**Indian Institute of Technology Madras**
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**NATIONAL PROGRAMME ON TECHNOLOGY ENHANCED LEARNING**

**Lecture -21**
**Materials Characterization**
**Fundamentals of Transmission Electron Microscope**

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Hello everyone welcome to this material characterization course in this class I would like to discuss about the sample preparation techniques for TM analysis you have to understand one thing the sample preparation is the biggest task as far as the tem analysis goes it takes lot of pains to prepare a suitable sample and getting a suitable sample is almost becoming you know it is a very long process because you need to produce a sample with lot of care because you want to produce a very thin section without I mean incorporating an external strain or I mean contamination in order to characterize the material in its original form.

So it becomes very difficult to prepare a sample and also it requires lot of patience and time so I will just go through some of the basic principles which has been I mean which is underlying these specimen preparation and also I will just show some of the laboratory demonstration in taking you through all the tools which we have in our electron microscopy laboratory so I will just briefly give a presentation on various techniques adopted for a sample preparation whether it is a metal ceramic or a polymer or a powder and soon so then we will take a corresponding individual case studies.

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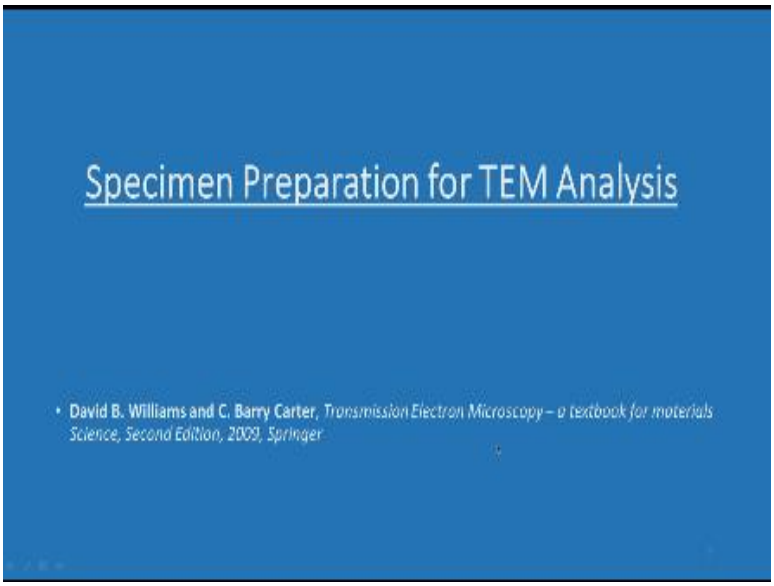


So most of the information is taken from this text book by David Williams and very Carter

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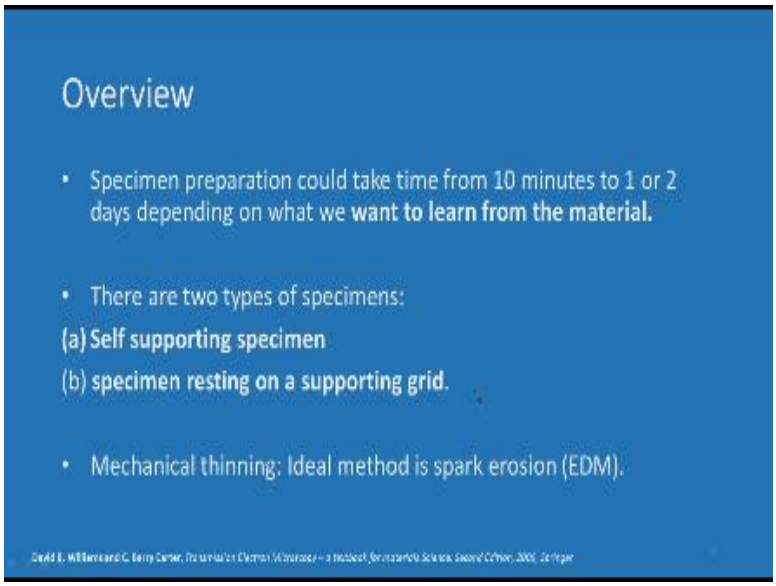


If you look at the overview of the specimen preparation specimen preparation could take time from 10 minutes 21 are two days depending upon what we want to learn from the material. There are two types of specimens self-supporting specimen resting on a supporting grid we will look at what is these two types and mechanical thinning when I say mechanical thinning I am keeping a material like metals and ceramics in mind.

So we will also show the procedures of what is mechanical thinning and the ideal way to do this job is to using the spark erosion or electric discharge machining instead of doing a manually this is also we will demonstrate.

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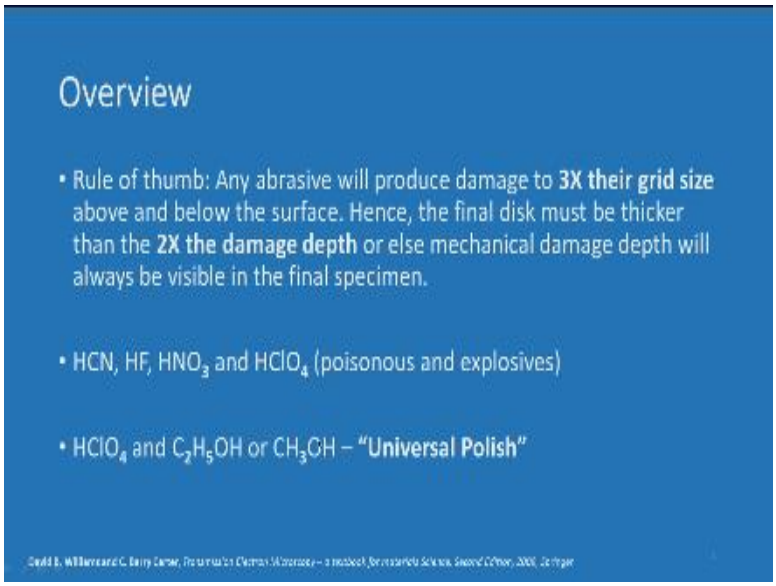


A rule of thumb is any abrasive will produce a damage to three times their grid size above and below the surface hence the final disk must be a thicker than that 2x the damage depth or else the mechanical damage depth will always be visible in the final specimen so what we are trying to say here is we are simply talking about a mechanical filling you will see that we will be demonstrating how to prepare a thin metallic sample or ceramic sample.

