Scanning Probe Microscopy

3rd year Physics Laboratories

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Contents

1 Introduction

The techniques used in Scanning Probe Microscopy (SPM) allow for the highest resolution 3D measurements of surface properties of materials to date. With the right vibration isolation and vacuum system a vertical resolution of 0.01 Å and horizontal resolution of roughly 1 Åcan be obtained. Compared to Transmission Electron Spectroscopy (TEM) with a resolution of 0.8 Å and Auger Electron Spectroscopy (AES) with a 30 Å vertical resolution and 100 Å horizontal resolution you can appreciate why SPM is routinely used by scientists worldwide.

SPM incorporates atomic force microscopy (AFM), which is sensitive to inter-atomic Van de Waal forces as well as electromagnetic forces to image a sample, and scanning tunnelling microscopy (STM) which utilises electron tunnelling to image the surface topology of materials. There are many imaging modes used to obtain better images for certain samples, however only the basic modes will be explored here.

In this experiment you will examine the surface topology and magnetic properties of a hard disk and other materials using both AFM and MFM modes of the NT-MDT SMENA scanning probe microscope. Furthermore you will examine several imaging modes of the AFM. The aim of this laboratory session is to give you an understanding of the forces involved in AFM and tunnelling phenomena as well as how SPM allows for the imaging of materials at high resolution in atmosphere and at room temperature.

1.1 A note about your findings in this experiment

This experiment is far more **qualitative** than anything you would have done in other third year labs, and maybe even second year. This means, with the exception of a couple of sections, you'll be talking about **comparisons** of data you've taken.

It is therefore extremely important to take good notes about any changes to parameters you make between scans. Note all of the variables for each scan, if you forget if you've changed a variable a meaningful comparison is impossible.

Strongly consider mapping out a table of changes you want to make before you start, or at least a few different starting points, then work out a table from there.

2 Atomic Force Microscopy

2.1 The Van de Waal Force

The atomic force between neutral atoms is called the Van de Waal force and the AFM is sensitive to this force. An expression for it can be obtained from the Lennard-Jones potential. This potential is typically used to represent the interaction between two atoms which are not chemically bonded to one another:

$$U(z) = 4\epsilon\{(\frac{\sigma}{z})^{12} - (\frac{\sigma}{z})^{6}\} \tag{1}$$

where for a heavy element like Xe, $\epsilon = 0.02$ eV and $\sigma = 0.4$ nm.

Question 1 *Calculate an expression for the Van de Waal force for Xe given that $F = -\nabla U$ and plot it. You will return to this plot later so make sure it's big and clear.*

2.2 Contact Mode - Fundamental Components

In its most basic form the AFM consists of the cantilever (1), a laser (2), a multi-section photo-diode (3) and a movement system (4). These are shown in figure 1.

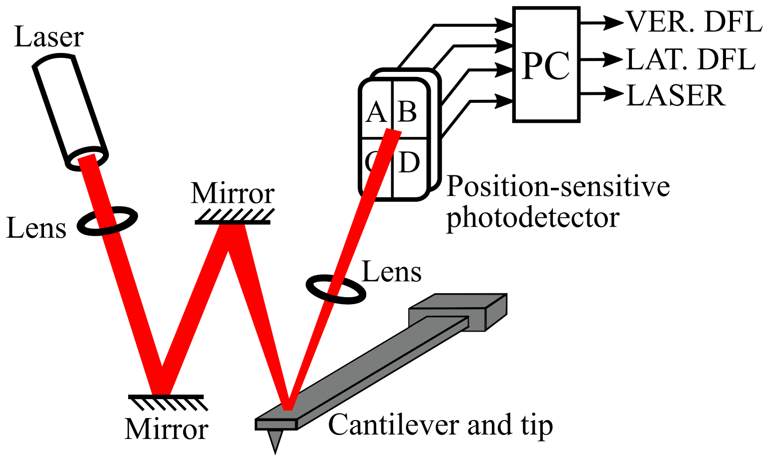

Figure 1: The basic components of an atomic force microscope.

When the cantilever of the AFM is brought close to the surface of a sample it responds to the Van de Waal forces by bending. The laser light is reflected off the back of the cantilever and is analysed with the multi-section photo-diode by comparing the photo-current in the different sections of the photo-diode. In doing this it is possible to determine the deflection of the cantilever. By scanning the cantilever across a sample and monitoring the deflection in this way it is possible to map the forces and the topography across the sample.

