

Techniques of Materials Characterization
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Lecture – 12
Basic Components of Electron Microscope - Continued

Welcome everyone to this NPTEL online certification course on techniques of materials characterization. We are in Module 3 that is week 3 and we are discussing about some general concepts of electron microscope. In the first lecture we discussed about some historical development in terms of electron microscope and then we discussed about how this electron microscopy, different components of electron microscopy.

And those are all general concepts about any kind of electron microscopy or for that larger sense this is general components for any kind of microscopy. And then we discussed about the electronic structure of materials, how it is important for this electron microscopy. And finally, we discussed about the relationship, we derived certain relationship between the wavelength of electron microscope or wavelength of electrons that is used as a source for electron microscopy.

And the accelerating voltage under which they are passing through the electron beam. And today we will be continuing those discussions about the basic components of electron microscopy.

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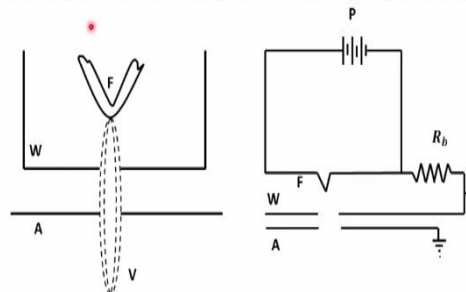
- Electron gun: Thermionic gun
- Electron gun: Field emission gun
- Comparing electron guns

So, now today we will be discussing some more. We will continue with the basic components and today we will choose a particular component which is the gun or the source electron source and there we will be discussing primarily of two different types of electron sources. One is called thermionic gun, another one is called field emission gun. We will discuss about the working principle, advantages and disadvantages. And finally we will compare them in terms of the size, the longevity, the price and all its various parameters at the end.

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Electron source (gun)

- Of the many ways of encouraging electrons to leave a solid so that they may be accelerated towards the specimen, two have proved particularly useful in the construction of electron guns.
- The most widespread system uses thermionic emission from a heated filament.
- At temperatures in excess of 2700K, a tungsten wire emits an abundance of both light and electrons.



- In a light bulb, only light is used but in an electron gun, electrons are accelerated across a potential difference of tens or hundreds of kilovolts to generate a beam of electrons of controlled energy and hence of known wavelength.



So, the first electron source or electron gun is the thermionic emission. The thermionic emission is like, so for any kind of electron, anything to work as an electron source the first requirement is that material should produce an abundance of electrons or as we all know that current. Current is a flow of electrons. So, we can imagine that we have to basically produce, producing an electron beam means we have to produce a current flow.

We have to flow and the direction if electrons are moving in this direction that means we can imagine that electrons are basically there is a current that is going on from this direction. If electrons are moving like this, then we can imagine the currents are moving in the opposite direction. So, we have to produce a current, we have to produce electrons somehow and we have already seen that if a material has abundance of electrons in the conduction band.

If a material is conductor, then it is quite easy to take those outer shell electrons out and produce an electron. It is now we have to just focus on how to get these electrons, free

electrons or delocalized electron how to get them out. So, there are two different methods to do. There are many different ways to do. For our purpose for electron microscopy purpose, there will be two different effects to consider.

The first of all is basically thermionic effect. Now, what is the thermionic effect? It is very simple it is using a thermal energy. So, you put thermal energy into the system in order to as a source of binding energy you provide that much of binding energy and then you extract those electrons out of the material that is it that is what is thermionic effect for at least for us.