Where we will be rubbing this specimen against an emery paper and an abrasive paper so you have to be very careful about this because this abrasive paper will have in itself you know it will produce a damaged up to three times its grid size so the size of the American / is important so you have to take a very fine emery paper if you have a very thin sample otherwise it will produce its own damage which is not a characteristic of a material itself.

So you have chemicals which we use in our specimen preparation you have hit $HCN_3$ of $_{HNO4}$ you have to be very careful because most of them are poisonous and explosives and then you have this perchloric acetic that is hitclo4 plus ethanol or methanol mixture is called universal polish that means most of the metals if you use this as an electrolyte that will polish so what we are now specifically talking about is a mechanical cleaning followed by an electrolytic polishing we will

get into the detail but since in earlier days the analysis was primarily done on a metallic sample and these are the techniques were developed in the beginning so now you have a sample preparation techniques for other materials such as ceramics polymers and biological and powder and so on.

So we let we will just also follow that order we will just look at first metallic samples and still more relevant people are using it and then this technique which is specific for a ceramics and polymers I will just keep mentioning it then and there.

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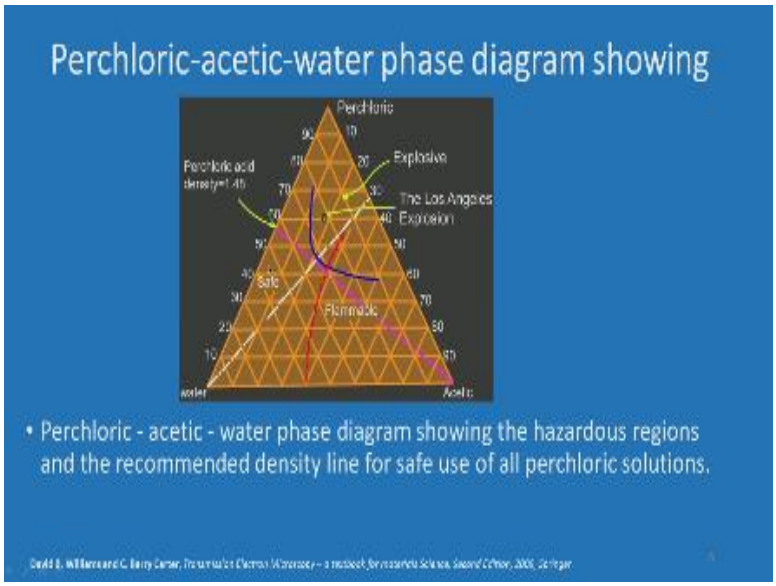


So the electronic mixture perchloric ascetic and water phase diagram is very important because I say mentioned in the previous like this is mostly used as a universal polishing electrolyte so if you see that a phase diagram this is perchloric acid water and then this acetic acid it shows the hazardous regions and the recommended density line for a safe use of all the flooring solutions so you see that you have a boundary with which if you mix these three assets it will be flammable or it could be an explosive also so you have a safe line under which you have to operate whenever you produce a electrolyte for the metal thinning or material thinning.

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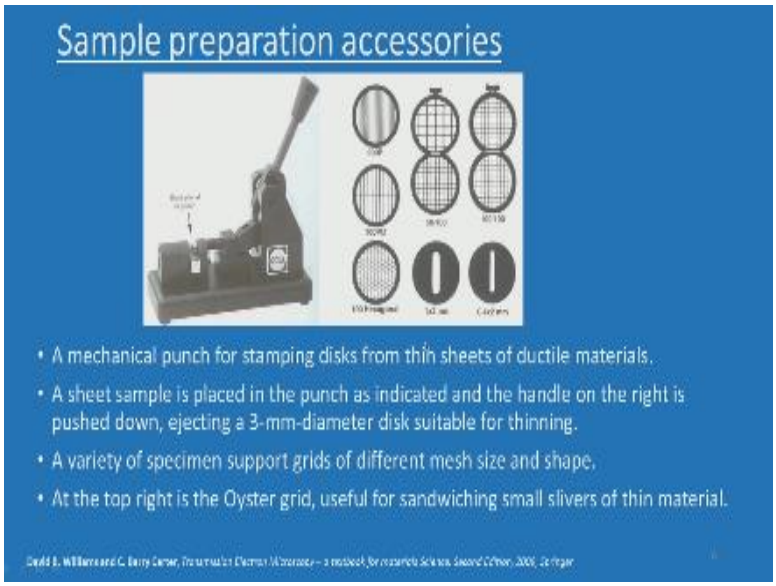


So just to give that point this slide is broad and some of the sample preparation accessories are shown in this slide this is a punching machine a sheep punching mission and then these are all a grits of various kinds and this is the you would say that no this is foldable grits where you can keep your sample inside and then if it is locked so that it is safe and intact.

So we will go through all this when we actually take up some practical examples a mechanical punch for stamping discs from this thin sheets of ductile materials so this punching machine will produce a metallic specimen of 3 mm discs in order to suitably placed in a sample holder in a TM so we will also show you the actual sample holder how it is being used a sheet sample is placed in the punch.

As indicated and the handle on the right is pushed down is acting 3mm diameter disc suitably fourth inning so it is I will show the live demonstration so don't worry about it and why 3mm because it has to suit the sample folder a variety of specimen support grades of different mesh size and shape are the top right in the oyster grade useful for sandwiching small Silver's of thin materials so this is firm sandwiching this sample.

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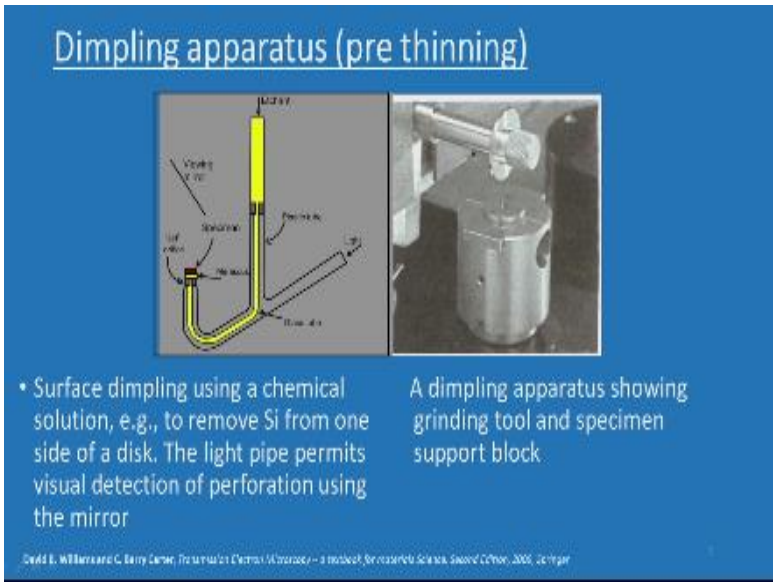


And then you also have the dimpling apparatus this is all coming under the pre thinning apparatus category we will we have this mission in our laboratory we will demonstrate but the principle is simple a surface dimpling using an chemical solution example to remove the silicon from one side of the disc the light pipe permits the visual detection of the perforation using the mirror.

