Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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Welcome to this class. In the last session, we briefly went through the sample preparation method by FIB. This is a focused ion beam microscopy method where we use focused ions. So, usually we use gallium ions, and these gallium ions at room temperature exist as a liquid, correct? Because their melting point is around 29.8 degrees Celsius.

So, we have briefly gone through the ion column. We started with the gun source, as I have told that time, that in the gun source you usually have a tungsten needle. Okay, a sharp tungsten needle, and there is a reservoir, okay? This is a gallium reservoir, and from there, the liquid gallium usually flows. To the tungsten tip, and it forms a Taylor cone, okay? Usually, the electric field—so you will have extracted plates, correct?—and this electric field. Pulls the gallium ions. Extracted plates here have a negative potential, and you will keep the positive potential, so your gallium ions get pulled from this.

Taylor cone, okay? So, there is also an opposite force due to the liquid tension, surface tension in the liquid gallium, and that acts against the electrostatic force. An electrostatic force, and when they balance together, there will be a formation of a Taylor cone, which is around 2 to 10 nanometers or 5 nanometers. With a tip radius, okay? So then, the electrons from the gallium tunnel through the tungsten tip, okay? This involves the field ionization of the gallium. To the gallium plus ions. This is called field ionization.

Then, due to the electric field near the tungsten tip or in front of the Taylor cone, there will be field evaporation. Okay? These are accelerated toward the column. So, this is your FIB column. Correct?

These are the extracted plates. Fine? So, now, What we will do is, we will also—so this is particularly what we call a gallium liquid metal ion source, okay. Usually, we call it a liquid metal ion source for ion emission.

So, now what we do is briefly describe the ion optics. What are the roles of different components in the ion optics in the ion column? Okay, so, as we drew in the last class also, okay, this is your cross-section. Fine, so, you will have an ion source. Then you will have a condenser aperture.

Condenser aperture. Then you will have a condenser lens. This is called a condenser lens. Sorry, this is not an aperture. Then you will have an aperture which has different sizes

holes, okay, to pass the beam fine. Then you will have a quadrupole. You will have a quadrupole lens. This is called a quadrupole lens, okay? Then you will have blanking plates. Blanking plates. These are called blanking plates. And then you will have an octopole. You will have an octopole, correct? This is called an octopole. And then finally, you will have an objective lens. This is called an objective lens, okay? So the ions get accelerated towards the bottom of the column, okay? These are the flight paths of the ions. Okay, so these are the ions which are accelerated through the difference in potential. Okay, if you zoom in on this position, this part, then you will have a source with these extraction plates.

Okay, due to the potential difference, these gallium plus ions get accelerated down the column. This is your column. Now, this particular ion beam will then go through a series of lens systems and apertures, like condenser, objective, then apertures. Correct?

Before it reaches a specimen. Before it reaches a specimen. Okay? So, what is the use of the condenser? So, as it may suggest, this condenses the ion beam.

Correct? And this forms the beam, which is then scanned across the sample surface. Correct? Across the sample surface by the octopole. Okay?

These are referred to as scan coils. So, by using these scan coils, the ion beam gets deflected and can be scanned on the sample surface. Okay? The function of the objective lens is to focus the ion beam. Okay, on the sample. Okay, and the astigmatism is

corrected by the coils which are present in the octopole. Okay, so what is the function of quadrupole lenses? Quadrupole functions.

is the—this uses the mid-column steering of the beam during alignment. Okay, so your quadrupole lenses and the octopole lenses, they are used to steer the beam during the alignment of the sample surface or the movement of the beam on the sample surface. So, one difference between the lens system in a normal electron microscope, like in SEM or TEM, and here is that in normal electron microscopes, you usually use electromagnetic lenses. So, they will generate the magnetic field. But here, you use the electrostatic lenses for the ion column.

So, there is a reason for that. So, in SCM and TEM, you use the electromagnetic lenses in SCM or any electron microscope. This difference is due to the difference in the source. So, in electron microscopes, you use electrons for your imaging or for the interaction of your electrons on the sample surface in SCM or TEM. But in the case of an ion microscope or ion beam microscope, you use ions.

