture we discussed about microscope basically the whole optical system. And we also discussed about the aberrations in optics what are the aberrations which there today we want to discuss something more practical in the sense that how we are going to use the microscope, how we are going to prepare the sample for viewing in the microscope? So, this is more like a practical approach how to use the microscope ok.

So, this is what we call as metallography or metallography practice what we have to do to ultimately see a microstructure. So, to start with let us see how I am going to prepare a sample for viewing into a microscope for getting a microstructure ok. And for that first we will we do what we call as a grinding process.

So, initially the sample may be given to you in whatever form it is not up to you sometime you have to take sample from a bigger some part ok. So, that may have a lot of undulations may be a different shape or it may have some curvature ok.

So, first thing we have to do is to make it a flat sample ok; using a very coarse abrasive particle which is what we have in grinding ok. So, the material removal rate is very high when you do grinding ok. So, the grinding process is basically we want to have a flat surface on which we will later on do the remaining polishing processes we will apply the remaining polishing processes.

So, first is grinding using a grinding wheel you have to be very careful.

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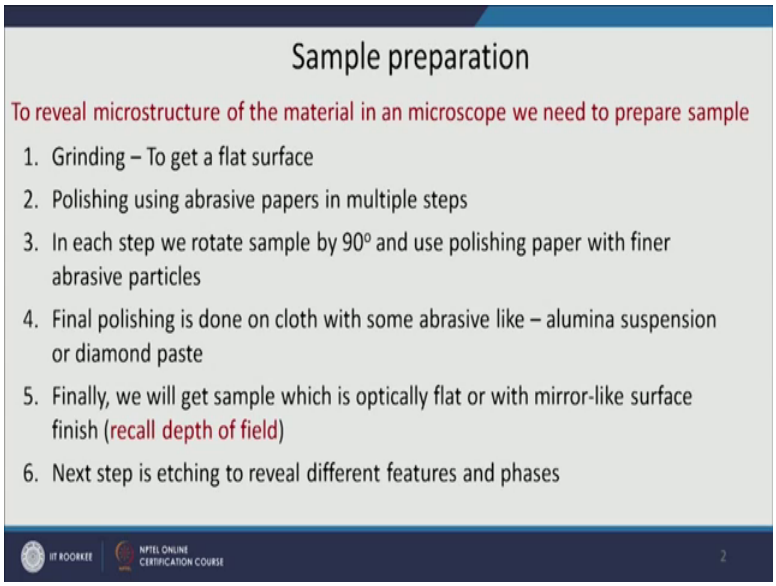

Because at that time you can introduce some more problem in the surface also may be you may create a curvature and all that; so, you have to have a very steady hand and you have to pay attention to the grinding process do not talk to anybody while doing the grinding process.

The next comes is because this is a very coarse abrasive particles will be there in the grinding wheel ah. We start with the next polishing process using polishing papers ok. So, you will have a A 4 size paper on which the grinding this abrasive particle will be pasted by some adhesive which you will get from the market ok. And then you start the polishing on this paper and that also first you start with a coarse abrasive particle and then you keep on going to finer particles ok; we will just see all these things in more details.

The next step is we by when you are doing polishing. So, first you will start with a coarse paper ok. So, suppose I am let us say this is my sample I am doing the polishing in this direction then I will rotate it by 90 degree ok; take the next paper which is of a smaller abrasive particle size and then again do the polishing. I will give the details in the coming slides.

So, we start with a coarse abrasive particle first is grinding then coarse particles in the abrasive paper. Then we take another paper with a small smaller abrasive particle size and this process can go depending upon what type of sample you have; what is your

polishing procedure you are applying you can have multiple steps of this paper polishing ok.

Then the step comes which is listed as fourth step here where we do final polishing using a cloth ok. So, usually these are velvet cloth or all the billiard you must have seen and this cloth on the pool table or billiard table that kind of cloth ok, which can hold the abrasive particle ok. So, then abrasive particle a very fine abrasive particle with a which is in suspension; we pu put on the cloth and again we do the polishing using the this abrasive ok.