So this is the setup so you have the etchant and then this is the jet and this is a sample script on this and you have the mirror to view it and the light will pass through this perforation once the etching is completed which produces  a hole in this . A dimpling apparatus showing a grinding tool and a specimen supporting block so you have the sample will be kept here typically a ceramic sample will be kept here and then this will grind this specimen and make a dimple that is called that is why it is named dimpling apparatus we will actually show you the demo how it is done.

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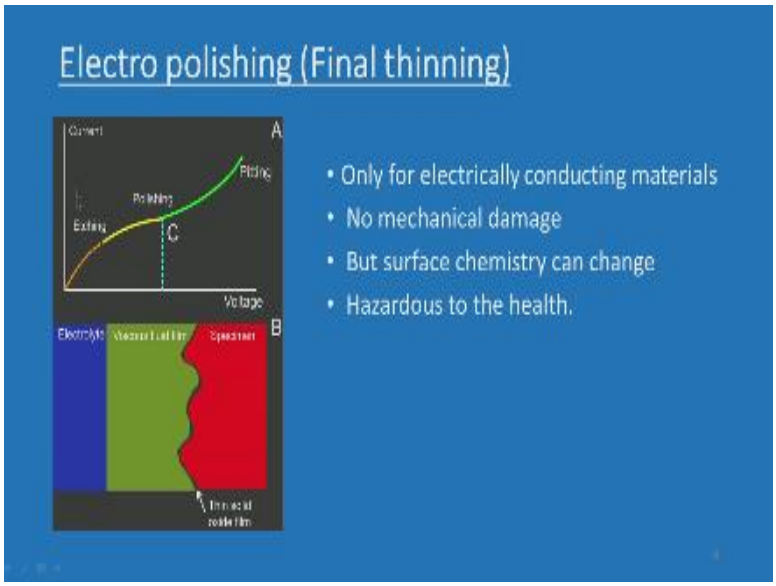


And then mostly the electro polishing is called final thinning so you have the typical I characteristics that is correct voltage characteristic of an electrolytic reaction you have itching region you have a polishing region and then pitting region so before you start any of this electrolytic polishing you have to characterize the electrolytic action so you have to choose an appropriate current and voltage in order to stay in this on region otherwise it is again a very tricky situation.

You can see that you have this kind of a layer which forms on the specimen you have the specimen here viscous fluid film and then electrolyte and you have a thin solid oxide film also forms on the specimen we will see how to handle this and one of the criterion for using this electro polishing technique is so only electrically conducting materials no mechanical damage but surface chemistry can change and hazardous to the health you have to be careful better to wear precautionary tools and safety devices before we engage in this exercise.

So what this curve shows electro polishing curve showing the increase in the current between the cathode as the applied voltage is increased polishing occurs on the platoon etching at the lower voltage and pitting at the higher voltage so the ideal conditions for obtaining a polished

surface require a formation of a viscous film between the electrolyte and the specimen surface. So this is an ideal condition for the specimen preparation so how do we make sure that so we have an in an automatic machine which characterize the ideal conditions for polishing i will show you when i demonstrate this action in the laboratory.

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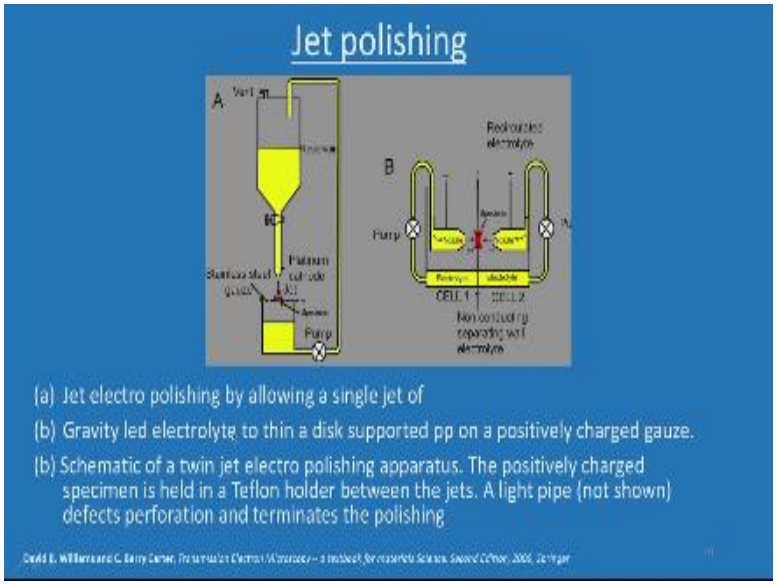


And the another type of jet polishing is shown here basically you have the two methods what we have in available trees this method you have a twin jet you are the sample in between and this is a research circulated electrolyte and then you have the pump which passes the electrolyte through these two nozzles and then producing the dimple on both sides and eventually make a perforation inside the sample and that forms your final sample for the a TM analysis.

So first one is electro polishing by allowing a single jet of electrolyte and this is a twin gentle tonight so this is a gravity let Delta light to thin disk supported on a positively charged gaze so this is one method and what we are going to demonstrate in the laboratory is this and as I mentioned this is a twin is called a twin jet polisher.

