Biomedical Ultrasound, Fundamentals of Imaging, and Micro Machine Transducers

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Lecture - 57

Hi, welcome to this lecture. We are going to talk about material characterization techniques, particularly STM and AFM. STM stands for Scanning Tunneling Microscopy, and AFM stands for Atomic Force Microscopy. Now, when we are going to see how sensors or transducers are fabricated, you have seen lithography techniques, PVD techniques, and CVD techniques, In that, when we deposit a film, we need to understand the material properties of this film. In the case of ultrasound, it will be a piezoelectric material. What is piezoelectric? When you apply pressure, there is a change in voltage.

To understand the piezoelectric properties, we have to use something called X-ray diffraction, which will be taught in a separate class. We also need to understand the roughness of the film using AFM (Atomic Force Microscopy), the tomography of the film, and the electrical properties of the film. All these material characterization properties are very important whenever you are fabricating any kind of sensor or transducer.

So today, let's focus on STM and AFM. STM, as I mentioned, stands for Scanning Tunneling Microscopy, developed by Gerd Binnig and Heinrich Rohrer at IBM Zurich Research Laboratories in Switzerland. You will appreciate that everything discovered in the field of optics and microscopes has generated valuable research, and this particular work resulted in a Nobel Prize in Physics in 1986. The instrument works by scanning a very sharp metal wire—a very sharp metal tip—over a sample, very close to the surface.

It scans without touching, maintaining a small distance between the metal wire and the sample. By applying electrical current to the tip of the sample, we can image the surface at an extremely small scale, down to resolving individual atoms. The advantage of this particular microscopy is that we can see atoms on an atomic level, essentially visualizing surface tomography at the atomic scale. There is a tunneling current amplifier, a distance control and scanning unit, piezoelectric tubes that help control the probe's distance, and we can scan the tip. In the context of quantum mechanics, quantum mechanics tells us that electrons have both wave-like and particle-like properties. Tunneling is an effect of the wave-like nature.

The top image shows that when an electron hits a barrier, the wave does not abruptly end but tapers off very quickly—this is a thick wall. For a thick barrier, the wave does not pass through the wall, which is obvious, and we can see that. However, in the bottom image, we see a scenario where the

barrier is quite thin. If it is very thin, part of the wave can pass through, and therefore, some electrons may appear on the other side of the barrier. You can see that part of the wave gets through when the barrier is extremely thin. The number of electrons that actually tunnel depends on the thickness of the barrier.

The current through the barrier drops off exponentially with the barrier thickness. To extend this to STM, the barrier is the gap between the sample and the tip. So, between the sample and the tip, there is a gap; the tip does not touch the sample.

This is the sample, and this is the tip. This is another tip that scans the sample. There are two modes of operation: constant height and constant current. In the constant height mode, the height remains constant, and the variation of current occurs with lateral distance and the surface density of states, allowing for faster scanning rates.

If this is the sample, we keep the distance between the sample and the tip constant. If there is a valley or a pit, or even a mountain or hill, there will be a difference in the current flowing through the system. That is one way of operating it. The second way is to keep the current constant, allowing the tip to scan across the surface. The changes in the height of the tip reveal the STM image.

In this mode, feedback adjusts the height to maintain constant current across the surface, ensuring a constant density of states. In both cases, software is used to add color and analyze the captured data. You can see here the image of a diffraction grating in 2D and 3D, and we can add different kinds of colors. This is how the UI looks, allowing you to visualize changes in the XYZ movement, down to atomic resolution. We can even see blood cells and materials like graphite. We can analyze different types of materials.

Now, what are the advantages and disadvantages of STM? The first advantage is that it offers high-resolution images with low power usage, and no damage occurs to the sample because there is no physical contact between the sample and the tip. The disadvantages are as follows:

First, a vacuum is required because the height of the tip must be controlled. Second, samples are limited to conductors and semiconductors because we apply voltage to the sample and pass current through it. Whether in constant current or constant height mode, we either look at changes in current with constant height or changes in height with constant current. So, the sample must be conductive or semiconductive. The cost is extremely high; the equipment is expensive. Surface preparation takes time, and maintaining the sharpness of the tool is another issue.

This is how the STM tool looks. As you can see, it is a very complicated tool, and operating an STM is not easy. It is also very costly, but the advantages are immense.

