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Lecture-42
Materials Characterization
Fundamentals of Transmission Electron Microscope

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Hello everyone welcome to this material characterization course. In the last class we just started looking at the sample preparation techniques for transmission electron microscopy. Then I just give a brief description of each techniques right from the mechanical thinning to jet polishing and then we have just gone through the complete procedures. And today we will continue that exercise and we stopped in the last class with the dimple grinder so the dimple grinder is typically used for ceramic materials are any nonmetallic materials. Where this is a pre thinning exercise before an ion milling operation.

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So I just stopped in this point yesterday somewhere here as you just see that your sample is just mounted on this stage. And then you have the ceramic slurry is there.

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And then you have the grinding be and then the disc is kept on rotating in order to produce a dimple in the center of the 3 mm disc. And the thickness of the dimple is measured in this dial gauge. So typically you remove every micron thickness you will be able to monitor through this dial gauge and then you have the control over the RPM of this the disc.

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Which is a grinding wheel and so on and this is some typical sample which is being demonstrated where you have the I think it is a metallic specimen just for a demonstration purpose.

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We have taken just see that this is a dimpled specimen this is an unspecified before just go back and.

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Then I would like to show the, the difference between the, the dimple specimen and dip under pool specimen so what you what the exercise what you are now just witnessed is the sample is stuck on a aluminum stub with a glue and the once a dimpling is done you have to reheat the stub in order to melt that glue. And then you remove the sample and you can see the clear difference between the undimpled sample and a dimpled sample it is the, the dimple region here is slightly offset that means the alignment. What we have kept on the stage as well as the, the disc is not exactly correct so it should be in the center nevertheless this can be used for further thinning in an iron Miller.

So that is the information I just want to give before I proceed to the next technique so the next technique which I am going to demonstrate is called ultrasonic a dis cutter this is called ultrasonic dis cutter this is one very specialized tool to prepare a 3 mm disc of a slice of ceramic material in a very rapid fashion.

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So can have a close look at it and the what, what this machine does is you have the, the tool it is a to blur tool which cuts the sample with the abrasive slurry and the high speed. So this high speed is achieved in this cutting tubular tool through an ultrasonic no ultrasonic vibration with the frequency of about twenty six kilo heads and so on. Typically it is a it is the vibration high-speed action or I would say the high-speed activation is done through a piezoelectric crystal and where it produces a high-frequency vibration and with the ceramic slurry which is there on the specimen on the glass plate here. This will just cut through the material with the localized corrosion action.

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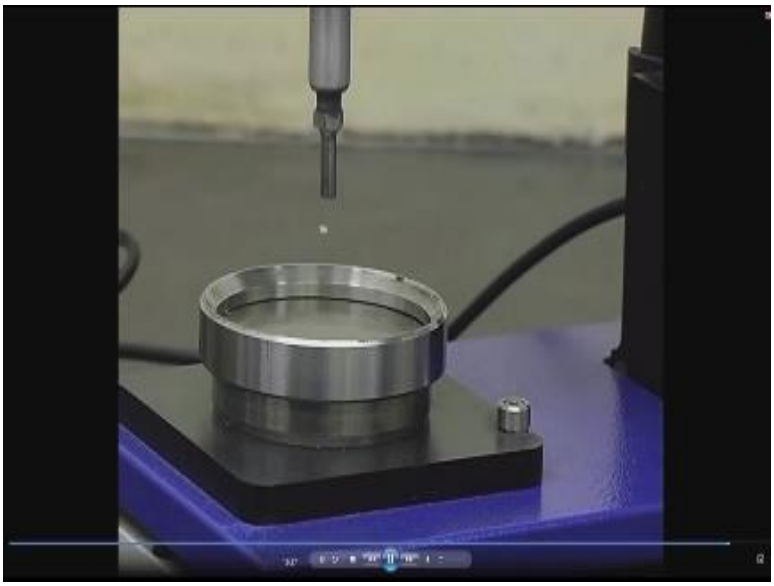

So that is the basic function and then you have the you have the facility to you know reduce or increase the frequency and this is the stage you can just move up and down and you can see that tool very closely so and here again you have the dial gauge you can prepare a samples with thickness to few microns.