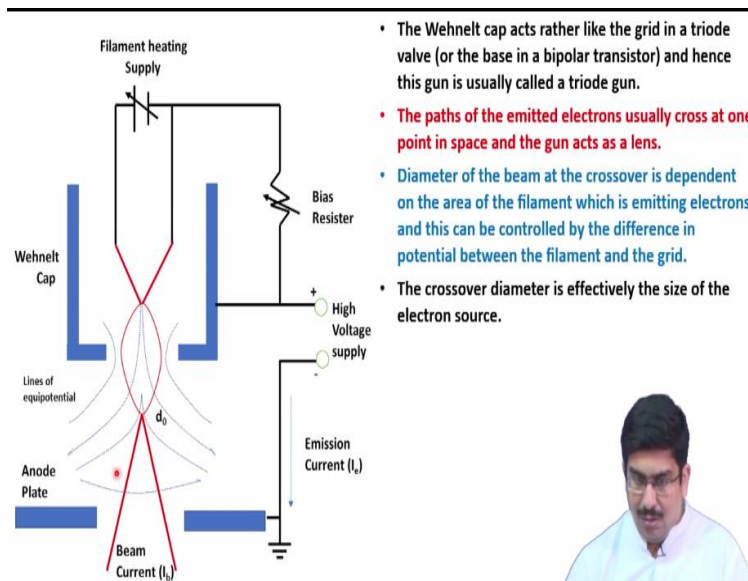
So, what happens in this case that if you heat up some material at a temperature normally in excess of 2700 K then generally that will emit or that will be giving you a beam of electrons from there. Typically, a tungsten wire is used for this purpose. Now, the tungsten wire that is used as a source in electron can also give you the light energy and the construction wise it is exactly the same as those good old day's tungsten halogen lamps.

So, they also used to have a filament. Here also the tungsten works like a filament, we will see that. The difference is that the light bulbs there mostly the lights are important. So, we are just dealing with the lights, we are looking to extract the light that is why we are putting the tungsten filament in a transparent, in a glass cage or in a glass bulb we are putting them so that the light can pass through that glass bulb.

There we restrict those electrons by using some kind of an inert gas inside so that the electrons get interacted, they lose their energy, they cannot come out. So that is what we do in a normal light bulb. In this case in as an electron source we just utilize not the light but the electron part of it and here the number of electrons that we generate is also very high. That means, we have to put a lot of energy in form of heat.

And also the amount of heat that we are producing is directly related to the binding energy of this outer electron and in turn this energy is deciding the wavelength of the electrons when they come out, just to come out after that they get accelerated and then those relation will be valid, but right now we are just talking about the electrons, how to take out these electrons from this tungsten filament.

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So, how do we do that? Basically, we put a piece of tungsten usually like a hairpin, the shape literally looks like a hairpin and it has a very sharp edge here from which actually the electrons are generated. And this sharp tungsten hairpin basically works like a cathode. So, this entire construction is like a triode valve and here this one works like a cathode which is producing the electrons.

That means if we consider in regular case, then the current flows from anode to cathode, here as I already said exactly the reverse, we can imagine the electron flows from cathode to anode that is it. Now, this hill of filament here which is the cathode and it is heated in excess of 27-2800 K and the electrons are generated here. Also, this cathode is kept at a slightly higher negative bias with respect to this anode plate here.

This is a slightly higher negative bias. It is kept at a slightly higher negative potential with respect to the cathode. So, electrons which are now thermionically emitted from this tungsten filament, they will all get attracted towards the anode and then in the process they will produce a very high energy electron beam. And this anode is usually kept like a plate and it has a very small opening.

So, all these electrons that is coming out of the cathode and that is getting accelerated towards the anode will now go out of the anode as a beam, as an electron beam, many electrons will come out and those electrons by the thermionic effect and those electrons

will finally produce a beam here. What also is added in this construction is this, this is called Wehnelt cap. So, what is the purpose of Wehnelt cap?

This one is kept basically at a slightly higher negative voltage than this filament. So, this anode is kept at a very high negative voltage with respect to the cathode that is why all the electrons are getting. So this difference, this is the potential or acceleration voltage or potential that I was talking about between the cathode and the anode. So, anode is kept at a very high negative voltage compared to the cathode.

Now, the Wehnelt cap is kept at a slightly higher negative voltage, not much, very less amount of negative voltage is given with respect to the cathode here. What it does is that whatever the electrons which are going out this actually repels this, Wehnelt cap actually repels those electrons and then make them to cross here, somewhere the path gets crossed here and that is how basically this number one it restricts that area from which the electrons are produced.

In this case only at this hairpin bend here because of that all other electrons basically gets repelled by this Wehnelt cap because it is kept at a slightly higher negative voltage. So that is why this one is kept or as I said the cathode is kept at a higher negative potential compared to the anode. This one is kept at even higher, slightly even higher negative potential than the cathode and that is why it basically repels all the electrons.

