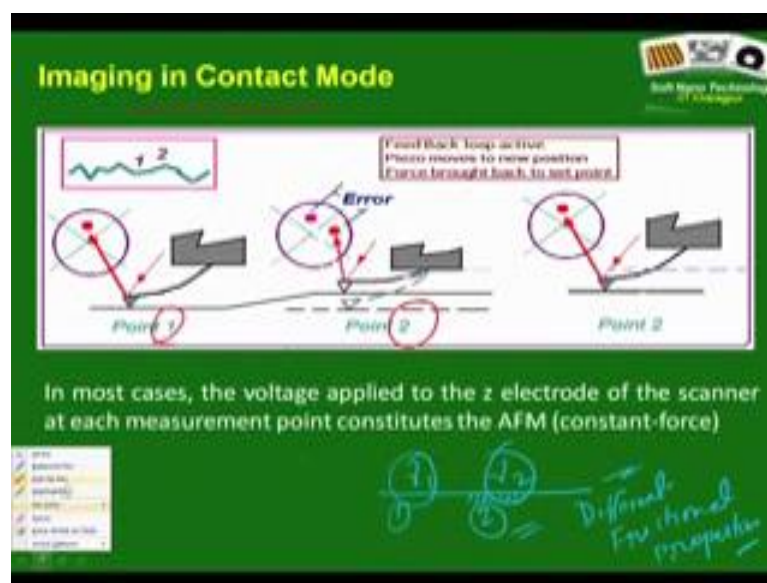


Soft Nano Technology
Prof. Rabibrata Mukherjee
Department of Chemical Engineering
Indian Institute of Technology, Kharagpur

Lecture – 29
Atomic Force Microscope – 6

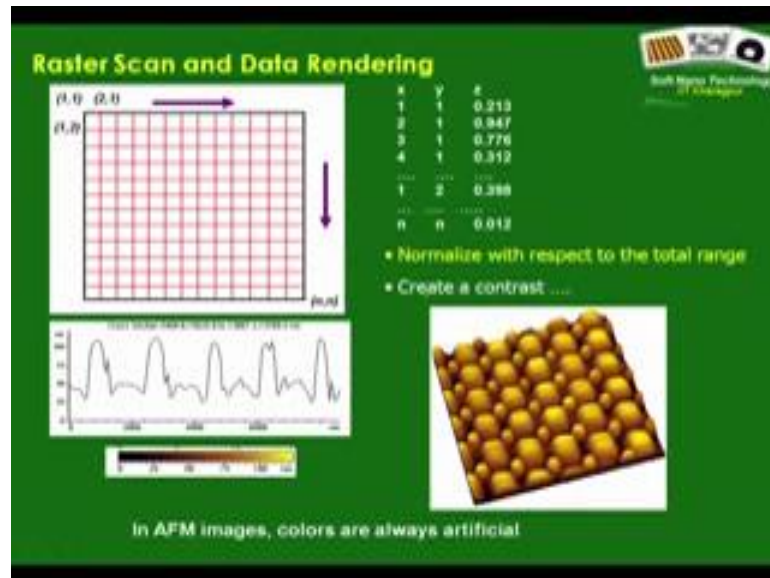
Welcome back. We continue our discussion on atomic force microscope. This should be probably the last lecture on AFM, there as I told AFM is a worst topic and it still developing and involving in various settings, but this is just a preliminary course to give you an idea what is an atomic force microscope several limit is my discussion essentially to the basic modes. Let us see if something continues will in the next lecture will just wrap it up.

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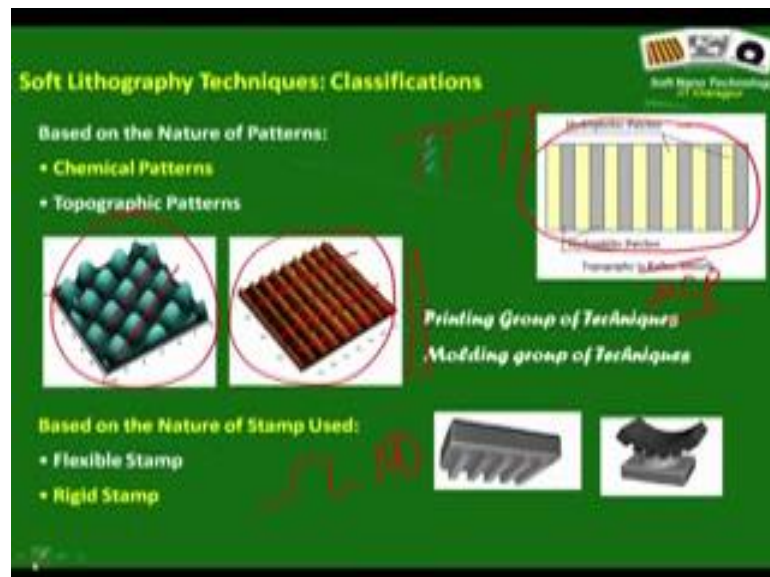
So, this particular slide should know make sayings you all of you, it is essentially some sought of a very enterprise schematic of a constant force contact mode imaging and this moves from point 1 to point 2 essentially in to the during the rastering. And we also now understand it is the piezo electrics scanner that essential takes care of this movement.

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So, this also should we should able to sought of understand. Now this is essentially details you how that restoring is done depending on which duration you restoring you can choose you fast of access and slow access and then you essentially get a data some will be plus and minus.