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To few one or two mm thick ceramic material can be made into three mm discs.

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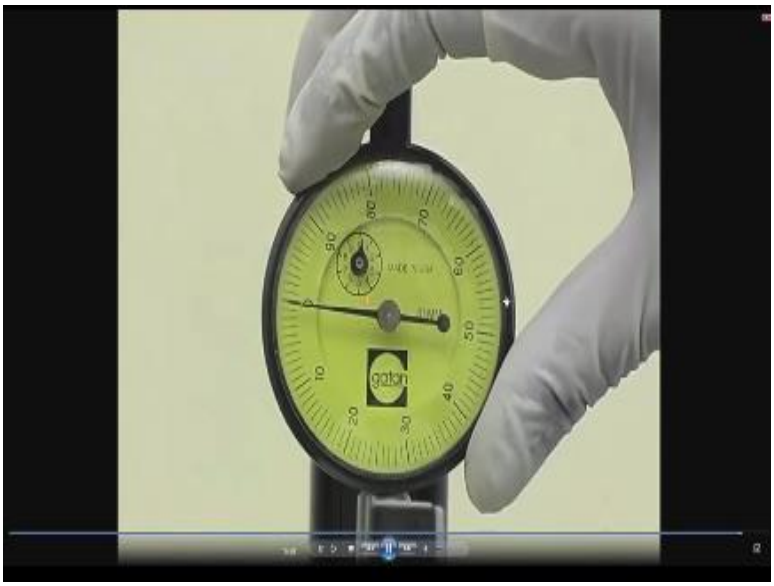

And this is a very important tool for ceramic material and in some cases people use it for metal as well but normally it is used for only ceramic because in metal you use it with the disc punch and each one is about a 10 micron division. So you have you can control the micron thickness so in order to do this cutting you put this ceramic it is typically a boron carbide and nitride or silicon carbide and then,

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Then what you, you should do is you take out the sample after that it means the local cutting width which produces a 3mm desk in a ceramic material. After that again you have to go to the dimpling action which produces further thinning and then it finally goes to the ion milling so now what we will do is we will go to an ion milling action.

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So we will take up the ion milling exercise they again I have taken only a metallic sample for a demonstration so it could be typical ideally it should be in a ceramic material. So to assume this a material of this 3 mm disc and then you can take it to the ion milling and before you put it into the iron Miller you should know how, how this specimen is being mounted into the specimen stage where it is being thinned by iron Miller. So we will just show you some of the details of how the specimen is held in a specimen holder it is called do post which is commercially available from the supply admission supplier.

So you have this, this specimen holder called video post I said and you see that there is a clip on the top of the you can see the both sides there is a clip and this is the holder.

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On which the do post gets inserted like this and then we will now show you how to load this sample in this do post and then later we will take this whole assembly into the iron Miller. So it is very delicate machine so it requires little more practice to use otherwise you will spoil this entire very sensitive region. And that is how the specimen holder is locked in the specimen stage now what we will do is we will put the 3 mm discs. What is being prepared from the ultrasonic disc cutter or a disc punch we will put the sample. And then you will demonstrate to you how it is being logged. So you place that three mm discs on this lever and then you can nicely are gently push this lever with the tweezers.

Which will just go above this locker I mean lockers locking pin and then you have to make sure that the specimen is being held by these two clips for that this is helps this, this knob helps where it can open and you can adjust it. And nicely you hold it with the clip so for that demonstration we will do it again. So now you tighten it so that it will hold this3 mm yeah now you can see that it is nicely lifted that means it is being held by these two clips and now you can remove this lever now this whole thing is ready for an ion milling system.