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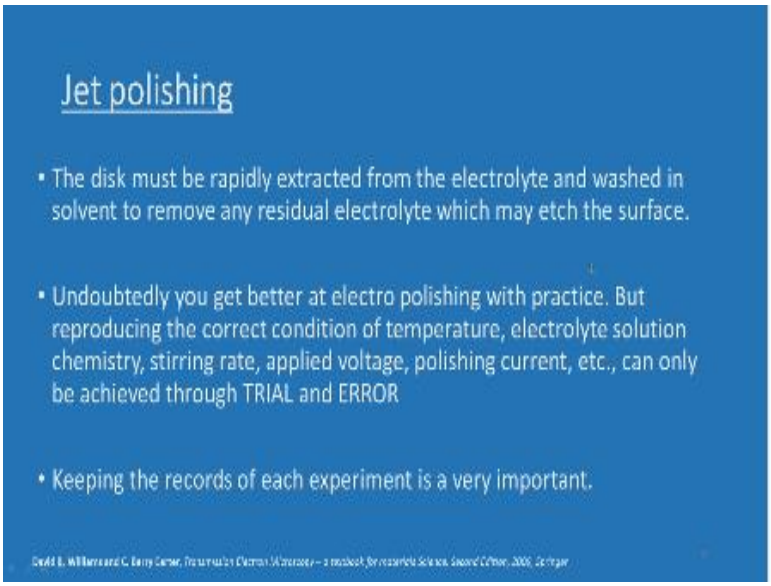


So the disc must be rapidly extracted from the electrolyte and washed in the solvent to remove any residual electrolyte which may make it has the surface. This is very important operation in the injured polishing once the sample is taken out from the jet polishing unit you see you have to remember that the electrolyte is still sticking to both the surfaces.

So you have to rapidly clean this specimen which is taken out of this electrolyte and thoroughly washed with the water not in one beaker or you have to keep three beakers with an alcohol and then you have to rapidly agitate the specimen to remove in order to remove the electrolyte from both sides so that the etching action is stopped and then you are which will eat away your material slowly which you may not see I twitch your naked eye but it will one you once you put it in the microscope you will see that that is more important.

I will also show this aspect I am telling this right now itself because this is one place where people fail and then they do not get the a good sample of after electrolytic polishing. So this is very important step undoubtedly you get a better at electro polishing with the practice obviously this is kind of you know it is a recipe everybody will have their own way of doing things because electrolytic reaction is depending upon so many factors which one has to look at the current the

voltage the specimen condition sample surface condition everything will vary a person to person or a specimen to specimen with which they are handling.

So even your pre thinning operations will have a significant role to play in an electrolytic polishing for example if you if you if you are not very careful with the abrasive thinning or mechanical thinning wear and then you may be using a glue to stick this foil under a bulk sample and if you do not remove the glue properly then also your electrolytic reaction will not be proper maybe once said it is stuck with the glue the other side is exposed to the electrolyte.

So it will be a non-uniform thinning will takes place and one very important aspect everybody forgets in this exercises suppose if you do the electrolytic polishing I mean before you come to the electrolytic polishing you better to look at the sample under the optical microscope both sides. Whether you have any contamination sticking onto your sample it could be a deputies from the emery paper or abrasive paper or it could be a glue which is still sticking at one of the edges are it should be if it is stored in an open space.

That dust particles or any other foreign material which will be sitting on the sample which you may not observed with the neck naked eye and it is essential that you observe this sample under an optical microscope before you come to electrolytic polishing then you will if you follow that procedure and then follow the jet polishing procedures I am 100% sure you will be able to produce a nice hole in the sample with a very large thinning a thin area.

So the importance I will just show you when we put the sample in the microscope and then you will appreciate what I am now talking about so it comes with the practice but reproducing the correct condition of temperature electrolytic solution chemistry stirring rate applied voltage polishing current etcetera can only be achieved through trial and error so this is another important point you have to remember you have to do few trials before you come out come out with the proper combination of parameters keeping the records of each experiment is very important of this to stabilize this jet polishing.

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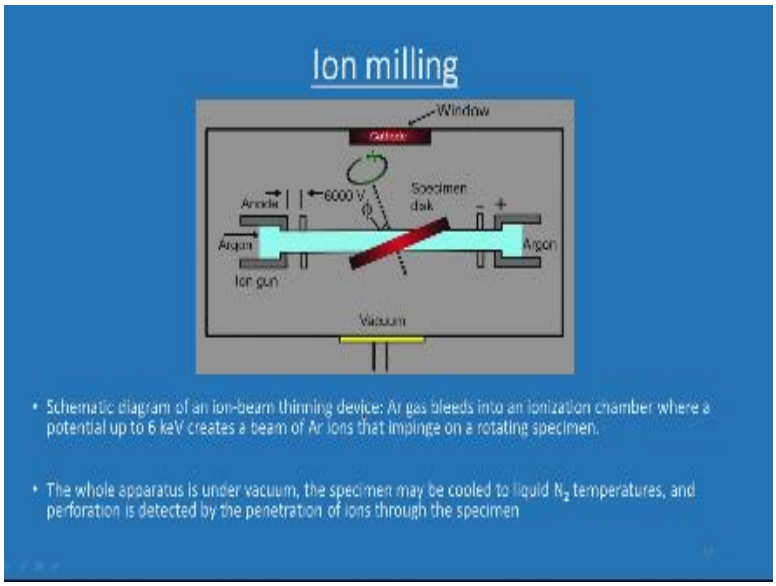


An another important technique is iron willing this particular technique is especially meant for a nonmetallic samples especially a ceramic samples. So we will briefly go through this schematic wear it demonstrate the how the basic functions of the iron milling machine you have the specimen which is kept there and then you have the circuit iron gun which comes and impinges on the specimen surface and it will start to become thin.

We will see what it is so the schematic direct diagram of anion beam thinning device will look like this argon gas bleeds into an ionization chamber where a potential up to 6qelectron volt creates a beam of argon ions that impinge on the rotating specimen so this specimen is rotating that is what shown in this arrow. So the beam of organs will fall on this specimen surface we will also shown the actual  mission how this operation is carried on. The whole apparatus is under back comb the specimen may be cooled to a liquid nitrogen temperatures and the perforation is detected by a penetration of ions through the specimens.

So in fact you will see that the specimen is being pinned and by both the direction so and also in some of the missions you have the cooling system which can go up to liquid nitrogen temperature and some of these systems will not have the liquid nitrogen a setup so the iron

impingement on the both sides will be there and you can see that and then how nicely the thinning takes place in this particular region where both the beam-in impinges on the sample on you come on the straight line.