Then the purpose of this polishing using different this different papers and then going to the cloth polishing is to get finally, a mirror like finish in this on the sample or this is also we call as optically flat sample.

So, I would also like you to go back to the previous lecture, where we talked about this optical system and we talked about the term called depth of field. And there was a relationship for the depth of field ok, if you put all the values in that particular equation you will see that the depth of field of optical microscope is very small in microns basically ok.

. So, you can understand that if my sample the surface is not flat optically flat then it will not remain in focus ok. So, to make a sample such that the whole sample or the whole surface remain in focus of the optical microscope, I have to prepare this optically flat sample ok.

So, all this polishing technique ultimately if the aim is to get a sample which is optically flat; the meaning is that I have a very small depth of field of optical microscope. So, I have to reach a condition where my flatness is in that micron range. This may not be may not be required in some other microscope for example, scanning electron microscope ok.

and the next step is the etching to reveal different features and phases. So, once I get this optically flat sample ok; now I want to introduce some roughness in the surface or some new features on the surface to start seeing the microstructure of the material ok. So, this is the in nutshell the whole process of sample preparation ok.

Now, we will look into detail each of these steps grinding of course, we will not see. So, step two and three where we use polishing using a abrasive papers ok.

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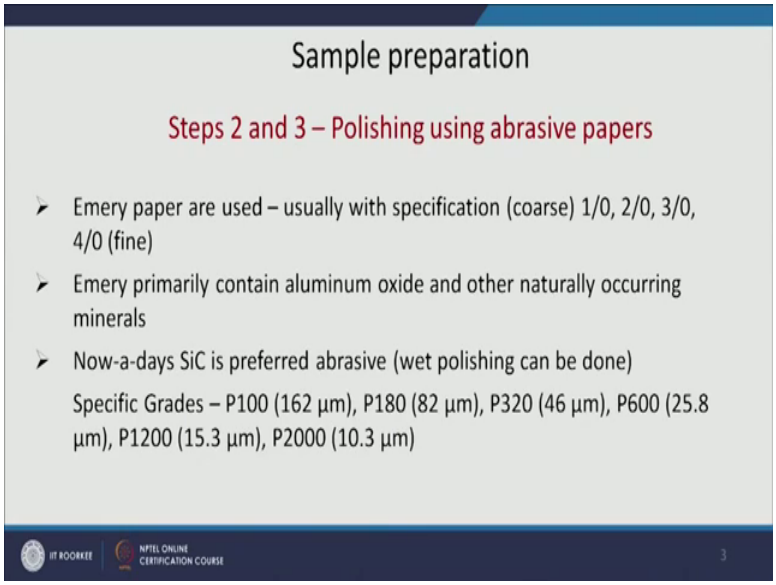

Basically what we do is we can use normally or popularly people know this polishing paper by name called emery paper they will just say that bring an emery paper.

So, emery paper were having abrasive particles as emery and emery is nothing, but aluminum oxide and other naturally occurring naturally occurring minerals. So, whatever you can get minerals from nature ok. So, it is a combination of different abrasive particles or different chemical compositions ok. So, it is not a single type of abrasive particle and that is why you can understand that it is not a it will not have a homogeneous property ok. So, a different particle will have different hardness and different capability of abrading the surface ok.

So, initially these were very popular because emery paper was very cheap. And their designation as you can see here is what is called as 1 0, 2 0, 3 0 and 4 0. So, 1 0 is the coarse paper and 4 0 is the fine paper. So, like that you can go from coarse to fine.

Nowadays of course, most of the places you will see that we are using silicon carbide papers ok; so, basically in this abrasive particle is silicon carbide ok. And the advantage is that you have a abrasive particle of similar hardness ok. So, you will not get different type of hardness the of course, the adhesive which is now used is also of much better

quality. So, you can also do polishing by you in wet condition basically you can pour water on the abrasive paper and do the polishing.