Let’s now move to another section, which is Atomic Force Microscopy (AFM). AFM is a tool used to understand the surface roughness of a film. AFM is a very high-resolution type of scanning probe microscopy, with a resolution in the order of fractions of a nanometer, more than 1,000 times better than optical microscopy.

The information is gathered by feeling or touching because the probe actually touches the surface. Suppose this is the sample, and the probe is present. The probe touches the tissue and scans across it, making physical contact. You need a tip, which can be a cantilever. You understand what a cantilever is, Think of my hand as a part of the cantilever, with a tip at the end.

When the cantilever moves across the surface, depending on the roughness of the film, the cantilever will vibrate. If we shine a light on the cantilever, the light reflects into a detector. There is a light source and a detector: the light comes, hits the cantilever, and goes to the detector.

As the cantilever moves across the surface, it vibrates, and the detector captures changes in light intensity. From these changes, we generate an image that helps us understand the roughness of the material's surface. AFM was also developed by great scientists who won the Nobel Prize in 1986.

We can investigate thin films, thick film coatings, composites, glass, synthetic and biological membranes, metals, polymers, and semiconductors. We can understand the roughness of almost any material using AFM.

AFM can also help us understand phenomena like abrasion, corrosion, etching, lubrication, plating, and polishing. AFM can image materials at the nanoscale. You can also measure force at the nanoscale, which is very important.

There are several applications of AFM, starting with life science applications where we can examine actin films, proteins, erythrocytes, bacteria, and DNA. We can also understand material surface science, for example, organic films, transistors, and ferroelectric materials, copolymers, polymers, polymer film engraving, anodic oxidation, DNA on mica sheets, and many other examples of AFM applications are available. As I mentioned earlier, AFM uses a laser for illumination. The laser is reflected off the cantilever. This is number 1, the source or the laser light that falls onto the cantilever. This is the cantilever, and this is the tip. Number 7 is the tip of the cantilever, or you can say the probe tip. The first component is the laser, and the second is a mirror. When the light reflects, it falls on the mirror and then goes to the detector. The third component is a photodetector, the fourth one is an amplifier, the fifth one is a recorder, and the sixth one is the sample. So, the probe tip actually touches the sample.

You have to bring the entire cantilever down, and the probe tip will touch the sample. You can then move the probe tip in either direction depending on how you are scanning the sample. Finally, the cantilever moves across the scanned sample and reflects the laser beam. These are different kinds of AFM tips. In lithography, what we understand and learn is very simple. You start with

the wafer, which can be oriented as 111 or 100. I will always prefer 100 orientation to create a diaphragm. You can create a diaphragm using micromachining, as we have learned.

If you have an oxidized silicon wafer, you can grow silicon dioxide (SiO_2) on it, and you pattern the silicon dioxide to form a window. You then etch this window, which means you are etching silicon. Silicon can be etched using either wet etching or dry etching. After that, you can further pattern the silicon dioxide such that it remains in only specific areas. You then etch the silicon again using wet or dry etching.

Let's say we are using wet etching. After this, you can remove the silicon dioxide and further sharpen the cantilever tip by etching it again. Then, you grow silicon dioxide everywhere. Afterward, you open and etch the silicon dioxide from certain areas and completely etch the silicon underneath those areas. After this, you can do front-to-back alignment and etch from the backside. This would be wet oxidation due to wet etching because of the angles created. If it were dry etching, you would have a sharp cantilever, and the angle created would affect the etching technique.

Finally, you can dip this cantilever in BHF (Buffered Hydrofluoric Acid) so that the oxide will be etched, and you will have your cantilever. There are different types of tips: normal tips, super tips, ultra-lever tips, diamond-coated tips, FIB-sharpened tips, and gold-coated tips, depending on the application. There's another process where you can have multiple tips simultaneously. This is an SOI wafer.

SOI stands for silicon on insulator. This consists of a layer of silicon, then a layer of silicon dioxide, and then another layer of silicon. The top layer is also SiO_2. The silicon sits on an insulator, which is why we call this wafer SOI, or silicon on insulator. You oxidize silicon on the insulating wafer, meaning there is SiO_2 at the front and SiO_2 at the back. Then, you perform lithography so that the silicon dioxide from the back is etched. After that, you etch the silicon, creating a window, as shown in the image. Then, you pattern the silicon dioxide again and sharpen the silicon completely. There is an etch stop here because when etching silicon, the silicon dioxide should not get etched, though practically it does, but at a much slower rate compared to silicon. This is why silicon dioxide works as an etch stop. After that, you can create the tips by using etching techniques. You then grow silicon dioxide again, create a window, and remove silicon dioxide from specific areas before completely etching the wafer. This way, you can have two cantilevers, cantilever number 1 and cantilever number 2. Multiple cantilevers can be fabricated using microfabrication techniques.