(Refer Slide Time: 01:37)



And sorry this will be 0 the first point this 0 that is the data anyway. Before I move on there the there are certain issues with their contact mode imaging and the most

significant issue is something I mentioned briefly, during the restoring you are actually driving the tip over the surface.

So, that is detrimental for both the tip and the surface because of the friction that tip sought of becomes blind which is soon. As well as if the surface is soft particularly for polymeric material. Contact mode is imaging often leads to scratches on the samples surface damaging it is so you cannot use. So, there has been development in this so called Non Contact Imaging Modes, particularly the intermittent and contact mode of the tapping mode. Before I move on I would just like to draw your attention to one un-visible topic which we discussed during on discussion on soft lithography and I have just borrowed this slide from all lectures on soft lithography.

So, now all this images in fact you understand, these are all atomic force microscope images and you have also understand whether it is green or brown does not make any sense because all color in AFM image artificial because it is only the color base you choose during the when you convert you data in to the grayscale that in fact matters. In fact, both the images to be honest study in complete because none of them comes with the vertical scale and therefore, when I was read the in this image, I have missed out write the information about the height of the structures; any way coming back to context.

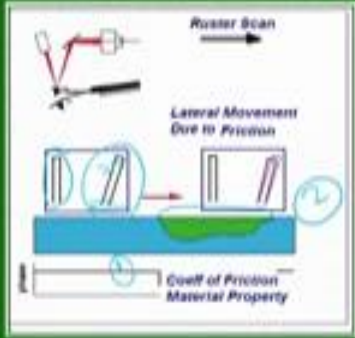
So, now topographic pattern we understand that they can be imaged very conveniently using an AFM, you can get all the relevant information like the profile as well as the feature height which is extremely important in the context of nano patterning you are any circular pattern in technique, but a question still remain unresolved we talk about this micro contact printed surface is right, chemically patterned surface and we are good that these surfaces has virtually no topography, but it has domains of different wettability like hydrophobic, hydrophilic or different wettability contrast regions. So, how does one image that or it is even possible to image that because as the tip moves from point 1 to point 2 it is virtually flat.

However what is the difference between let us a point 1 and point 2 in this type of a surface is these areas are covered with something different some species that exhibits a difference in surface energy and this difference in surface energy is of an associated with different frictional properties also to put it in very simple terms and

that in fact, one can very cleverly use in contact mode imaging to generate and image of a micro contact printed surface.

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Lateral Force Microscopy (LFM)



- Powerful extension of Contact Mode AFM.
- Goes beyond Topographical imaging to detect:
 - Friction
 - Adhesion
 - Mapping of different components of composites, block co-polymers etc.
 - Mapping of different surface energy domains.
- It measures the torsional deformation of the probe, rather than its vertical deflection.

So, let us see what we are essentially talking about. We are essentially talking about a surface which is physically homogeneous, but chemically there are different faces. So, it is a smooth surface, but there is some area which comprises of different material. So, here you see the material is different.

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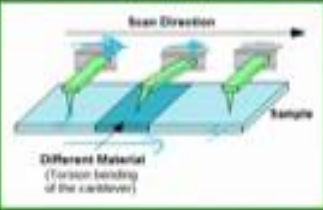
Lateral Force Microscopy (LFM)

During scanning in contact mode the cantilever bends not only along normal to the surface but also the cantilever torsional deformation occurs. (Twisting)

LFM measures the torsional deformation of the cantilever during scanning in contact mode.

The lateral deformation depends on frictional force acting on tip.

LFM studies are useful for imaging variations in surface friction that can arise from in homogeneity in surface.



So, now what happens? What happens is though you have been talking or even in this image shows that a tip sought of is moving in this direction just like that, reality is you are. In fact, the dragging your tip during this restoring action over the surface and please do not forget that the tip is in contact with the surface and it is the scanner because of the contraction or expansion of the piezo that wants to move.

So, we definitely moves an as we moves the tip is also move, but since the tip is add hearing with surface depending on the fictional property is or a (Refer Time: 05:40) properties of the surface there is some sought of an addition, which translate to some static friction, which the tip has to overcome in order to move from point 1 to point 2. Now if the material of the surface is unique form then this.

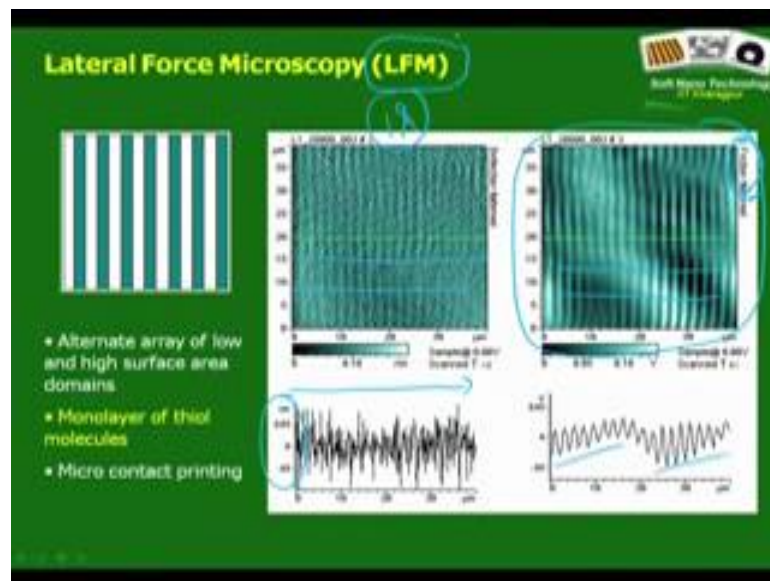
So, what is this static friction going to do? The static friction what does is the tip does not move like this. So, if the tip is moving in this direction, it under goes some sought of a torsion, some sought of a bending, right because of the fact this end is moving while this end is sought of still sack up on the substrate surface right.

