Advanced Material Characterization by Atom Probe Tomography and ElectronMicroscopy

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So welcome to this class. Last class we discussed just introduced the sample preparation methods. We just discussed about the electropolishing, double layer method and also the fine polishing along with the use of some inert material like platinum loop. So, now, but the only disadvantage of with those electropolishing methods is you cannot prepare the site specific lift outs where you need to do the field of operation from some interfaces, grain boundaries or some specific phases, heterophase interfaces.

So, now, we will just go through very popular technique which is based on ion beam techniques. ion beam techniques okay so in these ion beam techniques what we used is a focused ion beam with this method actually you can do the site specific lift outs means whatever the features You are interested. Those can be lift out. Those can be from those you can make a needle specimens and you can you can analyze by atom probe.

And these methods are used for the material which cannot be polished by electropolishing. As I told you, the material should be electrically conductive. So, focused ion beam can be used for those kind of materials. Usually, it follows the cutting, cut away methods or lift out methods. And once these are cut away or the lift out or the region which is interested that is lift out then these are sharpened until the cut out or the lift out sample becomes the sharp needle specimen.

Okay. And this is done by the method called annular milling. We will come to the milling basics just to introduce that these are called annular milling. So we use a focused ion beam. Focused ion beam—these are nothing but energetic ions that are focused into a fine beam.

Okay, so in this slide, you can see that if you have a cylindrical specimen—okay, actually, you can use energetic ions such as gallium. We will come to why gallium is used, but just for now, these gallium ions can be used by an aperture, which is called annular milling. And it can be converted to a thin specimen of radius up to 100 nanometers from a cylindrical sample. Okay. So this is called annular milling.

So with FIB, what can we do? With FIB, these energetic ions can be used to remove material. They can be used to remove material in micron to nanometer scale from the site-specific region. This means that it can be used for micro-machining. It can be used for micro-machining.

What is micromachining? You can use patterning from nanometer to micrometer scale. For example, you can use it for nanopores, generate nanopores, create microfluidic channels, prepare microfluidic channels, and also make 2D to 3D structures at micron scale. Okay, and this micromachining can also be used as fiducial markers.

Inverse sample, okay? So here, you can see that this is a secondary electron image, a secondary electron image, and you can see this is a material, and the material has been removed from this particular location, okay? Around the some specific site-specific region where the region of interest is located here, or actually, the material is removed around this site-specific needle by using high-energy ions. This is called micromachining.

The second thing that can be used is the cross-section. So this is your material, okay, and you can do cross-sectioning. Now, if you see the top surface, this is your top surface, you cannot see the material beneath or inside this material. Okay, so actually, you can remove the material so that you can see the cross-section of that particular material. Okay, so features that are hidden underneath.

Features that are hidden underneath. The sample surface, okay? So, these features can be explored and analyzed. Here, you can see there is a grain boundary, okay? And you can see that by cross-section. You can actually see this cross-section—how the crack is propagating along the grain boundary, which is not visible from the top surface. Correct? So, this is the cross-section sample preparation. Third is the site-specific sample preparation, either for TEM or for atom-probe tomography. Okay, so you can actually—this is your material.

You can cut the material at this particular angle, in these two particular angles, so that you can get a lamellar. And this particular lamellar— This is your side cross-section. If you see in this side cross-section, this is your viewing direction. And from the top—this is your top—you can see this particular lamellar which is hanging. On this particular location. And it is free from this side.

And you can use the manipulator. To remove this particular lamellar. And paste it on a silicon coupon. This will come later. And you can iron-polish.

To make a sharp needle. Or you can actually thin down this particular lamella. For making a TEM sample. This can be done in a site-specific sample preparation.

So, if you have a microstructure, if you have certain grain boundaries, you can locate the region of interest and you can take out the sample from that particular region so that you have that particular grain boundary either on the TEM sample or on the APT sample preparation. Okay, so more details will be discussed later. First, we will discuss the instrumentation. Instrumentation of the focused ion beam (FIB) machine. So, we will just cover the details of FIB. I hope that you are familiar with SEM,

scanning electron microscope, where you use a single column for imaging purposes, where it accelerates the electrons. Now, coming to the instrumentation. So, as I told you, a FIB is a focused beam of ions, okay? And this particular beam—this particular beam on the sample surface—this is your sample, this is the beam. This beam is rastered or scanned across the sample surface. Okay.