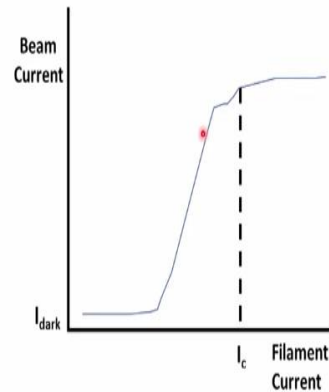
And those electrons will not be able to come out and that is how it restricts the portion or the area from which these electrons will finally come. Also, this will repel those electrons and it will do something like a focusing. So, it will make those electrons their lines, lines of forces, all of these directions will finally cross at a point in space and that spot will virtually form the source, that spot is basically called the crossover.

So, in that sense this Wehnelt cap basically works like a grid for a triode valve or you can also consider it as a base in a bipolar transistor and this entire construction is called a triode gun. So, the thermionic tungsten containing tungsten this thermionic gun is also called a triode gun. This Wehnelt cap as I already said make those electrons their path to crossover to cross at a point in the space and that point is basically the virtual source for all practical purpose.

So, finally, when we consider the size of the source of electron beam, we will not be considering this area, we consider only this area, the crossover because this is where the electrons after they are extracted from cathode they will meet here then further they will go away from this point only towards the anode. So, that is how is the Wehnelt cap works, the entire triode valve works.

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- A small current flows when the potential is applied but before the filament current is high enough to give rise to thermionic emission.
- This is known as the dark current since it flows before the filament is hot enough to emit light.
- As the emitted current increases so does the bias voltage and this suppresses further electron emission.
- This is known as the autobias mechanism and accounts for the characteristic shape of the emission curve.
- As the current through the filament is increased there is an initial rise in the emitted electron beam current.
- This eventually saturates and there is no point in passing more than the critical current through the filament since this merely increases the temperature of the filament (thus reducing its lifetime) without giving rise to any additional beam current.



Now if I look at this relationship between beam current and filament current, so what happens filament current means the current which is passing through the filament and heating it up, whereas beam current here means the electrons that is generated because that is what is current that is what is producing the current. So, you can also think indirectly it is the number of electrons which are coming up.

So, what is happening is that initially when I am hitting the filament, there is not enough energy and that to sort of extract all the electrons. So even there is some little amount of filament current is given no electron is extracting, no electrons will get out, only when they achieve the amount equal to their binding energy they will or that means the filament when it is sufficiently heated only after that the electrons will start coming out of it and they will start forming this beam.

Then the beam current will increase. So, this current up to which there is no electron or there is a very little electron that comes out is called the I_{dark} and this much of filament current needs to be provided for generation of electron beam minimum, this needs this is

the darker, So, once the filament is sufficiently heated up, once now these electrons get their binding energy, the energy, heat energy or thermal energy is almost sufficient for these electrons to surpass their binding energy and come out of this material.

Then the beam current will start increasing very rapidly. So, now many such electrons will be coming out of the specimen because of the thermal energy and that will increase the beam current or number of electrons will almost exponentially increase here. So, finally now we start getting a beam really literally an electron beam. After that, again once the filament current is reached to a sufficient value.

After that there is no outer electron available for generating this electron beam or for coming out to the electron beam. So, that means the filament current or the beam current is now reached as saturation value, after this if we pass further current in the filament, it will just be heated up. The filament will just be heated up, but no further electron will come out because we are now exhausted with all the outer electrons available in the conduction band.

After that there is only possibly inner shell electrons are left out which will not be able to come out because their binding energy is very high. So that is why after the saturation current there is no point passing filament current it will just heat up the filament, but without any and heat up means it will also reduce the life of this filament but without any real gain in terms of number of electrons in the electron beam.

So, this is one point we must remember that the electron filament should be operated somewhere close to this saturation current, then only the highest amount of beam current or highest number of electrons will be possible or will be present within the electron beam. So, neither we should operate the electron, the gun, the thermionic gun at this I dark current nor we should operate it beyond this saturation current.