So, if what I try to what I am try to emphasize, if the surface is homogeneous we exceptive of the topographic is the surface is physically homogeneous, we respective of the topographic this lateral disruption or movement of TP is uniform across the sample, but now you have imagine that, when you are surfaces different domains, different faces, this frictional resistance or the local addition extent of the local addition is going to be different and therefore, the lateral displacement let us say on this blue patches and this green patches are going to be different. By now you have probably understood that scanning pro microscopes are all about finding out some sought of a error.

So, you see here you can find out pickup of and additional error and additional difference in terms of the extends or the magnitude of the lateral displacement and that is exactly what is shown here for example, on this light blue patch the image is not fully correct once should shown this to be also partially distorted, but that is something missed out. So, you can considered the these areas have very low addition, which might be possible if you have a very low surface energy coating, but when it reaches this particular area you see the addition is more and therefore, the resistance in the form of static fiction is higher which interns leads to more lateral displacement of the tip.

So, you can essentially generate a sought of an additional contrast, not only in the deflection of the tip from point 1 to point 2 right because of the topography of the surface, but also depending on the chemical composition of the surface from point 1 to point 2. There can be additional lateral displacement or of the bending of the tip. And this you can pick up as a contrast and this you typically if you look in to an AFM scan you will find that there is a window which gives you the frictional map the frictional image this is nothing, but the lateral displacement of the lateral force.

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So, what you have are in fact seeing is a very interesting image, this is in fact the image of a micro contact printed surface. So, you see that topography is nearly flat, it look looks rough, but look at the vertical skill it plus minus point 0 minus dynamiter. So, all this roughness is confined to 1 angstrom which means that the surface is extraordinarily smooth, but when you look at the corresponding fictional image or the fictional image you find that there are difference domains and then these difference domains correspond to difference wet ability areas because of micro contact printing.

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Contact Mode

Advantages

- High scan speed
- Atomic resolution can be obtained on a hard surface
- Can image fairly rough samples

Disadvantages

- Capillary bridge formation very likely in ambient conditions.
- Chances of damage to the scanning surface in case of soft samples (Polymer and Bio-logical samples particularly)
- Chances of damage to the tip for hard rough samples

So, this is what is known as one of the secondary imaging modes, but it is Omni present along with whenever you are doing contact mode imaging this is only present this is what is known as lateral force microscope.

So, we are now about to finish contact mode imagines. So, advantages is we can do disenable high speed scan you can get the high resolution and can image fairly rough samples, but there are disadvantages since the tip is in contact with the surface and typically AFM does not require any sample preparation or imagine is done in ambient condition I mean you do not require any account chamber of something like that, but since a sharp trip is always in contact with a surface, there is always tendency if you lab environment is humid that a capillary bridge might form and the sought of reduces the resolution.

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Non Contact Mode

- In non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically from 100 to 400 kHz) with an amplitude of a few tens to hundreds of angstroms.
- Then, it detects changes in the resonant frequency or vibration amplitude as the tip comes near the sample surface.
- The total force between the tip and the sample in the non-contact regime is very low, generally about 10^{-12} N.
- In NC-AFM mode, the system monitors the resonant frequency or vibrational amplitude of the cantilever and keeps it constant with the aid of a feedback system that moves the scanner up and down.
- By keeping the resonant frequency or amplitude constant, the system also keeps the average tip-to-sample distance constant.

Something that I have already mentioned that, the contact mode always has a tendency or possibility of damaging the surface as well as tip on the rough or very hard samples.

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Approach

Set Point = 2 mV

Read after alignment. When light is falling on the center and reading is 0V (0 mV), it is equal to frequency.

At that time you have to stop the scanner.

Position of Reflected Laser at Jump to Contact

There is a non zero value of reading on the GPD.

Contact

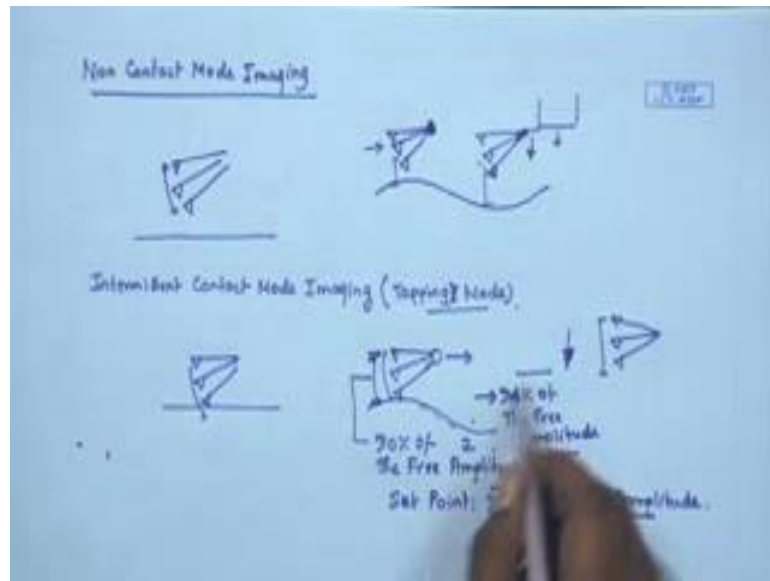
Straight

Convex

Sample Surface

So, the development essentially was the non contact mode image, we image in which I essentially pointed out let you do not go all the way here, you would like to keep your tip few nanometers away from the surface and do something.