For example, gallium plus ions. Okay? Now, in electron microscopes, you use electromagnetic lenses. But in an ion beam, you use electrostatic lenses. Okay?

So, why is this different? So, in electromagnetic lenses, Correct? Usually, the force which is exerted by the magnetic lens on a charged particle, or you can say an electron on a charged particle in the case of an electromagnetic lens, is directly proportional to the velocity.

Okay? And in the case of ions, so in electrons here, it travels very fast. Their velocity is very high, and the force exerted on the electron—or to the electron—by using the electromagnetic lens is directly proportional to the velocity of that particular electron. But in the case of an ion beam, these are much heavier, so the velocity of these gallium ions is much lower. As they are heavy.

So, if the velocity is much lower, then they need large magnetic lenses, which is practically not possible. Correct? So, control of electrons, or the focusing of electrons during electron microscopy in TEM or SEM, can be done by an electromagnetic lens

very easily. But in the ion beam, electromagnetic lenses cannot be used because their velocities are much lower. So, they need very large magnetic lenses.

However, in an ion beam, you are using the electrostatic lenses, which is why you are using them. Why? Because electrostatic lenses use an electric field to manipulate these positive ions. Okay? And here, the force is independent

independent of the velocity of that particular ion. Correct? So, by using the electric field or electrostatic lens, you can actually precisely control the ion beam. Okay? But not

In the case of using the electromagnetic lens, because the velocity of ion beam ions is much slower or lower than that of electrons, which are much lighter. So, their velocity is very high. So, the electromagnetic lens can be effectively used to precisely control the focusing of electrons, but not in the case of an ion beam. where we use electrostatic lenses, correct? Now, after the lenses, you will also have the blanking plates.

I talked about these blanking plates, correct? So, now during sputtering, when these ions interact with the sample surface, your sample is getting removed. But if you do not want the ion beam to interact with the sample at a certain instant, then you can use these blanking plates. These blanking plates can actually avoid the unnecessary exposure of the sample during the FIB operation. Okay.

So, they can block the ion beam. That is why they are called the blanking plates. So, this is a general ion optics we usually see in any focused ion beam microscopy of the ion column. Now, we also talked about the apertures. So, you have a source.

So, your gallium plus ions accelerate towards the column. Then, there will be a condenser aperture. This is your condenser aperture or condenser lens. Below the lens, you will have an aperture with different sizes. If you magnify this particular aperture below this lens, what you have is nothing but different-sized holes.

So, if you want a larger beam, the beam should pass through this particular large aperture. If you want a smaller beam or the smallest beam, you can change the aperture so that it adjusts to a smaller beam size. So, your beam size will be smaller here, and your beam

size will be larger here. Correct? So, based on the aperture, you are actually changing the current, the ion beam current.

Correct? So, your beam size will also change. In both cases, the beam will be focused, but the current—the ion beam current—will be higher for the larger aperture, and the ion beam current will be lower for the smaller aperture size, okay. So, this larger aperture and smaller aperture can control the current—the current of the ion beam. That actually directly controls the speed, or the velocity, or the rate of the sputtering process.

Sputtering process. Okay. So, the ion beam current can be selected by the beam-defining aperture below the condenser lens. Okay. And this is the strip.

This is the aperture strip with different-sized circular holes called apertures. And the larger the aperture, the larger the ion beam current, and the faster—or higher—the velocity of the sputtering process. Now, we have discussed the ion beam, the apertures, and even the ion optics. Now, we will briefly go through some of the physical terms. which are very important to measure.

Okay. First, we will talk about the ion beam current. Ion beam current. So, it is nothing but the number of ions that can interact with the sample during a time t, okay?

So, you have an ion beam, correct? You have a sample, right? So, this is the ion beam current is nothing but the number of ions that are interacting with the sample during the time t, okay? So, this can be given as n is equal to I cross t divided by c. Here, the n is the number of ions interacting I is the current in amperes and T is the time in seconds and C is the charge.