Somewhere in between we should operate it and this is a kind of a calibration that before the machine starts or before any filament. If you change the filament after that this exercise needs to be performed to find out that exactly where this saturation current is like, where this I dark is there, where is the saturation current because that will decide exactly how much filament current should be passed.

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Brightness of an electron gun

- In the context of electron microscopy brightness is defined as the beam current density per unit solid angle.

- Brightness is therefore a measure of how many electrons per second can be directed at a given area of the specimen.

$$B = 2 \times 10^5 T V \exp\left(-\frac{\Phi}{RT}\right) \text{Am}^2 \text{sr}^{-1}$$

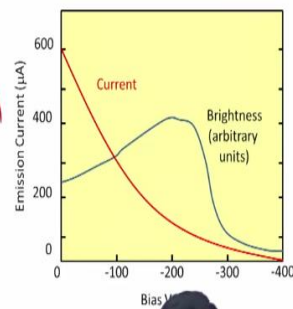
- B increases rapidly as T (temperature of the filament in Kelvin) increases and as Φ (thermionic work function of the filament material in electron volts) decreases.

- It is best to use a filament material with as high a melting point and as low a work function as possible.

- Tungsten has a high melting point (3653 K) and a work function which is much the same as most metals (4-5 eV) and is the most widely used filament material.

- Tungsten filaments give a brightness of about $10^9 \text{Am}^2 \text{sr}^{-1}$.

- The thermionic gun is satisfactory for many purposes but is limited in the brightness of the beam it can produce.



Now, we can look for another important factor which is called the brightness of an electron gun. So, what is the brightness? In context of electron microscopy, the brightness is basically defined as the beam current density per unit solid angle. What does this mean? This means it is basically how many electrons per second can be directed to a given area of the specimen.

So, how many electrons are there per unit second within any given area, within a given solid angle, unit solid angle the beam current density how many electrons are there. Roughly this is what the measure of brightness. Now, the relationship between the brightness of any material and the different parameters I will give you this relationship so that it will help us to understand exactly which factors are governing this brightness.

So, $B = 2 \times 10^5 T V \exp(-\Phi/RT) \text{Am}^2 \text{sr}^{-1}$. Steradian is the unit for solid angle. So, this is how we can express the brightness of this electron beam here. Okay let us go back to the laser pointer again fine. Here this is the brightness.

T is the temperature, of course the temperature expressed in Kelvin of the filament expressed in Kelvin, V is the voltage that you are providing here between the cathode and the anode and this Φ is the work function. So, voltage is basically constant, voltage between cathode and anode this you usually keep it constant, for a particular type of brightness for a particular type of beam, this potential difference is very much kept constant.

Because this ultimately controls the wavelength and this ultimately decides the wavelength. So, we do not do much with this voltage here. Φ , this one is the work function. That means how much or what is the binding energy, how much energy, thermal energy you have to supply to take out the electrons out of the material. So, brightness of course then increases very rapidly with this almost linearly increases with the temperature. So, as the temperature increases brightness also increases.

Also, it will depend in a complex way, in an exponential time it will also depends on this Φ that is the binding energy thermionic work function of the filament material. So, ultimately the brightness is a very complex function of both of these parameters, the T that is the temperature of the filament as well as the thermionic work function of the filament and this basically decides that what should be the best filament material.

So, you can see that this brightness varies in a very complex manner with the bias voltage and emission current, the emission current means again number of electrons and the bias voltage is the voltage given for as an acceleration. But this one says basically that it is best to use the filament material which is having as high melting point as possible because so that you can then use this material at a very high temperature.

Also this material should have a very low work function so that electrons can be taken out. Now, these two sounds a bit oxymoron, something which has a very high melting point should also have a high work function because this is basically somehow tells you the nature of strength of the bonds as well. But as I already said that the work function is also related to the conduction band, that kind of conduction band you have.

So, that is why this kind of material also exists and tungsten is the best example for this which has a very high melting point and a very low work function, moderate work function I would say of around 4 to 5eV and that is why tungsten is typically used for filament material. Tungsten has some other problems which I am not going into that because of the time restriction.