Question 2 *Re-examine the plot you made for the Van de Waal force, at what distances would we obtain the greatest resolution? Why is the resolution greatest there? Indicate on your plot from the region where the contact mode operates. Make sure you are clear on what 'contact' actually means.*

2.3 Cantilever Basics

The cantilever typically has length of about a hundred microns and a tip height of around tens of microns, see figure 2. The cantilever is flexible and its behaviour can be described with Hooke's Law:

$$F = -k\Delta z \tag{2}$$

where k is the spring constant for the cantilever. The action of scanning the cantilever tip across or near the sample surface causes the sample to experience downward forces which can and does result in distortion of the image and damage to the sample. To counter or lower the effect of the sample being damaged or deformed during scanning it is desirable to use a cantilever that is highly flexible. To estimate the required spring constant that will not damage the sample consider the resonant frequency and mass of the typical atom, approximately 10^{13} Hz and 10^{-25} kg, respectively. If you substitute these values into equation 5 then a typical atom has an effective spring constant of the order 10 Nm^{-1}. Thus manufactured cantilevers should have tips with spring constants lower than this to minimise damage to the sample.

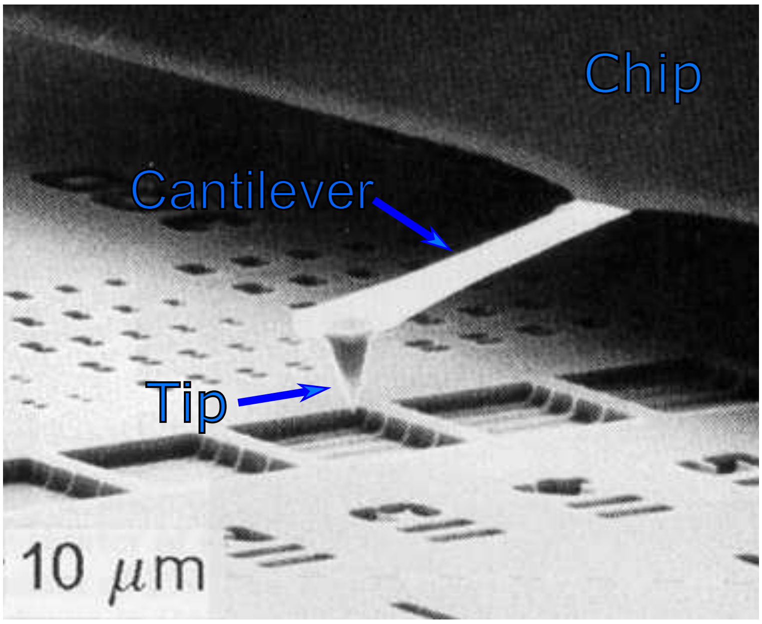

Figure 2: A scanning electron microscope image of a AFM cantilever.

Question 3 *Do we need a small or large spring constant for good sensitivity? Think about the motion of the cantilever relative to the surface.*

Question 4 *Texts quote that a vertical resolution of 0.01 nm is needed to image single atoms on the surface of a material. Given that all atoms are larger than a hydrogen atom why does the resolution need to be less than a Bohr radius? Think about how chemical bonds work.*

Question 5 *Assume the 1D thermal vibration is described by the following equation:*

$$\frac{1}{2}k < \Delta z^2 >= \frac{1}{2}k_B T \qquad (3)$$

where $\sqrt{< \Delta z^2 >}$ *is the RMS vibration amplitude and* k_b *is Boltzmann's constant.*

Use the Manufacturers specifications for both the contact and non-contact spring constants, to determine the theoretical limit to the best resolution obtainable at room temperature. (Further on in the notes you will be required to calculate the non-contact mode spring constant and you should recalculate the theoretical limit of the resolution at room temperature.)

Question 6 *Would a large or small spring constant be better for reducing the amplitude of thermal vibrations? This is kind of a partner question with the one about good sensitivity. Maybe it would be good to have a table listing pros and cons of both large and small spring constants.*

2.4 The Tip of the SPM Cantilever

2.4.1 Design of SPM Cantilever Tips

The cantilever tips are usually made from either Si or silicon nitride (Si_3N_4). Silicon nitride cantilevers can generally be manufactured more thinly than silicon cantilevers and as such are more flexible. The drawback with silicon nitride cantilevers however is the lower quality of the cantilever tips compared to the silicon cantilevers. Typically the contact mode cantilevers are triangular-shaped so as to minimise lateral distortion or deflection. [1] Non-contact mode cantilevers, on the other hand, are rectangular. Although the value for the spring constant of the cantilever is important in achieving good sensitivity, there are several other characteristics of the cantilever that also have a significant effect on the acquired images.

