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**On**
**Technology Enhance Learning**

**Course Title**
**Advanced Characterization Techniques**

**Lecture-03**

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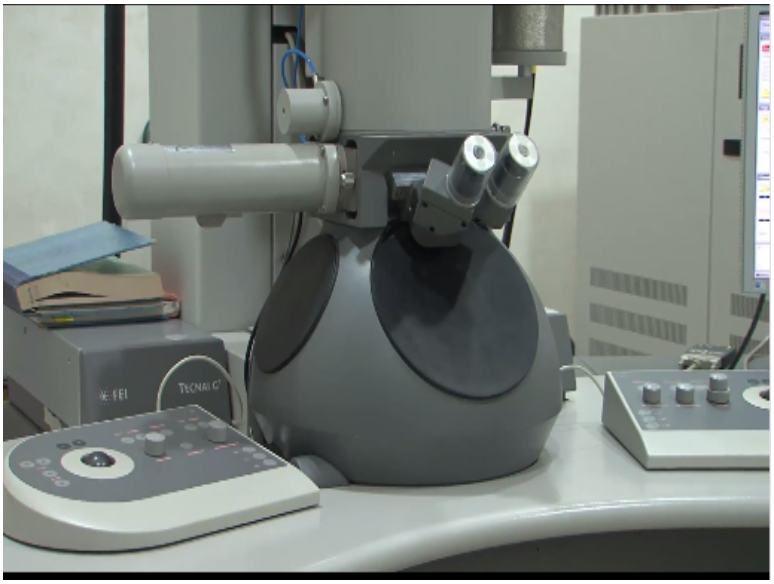


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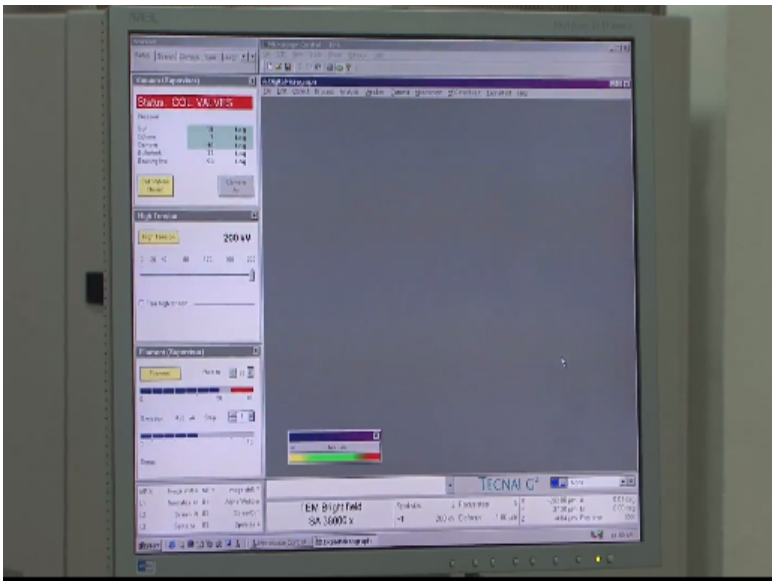


Okay, so with the last class I have shown you the efficacies of transmission electro microscope in which I discussed about the resolution and the depth of field. I have discussed how to use microscope to obtain different kind of images. Today I am going to show you the real microscope. This microscope is in-housed in the department of material science engineering of IIT Kanpur.

We have been using this microscope for the last five years. So I am going to show you first actual microscope different parts of the microscope, and then describe how we can use it. So in transmission electro microscope is basically and versatile in equipment, in the sense that we can use this machine for many such analysis of materials, and get information from starting from the diffraction, to composition, or spectroscopic analysis, to analyze in microscopy.

So obviously microscope will have a basic features and lot of attachments to get information's on the spectroscopic related things and also the other aspects which I will discuss slowly. So in a real microscope we start from the top, basically in a electro microscope the source is electron. So electrons are to be generated by certain means, electrons can be generated by many means one way of generating electron is to use thermionic emission of tungsten filament or the lanthanum hexaboride filaments.

Otherwise, we can use field emission guns. So all the modern day have in microscope has field emission guns. In this microscope the top part is basically the gun, you can see there is a power cable coming and sitting on the gun, and that assembly actually contains a hairpin tungsten

filament. This is nothing but just like a tungsten filament in an in conditioned bulb we see in the real homes.