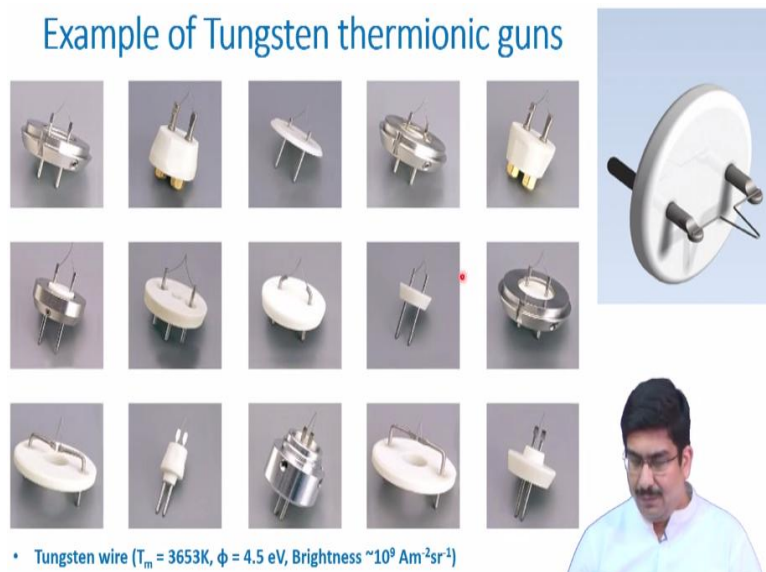
If anybody is interested, please drop a question, we can discuss about this the problems with tungsten filament. Tungsten filaments when you operate them at a typical voltage

and typical current in excess of 2800 kilo Kelvin this gives a brightness of around 10^9 $\text{Am}^2 \text{sr}^{-1}$ and this much of brightness is okay, sufficient for many purposes like imaging purposes and so.

But this one is definitely not sufficient for doing high resolution work where you need a lot of aberration correction and we have already discussed for example spherical aberration correction, the best way is to restrict the electrons close to the optic axis. That means you have to chop out most part of the electrons, most of the electrons will be ultimately removed.

So, if you do not start with a very high intensity of the electron, very high number of electrons in the beam then you will not be able to perform this aberration correction properly. So, that is the number one problem of the tungsten filament or thermionic effect for that matter in the larger sense and also diffraction experiment, I am coming to that.

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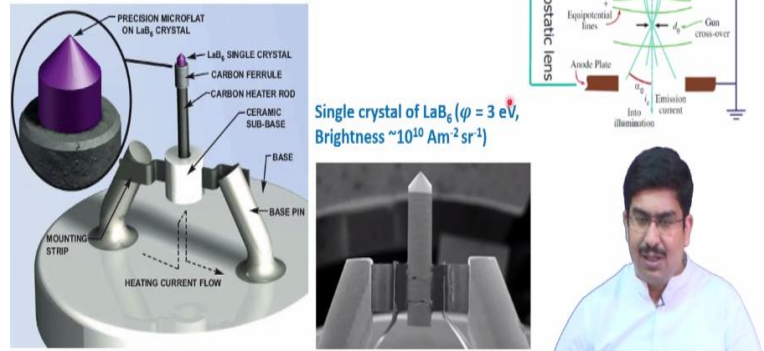


So, these are the example various configurations of tungsten thermionic gun but as showed here most often this is used like this kind of a hairpin and this is very easy, very cheap, most of this it comes not more than I would say a few 1000 rupees and then this entire thing can be very easily replaced in a thermionic gun, once trained operator should be able to do that.

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LaB₆ thermionic guns

- The brightness can be increased by a factor of 10 or more (from Tungsten) if LaB₆ with a work function of 3.0 eV, is used ($B \sim 10^{10} \text{ Am}^{-2} \text{ sr}^{-1}$).
- Electron guns which use LaB₆ are quite common on microscopes which are used for analytical or high resolution work since in both of these fields a high brightness is desirable.