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So the do post assembly is taken with the sample mounted on the clip now we will see how we can load this into the iron Miller.

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So this is the typical ion milling equipment where you have the stereomicroscope through which you can monitor the, the milling action and you can have a closer look at it how it appears and you have the two bands left and right hand side hand. If you recall the presentation which I made on this iron Miller you remember that there are two bands one is top and one is bottom so you have the left and right. Then these are the two bands and some of the instrument instrumentation details we will go through. So that you will have a feel of what is happening in the iron Miller so you have that a specimen stage on this.

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And then you have the left and right gun current indicator here so now we will try to load the sample into the machine. So before we do that exercise we should also remember that for this kind of milling the, the thickness of the specimen which is after the iron Miller are sorry after the ultrasonic discard and dimpling it should be less than 40microns or 30 microns. Then only this system will work if it is too thick it is going to take a lot of time in fact that is not correct to you the correct way of using this equipment. Please remember you have to have very thin specimen after the dimpling at least 30micron to 35 to 40 microns maximum.

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So you have the ion gun gas flow control here then this is say the current indicator of the two guns and then you have the beam energy also being shown in the display. So what we are going to do is this is the specimen stage and which is completely kept under the vacuum so you can just have whether it is before loading and after loading you have to control this vacuum. And this is the airlock control for the specimen stage you can bring it up for the loading the specimens are bringing down to miller the specimen and so on. And this is for a raccoon and this is for windy before we open the chamber we have to vent it.

And you have the RPM which can be controlled that is a specimen rotation and then you have this is a time clock you can program the whole milling exercise.

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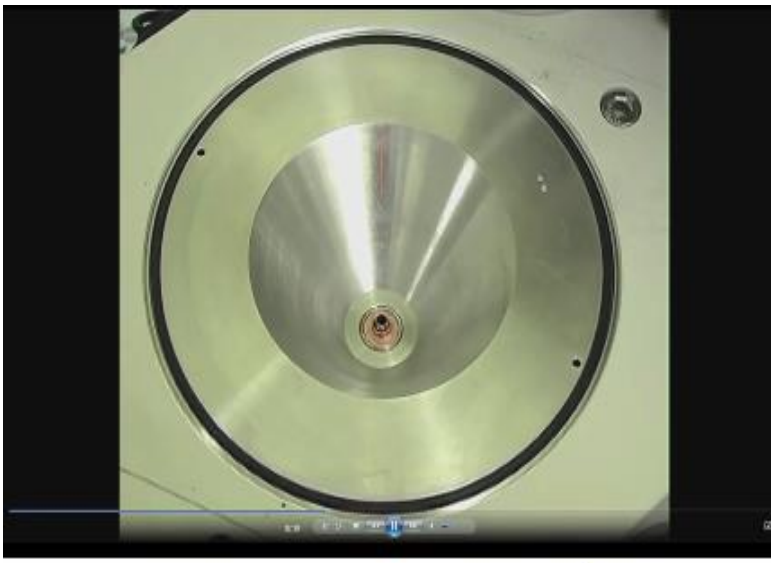

And this is how the, the specimen chamber will look like in a iron Miller so the do post what which is holding their the, the dimple grind ground specimen. Which will be mounted on this stage and this disc will rotate that is the RPM which I just showed will control the rotation here. So now you are seeing that the do post is going to get fitted onto this holder it requires little more practice before you get used to doing this whole exercise without damaging the sample as well as the equipment and so on. They are very delicate small devices which requires quite a bit of a cat to use them as well as maintain them because they are also very expensive.

So you need to have extra care to handle this. So finally we are loading this sample that the days of post assembly like this okay. Now it will be enclosed from the top for the vacuum and you have the glass window to see what is happening to the specimen.

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So now you pump it and then it will get locked so now you can what we are doing this pumping and once the vacuum is then it becomes tight. You see that you are not able to move it so it is ready for the milling now, now we can set your parameters what is the time and what is the current or what is the angle of the beam whether you want use a single gun or double gun both all those things to be decided beforehand. And in this exercise we will see we will use both the guns now you see that specimen stages gone down by using that air lock knob switch okay.