So tungsten in the form of wire based on 0.1 millimeter is bend to create a hairpin like structure, and then if we apply a voltage, or if you heat your tungsten filament it emits electrons. So therefore, that is the main source of electron, the problem is in thermionic emission is that, because it is a tungsten filament, so therefore, the electrons which are coming on the surface may have different energies.

So that is why many times we use another kind of hairpin filament known as lanthanum hexaboride. Lanthanum hexaboride crystals are actually grown along 110 directions, and these crystals then can be used to get emission by heating. So they are much better than tungsten in the sense that the emission is much more stable, analysis speed of the electrons is less, and the brightness of the beam is higher.

And in modern day technologies we use the field emission guns or called FEG. So field emission guns are again tungsten filament grown or single crystal tungsten grown along 100 directions, and then we have a very fine tip less than about 0.1 millimeter, and if we apply a very high electrical field they are out of $10^6$ volts per centimeter between this filament and another electrode, the anode.

Then we can actually force the electrons to tunnel through this electrode and come out as electron. So these are the sources which is normally use, in this microscope we use tungsten filament or lab6 depending on our uses. So in FEG we need to have a very high vacuum system, because FEG filaments needs to be very clean contaminated free. So therefore, if you want to use the FEG filament these top portion needs to be evacuated to a level of $10^{-10}$ torque.

So that is basically additional cost that is why many of the microscopes normal center microscope do not have FEG column. Otherwise, one may heat up the tungsten filament in a FEG microscope, so that any oxide which forms the surface can be removed. What are maybe the way the cost is very high for tungsten filaments. Now once I let down to generate it then need to be focused, they are focused by set of condenser lens which are situating here.

So normally a normal microscope there will be two condenser lens in an microscopes where you need to have a converge electron diffraction, you need to have three lens what is known as a

extra lens is nothing but a condenser mini lens, which can force the electrons to get converge into a small spot, and that is required for scanning tungsten electro microscopic purposes. We have built in here.

And in the microscope normally the most important part followed by the illumination section that is the condenser lens, source of the condenser lens is the objective lens. And you can see here this is the sample holder, and this is the objective lens and the objective lens is basically just like a twin lens here it say alter 2 in lens or inurn lens if sample is inside between these two twine pole piece and then the beams which falls a sample are passing through the sample they are actually focused by the objective lens to either generate image or generate the refraction pattern by a set of lens which is sitting here they are called in terminate lens and the projector lenses.

Obviously electrons cannot be seen in a micro scopes so one needs to have beam scheme and you if you look at any micro scope this is the view screen with the binoculars as view screen is nothing but a florescence screen made of zinc sulphate on which the electron falls can create light and our eyes human eyes can only see light cannot see electrons in the electron falls in eyes in fact we will be blinded.

So that is why this is fully protected there is a glass laid by chalet which will not allow anything come out on the sample so remember this is very higher assuming unless until dis protected so that is why all these things are actually nicely covered and you only you cannot temper about you only view the screen and do all kinds of analysis and in a normal modern microscopes the panels are like this the very small in which you can have very less number of buttons which can allow us to control the microscopes and it is attached to a computer.

Computer controls all the mechanism in the microscopes in fact microscopes control like the mouse vacuum systems and the high dense and everything is controlled by that so let me also tell you that as a electrons comes from the source they need to accelerated for any electron microscope because that only energy of the electron can be increased this is done by a high dense in tank.

So high dense in tank actually steps up the voltage from normal supplied voltage 220Kw in our country to our 200Kw in the microscopes + microscope which can actually use higher voltages

like 300, 400 or may be 1000Kw so depending on the kind of microscope which you have you need to have  a very large tank in this microscope which is basically dedicated high microscope we have object lens which is separately cooled so that temperature of the till lens cannot be increased so much that focal length can be changed.

Other than this lens we have apertures basically used to select particular you know beam or basically to dictate the side of the beam where aperture have just in below the condenser lengths first condenser lengths which can actually make the spot size very precisely so one can change the aperture set a different level depending on the intensity level of what we what then you have aperture in the objective lens column which can allows us to select the basically the weather we can we want to image using remissive transmitted beam of all.

We want to image using refractive beam or you want to do high less in electron microscopy which I will show just within few minutes time then you have also have a aperture in SAD all called selected a refraction basically if what to select a particular which of the sample and do refraction analysis you need to use in this aperture.