There is another variation of thermionic effect by using a suitable or better material than tungsten that is called LaB₆ and LaB₆ is lanthanum hexaboride. This is a boride, this is a non-oxide ceramic material which can be also used, by using this brightness can be increased by another one more order of magnitude. So, you will be getting something around $10^{10} \text{ Am}^2 \text{ sr}^{-1}$.

This much of brightness is possible because it has a work function even smaller than that tungsten, it has almost 3eV, whereas tungsten has around 4 to 5 plus. Because of it is non-oxide ceramic material it has also a very high melting point. Problem is of course since it is non-oxide material keeping it or prevent it from oxidization is an important factor, so you have to maintain a very high level of vacuum for this.

But this kind of microscope you can of course use for doing a little bit of high-resolution imaging. Problem is that with time, this LbB₆ the maintenance cost of LbB₆ is really high and the installation cost also is pretty high. Making this LbB₆ filaments with this kind of a shape with a very precise end here very small area, again you remember we discussed about that that you have the Wehnelt cap and then you try to restrict the flow of electrons from one single point.

If the electrons are coming from different parts of it, you will have a completely different energy and a very completely different wavelength for this electron. So, finally to maintain those electrons within a narrow wavelength range and the narrow wavelength

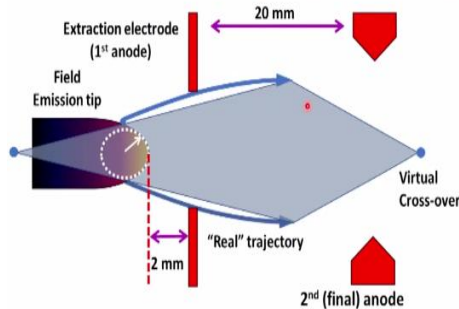
range, I was coming to there, narrow wavelength range is helpful for reducing chromatic aberration. So, we discussed about chromatic and spherical aberration.

So, if you have very narrow wavelength range, you can restrict chromatic aberration as well that is why you tend to keep the electrons confined from a very small part of this filament. In LbB_6 the preparation is difficult, maintenance difficult and the advantage that it offers also is not a big game. So, LbB_6 filaments are not that popular and these days. There are very few microscopes which operates with a LbB_6 thermionic gun.

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Field emission guns (FEG)

- If still higher brightness is required then the field emission gun is used.
- If a metal surface is subjected to an extremely high electric field ($> 10^9 \text{ V/m}$), there is a high probability that electron can leave surface without needing the amount of energy represented by the work function.
- This is because of the effect predicted by quantum mechanics known as tunnelling.
- The result is that many more electrons can be drawn from a piece of tungsten than is possible using thermionic emission and the brightness can be increased in excess of $10^{13} \text{ Am}^2 \text{ sr}^{-1}$.



Now the next option that you have if you really want and these days almost every electron microscopy wants to have this as an electron source if they want to work high-resolution work or electron diffraction work that is called field emission gun or FEG source. This gives you a very high brightness compared to the thermionic guns, almost like a few several orders, three four orders of magnitude higher brightness.

It can give you around $10^{13} \text{ Am}^2 \text{ sr}^{-1}$. So almost four orders of magnitude higher than tungsten filament thermionic guns. What do you do here is that you use or you subject a metal surface or a conductor you need. So, you subject the conductor surface which has lots of this conduction and lots of free electrons in the conduction band.

You subject it to a very high electric field in excess of around 10^9 Vm^{-1} that kind of a high electric field if you apply then there is some kind of quantum mechanics, some kind of mechanism possibly in quantum mechanics called tunneling. So, because of this

tunneling mechanism the electrons can leave the surface of this conductor even before reaching this.

It does not need that much of energy which dictated by work function, even before reaching that much of energy the electron can come out because of this actually very high electric field and because of this effect it can generate a very strong electron beam. Because if you can use now, if you look at this configuration of this field emission gun, what you have is that this tip again a very sharp tip here and then you have an extraction electrode.