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So, if you are few nanometers away for example, for here the tip will have a configuration like this and you can raster it and try to track if there is any change. That is seldom done what is done in non contact mode imaging is, one takes advantage of the fact that can to nearly behaves like a spring. So, there is an in fact, and additional drives. So, which oscillates the cantilever?

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Intermittent Contact or Tapping Mode

Tapping mode: frequency

Resonant excitation frequency:

$$\omega = \sqrt{\frac{k_{eff}}{m}}$$

k_{eff} effective force constant in cantilever mass

$$k_{eff} = k - \Delta F$$

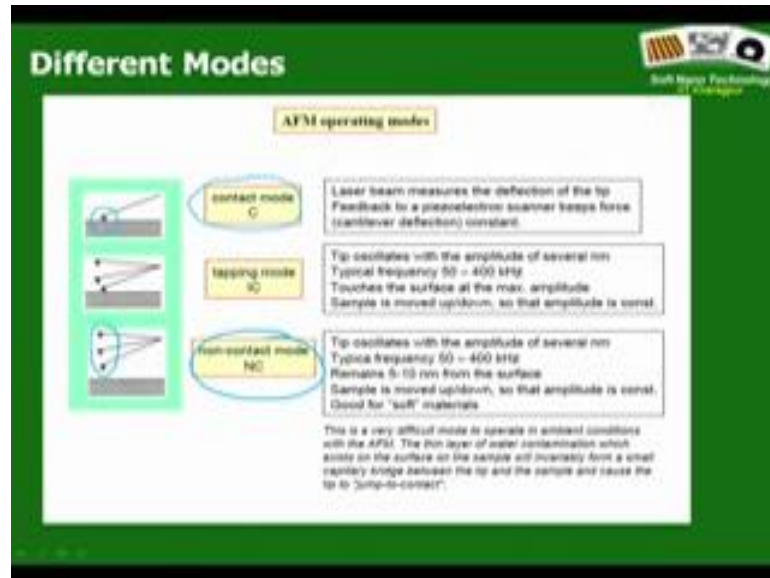
k : force constant of cantilever
 ΔF : change in the external force

movement

- $\Delta F < 0$, k_{eff} increases, ω increases
- $\Delta F > 0$, k_{eff} decreases, ω decreases

Typically, the oscillation is done at the resonant frequency of the cantilever spring, why? Because at the resonant frequency the oscillation amplitude going to maximum for a given amount of power that you are given.

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So, contact mode you already know where you there is no oscillation, you simply bring in the cantilever in contact with the sample surface. Typically in non contact mode classical non contact mode, what you do? You bring the cantilever very close to the surface and then start oscillate you need at in resonance frequency, you can choose the amplitude, but you choose the amplitude in such a way that the cantilever does not touch this surface right.

So, what happens is here is a surface, you been here cantilever here, you somehow stop here approach here, how you stop it sable discuss and then you start oscillate. Frequency let me remind is a resonant frequency is a cantilever. So, that is why this sort of a material property, but the amplitude you can choose. In typical non contact mode I will let me repeat you choose amplitude where the oscillating tip does not touch your sample surface.

So, now what happen if you now do recentering under this condition? So, what happens is that, as you go away the net force sort of changes and as the net force changes because the force that is acting between the tip and the cantilever is low or here and we have look at the resonant frequency of the cantilever. So, which is the effective because what is

important is the effective force constant in fact changes because the ΔF is a the change in the external force and which sought of changes and you would like to keep the amplitude constant.

So, what happens is as the effective frequency changes the amplitude changes and now you what you need to do your piezo gets activated. So, it now again becomes lender and pushes this little bit down ward. So, that your amplitude matches with the amplitude at approach. So, this is in an assail, the typical non contract mode imaging, but getting a good resolution is very difficult because the tip does not touch. Off course it is scientifically very reach because you are in the attractive reign you never touch and you relive entirely on your forces, unlike here in fact, in the deep preposition you are not really reline and on an attractive interaction between the tip and the sample surface.

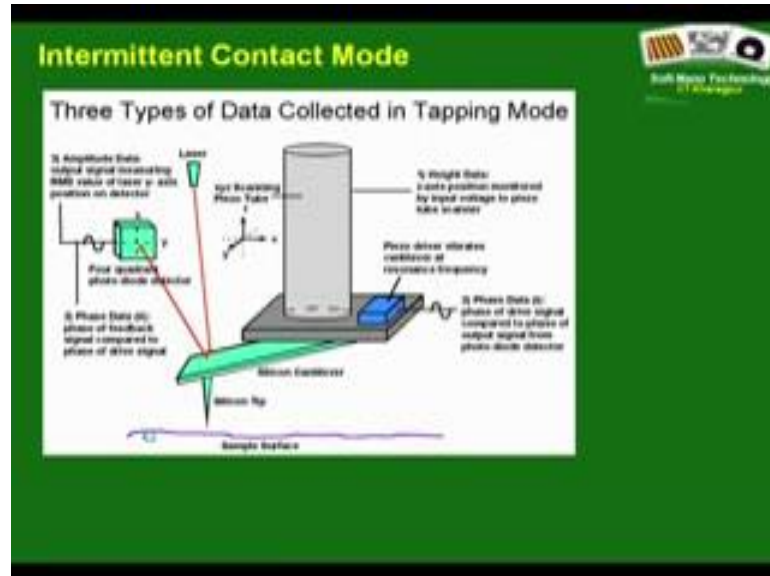
So, I am deliberately sought of rushing through non contact mode imaging because that is not an industry standard any more. What is the industry standard is essentially concept that combines the advantages of non contact mode imaging as well as mode imaging. That is you use the cantilever as a spring oscillate is, but do not oscillate it at a zone where it does not touch the surface. You let it oscillate, you when it a big closer start oscillating and let it up the surface like this it goes on tapping the surface like this. So, what is the advantage? advantage is under this condition let us a approach here again I will discussed a bit on how to approach in a intermitted contact more and what happens is suppose this was the fee amplitude of oscillation and when you bring it in close proximate to the surface this already gets truncated.