There are actually 3 or 4 apertures depending on the kind of investigation you would like to do one can select this apertures and then get a refraction information remember in a electron microscope refraction pattern forms on the back total frame of the objective lens so depending on the your wish to weather to use the refraction pattern or the image you can actually energies the intimidate lens more or less and to get other refraction pattern on the screen or the image.

And that is normal routinely done by the microscope system we do not need to bothered we have seen in the microscope and do it another than that this microscope has basically you know to make vacuum system clean we need to have liquid nitrogen cooled set up where there is the liquid nitrogen chamber which can take care all the contamination on the sample because most of the samples will have contamination not only when the electron may falls to the sample it can generate contamination.

Those contamination cannot be allow to go to the vacuum system or it can actually create the system vacuum system to be you know to contaminated so to negated anti contamination free we have anti contamination device like a liquid nitrogen cool trap which can take care all the electric

all these contaminations so these liquid nitrogen tank needs to be refilled intermediately to get a very good vacuum inside that column of the microscope.

Well there are several attachment with microscope the first one is this attachment which is known as energy disposes spectroscopy or reads which is again basically we thin wind so we can use the as the electron falls and I have discussed in the last class it generate x- rays and this x – rays can be used to basically qualitatively or quantitatively determine a particular electron and element also figure out amount of the particular element present in the sample.

So this is done by the which will be which are already been discussed in your metal characterization course so I will also discuss after some time in a course also another important thing which we have a basically here known as hard if or high angle detector which is sitting over there this detector is basically mean to get j contrast images as I discussed in the class the electrons which as a falls in the sample very thin task by sample it get detracted and this fraction play power of the you know materials depends on kind of the element presents the material if the sample condense is very heavy element they will diffract strongly.

On the other hand light elements like Lithium, aluminum, silicon as compared to palladium they will diffract very slowly, so therefore we can actually get this define diffract in different it is okay we can actually get the diffraction beams which are kind of a sample collected by this detector atom unless space or at an angular place form the transmitted beam and by suing this detector one can actually see the actual you know z contrast image on the sample this is just like in a scanning electro micro scope.

As we use basket an imaging more but here we do not use the same technology of same concept like an scanning electron micro scope whether we using a diffraction contrast imaging which is basically governed by the active number of the element, so that is attached to the microscope other than that one can have other many other things which includes are as well all spectroscopes analysis which can be attached at the bottom of this microscopes are one can have different video ports to do instead of microscope.

Remember who do not have in set of microscopic stage here but one can attach these to this microscope incentive microscopic means we can put the sampling in cell the microscope then we can heat or cool it down and observe the sample of what are the kind of the changes happen in

the sample and this can be recorded in a video by attaching a video port or this can be recorded in normal mode also which I am going to discuss in it is time, so therefore depending on our use we can attach the different kind of things.

In the microscopes one can actually even attach other things like people sometime use what is called the coal stage microscopy where the holder itself it is a coal stage and this kind of stage is actually used for biological sample be even, so depending on the use or depending on a need a thermo electro microscope can be used for any purposes now as far as recording is concerned because images needs to be recorded are this whatever you are feeding on the scheme is to be recorded earlier days people use to record using a photographic plates just like an normal photographic plates so we do but this place are very big and long so they can be pushed at the and replaced and they just below this.

The way in the screen and because Trans electro microscope has very large depth of field so therefore whatever we will focused on the screen can be assumed to be focused on the phonological plate and along this photographic plates are exposed by the electron beam they can be just develop and the images can be obtained but those days are over now normally in our days we were used digital camera which is there then bottom of this things this is basically digital camera found graph in corporation.

And this digital camera actually very sensitive to electron beam so depending on the imaging conditions we can actually collect this images directly on the digital camera and then we view it on the screen okay so depending on that so I cannot show you one such image which is we have collected in the microscope, so you can see here.