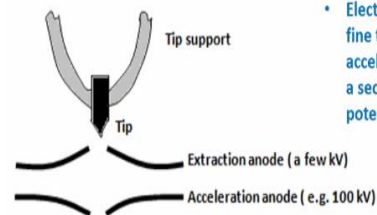
That means you keep another plate, so there are two different anodes here. The first anode is just there to put or just there to apply a very high electric field on this tip and remember it is electric field. So that is why you basically tend to have a very sharp tip here so that ultimately the electric field increases, you cannot increase un-definitely the potential difference, so you reduce the size of this tip here.

And that is how you can increase or you can reach at least this high amount of electric field that is what So, you have the first extraction plate which is called extraction electrode which is kept at a very high potential difference, and then once the electrons are generated you will have the same kind of configuration you will be having a second final anode which is kept at a very high negative.

Cathode is kept at a very high negative bias compared to this anode, finally the electrons will be accelerated through the anodes and they will again have a virtual crossover here also. The biggest advantage as I said in this case is very high brightness.

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- The field emission current depends very strongly on the applied field.
- For a field in excess of about $5 \times 10^9 \text{ Vm}^{-1}$ the current emitted by field emission at room temperature exceeds that which can be thermionically emitted: Cold FEG
- In order to apply such a high field the emitter, usually tungsten, has to be prepared in the form of a sharp point.
- The diameter at the point must be about $0.1 \mu\text{m}$, which is orders of magnitude finer than a pin, so the emitter is a rather delicate structure.
- For this fine point to be preserved in use it must be operated in an environment with very few ions and this dictates the use of ultra-high-vacuum (UHV) techniques.
- Vacuum in the gun must be lower than 10^{-7} Pa , which is rather better (and more expensive to achieve) than 10^{-2} or 10^{-3} Pa which is common in thermionic guns.



- Electrons are extracted from the fine tip by a first anode and then accelerated down the column by a second anode at a much higher potential.



Now, the field emission current as I said depends very strongly on the applied field and that is why you tend to have this as sharp as possible. And at times if you can properly prepare this tip here, the field can reach as high as $5 \times 10^9 \text{ Vm}^{-1}$. Then the current that emits from this cold emission the number of electrons that can emit from a field emission source at room temperature.

You do not need any kind of temperature to pass through this source, that can be easily surpass or much better almost orders of magnitude higher than the thermionic emissions. And for thermionic emission you need the thermal energy, here in this field emission effect even in cold condition even without any kind of a heating you can have almost four orders of magnitude higher brightness than a thermionic gun. This is the biggest advantage of this FEG source.

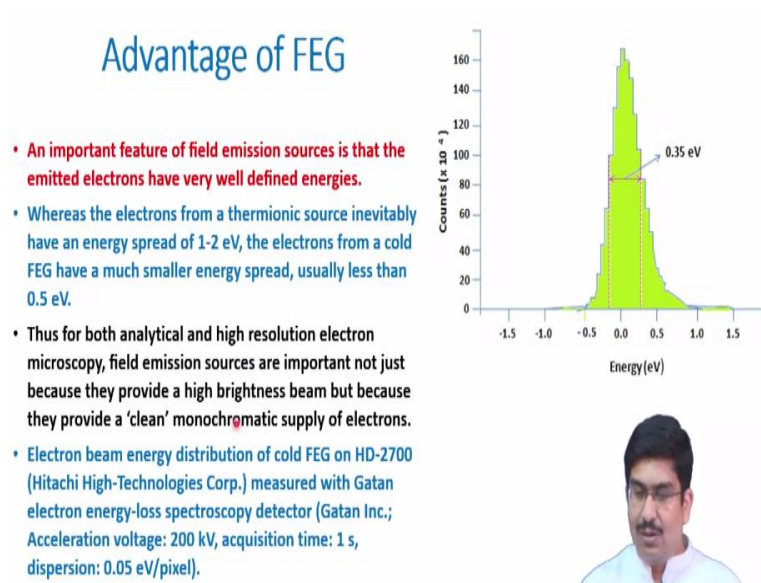
And as I have already discussed because of this high brightness very easily you can restrict spherical aberration by using a very small aperture. Again, the material that is used here is tungsten because the tungsten is again have a very low work function and tungsten is a conductor and also preparation of tungsten is comparatively easier than other material. The diameter or tip diameter is often of the order of $0.1 \mu\text{m}$ basically the radius of curvature here is around of that range which is very fine than a pin.