Please remember the pre themed samples are only put on the ion milling machine not the bulk sample which is very important so this is again very delicate to handle it because if you are not performing the pre thinning exercise much more carefully again this and milling is not going to help you which is going to have lot of problems which we will demonstrate in a laboratory.

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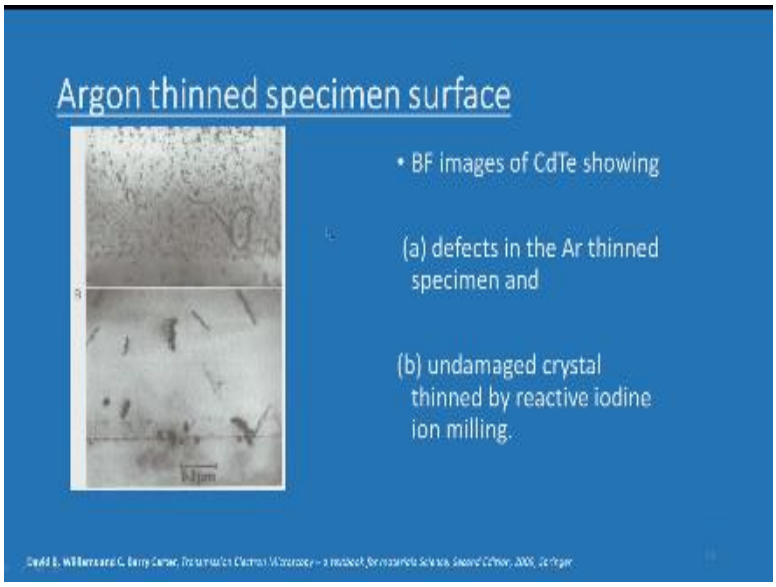


So another important aspect of this organ thin specimen surface you can see this micrograph these are all bright field images of cadmium telling the defects in the Argonne thin specimen and this is an undamaged crystal thin by reactive iodine iron willing. So you have to be extra careful about this ion milling is going to create a lot of surface damage and in order to use this you have to you have to keep that fact in mind it is going to create a lot of defects.

So if you do not choose an appropriate parameters for this thinning here again you are going to spoil your sample are you may be without noticing this if you start doing the microscopy you

will be wrongly characterizing the damage which is generated by sample preparation which you will interpret as the material characteristic which is quite dangerous so you have to keep that in mind.

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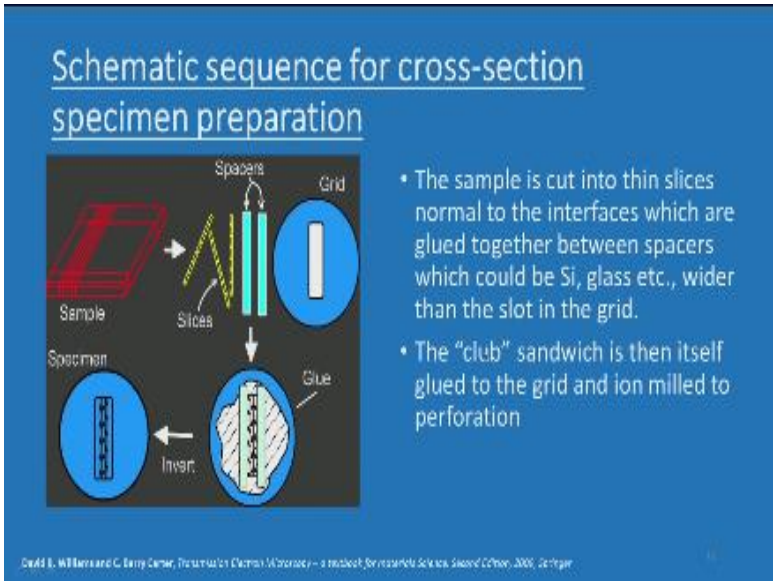


So if you want to produce a cross-section specimen then the here are some of the procedures which is shown here the sample is cut into thin slices normal to the interfaces which are glued together between the spaces which could be silicon glass etcetera wider than the slot in the grid so this is the spacer so you put all the slices and then put it inside the club sandwiches then itself glued into the grid and iron willed to the perforation. So the whole thing is put into this spacers and then and then it is slightly glued into the grid like this and then finally iron-willed. So this surface get iron-willed and then you will be able to see the cross-section specimens so it is very involved procedure here again.

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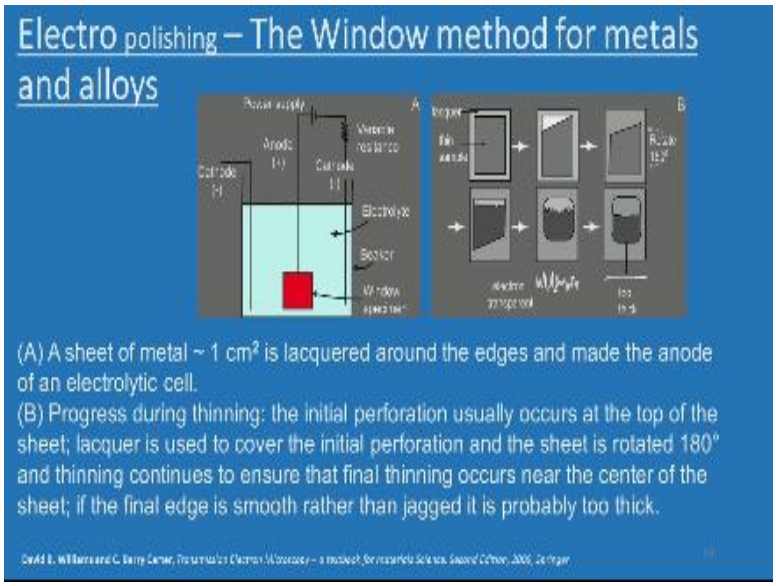


And one of the most conventional way of preparing the sample was through electrode electrolytic polishing is the window method of for metals and alloys where you have the electrolyte set up for polishing electrolytic polishing and you have the sample window which is completely covered with some kind of Lacer.

So and then it will go through the sequence of electrolytic H like this so we will go through the remarks a sheet of metal one centimeter is lacquered around the edges and made the anode of an electrolytic cell and if you look at the progress during the thinning the initial perforation usually occurs at the top of the sheet like this latter issued to cover the initial perforation and the sheet is rotated 180 degree like this the thinning continues to ensure that final thinning occurs near the center of the sheet if the final edges smooth rather than a jag it is probably too thick.