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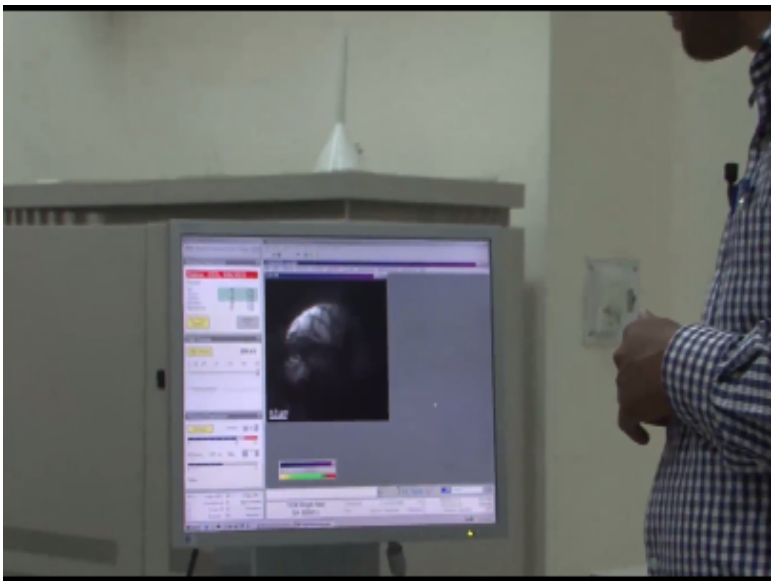

This is the image which is collected by this from this microscope so there are many features so one can actually do this image collection directly only camera and camera technology as improved extensively for the what the time scale as far as reach on the camera sensitivity of the camera also the exposure to the camera all has been developed to the optimum level and now e days we can have a very high levels in camera which can even grab the high images to the exact resolution what we seen on the screen.

So but still many people use both the imaging systems and for day to day life the another kind of meaning system which has been developed now a days which we are used this called photographic plates with the sensitive light, so those photographic plates can be use for recording and then image can be transferred on a computer for the photographic plate and then this images can be erased or the photographic plates then remember you to photographical can be erased and they can be reused they are very sensitive with the lights, but they are not used for the normal microscopes.

All if are very exceptional cases where you want to record diffraction patterns which are energy filtered that can be done, well that is basically the basic feature electron microscope here but normally in the electron microscope there are many other instrument behind as you can you may not be able see from the video but there will be UPS the uninterruptible power supply that is needed or there will be chillers because this lenses which are basically used to image the that of the sample or in the microscope.

Need to be water cool because they have electron magnetic lenses and this water cooling is done by chiller which is normally kept outside this microscope rooms and then you can also have sample cooling system before we put the sample of one can clean the sample using plasma cleaner now a days we have we also the plasma canal which will show you just written after just a finished the discussion here.

So using the plasma cleaner one can clean the sample very nicely you show that one can get a very nice station of the sample and view it. Well these are all background things what you needs to have for a microscope so there so I other that the microscope needs to be kept in a very neat environment free from any kind of vibration free from any kind of magnetic field also free from noise are probably you will see sometime down the line.

If I have the opportunity I will show your microscope which is the best with the best possible resolution call Titan which will be install in our in IIT Kanpur, in a normal Titan microscope even the person who is sitting in the front of microscope cannot speak a wide or even cannot even you know we want by doing so you can actually create the problem in image because of vibration which is generated by that can actually reduce the solution of the microscope, that is why those microscopes are emboli control you can install the sample then go out to the separate room and emboli control.

So but here does not the case because it exist of the microscope is not that so in those cases not only the microscopes are to be kept in a very clean and neat environment but also the environment has to be free from all kinds of noises even in the noise from the area of conditional can create problem. So these are the way halation microscopy has developed over the timer scale from 2006 one over the Titan microscopes has come up in the world and this has change the whole concept of electro microscopy ultra world.

In the class I will show some of the images of the microscopy, okay now let us switch on the b1 the microscope  and just see that like up original, so I just taken on this chair and just switch on the bam here by using the computer which we may not will to see and one phase switch on the beam one side switch on the beam in the microscope I can clearly see that the flow sense scheme lights up and that means I have the beam of the line on the micro scopes remember they for a normal microscope solution microscopy the electron beam needs to be align properly.

So I just then bring the sample in another microscope called in to the impelled of view and then I will just set are the microscope, I will now show you the basic features of the microscopic investigations we have already sample in stated of the microscope and I want to show you most of the things on the screen. So that you can absorb with because seeing here is so lance difficult task on the screen this is small area so I just what I deal is basically I put the sample n and focus the beam and just took the apertures both the objective aperture and the selected apertures using that again form a bright full image.

Which I will show you in a moment's time, so please do it on the screen here so you can see basically the image on the screen, so this is typical white film image in a electron microscope at the field of the way is bright and one can see that there are different features on the image, some of the features actually tell, each features will tells us the contrast and basically these kind of image is based on the diffraction contrast on the electronic microscopes, as the electrons fall on the samples, it under goes diffraction.