You can now see that we are showing that action moving this do post assembly to the top as well as bottom so now it is taken inside and in fact you can completely view the whole thing through a stereo microscope which is mounted on this we will see that I will show you how to use that. So now the specimen is going to think and now try to we are trying to set up the time as well as the gun angle please remember the very important aspect of the gun angle is depending upon the angle your specimen thickness I mean the thinning region will be decided if it is too high an angle it will be a problem. You will have a narrow theme region will be produced if it is a very small angle it will produce a large thin region that is to be understood.

So this is just to show you the shuttle action because with specimen we did not show that shatter so once the specimen goes down the shutter will close this chamber and you can. And if you if you want to monitor the action of the thinning then you open the shutter and then look at the specimen situation through the stereo microscope.

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So again we are doing backbone specimen is going down with this airlock control. We are switching on both the guns left and as well as right there so immediately you will see the corresponding the current and we are setting it for some time and we can adjust this to require thinning normally you give a few minutes off an hour is typical to give and depending upon this specimen nature. If we take a few hours to I mean half a day or so. So now the milling action started and then now you will see the current of these two guns and the beam energy this also can be controlled and typically this is the KV you use for thinning a typical metallic sample.

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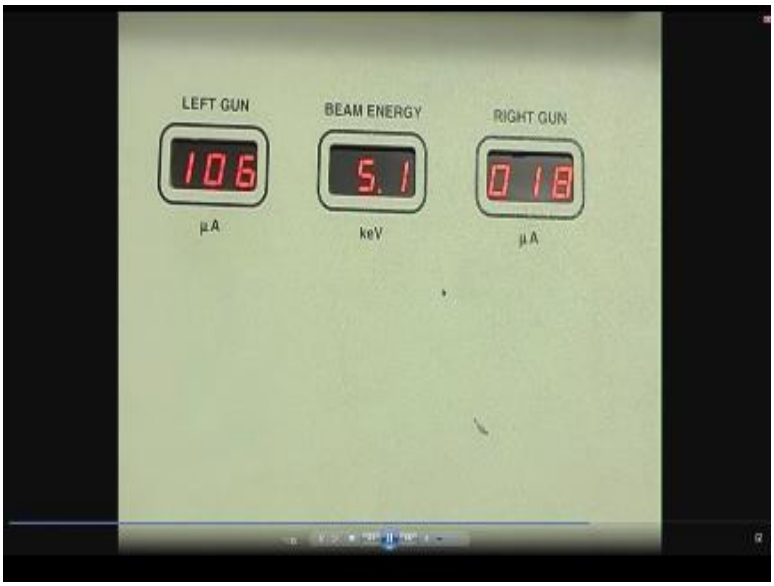

And metallic samples are used in this used in this milling because some of the you know alloys will have very sensitive to electrolytic polishing. Where you have the second phase particle which may fall out during the electrolytic thinning they are being polished with this iron Miller so now you can see that the two beams are falling on this yeah.

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Now all the lights switched off you can see that iron beam which is being, being bombarded on to this sample on both surfaces from top and bottom. So from this camera you can only look at this angle but if you actually look at the stereo stereomicroscope from the top you will be able to see both and bottom and top beams which is coming. So now we I think you have cut short that the full time. And we will now try to remove the sample and then show you what, what kind of precautions you have to take before you remove the samples that we will see. In fact this milling time suppose if after 30 minutes you can also give some additional milling time by changing the angle in order to remove the or clean up some of the debris which is being produced.

So higher KV you must have chosen but you can do a lower KV also and you can clean the sample for a few minutes with changing the angle it is normally done. And if you look at the sample which is being built now it is only a metallic sample and I will just try to take out the sample you are bringing the stage up.