Correct? So, this is the ion beam current. Another term is the ion dose. Correct? So, there are two terms here.

One is dose, and one is fluence. Okay, so there is a difference between them. So, what is the fluence? Fluence is the number of ions that pass through an area prior to interaction with the sample. So, if your beam is there, and you have this particular sample, fluence can be described by this particular area.

Just before the interaction, the number of ions that pass through an area. Correct? That is called fluence. And dose is the number of ions that have traveled into the sample surface. Into the sample surface.

Okay? So, dose is the number of ions that travel into the sample surface. But the fluence is the number of ions that pass through a cross-section prior to the interaction with the sample. Okay.

So, the dose is given by N divided by A. Okay. N is the number of ions. A is the area in centimeter square. Okay.

So, there is another term called ion dose rate, which involves time. Okay. So, the dose rate is described as the number of ions passing through a specific area per unit of time. So, dose rate is given by N divided by A multiplied by T. Time is in seconds, area is in centimeter square, and N is the number of ions. So, these are the three terms you usually encounter when using the focused ion beam for sample preparation.

So, you need to control the ion dose rate, ion dose, and also the ion beam current depending on your sample. Correct? So, these are the three physical features or three parameters which we usually change during the sputtering process. Now, after describing briefly the section of the ion column, what we now go through is the interaction of the ion beam with the sample. So, what type of radiations or what type of electrons or ions, what type of emission of waves will take place when your ion beam interacts with your sample surface? Imagine you have a sample surface

This is your sample, and your ion beam is interacting with the sample. So, you have a certain structure of that particular sample on the sample surface, correct? This is your sample surface, and these are the atoms which are arranged inside the sample. Fine? Which has a definite structure. Now, when an ion beam...

interacts with the sample, the first emission, the most common emission, is the secondary electrons and secondary ions. Okay, so what are secondary electrons? These ions and electrons can be used, can be detected by different detectors. Okay?

And these two secondary electrons and secondary ions can be used to form the image of the sample surface. Okay? The second interaction, which is the main one for the focused ion beam, is called sputtering. What is sputtering here? Sputtering is the removal of sample surface atoms.

Okay, so this sputtering process When an ion beam interacts with the sample, the surface atoms can be removed based on the dose rate and the ion beam current, correct? So, this sputtering process is used to cut structures, cross-sections, and features from the sample, okay? The third thing is, when the ion beam interacts with the sample surface, it may modify the sample. Modifying the sample means it can generate vacancies, dislocations, and also interstitials.

Okay. Now, the interaction of the beam takes place as the ion beam travels into the sample surface until all its energy has been consumed. All the energy of the ion beam is consumed. So, it means that it has a certain depth, which is called the projection range. This depends upon the ion beam current you are using and the material under investigation.

Okay, so material for the sputtering process.The ion beam travels across the cross-section of the sample surface up to a depth, which is called a projection range, now refer to as projection range. Now, we will briefly describe the secondary electrons and secondary ions, correct? So, secondary electron emission, okay? This is just the removal of valence electrons from the surface atoms. These are used for the SE image, okay?

Which is similar to what we use in the SEM, Scanning Electron Microscope, correct? In the Scanning Electron Microscope, the electrons interact with the surface atoms, and there is a removal of valence electrons. They provide information related to the surface topography or the surface, correct? Similarly, by the interaction of the ion beam with the sample surface, you can generate these secondary electrons, and they can be used for imaging. Correct?

Here, the yield of SE, secondary electrons, is much higher than in the electron microscope. Here, for an ion beam, per ion, you can generate 1 to 10 secondary electrons.

But for an electron beam, Or in an electron microscope, for each electron, you can generate only 0.1 to 1 secondary electron. Okay.

So the yield of secondary electrons is much higher when using the focused ion beam. Okay. Next are the secondary ions. Okay. So these secondary ions are produced due to the high-energy collision of the ion beam.