So by using this diffraction information we can form different kind of images, so the first kind of image is formed known as the bi fill image in which the transmitted way or the powers scatter beam is used to image so whatever the information are there in power scatter image we will be seen on this image. On the other hand if you say that defected beam then we can see the information regarding dark field.

The most important thing in the microscope when somebody sits is the diffraction pattern, because diffraction pattern is basically form by the diffraction of the crystals in the sample during the as electrons forms as a sample. So electron getting diffracted can lead to diffraction patterns and they contain all information regarding the samples type regarding the feature phase sample whether the defects are not or not even that.

Depending on the diffraction this can be changed, so that is why the diffraction pattern is something which is known as the most important in the micro scopes, by using the diffraction pattern subsequently one can generate a either white full image or the dark full image or then one can use this as high generated microscopic. So this is the very power full process, so that one can actually analysis almost all features which are present in the sample at the fine scale to the level of Armstrong.

One can actually gather information that is the reasons I have told you in the last lecture that microscope is basically versatile equipment can be used for many kind of analysis of the samples. So let me just now go back to the microscope and try to figure out the diffraction pattern and this. So this is very microscope very typical they can have one day nowadays everybody.

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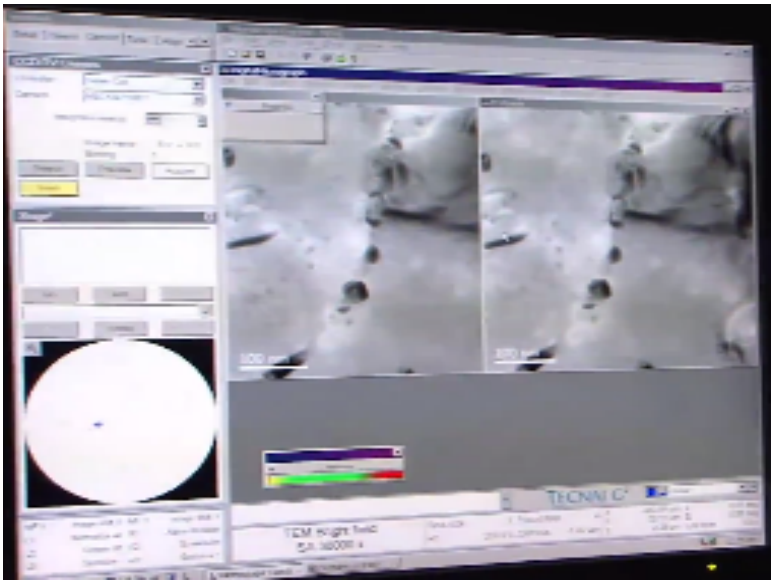

Grab the images on the computer screen; you do not come into the picture okay so we can actually gather diffraction pattern by using the selected aperture select the particular region as sample and this is once as the diffraction pattern you can see on the screen so diffraction pattern as a transmitted beam on the scatter beam and the diffracted beams sitting on the different places, so if I put aperture here.

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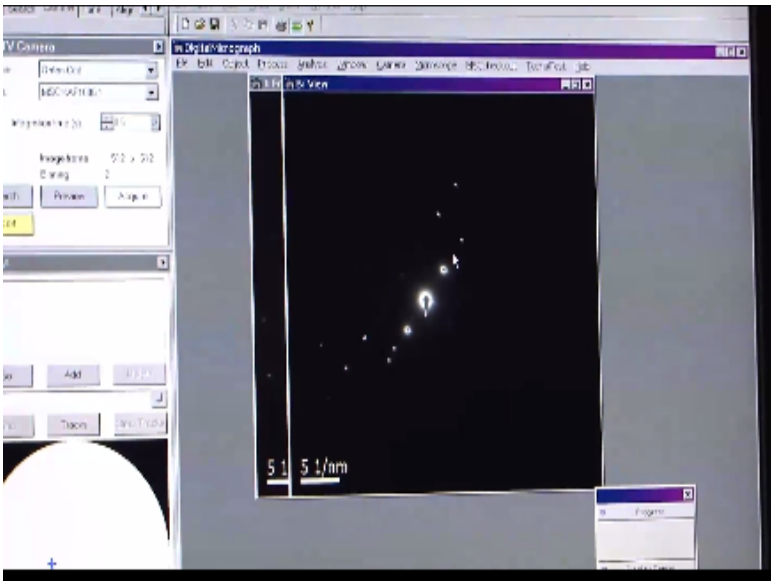


On the transmitted beam then I can basically create the white full image the one which is shown you there okay here, now if I select any of the diffracted beams and put the aperture in the diffracted I can get the black film image. On the other hand if I select the large number of the diffractions spot by using the bigger objective aperture I can get basically I can make them interfere and get the interference pattern which is nothing but as image.