And this beam is of highly energetic ions. So when these highly energetic ions hit the sample, actually what you can do is if you have a sample surface, if this highly energetic beam hits the sample, that particular sample or the material atoms, the energy is transferred from these particular ions, the energetic ions. So, these atoms on the surface can be removed. It is just like trenching.

It is just like trenching in sand. Correct? So, this is called milling. Okay, so the FIB, the focused ion beam, can actually remove the material, can remove the atoms from the material so that it trenches the region of interest. Okay, and this cut or trench or milling, so this can be termed as cut, trench, or milling, can be done in few nanometer scale to micrometers.

This is the range of these focused ion beams. Okay, so in the construction, what are the basic things to be remembered? So, you will have an electron gun or an electron column, an electron gun column. Okay, you have a certain chamber. Fine, now, you have a FIB column.

This is your electron gun column. This is your FIB column, and there will be a gas injection system. This is called the gas injection system. Okay? And these gas injection system and FIP column are focused in such a way that they focus on the sample stage.

This is your sample stage, and this is your sample. Correct? And this is a complete chamber of your cross-section, a chamber cross-section of a typical dual FIP focused ion beam and SEM machine. Okay? So,

The first part is the electron vertical column, which is used to image secondary electron images and backscatter electron images. The second part is the ion gun. What is an ion gun? This is called an ion gun. And this particular ion gun generates highly energetic ions.

which actually travels and hits the sample surface. Okay? The third important part is the gas injection system, or we can call it a GIS. This is used for welding and patterning on the surface. on the sample surface, okay?

And fourth, we will describe the function of all these parts, okay? And the fourth is the manipulator for in-situ lift-outs. So, the manipulator is just located here, which is a needle. This is called manipulator, okay? And along with this, you can also attach several types of other detectors, such as backscattered imaging, EBSD, and EDS detectors.

Okay, so all these things can be simultaneously attached to this particular chamber of the FIB. Fine? So, the electron gun's function is to generate electrons, and these electrons are accelerated across the column and hit the sample surface. So that it generates signals which can be used for imaging. What signals can it use?

So, the signals which are produced are these electrons. If you hit the sample surface, they can produce secondary electrons from the surface of the sample and backscattered electrons. These are the incident electrons which travel across the sample and come out. That is why these are called backscattered electrons, which give your Z contrast. So, the electron column is used for imaging purposes. What is the function of ion gun?

Ion gun function is to cut trench or milling. It means that it produces a high energetic field ions we will discuss about the gallium gallium ions which are very heavy ok and these gallium ions accelerate across this column and hit to the sample surface so that the region which has to be cut trench and mill can be can be analyzed ok so gas injection system it is just a particular gas the gas it is usually it is a chemical gas And this gas injection system is used for welding and also the patterning on the surface.

And to lift out the samples, whichever the cut or trench samples which you want lamellar, you have to use the manipulator. So, we will come to each section separately. One by one. Okay. So, first we will describe about the ion column which is the basic for this particular sample preparation.

Okay. So, we will describe the ion column. Okay. So, this is your column. Okay.

Okay, so this is the cross-section of that particular ion column. Okay, which, if schematically I show you, is your electron column. The ion column was here. This is your gas injection system. There is a manipulator and there is a sample.

Okay, so we are schematically drawing this particular column. This is your ion column. This is your electron gun. We are schematically drawing the cross-section of this particular column. First, you have a source.

This source is called a liquid metal ion source. Liquid metal ion source. Okay. Then there will be a set of condenser lenses. This is a condenser lens.

Okay. Then there will be an aperture with different holes, different-sized holes. An aperture is nothing but a plate which has different-sized holes. Those holes, in those holes only, the iron, gallium ions, or the ions which are produced from the liquid metal ion source are accelerated.

These are called apertures. Or we can call them beam-selective apertures. Then there will be a set of lenses which we call quadrupoles. These are called quadrupoles. which has a hole underneath.

Okay. So, there is also a pair of blanking plates. Okay. There is another set of lenses like a quadrupole. There is an octopole.

There is an octopole. Okay. And there is an objective lens. This is called an octopole. And this is called an objective lens.

Okay. Then, this is an objective lens. So, the ion source—this is called an ion source. The first thing is, it creates the beam. Okay.

Okay? And the electrostatic lenses, as I told you, are electrostatic lenses. What are the electrostatic lenses? Such as the condenser lens, objective lens, all these electrostatic lenses that control the shape of the beam, the ion beam. Okay?