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And then you have to went so that the you can remove will be able to open the chamber you have to be extra careful with the tweezers you are to remove this do post which will be little tight enough because you have you have put that into the hold and also. With lot of stress okay so now it has been removed and you can see and there again if you want to remove the specimen from this do post you have to go to that a specimen holder.

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Initially how we loaded this foil a similar fashion you will be able to remove them using that stage and you can see that there is a perforation in the middle of the foil. So now you can just put that sample into the microscope for the examination and you can also do after this plasma cleaning which will also clean up the surfaces and which will be finally ready for the examination okay. Now I quickly what I will do I will show other important sample preparation technique called ultra micro microtones.

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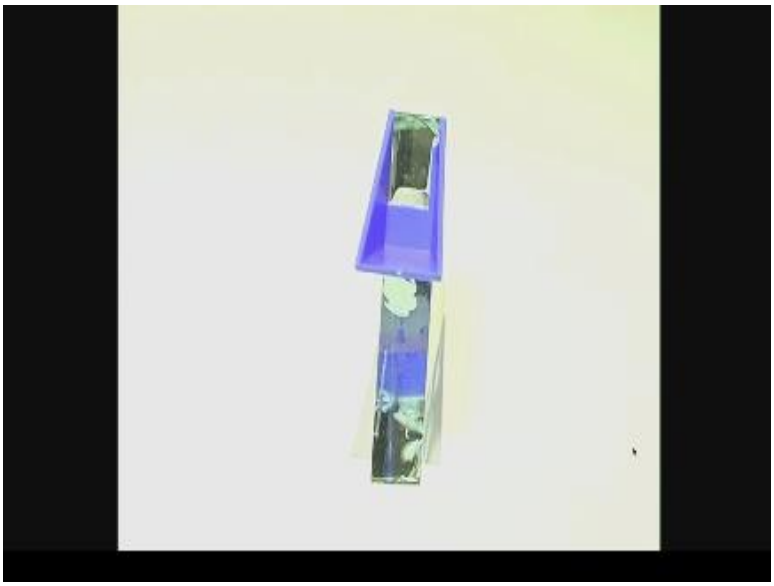

And as I mentioned in the presentation this particular technique is used for polymeric materials and this is the.

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This is how the equipment would look like this is a ultra microtome it is very, very useful equipment for a polymeric sections preparing a polymeric thin sections again you have the stereomicroscope on this top and thi.

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This has got two important part of this machine this is the specimen holder on and this has got a stage where you can move on this lever.

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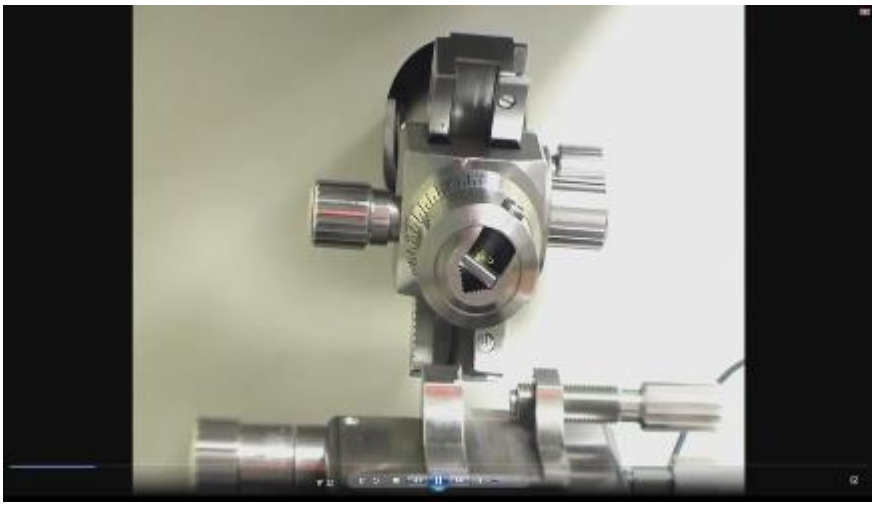

And you have the specimen stage here this is a specimen stage and you have all the gauges to control the stage movement left right and top bottom and so on. And this is the specimen holder you can open this and then put the specimen corresponding specimen inside and then lock it up into the arm. We will show the typical action of the cutting and then we will proceed with the actual demonstration.