Now days one even do not need to knew to do that because the lens in particular is so good actually generalizes the high evolution image in a very high magnification just to met so this is dark fill image which can be actually obtained by using one of those defected spots in the microscopes and it will light up the regions.

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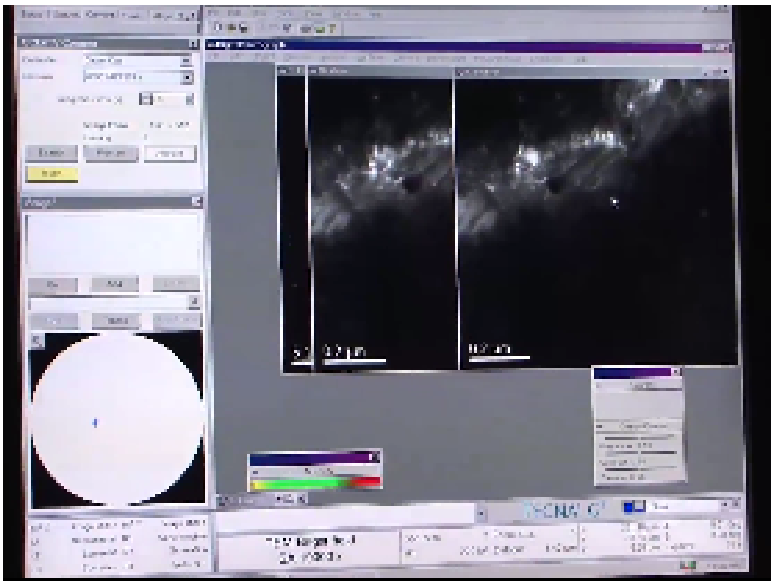

Which are reflecting strongly in the microscopes so these dark field image can be obtained even if that is called scanning transmission electro micro same mode also and the dark field same module give you again some kind of j contrast if you use hardly okay in a dark filed that is called high angle analog dark filed image which can be done so absolution linear microscope nowadays but one is to install this particular detector inside the column together the information.

So other than that one can actually select these area of these regions of the sample and then do the enable regions so sample here and do the spectrums to be analysis using the read axis or one can use another law spectroscopic pictures to basically do the computer analysis of the sample but those are very informal techniques so in the microscope requires lot of time so show you in front of the microscopes.

But I will discuss in the class and subsequently lectures and when I discuss about and the stem I will show you the basic pictures and I will show you some examples of our one study which we have done in the process of our own deserts even using this microscopes or may be some other microscopes which I am going to show in the class

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Modern ways electron microscope requires sample preparation is one of the biggest bottle neck of the getting higher relation microscopic images so that is why sampling friction technique as key chain over the time so I will not be able to discuss exactly difference but off let even after sample is accurate and theme down to the electro task able the samples have to scan of oxides or other contamination to make it contamination free is for the normally for the analytical microscopic purpose.

And also for the purpose the machine which is used is known as plasma clean up it generalizes the plaza using the gas or gas mixture like hydrogen nitrogen oxygen inside this machine and we can actually load the sample net ampere and sample holder and inside these one and clean it using a plasma this is very fast and very what is called authentic technique to get sample continuation free this is very important for many analysis as I said not only high illusion that also computer and also stem because in stem we use to focus or convert beam rather not for convert beam.

And as the sample as continuation it converts interact the continuation in sample will be basically more contaminated as a investigation goes on thus so and all normal days all the microscopic samples needs to be first cleaned in a plaster inner before you can inside the TM column that also steps the backgrounds of the TM column so this as been an new feature in fact many people nowadays use something know as nano meal where again after the normal theory of the sample is teamed by using very low energy organs beam in nanometer very prissily.

So the surface oxide layer are removed and very control manner and so that we can get a actual higher image in the microscope so following that this machine can actually remove all the further confirmation at all there but the nanometer is very expensive when free people live in this world can buy it so we do not have so many people in this world actually the plasma have to clean the sample okay.

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