The third thing is the beam selective aperture. Based on this aperture size, you can vary the ion beam size or you can call it the current, the ion beam current. Okay. So, based on the aperture size, you can select whether you need a higher current or a higher beam size, or a lower beam size or a lower current, based on this aperture. And the fourth thing is the blanking plates.

Blanking plates. So, these are used in the FIB column to push the beam away whenever it is not used, whenever it is not required. Okay. So, if you have a sample here, your beam is generated in this particular source. It gets accelerated.

And these condenser lenses—condenser lenses—mean that they condense the beam. What is the function of a condenser lens? It condenses the beam. So the beam, which has a spread, gets condensed, and by selecting this aperture, you can actually select the beam current or the beam diameter. How much beam diameter has to pass through? Then it goes to this quadrupole, a set of quadrupoles and octupoles, so that they control the beam shape.

They will also control the beam shape, and it goes toward the objective lens, gets accelerated, and falls on the sample surface. So whenever you don't want to mill it, Milling can be done depending on whether you want to mill or not. So, whenever you do not want to mill it, these blanking plates can be used to divert the beam away from the optical axis so that it will not fall on the specimen. Okay.

So, that is the role of blanking plates. So, this is the typical reconstruction of the ion column. Now, first, we will discuss the ion gun. Okay, so first, we will discuss the ion gun. As I told you, this particular ion gun is nothing but a liquid metal ion source, which is called LMIS.

Okay, and most of the FIBS, focused ion beams in the present scenario, this liquid metal source is the gallium. Okay, so this is the most common in microscopy applications. Okay, and this instrument, by using this gallium plate, generally operates with an energy range of 500 electron volts to 30 K electron volts. With the beam currents of 1 picoamp to 65 nanoamps, which is a very high current. And this change of current can be done by apertures, which we have discussed just now.

Correct? And with this gallium-ion feed, you can get a resolution, resolution of trenching or resolution of imaging up to 5 nanometers is possible. Okay, similarly, in the new FIBS, in the focused ion beams, they also use gas-filled ion systems, which are called GFIS. For this, you can use helium or neon instead of gallium ions. Now, this is one type of source which is very common in several institutions.

There are also some microscopes which use gas-reliant sources. There is a third type of source which is called an inductively coupled plasma source. Okay. And these are used; this uses noble gases such as xenon. Okay.

So, based on the time, based on the time for milling, you can actually use these gas-filled ion sources or inductively coupled plasma sources where the time for milling is much less, and you can actually mill the samples on a millimeter scale by the plasma FIB sources. Correct. So, liquid metal ion sources, we can use the gallium ions for a few microns, and the resolution is 5 nanometers. But for the gas field and plasma sources, the resolution is much lower. So, it means it can be a few hundred nanometers.

But the rate of milling is much higher for these sources. Okay? Now, This particular class, we will just focus on the most common, which is the liquid metal ion source. Okay.

And the principle of the column and the principle of the ionization is similar to the gas field and the coupled source. Okay. We will just discuss the LMIS, which is called the gallium liquid metal ion source. Okay, if you see the cross-section, we have shown that this particular liquid metal ion source, if you zoom into this source if you see the cross-section, you will have a structure like this.

There is an insulating mount. Okay, there is a tungsten needle, okay, and this is connected to a heater coil. Okay. And this particular part is the reservoir. Okay.

And these are the set of suppressors. We call them suppressors. Okay. So this is a coil heater. This is your tungsten needle.

This is your gallium reservoir. Okay. Okay. And from this gallium reservoir, actually, the gallium metal. So gallium metal, as you know, is usually liquid at room temperature.

Okay. So this is called a liquid gallium reservoir. And from this gallium reservoir, the gallium flows along this particular tip and forms a sharp cone. This particular cone is called a Taylor cone. It has a radius of 5 to 10 nanometers.

And beneath the Taylor cone, you will have an extractor. This is called an extractor, which has around minus 15 kV. It has a negative bias. Correct? Because it has to accelerate the positive ions from the source.

So, it has a negative bias. These are called extractors. Okay? So, as I told you, this particular cone is nothing but a cusp, a tiny cusp. Okay?