So if you have a very thin region you will your final specimen will appear like this if it is a too thick it is it will be like this it is true for even a twin jet polishing also if you have a thin specimen you are what final surface will look like otherwise it will be like this see one important advantage of doing this window technique is you will have large thinning region as compared to a twin jet polished three mm discs and here it is not 3mm risk it is a big window you are getting.

So you will get a very large area of thin region to examine which is a great advantage for a TM analysis.

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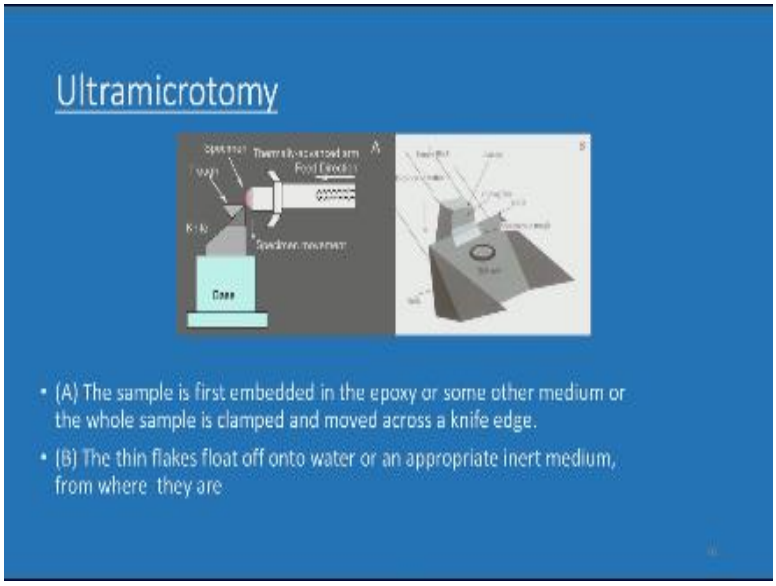


The another important technique is ultra micro to me this is primarily used for a polymeric material soft material and we will just go through the principle and then here again we will show the actual demonstration in the laboratory how to do this. You see the basic operations shown in the schematic you have the base with the knife and then you have a small truf we call it as a boat sometime and the specimen is kept in are stuck with then a moon.

I mean you can this is a mobile arm I would say that thermally advanced arm and this is the feed direction like you were a lathe and that is how the specimen moves up and down and this is a knife that dark triangle and this is at rough so the specimen will impinge on the knife edge and then it will be coming out like this so this is the rough in the other view enlarged view and this is the knife and this is your arm this is how it look like so it will cut through like this.

The specimens going to just float on the tough because the tough will be filled with water and then you have this is a cutting surface and you have the sample here it is some PDP sample block and the direction of motion is this up and down so it will be taken as a simple thin section of a desired thickness which can be taken with the TM copper grid you can just fish it out and then directly look at under the microscope.

So the sample is first embedded in the epoxy or some other medium or the whole sample is clamped or moved across a knife and see if your sample this all depending upon the sample dimensions if you have the very small samples you have to stick it with an epoxy or something else or your sample itself it is a bulk you can directly clamp it onto the arm and then perform this operation.

You produce a thin flakes of that floats off onto the water or an appropriate inert medium from where they are taken onto the cop grid like this.

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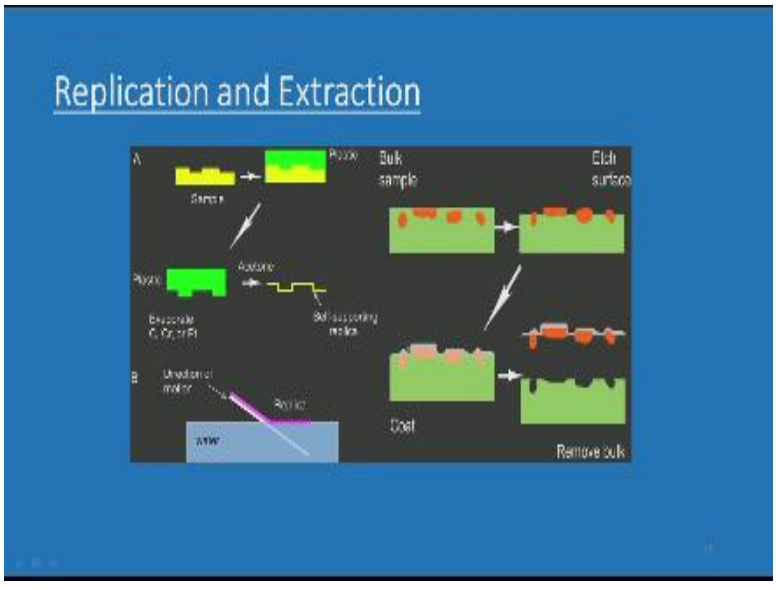


See another important technique is a replication or an extraction technique which is very important for some of the analysis of a second phase particle in the metallic specimen for

example if you have very you know suppose if you have a magnetic sample for example iron or steel and soon and then you have the second phase particle which is there in the material in order to obtain the information from the second phase particle alone and if you put the metal foil the metal foil also will influence the electron beam and because of that you will not be able to focus or obtained specific information from the second phase particles.

So in order to take the you know the information from this second phase particle alone and this extraction elliptical a replica technique is being used see the one of the bay they have to produce a replica is shown in the schematic so you see the bulk sample which has a second phase particle like this so after etching you are going to clean this and then it will all your second phase particle will come almost to the top surface and then you do coating with typically with the carbon or anything in any thin film you coat it and then you let us just extract this the carbon film from the bulk.

So that will have this kind of a situation where all your second phase particle will stick to this carbon or any thin film which you code and if you are able to extract this I mean since the film is carbon film is extracting this article is from the bulk material that is why it is called carbon extraction replica and this replica can be put on the grid and then you can you it under the microscope.

So here it is shown with some kind of a typical plastic and wit which will take the impression and it will with an a chemical etching you produce this kind of a self-supporting replica then you can view it this is this kind of technique is for some fracture surface you can analyze you take the impression of the fracture surface and then look at the features carefully and once the replica is produced then you can just slide it on the water then it will float like this.

So that is also clearly demonstrated so this is very important technique if you want to take the information T or if you want to perform a TM analysis on a second phase particles without the interference of the matrix then you will be able to use this technique.