Now, let's briefly recall what we have learned about AFM. Atomic Force Microscopy (AFM) is a technique used to measure the roughness of a film. It involves a cantilever for probing the sample, a laser diode, a mirror, a photosensitive detector, and an amplifier. Errors can be measured by comparing the actual signal to the set point. A piezoelectric scanner is used, and a computer records the data. The same setup is shown in both images, but image 1 is a more detailed version.

The principle of AFM operation is simple: it consists of a cantilever with a sharp tip at its end, which scans a specific specimen. The cantilever is typically made of silicon or silicon nitride. When the tip is brought into proximity to a sample surface, the laser beam activates, and the force between the tip and the sample causes the cantilever to deflect. The forces measured in AFM include mechanical constant force, Van der Waals force, capillary force, chemical bonding, and electrostatic force.

AFM scanners are made from piezoelectric materials, as these materials vibrate, allowing the corresponding changes in voltage to be measured. Piezoelectric materials can include barium titanate and zirconium titanate. Whether the material elongates or contracts depends on the applied voltage. Traditionally, the tip of the sample is mounted on a tripod of piezoelectric crystals, which allows scanning in the X, Y, and Z directions (but not in theta). Sensitivity varies from scanner to scanner depending on material differences or size.

AFM can be used for measurement, imaging, and analysis. You can see an image from AFM, which shows the change in the Z-delta, the variation in the film height relative to the distance. You can obtain both 2D and 3D images using AFM. The tip is brought within a nanometer of the sample, and Van der Waals forces act. The radius of the tip limits the accuracy of the analysis: the sharper the radius, the better the accuracy. Stiffer cantilevers protect against sample damage since they deflect less in response to small forces.

We can perform morphological characterization, surface roughness measurements, and analyze physical properties such as swelling, cohesiveness, and smoothness. There are different methods to measure the roughness of a film.

The first method is contact mode, where the cantilever makes hard contact with the sample. This mode is stable for samples in air or liquid. The second method is non-contact mode, which is non-invasive, meaning the cantilever does not touch the sample. In the third method, tapping mode, the cantilever oscillates vertically. Tapping mode is useful for overcoming problems associated with friction, adhesion, and electrostatic force. This mode is also more effective for scanning larger sample sizes quickly compared to contact mode.

In contact mode, the repulsive forces between the tip and the sample are measured as the tip makes physical contact with the sample. However, excessive tracking force applied by the probe can damage the sample, especially when the material is delicate. The second method, non-contact mode, measures the attractive forces between the tip and the sample without the tip touching the surface. This mode works best for soft and biological samples, although it cannot be used in fluids.

In tapping mode, the tip oscillates vertically at a frequency between 50,000 and 500,000 cycles per second. The oscillation amplitude decreases as the probe contacts the surface due to energy loss. Tapping mode solves problems related to friction, adhesion, and electrostatic forces. It's faster and better suited for larger scans compared to contact mode. In the image, you can see the

difference between contact mode and dynamic mode (non-contact mode) in terms of how the tip interacts with the surface tension and the amplitude changes when a fluid layer is present compared to when there is contact.

The tapping mode has its advantages. But in general, if you want to understand the advantages and limitations of AFM, the following are the advantages. The sample preparation is very easy compared to STM. It works in vacuum, air, and liquids, providing accurate height information.

We can do live sample analysis. We can use 3D imaging. We have a dynamic environment, and surface quantification is possible. The limitations are limited vertical range, limited magnification range, data not independent of the tip, the tip or sample can be damaged, and limited scanning speed. The AFM is a versatile tool in general that can be used to understand the topography, properties of surfaces, properties of single molecules, and forces within molecules. However, you always have to consider that there will be a lot of artifacts in experimental conditions, which need to be carefully addressed. The future of AFM, which is actually now (as this slide is a bit older), lies in the fabrication of sharper tips using microfabrication processes. Another development is a more flexible cantilever spring and less damaging, non-sticky probes, which, if we start using them, can further enhance the image quality of the cantilever.