And near this tiny cusp, you will generate a very high electric field. For field emission. Now, I hope that you must be thinking about the atom probe. So, in your atom probe needle—in your atom probe needle—the needle is kept at a positive voltage. There is an electrode that is kept at a negative voltage, and due to the application of DC voltage, what will happen?

The atoms on the tip surface get polarized and ionized; they will become positively charged and these positive charges are accelerated along these field lines toward the negative charge—the negative bias, correct? This is the same concept used in the focused ion beam, okay? So, here, this tiny cusp has a very high electric field, and this is for field emission, which is around 10 raised to the power of 10 volts per meter.

This electric field density or electric field concentration is enough to field-evaporate the gallium—the liquid gallium—into gallium ions. Correct? So, now we will talk about the gallium ions—the gallium ions. So, the gallium reservoir—this is the gallium reservoir. The ions—the gallium ions—are extracted from the Taylor cone.

So, the ions are extracted from the Taylor cone by the application of an extraction bias, which is around negative 15 kV, and these are accelerated down the column. So, this is your column, and this is the extractor, and these gallium ions are accelerated toward the column due to the application of this negative bias. So, these are accelerated down the column. So, there is a field emission of gallium plus ions from the source. Now, we will talk about something related to the gallium reservoir.

Gallium is mostly used for—as I told you—at room temperature, or near room temperature, the gallium melting point is 29.8 degrees Celsius. Okay, so it can be a liquid at room temperature. And gallium is a very heavy element. So it has a high mass, correct? So, because of the mass, the density is very high.

And also, the gallium—the interdiffusion, the interdiffusion of gallium to tungsten—is very minimal in the liquid state. So, the gallium is heavy enough that it can be used for sputtering. Sputtering means it is nothing but the removal of atoms from the surface. from a wide variety of materials. Okay?

So, this is the cross-section of the gallium. Now, we will just talk about the field emission, which we have just introduced. Correct? Now, if you particularly see this needle, if you see this needle with the Taylor cone, correct? So, I am just drawing that particular needle.

Okay, and the liquid gallium will flow and form a Taylor cusp. So, this is called the Taylor cone. Okay? And this is a tungsten needle. So, when you apply an extractor here, which has a negative bias voltage, what will happen? The first thing is, it will induce an electric field.

It will induce an electric field across this, in front of the Taylor cone, which is nothing but liquid gallium. And this electric field pulls the gallium from the needle tip. But also, due to the presence of the tungsten needle and the surface, it has a certain effect of surface tension. This surface tension can put up a force against the pulling of these gallium ions towards the extractor. Okay, so once the balance is achieved, once the balance is achieved,

Between the surface tension of the gallium and also the pulling force of the bias voltage. Then the liquid gallium forms a conical shape, which is called a Taylor cone, and the tip radius is between 2 to 10 nanometers or 5 to 10 nanometers. Now, due to which the electrons—the electrons— From the gallium atoms, the electrons from the gallium atoms at the cusp can tunnel toward the tungsten tip. Okay, in this particular direction.

Due to this tunneling of electrons toward the tungsten tip in this particular direction, usually what will happen is called field ionization. Of gallium atoms, once this field ionization of the gallium atoms happens, these gallium plus ions get reflected. And it accelerates toward the field lines, which is called field evaporation. So here, there is a combination of field ionization plus field evaporation. This is how the gallium plus ions get accelerated toward the column. Okay.

Due to the extractor. Fine, so. So, we have just gone through the cross-section of the ion column. Okay, so you can see this particular region. This is an ion column, and if you see the cross-section of the ion column, there is a set of lenses. There is a source, there are blanking plates, there is an aperture to control the beam current, and there are a set of lenses which are quadrupoles.

and octopoles which can control the beam shape and size, and also the objective lens so that it accelerates towards the sample surface. Correct, and this liquid metal ion source, the most commonly used is gallium. because it is a liquid near room temperature, with a melting point of 29.8 degrees Celsius, and also it is stable enough to be used for the sputtering process. And the extraction of gallium ions is a mixture of two basic principles, which are called field ionization and field evaporation. Field ionization occurs by the tunneling of electrons towards the tungsten tip, which leads to the formation of a Taylor cone of gallium plus ions.

And due to the electric field and the extractor, these gallium ions get field-evaporated from the liquid and accelerate towards the column, towards the specimen surface. With this, I will end this class, and in the next class, we will go through other aspects. Other than the ion source, we will talk about the ion column and the other parts present in the ion column, which are related to the ion optics.