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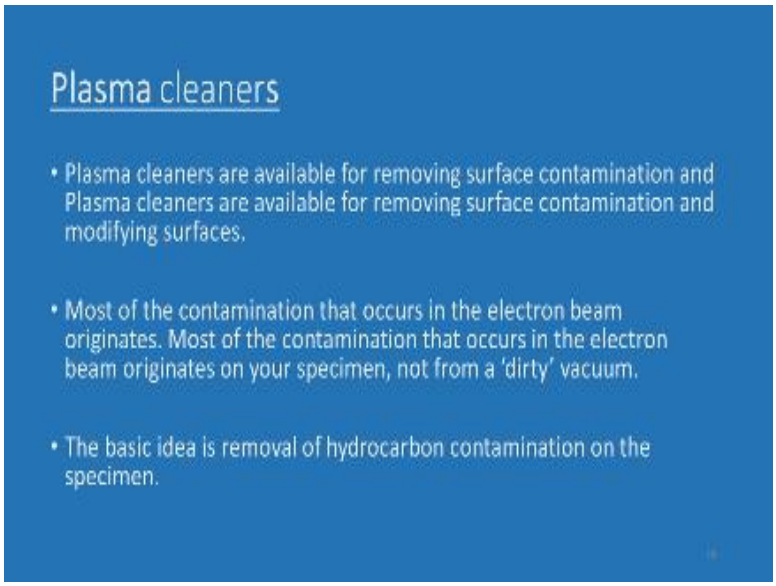


And finally you see that after doing all this jet polishing or final polishing then if you want to clean the surface of the specimen because after all these things your specimen is highly corrosive or amenable to all the oxidation and soon then it is better to clean that surface before you put the specimen in the TM column so in order to clean that you have something called plasma cleaners.

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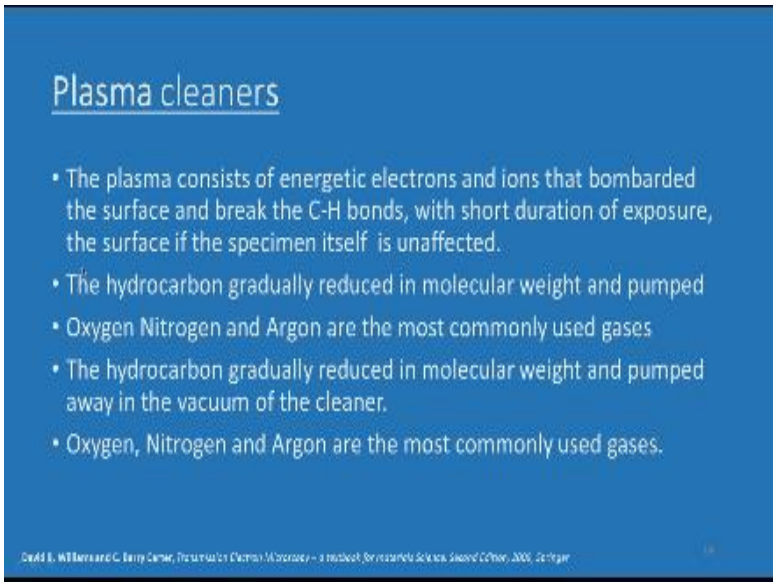


The plasma consists of energetic electrons and ions that bombarded the surface and break the carbon-hydrogen bonds with the short duration of exposure the surface if the specimen itself is unaffected the hydrocarbon gradually reduced in the molecular weight and pumped oxygen nitrogen and argon or the most commonly used gases the hydrocarbon gradually reduced in molecular weight and pumped away in the vacuum of the cleaner. Oxygen nitrogen and argon are the most commonly used gases for this kind a operation.

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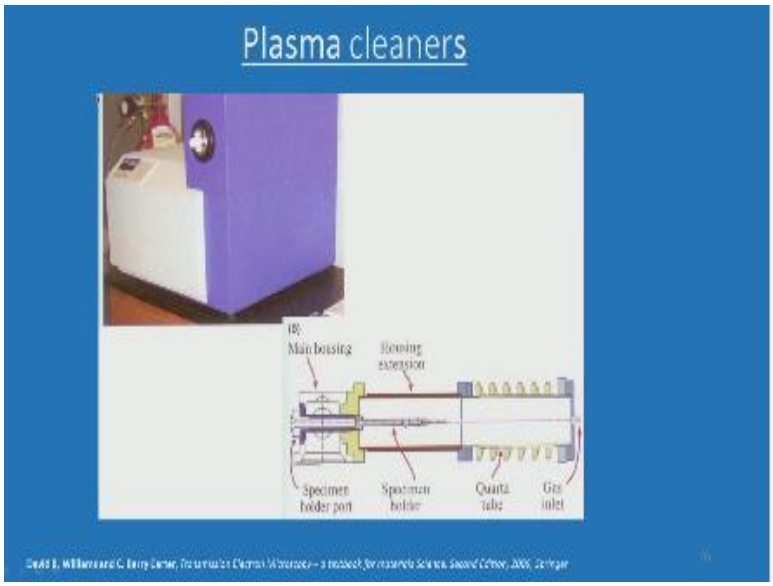


So this is the typical plasma trainer this is how it look like so the instrument and cross section details are shown in this schematic you have the this a main housing and this is the specimen holder which after it inserts into this chamber how it look like. So you have the course tube and this is a gas intellect and which comes Andy althea vacuum the specimen this is the warring with which you know you the specimen will be inside the vacuum of the microscope similar chamber is here and then the cleaning operation is done.

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So now what I will do is I will just quickly go to the laboratory demonstration and I will just start with a simple low-speed saw and this is atypical diamond blade which is being used to produce any thin section.

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So we will start with from this how we proceed with the cutting action so you have the specimen you can put it in the diamond saw slow speed diamonds are the advantage of this mission is you can produce a sections of very thin sections you can produce typically point 1 mm and these are the details of the equipment.

You have the equivalent for the slow speed XO some of the coolants are commercially available so we can directly use it and the material which is being taken for this demonstration is some aluminum alloy and you see that it is now almost producing a thin section and you can and the specimen thin section is I will the thin section is further polished by mechanical thinning for that yeah it has-been pasted on me a bulk sample like this.