This is an example of a piezoresistive microcantilever. So let us quickly understand this particular example. You have a piezoelectric material that you are diffusing into silicon. These are contact pads: contact pad 1, contact pad 2, CP1, and CP2. These are gold. So, as you see, this one and this one are the contact pads here. The big one goes all the way out. So, in this SEM image, these two are contact pads 1 and 2.

You can see there is an SU8 tip, and you will appreciate the fabrication technique because we have used deep reactive ion etching. You can see a completely released cantilever. You start with an SOI wafer, which is silicon-on insulator. There are specific colors: silicon nitride is purple, silicon dioxide is green, silicon is gray, gold is, of course, gold-colored, boron resistors are yellow, crosslinked SU8 material is blue, and boron contact is orange. Orange or light pink, however, you want to describe it. So, we start with the silicon dioxide wafer. We grow silicon dioxide on SOI and then create a window. Then, we diffuse boron, which is a p-type material, into the silicon. After that, we again create silicon dioxide. Then, we create a window and diffuse high P++ boron as a contact pad. Here, contact pads 2 and 1 are P++. The area where the cantilever is located, which is here, is P-type.

Another question is why we have contacts of this cantilever with P++ while the cantilever is P-type. P-type is a piezoresistive material, The resistor is P-type to ensure that we can extend the contact further, as seen in this whole contact pad. The contact pad is gold, so to ensure ohmic contact, we need to make sure that the conductivity of the contact pad is high, which is why we use P++. After that, we create a gold contact to take the contact pads outside. Then, we deposit

silicon nitride using PECVD. PECVD is a technique used for growing silicon nitride and silicon dioxide at lower temperatures. After that, you create a window, as you can see here, here, and here. Then, you can etch silicon after etching silicon dioxide.

So, when you create a window, you see silicon dioxide. This silicon dioxide can be etched using BHF. When you etch silicon dioxide with BHF, you encounter another layer of silicon dioxide. Then, you coat SU8, spin coat SU8, and pattern SU8. After spin coating the SU8 and patterning it on the backside, you do front-to-back alignment and pattern the backside so that you can first remove silicon nitride followed by silicon dioxide. After that, you can etch the silicon using DRI, so that you can get the final cantilever, which is shown in this SEM image of the piezoresistive microcantilever.

Let's repeat it once again. The first step is SOI. The second step is to grow silicon dioxide. The third step is to create a window for the piezoresistor. The fourth step is to dope the boron resistor. The fifth step is to grow silicon dioxide again. The sixth step is to create a window for the P++ contact pad of the piezoresistive microcantilever.

After opening the contact, the next step (step 7) is to dope P++. The eighth step is where we add the gold contact pad. The ninth step is where we grow silicon nitride and then pattern it. In the tenth step, we remove or etch silicon dioxide and silicon. The eleventh step involves spin coating SU8 and patterning it to form the cantilever tip. In the next step, we perform front-to-back alignment and then etch silicon nitride from the backside.

From this side and also silicon dioxide, we can see the silicon. The final step is to use DRI to etch the silicon. This is how the piezoresistive microcantilever can be fabricated. I hope that now you understand the importance of AFM and STM.

We can also fabricate piezoresistive microcantilevers. Since we are discussing resistive materials, when you apply pressure, the resistance changes. This can also be used to measure the roughness of the film. For example, if there is a piezoresistive cantilever, and this is the cantilever (my hand with a tip), and you are scanning it across the sample, depending on the roughness, the cantilever will bend. When this bends, the piezoresistor embedded or diffused into it will change its resistance due to the pressure. The bending creates stress in the piezoresistor, which causes the resistance to change based on the stiffness of the material deposited. This technique can be used to understand cell analysis, tissue elasticity, and so on.

The beauty of AFM is that not only can it be used with cantilevers that work with a laser diode and detector, but we can also modify this technique to use piezo resistors. In this case, instead of light deflection, the output change is in resistance when you apply pressure. With a piezoresistor, applying pressure changes the resistance, while with a piezoelectric material, applying pressure changes the voltage. With that, I will end this lecture here, and I will see you in the next class. Until then, take care. Bye for now.