The advantage is that it will minimize the heating of the sample ok. So, suppose heating may destroy some features or may alter some features of your sample that can be reduced if you use wet polishing. And in this the grades are given by some designation is like this P 100 P 180 P 320 you can go to P 2000 4 each grade; the size of the abrasive particle is also given in the bracket.

So, for example, in P 100 the particle size of the abrasive particle will be 162 micron around 162 micron basically and you go up to final polishing it for P 2000 the particle size will be 10 microns. So, you can now understand that as I told you depth of it is in that micron range by doing this type of polishing we are reaching towards that range by doing this type of polishing. So, this is what you do in a in a abrasive paper polishing and this is the polishing technique which you use ok.

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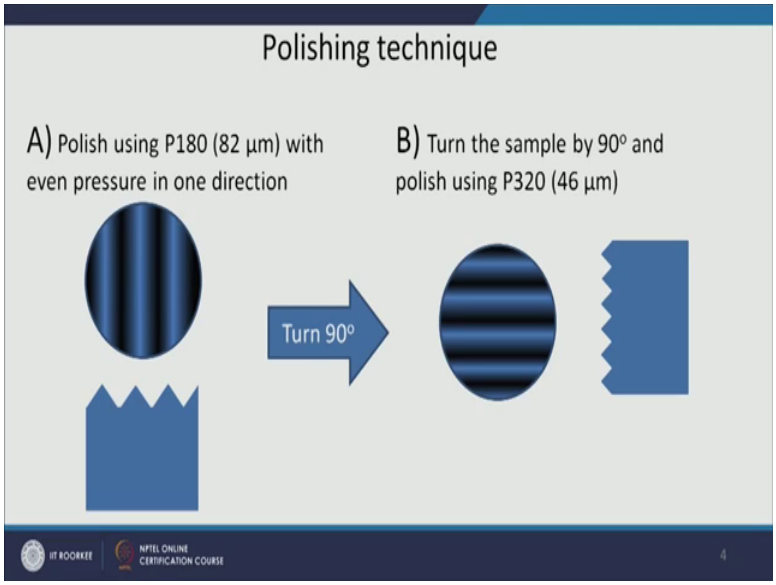

As I told you I will tell you this thing in detail ok. So, suppose I take a polishing paper this is an example here given ok. So, first we will do on a coarse paper. So, we are first doing let us say on P 180 paper which has a 82 micron of particle size.

So, I will be doing polishing like this in a single motion only in one direction care must be taken that we should not put too much pressure the abrasive particle itself we will

abrade the surface. So, we do not have to put too much pressure here and we have to do it in one direction as like this.

. So, that you get marks in one direction is shown in this slide here that all the marks are in one direction ok. And if you see from if you see have a side view ; the ridges and the asperities will be something like this it may not be that regular, but this is just a schematic can give you an idea that how the roughness will be there on the sample.

Now, when we go to the next paper which will be of course, having finer abrasive particle let us say we are taking P 320 here ok. So, now, I will rotate my sample which I was doing like this by 90 degree. And then I will again start polishing ; the idea of 90 degree rotation is that we should be able to remove all the roughness or a scratches which we introducing the previous paper to so, that we make sure that it is all removed.

When you may cover 90 degree I am I am sure that the earlier ones were removed because I will see a new direction of scratches. So, if I am not able to see the see the earlier scratches ; that means, those are removed if I do in the same direction I will not be sure that the earlier ones were removed or not ok.

. So, progressively we want to have a scratches of finer smaller wavelength and finer this amplitude of the if you say it a it think of it as a sinusoidal wave ; a smaller amplitude. So, you can see I have done the sample by 90 degree then I polished it on the P 320. So, now, my scratches are finer the size is smaller the wavelength is smaller and we can keep on going like this up to let us say P 2000.