And then being rubbed on the very fine emery paper and then you just measure the finest with a micrometer the thumb rule is should be below 100 microns you have about 67 micron thick foil and then you take that foil into the punching machine so this is how the 3mdisks are produced in this using the punch mission you can clearly see the punching action with the punch on the die and your 3 mm discs will be collected in this a container which is kept below.

This so after punching this you can produce as many number of three mm discs from the a large thin disc which you have me chemically polished and then you can produce as many specimens as possible from this you can punch out and then you will do this jet polishing okay I will stop here.

Then I would like to tell you something before we proceed with this jet polishing demonstration in the if you do not produce this thin section with the slow speed hacks are properly then you will have the problem throughout the subsequent operations for example if you want to produce a parallel thin section it is very important that you remove one section by the diamond blade you remove that.

Do not take the first because you may not know whether whatever the sample you are putting for the first cut is it parallel the surfaces may not be parallel so in order to produce a parallel surface you remove first section do not use it from the second section you start using it the approve you can go up to very thin section depending upon your sample availability.

So take that and also make produce their section with the proper cutting parameters for example I did not talk about the kind of load you can use for the cutting the speed and the coolant flow everything is important and if you are not following this procedure then you may spoil the specimen there itself or if you produce a tapered section from the slow speed saw itself then that will carry on you will have a problem until the final jet polishing.

So a slow speed AXA if you if you have if you know how to use it you better learn it and then produce a parallel a uniform theme section and then that parallel uniform thin section should be maintained during the your mechanical polishing you should not apply a lot of mechanical force because you will be generating a damaged by your own force so you prepare a very thin section and then get into this 3 mm disc.

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So now you continue this we will continue this jet polishing unit so this is the specimen holder in a twin jet polisher this the 3mm desk is kept in the specimen holder and then now this will be put it inside d to inject polishers. So this is a liquid nitrogen bath we are going to our now this is an electrolyte and then this will be kept in a mixed with in liquid nitrogen bath and then the setup will be immersed in that.

So the electrolyte is being cooled so the whole the electrolytic thin jet unit is being is kept on the birth and then you insert the sample in the slot and now you can look at the polishing recipe on the digital display and here you can have a standard recipe which is provided by the machine supplier or you can have your own recipe as I mentioned in the representation the IV characteristics of the sample should be first established and you have to choose an appropriate electrolyte voltage temperature polishing time and so on.

So the two in depending upon the specimen and the thickness the machine gives a kind of rough range of current and voltage optimized and then you can choose that guideline and then further proceed with the polishing machine so what is being now done is just first do a scan that will

give you an idea depending upon the thickness of your sample it will give you the appropriate thinning region so that is called s scan or it can say that a simple scan.

It will show some air rough idea of the polishing IV characteristics so if you follow that method you will avoid the spitting or not pitting region or as well as our if it is not good enough for polishing so in this case this point has been identified as the polishing region then you go ahead with the actual electro catalytic polishing.

I repeat the recipe what we are following in this electrolytic method it is not done in one go it is done with trial and error you have to do two three times to see whether you are getting the kind of specimen thinning which you are wanted so what i will do is i will just go to the thinning after thinning this the important step once the thinning is complete it gives the beep sound because it will sense the light through the specimen.

Then you have to remove this very quickly and then you have to wash it in a an alcohol much more rigorously or to wash three four pate read is you can keep it and wash it thoroughly and then only you will be able to look at you can see that a small hole and this is going to be put on the TM holder this is a 3 mm disc which is being loaded onto the TM specimen holder yeah this is the single tilt holder and now before we put it into the microscope this is being cleaned by a plasma cleaner.

So this is a plasma cleaning unit again it has got its own recipe you have to follow procedures and since it is done in a vacuum again you have to check with the vacuum and like a jet polishing machine this also has the ready-made recipe is available for type of specimen or type of operation. So now the vacuum is on so the pumping is being done and soon you will see the so these getting ready now so this cleaning is done typically for a few minutes.

So there is a recommendation depending upon the sample so typically it is four minutes in this case and then you see that the indication with the glass globe in this schematic and you can also view through this window how this plasma is getting I mean specimens getting clean by the

plasma and then it is all very simple automatic process then now your sample is ready for the viewing in the TM.

So now so that metallic part is over now what we are now going to see is suppose if you have the nonmetallic sample in case of a ceramic sample typically a ceramic sample now I will just demonstrate how a ceramic sample is being pre thinned for example it has to go through a dimple grinder which I just showed in the presentation and for this dimple grinding this the simple video which we are not going to show this is a stub and this is a glue typical a polymer-based blue.

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Which is commercially available so you put this glue you melt it on the hotplate and put a glue on this stub in this case for just for demonstration purpose we have not taken a ceramic sample but it is again a metallic sample just for a demonstration but typically a ceramic sample would be it should be used for this kind of thing so the specimen is being stuck on the sample stage and this is a magnetic stage.

Then you have the a simple microscope to look at the specimen surface and alignment you can see that so it will it will be done very fast so you see that now the microscope will be mounted on the specimen stage so that you can just look at the thinning action some pre thinning action I would say so now you can look at this then align the specimen stage in order to produce a proper dimple in this center of the 3 mm dis.

So that is very important you have to produce a dimple in the center of the sample that is why this microscope issued then you choose again here an appropriate parameters so you have the grinding wheel which will touch on the sample and that this is the dial gauge which controls the thickness so you can control the thickness in microns and then speed of the grinding wheel is controlled.

You here again you have specified recipe or you have to arrive at your own recipe for thinning it and this is in a ceramic abrasive glue which is placed on the specimen and then now the grinding action will takes place that is dimpling action you can see that the specimen stage is also rotating in order to produce a uniform a dimple and the center.

So this is how the dimpling is done so it is very important that the dimpling is done in the center of the specimen and after this dimpling you can you can take this sample for the further thinning in the iron Miller so this is après thinning typical p thinning operation for s as i mentioned it is a ceramic sample so after this sample is taken to the iron Miller or if you are dimpling in a in a bulk sample.

You have to produce a 3 mm disc 3 mm disc cannot be produced with the disc punch which we have shown because only metallic specimens are prepared by that disc punch so for a ceramic material you have something called an ultrasonic disc cutter with that you prepare a 3 mm and a disc of specimen from the ceramic sample then you can perform this dimpling action then go to iron Miller so there are three stages the is the next class I will demonstrate the how the ultrasonic disc cutter functions and then followed by the ion milling equipment and then we will move on to the next technique. Thank You.

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