Okay? And the sample atoms get ionized or polarized on the surface. Okay? So, and this is followed by these ions being knocked out due to these secondary ions. Correct?

And these secondary ions usually carry chemical information. Correct? about the specimen surface. Okay, so it is nothing but, as in the atom probe, you are depending upon the chemical species. Actually, you can have atoms that are polarized and convert to ions, which can accelerate towards the detector. Similarly, these secondary ions from the sample surface carry chemical information. Correct? So, these are called secondary ions.

Now, there is also backscattered ions. So, it might possible that the incident ion gets travel into the sample surface and the same ion beam can actually backscatter. It means that the same primary beam can bounce back. These are called backscattered. And these backscattered electrons or backscattered ions are nothing but the gallium plus ions if you are using the gallium ions as the source.

Correct? And due to the interaction of these gallium ions with the sample, you can also generate heat on the sample surface. These are termed as phonons. Okay? So, these can create atomic vibrations due to the interaction or waves or we can call as atomic vibrations or waves with the samples which is having a certain crystal structure and the

they can represent the form of heat, heat generation into the sample surface, correct? So, these are the basic radiations or basic emissions while when your ion beam interacts with the sample surface, okay? So, during the interaction of the gallium with the sample surface, We also encounter an issue which is called ion implantation. As I told you before, if you have a sample surface, okay, so and if you have a gallium feed or the gallium ions which are accelerated towards the sample surface, this is your cross section, okay.

They can travel into the sample surface until the moment when they lose all their energy. Fine, and if they lose their energy inside the sample, these gallium plus ions will remain in the sample. Okay, so this is called ion implantation into the sample surface. Usually, the gallium ions can implant up to a thickness of around 10 to 12 nanometres. So, the gallium ion penetration depth is around 10 to 12 nanometres.

However, in the case of electrons, it is mostly in the range of 18 to 2000 nanometres. So, electrons can travel much farther into the sample surface, but the gallium interaction is around 10 to 12 nanometres. And on this surface, there will usually be gallium implantation because some of the gallium ions lose energy and stay in the sample surface. Okay. So, the average depth, as I told you, the gallium goes into the sample and comes to rest, which is called the projection range.

It's called the projection range. R project, correct? This is called R projection. So, in the case of gallium, when the gallium goes and gets implanted into the sample surface, it can change the physical properties of the sample on that layer, in that layer, the physical properties of the sample, which may be the crystal structure.

You can, if there is a presence of grain boundaries, then the grain boundary chemistry will change. And some other, you can call them characteristics of the specimen. So, the local composition may change in the solid solution. Correct? So, this is what we call gallium damage.

We usually call it gallium damage due to the gallium implantation into the sample surface. Correct? So, we will just briefly describe the sputtering process. Then we will just introduce the sputtering process. What is the sputtering process? It is nothing but the removal of atoms from the sample surface.

Okay, so sputtering occurs when atoms are actually ejected from the sample surface. Okay, so it means that this is a type of displacement where the atoms near the sample surface get displaced into the environment, and the more collisions occur, between the ion beam and the sample surface, the more likely sputtering will occur. More sputtering means more atoms can be removed. It depends on how much the ion beam locally

interacts with the surface atoms of the sample. Okay, so sometimes it happens that atoms located deep in the sample are less likely to be sputtered out.

Okay. So, only the surface atoms are more prone to the sputtering process, which is the removal of atoms. So, this sputtering process or the rate of the sputtering process depends upon the surface atoms that need enough energy for the sputtering to take place. Okay. And also the

the surface atoms' binding energy, okay. So, third is the incident angle, and the fourth thing is the initial ion beam energy. These are the four conditions which usually control the speed of sputtering or the sputtering process by these four factors. Okay. So, with this, I will end this class now. And in the next class, what we will do is we will briefly go through this sputtering process

and how it depends on the incident angle and what are the different aspects of FIB, which we need to carefully look into. Okay. Thank you.