So, this is 90 percent of the free amplitude. Now you move on to a next point, this is the point 2. You see the surface is the point two as at lower elevation as compare to point one right. So, what happens is this amplitude let us a now become. So, over here 94 percent of the free amplitude, because there is simply more space and now what you do? You essentially choose a set point let us a just a the way you have a four set point in contact more you choose a set point which is 90 percent of free amplitude and now you know what to do right what to do you this is a set point now you free amp free amplitude has gone up to 94 percent.

So, what you do? This information in the form of the error is fade to the piezo through the feedback look the piezo sought of will change it is geometry pushed down the holder

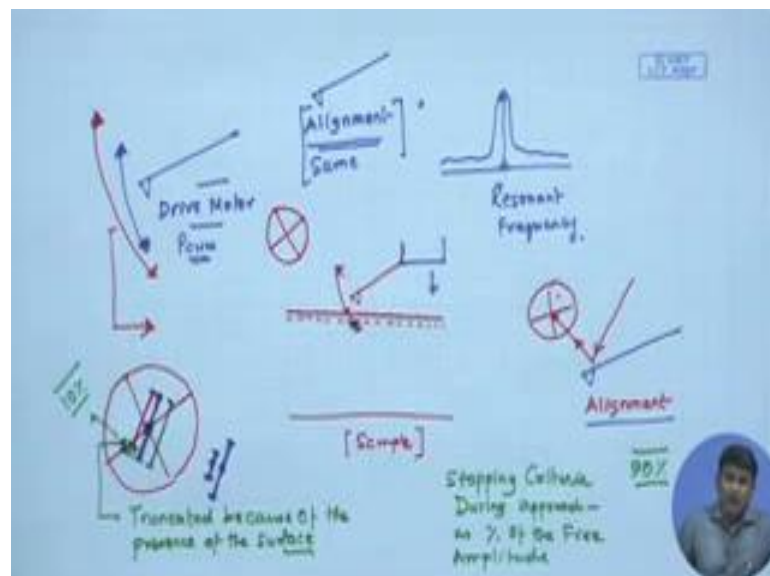
or pushed down the location of the cantilever it comes down. So, that you are amplitude is back again to the 90 percent of the set point amplitude.

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So, this is something I will discuss in a bit more detail is intermittent contact or the tapping mode imaging. Geometry is roughly same, as a system is same only excepting the fact that there is an additional drive at to produce the oscillation of the tip. Most commercial AFM come with the option that you can do a frequency swipe and find out what is the resonance frequency.

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So, what you do is simply do a frequency sweep and you find out the resonance frequency of your ca tip you're using. Typically the manufacturer specifies something, but every tip because it has different dimensions, different geometry due to manufacturing tolerances. So, it is a good practice to find out and any reasonable instrument will allow you to do that to find out the resonant frequency once you find out have found out the resonant frequency.

So, before the tip still up to the alignment is same, you would like to align. So, I think I write here again. Anyway sorry, picture is become a bit bad, after alignment the laser spot falls at the center of the cantilever. So, after you have aligned you have essentially do your frequency sweep and choose. So, there is a drive motor, additional drive motor which will isolate your cantilever tip and you will choose certain fraction of the power, some power for the drive motor. So, what will this drive motor do oscillation, the amplitude is chosen by the resonance frequency this drive motor will decide whether would you like have higher amplitude or lower amplitude.