So, this is the paper polishing care must be taken if you do not do these steps religiously; you were not you are not going to get a good microstructure later on. These steps are very important ok; so, that each step should be followed religiously and you remove the damaged layer which is introduced because of the polishing you can understand we are abrading the surface. So, it will also introduce a damaged layer in the sample that has to be removed in the next paper, then that has to be removed in the next paper and so, on.

The final polishing is cloth polishing so, that now the particle size abrasive particle size is in even smaller it is in submicron size ok. You can get again of different particle size ; so, different materials for example, seat steel samples if you are doing usually we use alumina which is in suspension ok.

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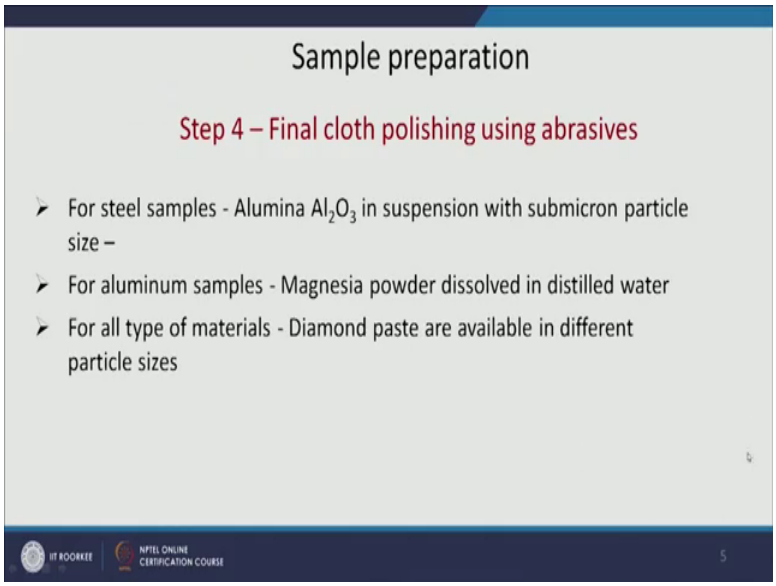

So, you can use water to suspend it and it has a submicron particle size that you keep on pouring on the cloth and the polishing wheel will be there which is rotating continuously ok. And your sample will encounter every time a new abrasive particle to abrade the surface. This is the slower process my particle size abrasive particle size is smaller ok. And that will make sure that you do not damage the surface of the sample ok.

For aluminum samples for example, I may use a magnesia powder dissolved in the distilled water to get a good surface finish. Nowadays diamond paste is also very popular you can use it in almost all type of samples and you can get a very defined size of particle in the diamond paste ok.

So, it can be 9 micron, 3 micron, 1 micron less than 1 micron 0.25 micron and so, on ok. So, you again in this case also I can keep on going to finer particle size of course, you have to use different cloth for each of this different particle sizes otherwise if you use the same cloth then it will get mixed you will get these scratchers from 9 micron as well as one micron if you are using these two.

So, for each diamond paste you will use a different cloth clean the your sample polishing unit completely put the new cloth on a disc polisher disc polisher are basically discs on which you put the cloth and this disc rotate ok. So, it is a kind of a semiautomatic polishing technique.

And that is how you do the polishing using cloth then once I have got ah nice polished surface I have to now see the microstructure. To reveal the microstructure in the material the process which is there is called etching.