So, this is a real delicate structure, this is the problem of FEG source. Two problems are happening in FEG source. Like LaB_6 to make this sharp tip itself is very difficult that is why tungsten is a popular material and this is usually done by a method called FIB,

focused ion beam milling. By that method you prepare this sharp tip and to maintain this sharp tip is again very difficult because they tend to oxidize and then they tend to lose the shape here and it sometimes breaks also because of oxidation.

So, a very high vacuum is needed, ultrahigh vacuum of the order of 10^{-7} Pa that kind of a high vacuum is needed for maintaining of this. So, maintenance cost is high, initial investment is high but the advantage this FEG giving is for high resolution work, for electron diffraction work is amazing.

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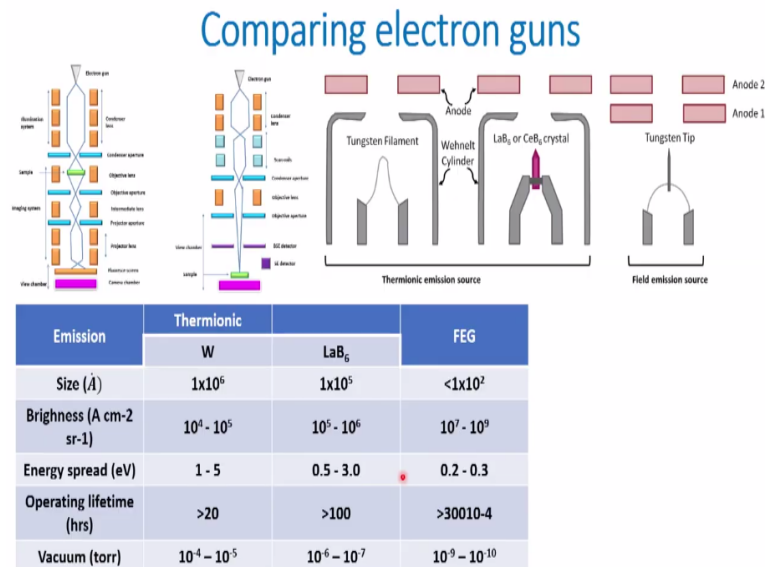
Another big advantage of FEG is that the spread in wavelength. When it comes out of the filament the spread in wavelength is much lower than the thermionic emission. Thermionic emission pretty much random process. So they are the spread, the electrons are coming out with all possible energies and all possible wavelengths. Spread is often around 1-2 eV for thermionic beam whereas for a FEG beam it is hardly around 0.5 eV.

So, you can see this is a real data where it is captured from a cold FEG and here you can see this spread is hardly 0.35eV. So, this is another big advantage of FEG. It is kind of a clean monochromatic beam, which is very good for chromatic aberration correction. So, this is another advantage for FEG source. FEG source is good for both chromatic aberration correction as well as spherical aberration correction.

And that is why FEG source is the most popular one for doing high resolution electron. Also when we discuss about diffraction phenomena, we will see that this monochromatic

supply, monochromatic beam is very essential for doing any kind of diffraction experiments. So, there are also the FEG source is a big advantage.

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So, finally if we compare all these three different types of electron guns, what we can come out is that the resolution is definitely much higher in case of FEG, although the size is bigger. The brightness is again quite much higher in case of a FEG source. Then the energy spread is much lower in case of a FEG source. Operating lifetime, this is wrongly written this should be around 300.

This is 300 hours, but generally the lifetime of FEG sources is much higher and many of the electron microscope manufacturers they give a something like a lifetime of around 3 years or even more a guarantee of 3 years. That means even they are very assured of producing this FEG source to work for more than 3 years. They can very safely give such kind of a lifetime guarantee and the vacuum that is needed for FEG is also pretty much high.

So, with this we are done with the electron sources and in the next class we will be discussing about another important component of electron microscopy that is the lens and we will be seeing that how this electron source and electron lenses are working and how they are, what help they do in reducing this aberration and making the resolution better in electron microscope. So, till then thank you and goodbye.