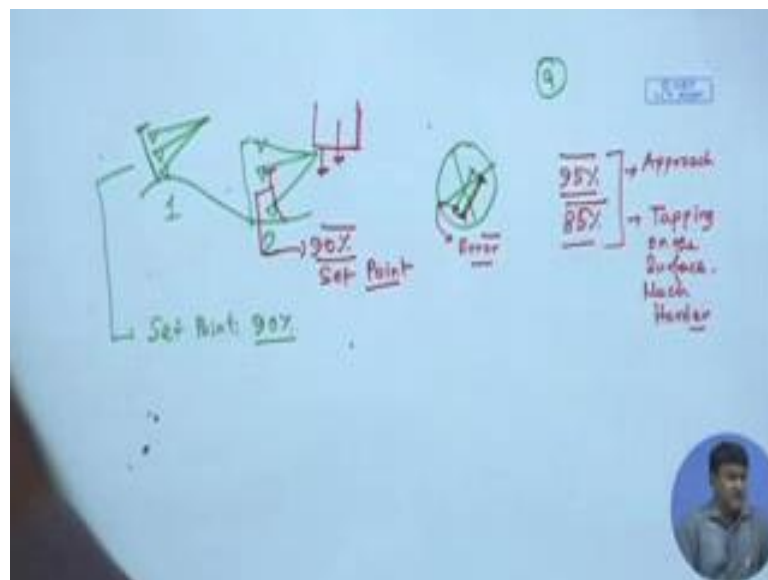
So, for example, this if you are amplitude is high it is obvious that you are going to choose higher drive percentage, if the amplitude is low it is obvious that you are going to choose lower percent. So, well now what happens? So, sample surface is somewhere over here and you have a cantilever that is oscillated. Now as a consequence of this isolation what in fact, happens at that QPD, that is an interesting question to ask because unlike the contact mode where the cantilever was stationary. And therefore, at the beginning of the approach sequence like it was anyway falling or the same time of the QPD, but here may be I will draw a bit (Refer Time: 20:42) you for easy understanding after alignment like it was falling here, but the movement you time known your oscillation, what starts you happen with the laser spot also sought of start to oscillate over the QPD like this.

So, there is a span and let us this span depending on the magnitude of the amplitude you have chosen. So, this range what I mean to say is if you have chosen a smaller amplitude this is going to be range if you have chosen a larger amplitude this is going to be the range right and it has to pass through the center. So, now, the tip is oscillating and the approach sequence again with the help of the steeper motor begins. So, you are now beginning the sample and tip close to each other again is the same you can either take the sample a fortune pushing down, but when it is close to the sample let us see what happens? What

happens is first is start to tap and then as you bring the sample closer and closer let us a this is the configuration if now, since it is tapping the sample surface it fails to execute the whole entire oscillation during it is amplitude right.

So, what happens now on the QPD you see the oscillation is sort of truncated, like this portion is truncated because of the presence of the surface right and now you simply choose your set your stopping criteria during approach as a percentage of the free amplitude. So, let us choose 90 percent of the free amplitude as the stopping criteria. So, when this area becomes 10 percent of the entire oscillation amplitude the cantilever will, the approach will stop. What you now all understand? What is mean by approach will stop the piezo. In fact, gets regard and then you can start scanning.

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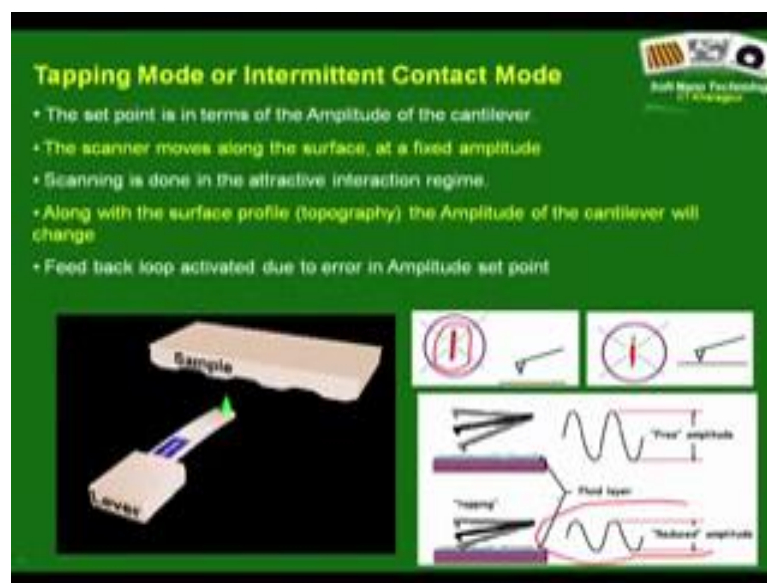
So, now from point 1 to point 2 I have already discuss what is going happen. So, you have in isolating cantilever at point 1 which now you to the restoring, goes to the second point, let us a you have chosen set point to be 90 percent, you move on to point 2 which is at a lower elevation. So, it is certain lower level. So, essentially the free amplitude the amplitude not the free amplitude the truncated amplitude which was 90 percent here let us it has gone up to 96 percent.

So, that 9 whatever is the equivalent that difference between 96 percent 10, 90 percent on the QPD essentially it translate to some amount of voltage. Suppose this was 90 percent, now you have gone up to 96 percent. So, it has increased please remember it has to pass

to the center I am just drawing them site by site. So, this is now the error and this error is fade to the scanner to the piezo scanner. So, that the can take necessary reactions and again once it is pushed back here, it is again that to the 90 percent or the set point value.


So, one can ask you whether you stop at 85 percent versus 95 percent of the free amplitude what would be the implication? Implication is here the approach is a must softer approach and here you are essentially tapping on knocking on the surface much harder. So, that is actual the tapping mode and you can see these pictures.

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So, this now should be clear to you, this the zone corresponding to the free amplitude this gets truncated, actually get truncated from one of the site. So, it is reducing amplitude and you have a set point and based on that some point to point the amplitude is maintains to be constant.