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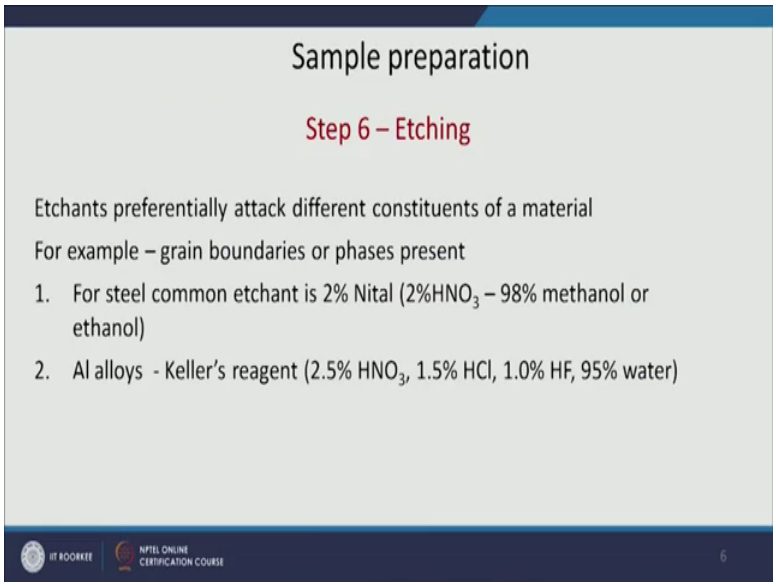

So, etching what it does is it reacts with the different constituent of the material on a different way. So, there may be a grain boundary in the in the sample ok, there may be different phases in your sample ok. So, this etching or the etchant the; it reacts with this in a different way ok. And when it reacts a with in this constituent in different ways ; it give rise to some at micron range only some undulation on the surface some variations will be there on the surface ok.

Now, the microscope will kind of capture those variations and by capturing those variation ; it will be able to give you that what is there in the material what is there at the micron scale the why we call this as microstructure because we are viewing the thing at micron scale.

So, that is why it is microstructure for different materials again there are actually if you if you are interested, you can go to there are handbooks available for each material there are maybe some time for some material there can be 10, 15 etchants different type of etchants.

And there are 100s of etchants for all kinds of materials and each etchant serve a certain purpose. It may reveal your grain boundary, it may reveal your different phase ; if there are two three different type of phases in the microstructure, it will reveal all these phases in different way maybe some time different colors.

Now, right now the problem with this particular lecture which I am taking is I am using lot of terms here which are not still you are not familiar with that; for example, grain boundary or phases. So, right now you are bear with me because those will be covered in the subsequent lectures advantage you have is that all these are video lectures.

So, I you can always go back to the this particular lecture later on so, that when we cover the phases grain boundary and boundary return and so, on. When you get familiarize with that you can again come back to this particular lecture to see that what we were trying to tell you ok.

Because any sequence I take something will be missing in that particular lecture ok. So, you can always go back and forth to clarify your concepts. So, for if I want to have some I want to see some features in for example, is steel sample a sample of steel then I can use a etchant very popular etchant which is called 2 percent nital ok.

So, it contains some HNO 3, 2 percent HNO one three and 98 percent methanol original usually the handbook says that we should use ethanol ok, but use of ethanol is kind of not allowed you have to have a license to deal with ethanol ok. So, usually in India we use methanol instead of ethanol for aluminum alloys Keller's reagent is there one of the popular one there are others also I am just taking here as an example some etchants ok.

You have to also understand this that polishing or metallography or preparing sample for seeing microstructure is kind of a you can say art it takes time it takes time to understand that how you do polishing. Just if I want to tell a story initially when I was doing when I was start when I started my PhD it took me 3 months to get a very good microstructure in the aluminum lithium alloy and which I was working. I was a struggling with the polishing and then I was doing etching.

I was doing one thing wrong in that which friend of mine told me that you instead of immersing the sample in the etchant in the that chemical for example, here it is nital in case of aluminum; I was using Keller's ok. So, instead of immersing it you just swab it

with a cotton ; so, you take a cotton dip it in the etchant and just swab it like this. And what I was struggling for last 3 months I could get in like half an hour.

So, my point is that something you have to whatever is given in the handbook or somebody is telling, you also have to try different things maybe immersing may give you the micro structure, may be swabbing will give you the micro structure, maybe slightly changing the composition may give you a good microstructure, maybe you want to modify this Keller's reagent ; you are allowed to do that these are not etchants or composition set in stone you are allowed to play with it.