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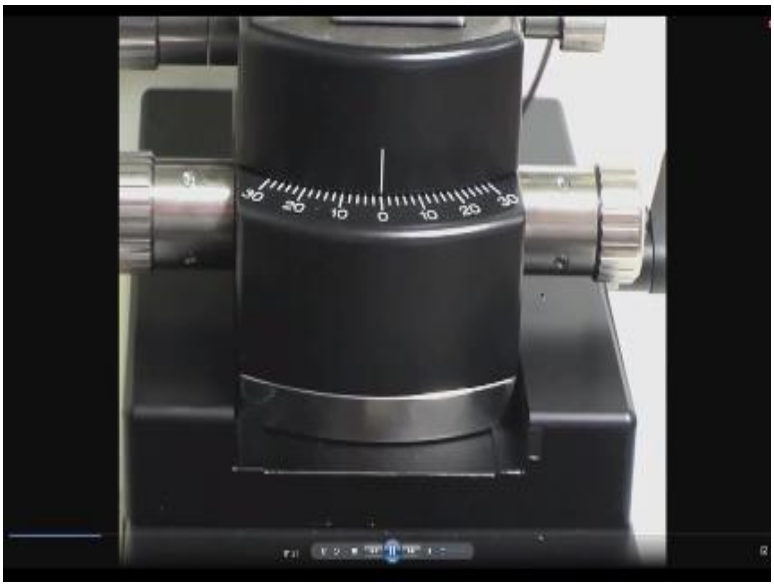

Please remember in order to use this equipment either you if you have a bulk sample you can directly machine it to such a way that it can be held in this holder directly. Or if your specimen is too small you have to mount it on a polymeric material.

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And then fix it I will we will talk about it in a few minutes and then you have the, the controls control panel you are seeing you have since it is a kind of a cutting tool you have the, the speed and the feed of the material cut to be controlled. That is what just we have seen in the display panel and you just see that this is how the, the cutting action will go you see that it just has the free play and then finally just press it with a controlled manner.

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Then the arm will just cut against the, the knife then your thin section will come out so this is the knob with which you can control the action of the specimen arm and also the feed and the speed of this arms can be controlled with this. So they these two movements have synchronized so this is how the cutting action will go on of the step size in hundred nanometers jump and then you have the speed mm per second and feed of the material what is the you know material you want to cut a tone time. That those things can be controlled by this and also you have the presets you can choose any one of them it will cut directly.

With that for example 1 mm per second will give you 50nanometers at 70 nanometer 200 nanometer1 micron and so on so you have all this set available to choose and yeah.

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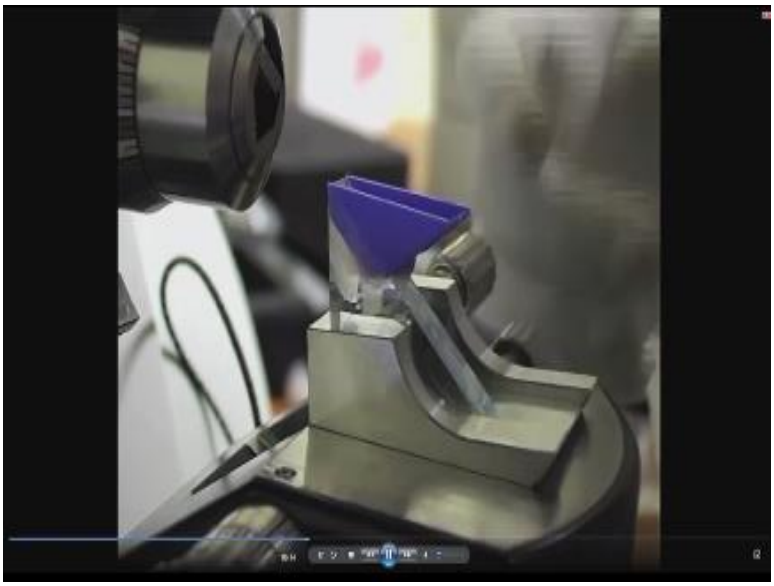