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Tapping Mode

Advantages

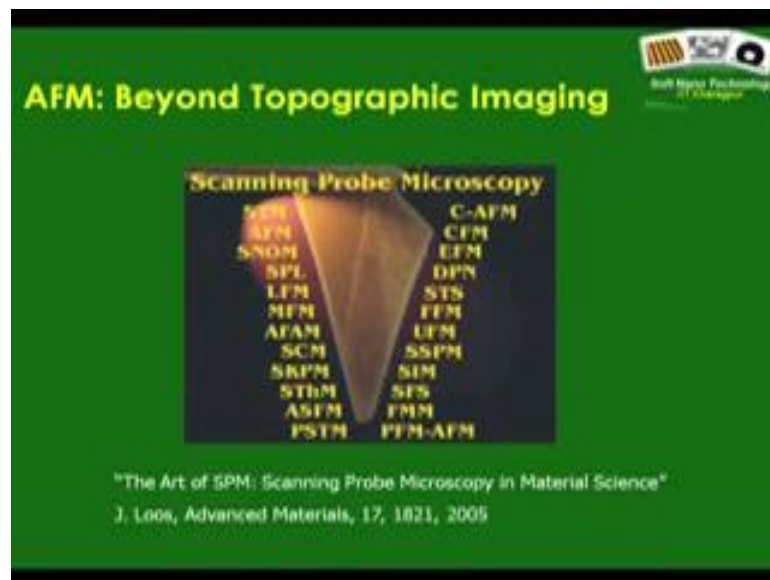
- High lateral resolution
- No damage to sample and the cantilever tip
- Can image fairly rough samples
- Ideal for sampling in liquid cells.

Disadvantages

- Resolution is a shed poorer than contact mode.
- Lesser scan speed than contact mode
- Micro ripples can be generated while scanning under liquid

So, advantage pretty high lateral resolution no damage to sample and to the cantilever tip can image fairly rough samples and ideal for sampling this is wrong; in fact ideal for sample sampling soft images.

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AFM: Beyond Topographic Imaging

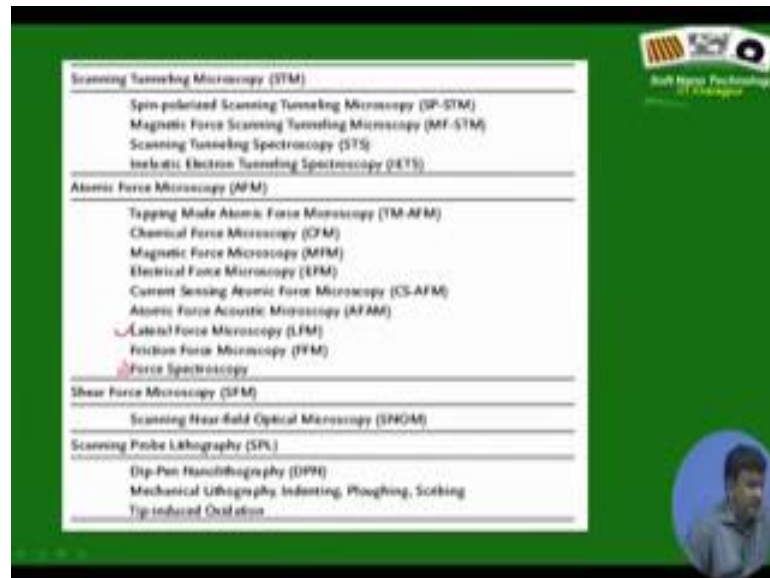
Scanning Probe Microscopy

SPM	C-AFM
AFM	CFM
SNOM	EFM
SPL	DFM
LFM	STS
MFM	IFM
AFAM	UFM
SCM	SSPM
SKPM	SIM
SThM	SFS
ASPM	FMM
PSTM	PFM-AFM

"The Art of SPM: Scanning Probe Microscopy in Material Science"
J. Loos, Advanced Materials, 17, 1821, 2005

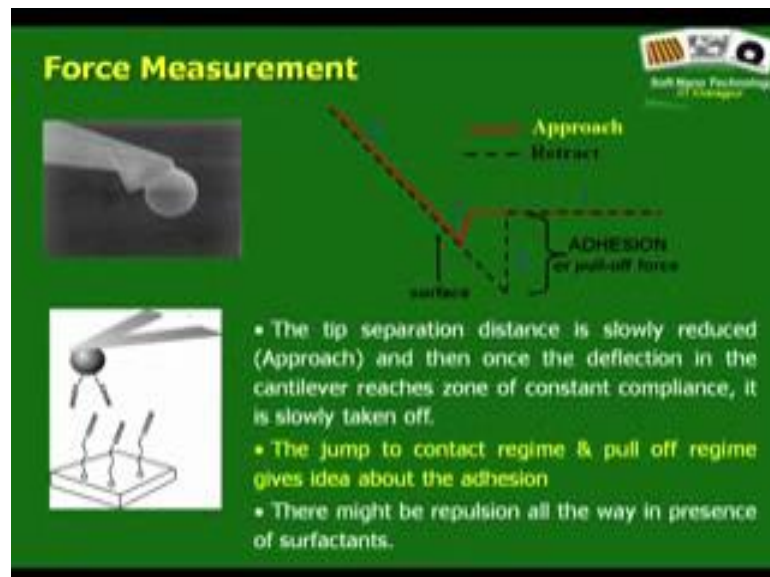
Resolution is a bit poor. So, it is almost time to rape up AFM, just one quick slide I would remained you that AFM can be used for doing a whole lot of additional measurements like let us ASTM we have already talk about.