But after getting whatever sample you are working on you should be able to understand the microstructure, it should not happen that something you are getting you are not able to then explain what you are seeing there ah. Similarly with steel also you can work with. So, many etchants are available there.

So, do not get fixated with one etchant or one procedure when you are not getting a good microstructure or not able to reveal the all the features; try a different things ok, try a maybe changing slightly the etchants the way you do etching and so, on ok. And you will get definitely get the microstructure and that is one of the beautiful feeling when you see the microstructure in the microscope for your material ok.

Now this how we see the microstructure? How microscope is able to capture all these ah features in the sample ok?.

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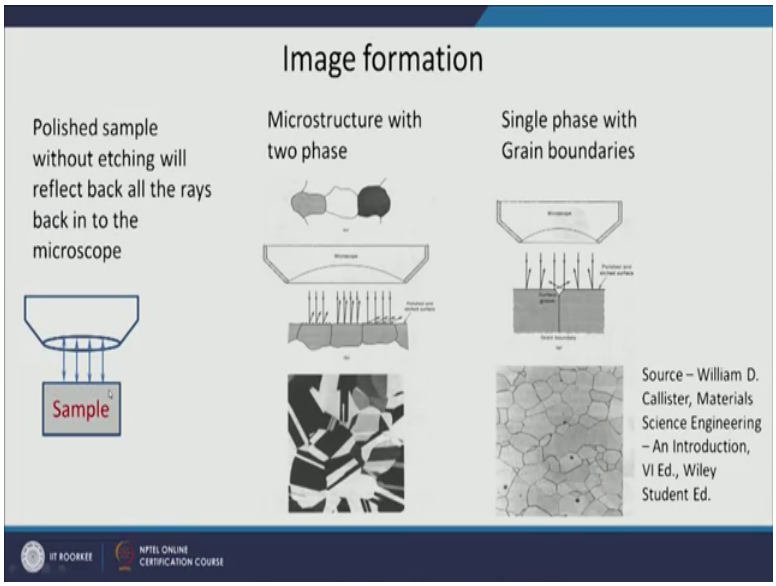

For example, if I take sample just polish like the technique I was telling you ; I have an optically flat surface which is a mirror finish and my rays are coming from the objective what will happen? It will get it will reflect and again go back into objective. So, the ray and because this surface is at 90 degree to the rays coming, it will again go back into the same path. So, the ray which is coming like this it will again will be reflected back into the objective and so, on ok.

So, if I take just a polished sample I will not see anything on the on the sample on the surface of the sample ; I will not see anything in the microscope it will only be a kind of a if it is a good polish to surface; you will only see a even brightness from every part of the sample ok.

But suppose I have a my sample which has a microstructure which has two phases for example,. So, what this is etchant does as you can see in this particular slide here ok; you have one phase which is here, another phase which is here then again same this phase is again repeating here ok. You can see this phase has reacted with the etchant and by reacting what has happened it has got this roughness on the surface because I think when it reacts; it is not a uniform process it may give us some kind of roughness on the surface ok.

This phase it is it is may be in order to that particular etchants; it has not reacted with that etchants. Again with this say phase when it is repeated here again it has reacted and you can see the roughness on the sample.

So, now what will happen my rays are coming from the objective in the case of the phase which has not reacted the light will come and go back into the objective. So, I will see because I am now getting a reflected light ; I will see that that part is looking bright.

The another phase which is which reacted with the etchants and has this kind of roughness the light from the rays from the objective is coming and because of roughness the rays are getting reflected all over the place they are not going back into the objective. And because it is going all over the place and I am not seeing any reflected light back in the objective in the microscope ok; I will see these particular phases dark ok.

So, now what will happen? The phase which has not reacted will look bright the phase which has reacted will look dark ok. And that is how I will be able to see a microstructure like this which is shown here that one phase is dark, one phase is bright and so, on ok. So, these are the two phase microstructure; I will be able to see that what is the how the two phases are distributed in the microstructure what is their size and so, on.