So this is how the specimen cutting tool is fixed this is a glass knife which has got a boat fixed on this cutting edge. So you can now see it yeah this is the glass knife and now we are trying to fix the sample in the specimen arm so what you have to appreciate here is here the sample is mounted with the polymeric Racine. So this the inside is the sample which is having a different color and it has to be made a cone shape like this in order to you know facilitate the cutting a thaw the specimen will be cut with this glass knife.

Basically you have two types of blades one is glass knife as well as a diamond blade and the glass knife is you can always make yourself and now you will see how the cutting action proceeds with this micro Tom please look at this.

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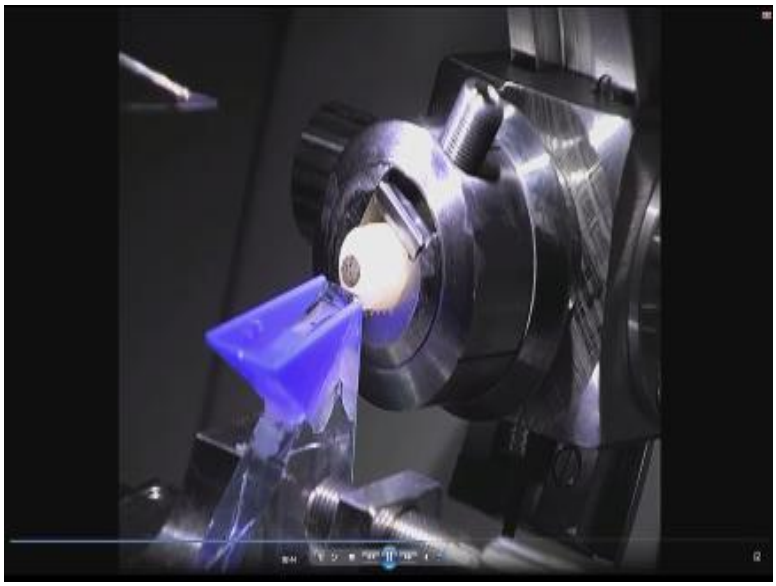

This is a boat with the water so this is the cutting edge of the clasp knife the material is going to get plunge on to this knife. Depending upon the feed rate of whatever we have chosen it will be your thin sections will get cut and then it will float on the water. And the specimen will be taken out by fishing by a copper grid from the bottom with the help of a teaser. And that can be dried after that and then it can be used for the microscopic analysis afterwards. So that is how it goes but you will now witness the technique cutting action you yeah.

Now one section is ready the next section is ready this current section has got the thickness of 100micron which you will not see in the with your naked eye but if you look through the stereo microscope. You will see that the thin section which is coming and floating on the blow both so how to prepare this glass knife the glass knife will have some limited life.

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And this is a knife maker we will also show you some of the actions of how to prepare a glass knife so that it is a regular consumable. So you, you get a lot of glass strips from this supplier so this is a very simple device which perfectly makes the glass knife.