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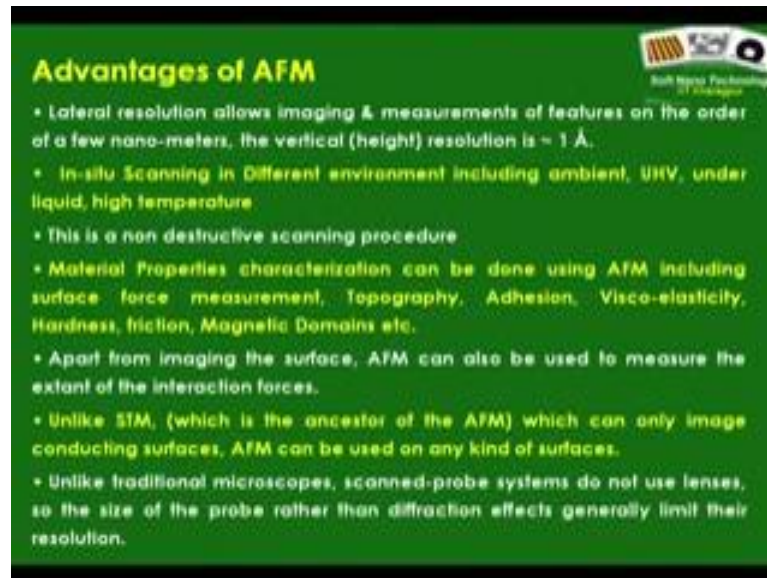
One can do magnetic force microscope electrical force microscopy atomic lateral force microscopy something in you have already told we can also measure the interaction force between that tip and any location of the surface. This particular transference will be available to you as a resource.

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There are some bullet us on some of these advance techniques, but I would not discuss them because I do not want to make this course too heavy and therefore, I stop my discussion on AFM right here.

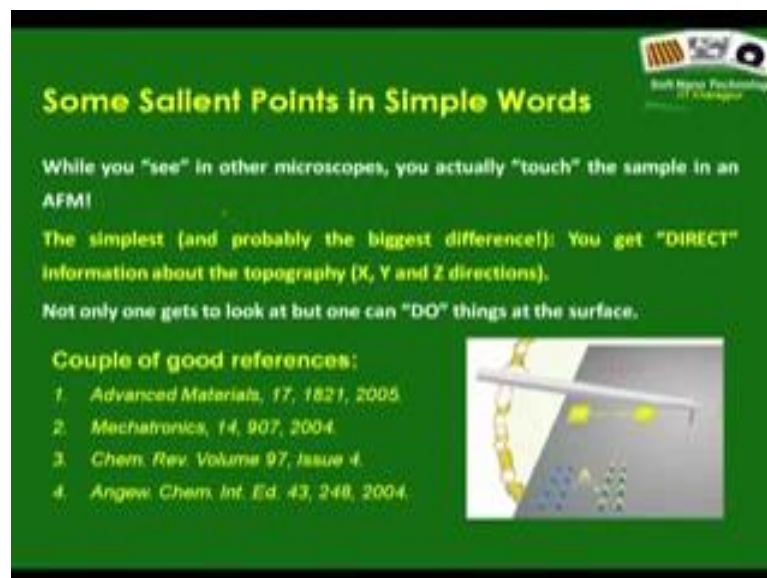
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Advantages of AFM

- Lateral resolution allows imaging & measurements of features on the order of a few nano-meters, the vertical (height) resolution is ~ 1 Å.
- In-situ Scanning in Different environment including ambient, UHV, under liquid, high temperature
- This is a non destructive scanning procedure
- Material Properties characterization can be done using AFM including surface force measurement, Topography, Adhesion, Visco-elasticity, Hardness, friction, Magnetic Domains etc.
- Apart from imaging the surface, AFM can also be used to measure the extent of the interaction forces.
- Unlike STM, (which is the ancestor of the AFM) which can only image conducting surfaces, AFM can be used on any kind of surfaces.
- Unlike traditional microscopes, scanned-probe systems do not use lenses, so the size of the probe rather than diffraction effects generally limit their resolution.

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Some Salient Points in Simple Words

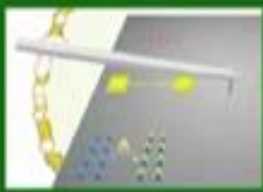
While you "see" in other microscopes, you actually "touch" the sample in an AFM!

The simplest (and probably the biggest difference!): You get "DIRECT" information about the topography (X, Y and Z directions).

Not only one gets to look at but one can "DO" things at the surface.

Couple of good references:

1. *Advanced Materials*, 17, 1821, 2005.
2. *Mechatronics*, 14, 907, 2004.
3. *Chem. Rev. Volume 97, Issue 4.*
4. *Angew. Chem. Int. Ed.* 43, 248, 2004.



There are certain advantages of the AFM, but salient points in simple words that I would like to highlight are unlike other microscope AFM does not take a picture. It actually goes there is a probe. Firstly, that goes to the surface and does some measurement and based on that measurement. In fact, you generate or generate and artificially later ender picture. So in fact the way you visualize and AFM is completely different than any other conventional microscope.

So, in very simple terms while you see in other microscopes, you actually touch the sample and therefore you get a much better idea about the surface using an AFM and since you are touching just the way you can touch something and you can feel whether it is hard, soft etcetera. The AFM tip can also perform those functions for you and can give lot of information.

So, thank you very much I hope you have some basic idea about that atomic force microscope now and those of few who want to do further reading on this all most welcome there is a huge amount of literature that is available in an AFM and if you really take of nano technology as a serious carrier option in research, I am sure you will come across the AFM again and again. And these lectures when you are few will then remember sought of give you first gleam of the utility of an atomic force microscope.

Thank you very much.