Let us say if there is no this is not a material with 2 phase only single phase is there ok. So, now, there is no 2 phase here again when you your sample reacts with the etchant the grain boundary are high energy sites; we will see that when we cover the grain boundary part. And they will react more vigorously with the etchant than the grain.

. So, what will happen? The grain where the grain boundary is because of this surface tension also and because this is reacted with the etchants you will get a ah curvature like this ok. So, now, when the light is rays are coming from the objective the where the grain is there where the flat surface is there it will be reflected back into the microscope. And where the lights are interacting with this curved surface; it will get reflected in this different directions.

So, this will appear dark this will appear bright and that is what you will see in the micro structure. You have grains you have grain boundaries and so, on the source is also given

from which I have taken this particular figure. So, this is how image formation takes place in the microscope.

Now, just let me quickly tell you that how you can use the microscope ok.

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So, as we in the previous lecture; we saw the whole optical system I just wanted to show you the actual microscope and how this optical system or different parts of the ah microscope are there. So, we have a light source here from which ah the visible light will be coming, it will go through different filters here you can insert filter or you can remove it is up to you. Then there somewhere here it will have that half silver mirror ok. And then the light will go through objective and it will be falling on the sample and then it gets reflect it will to be reflected back.

So, depending upon the feature on the sample it will either reflect back or may not ok. So, according to that you will see either bright feature or a dark feature. So, let me just tell you that how I will like to use a microscope ok; some precautions are there. And after the light goes through the objective it will come into the eye piece and you can see the microstructure.

So, when you prepare the sample you polish the sample then you will do the etching after etching always remember to do very thorough washing of your sample in a running water ok. The idea is because the all the etchants have acids ok; so, if it acid remains on

the sample you bring it here it will also react with your sample and the fumes of acid may also damage your optical system the lenses and so, on ok.

So, do a very thorough of washing dry it in a air blower not your hair dryers a hair blower is a blower which gives air at a very high velocity. So, just put it in front of that; so, it will physically remove all the water from the surface and dry your hands also and then you come to the microscope.

So, microscope there are some adjustments here you can see this is a coarse adjustment and in a smaller nob has fine adjustment. And this is a stage where you will keep the sample it also moves; so, you have a x and y variation. So, that I can watch the sample if I want to measure certain feature on the sample I can do that measurement also because there is a vernier scale on the stage.

So, first I will what I will do there are three objective on this turret. So, I will always start with the lowest magnification objective. So, right now it is 10 x here and then I will place the sample here which is dried completely and first I will do the focusing using the coarse setting ok.

So, that I can quickly find out where my focus for this sample is there and once I find out that then I do not have to touch my coarse adjustment anymore because other when I go to higher magnification only a fine adjustment will bring the ah image into focus.

The reason why I am saying that do not touch coarse adjustment after you have done focusing at a lower magnification is because you can see that the next one is twenty x and this final one is 50 x. So, if I can if you may be able to show see it in the this particular video that in this case my working distance is suppose here very close to the sample whereas, in the coarse 1; the working distance is for ten x it is very far. You can see that there is a distance more distance between the sample and the and the and the lens.

So, when why we avoid using coarse adjustment for high magnification lenses is because working distance is very small and make your sample may touch the lens in the process may damage the lens and which we do not want. So, do a coarse adjustment of focus at lower magnification and after that you just use the fine adjustment to do focusing at higher magnification lens.

So, start with the lowest one and then you can change go to 20 x for example, here and then you can go to 50 x and so, on. And you will see a very nice microstructure going from 1 magnification to the next.

So, I think that is a small demo of a microscope; how to use it? How to take care of it ? And always cover the microscope when it is not in use because the dust gets collected on the lenses. And sometimes there is a fungus fungal growth also on that dust and we should not we do not want that. So, for a microscope the optic or lens is the heart of the microscope and we should take care of that.

Thank you.