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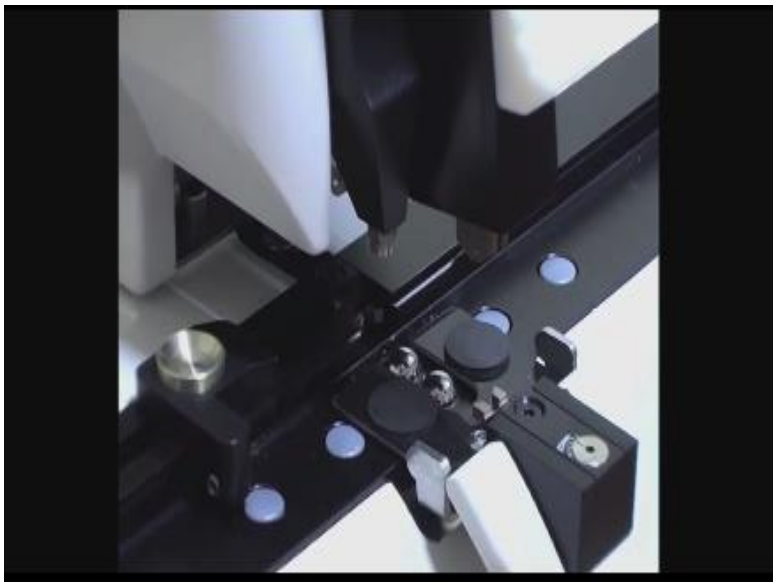

So we will also show you how to do that so take a strip like this so here we are going to clamp this into this machine this knife maker. So that will be very interesting to watch. So you basically tap it and then make sure that it is aligned properly and now you make a perfect scratch on the surface and then now apply a load then it breaks that glass perfectly into two pieces with the sharp edge. You can see that yeah now you can see that it is very sharp what we require is a triangular knife for that you need to cut this strips into a very perfectly square piece then the square piece can be cut in diagonal, diagonal manner to in order to get the knife of the required shape.

So this is the same action you can see that it may produce a short stretch and then, then load and it becomes too now one of this pieces will be used to produce a final glass knife which being used for the sectioning the polymeric material yes.

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Now you got a sharp edged knife which can be used so after few experiments this edge will lose it is sharpness that is why it is a consumable. So every time you feel if you want a very sharp knife you prepare it like this and, and you can produce a very beautiful polymeric sections using this tool and that is very important and another important aspects. I would like to mention here is after sectioning this polymeric material as some of the material requires some staining chemical staining in order to produce better contrast even for optical microscopy people use these sections.

And some of the polymers require staining operation which I am not going to demonstrate but just for our information for the completion of this presentation. So you have to look at the requirement of what kind of information you want in what kind of a polymeric material. So the staining is that another important specimen preparation technique which is required for microscopy analysis. Now the all this sample preparation techniques are very important techniques and very I mean I would say that these are the state of the art technique available today for the transmission electron conventional transmission electron microscopy studies.

And if you now be out what you have to understand is how this specimen is loaded into the TEM and for that again I would like you to go through the laboratory demonstration which we will do

it and for that purpose what I have done is I have requested our senior the technical officer in our transmission electron microscopy Mrs. Concha la Malashe is a has a tremendous experience in operating this equipment. So I just requested her to give a demonstration instead of me doing because she is she also like to do this and she is very good at it.

So I wanted to give her credit so what I suggest is after this the TEM demonstration you go through Mrs. Concha la Malashe demonstration which I will give it after this video and what I suggest is you just go through that video and carefully observe how the TEM is operated to in order to obtain a bright field image a dark field image a diffraction pattern. How the dark field image is produced using a gun tilt and all these basic informations you just observe in that video. And also you just if you have any doubts you note down and you can always contact me about the Machine operation details and in that demonstrations.

We have also added too kind I mean another microscope one is120 KV microscope the another microscope is 200 KV microscope where you will be shown demonstrations we are very Nano materials for example if you have the carbon materials where you like to see the, the lattice fringes or that the carbon tube multi-ball characteristics and so on. Just to give you a flavor of how the high resolution images are produced in a conventional transmission electron microscopy. So I reckon I request you to go through both the sections of that demonstration and get back to me if you have any doubts through email and so on. So with that I will I will finish all the laboratory demonstrations. I hope you will find it useful and you will have will be immensely benefited from this all this laboratory demonstrations thank you.

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