Question 7 *Consider two types of tips on the end of the cantilever, a hemispherical and a triangular shaped tip. On a diagram draw the resultant path of these two tips as they scan across a sharp spike protruding from a flat surface, similar to figure 4. You will find that the path of the tips will trace out the inverse of the tip shape. If the size of these tips increased or decreased then the resultant image itself would also increase or decrease in size. Clearly, the resultant image acquired from the SPM is a convolution of both the shape and size of the tip and also of the surface structures themselves.*

Make sure you understand this effect moving forward.

2.4.2 The Tip Broadening Effect

The finite dimension and size of the cantilever tip has an effect on the appearance and size of the images. Tip broadening will occur when the radius of curvature of the tip is of a similar or larger size than the feature on the surface. Figure 3 shows that as the tip scans across the surface feature, in this case a sphere, the sides of the tip make contact before the apex of the tip causing the tip to respond. The amount of broadening that occurs is dependent on both the dimensions of the tip and of the object. In the case of a hemispherical tip with a curvature of radius, R_t scanning across a sphere of radius R_s on the surface of a substrate the apparent width of the sphere will be equal to $4\sqrt{R_t R_s}$ where $R_s < R_t$.

Question 8 *Derive the above relationship describing the tip broadening effect due to the finite dimensions of both the tip and the object. Calculate the apparent width of a spherical object with a diameter of 60 nm for the contact and non-contact mode cantilevers, using the manufacturer's specifications for the radius of curvature of each tip. Note that the broadening effect becomes an important factor when resolving sub-micron and atomic scale dimensions.*

This should also give you some idea of how degraded tips give much lower resolution images.

[1] However, 'recently' John Sader from the University of Melbourne's Maths Department has used established mechanical principles to prove that the V shaped cantilever inadvertently degrades the performance of the instrument, and is actually more sensitive to lateral distortion than a rectangular cantilever.

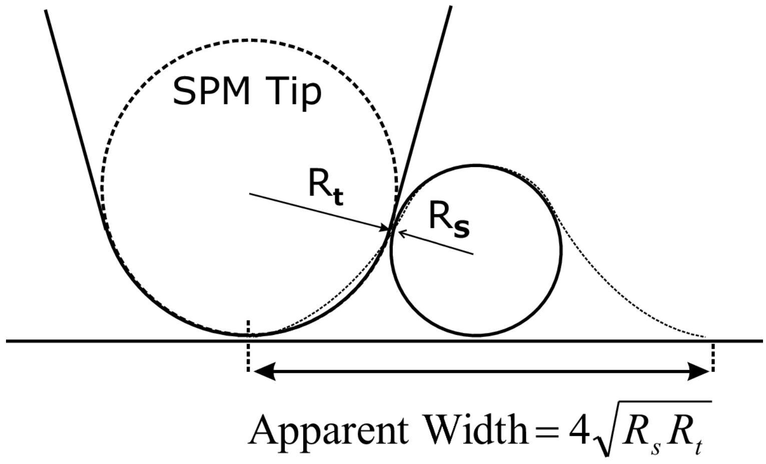

Figure 3: The hemispherical SPM tip is scanned across the surface of a sample from left to right, and the dotted lines represent the actual path of the tip as it comes into contact with a surface object spherical in shape.

2.4.3 Rayleigh Criterion for the Lateral Resolution of the AFM

There is no well accepted definition for determining the spatial resolution of the SPM. In fact, each surface feature must have the resolution independently determined. Above you saw the example of the relative sizes of the tip and the object determining the resolution, in the following example you will see how the height difference between 2 surface features affects the lateral resolution. It can be shown that the minimum distance, d, that you can laterally resolve two sharp spikes of unequal height, Δh on a flat surface for a given noise level of Δz, and for a hemispherical tip of radius, R is:

$$d = \sqrt{2R}\,(\sqrt{\Delta z} + \sqrt{\Delta z + \Delta h}\,) \quad (4)$$

for d $> \sqrt{2R\Delta h}$. Note that equation 4 assumes that the two surface features - the sharp spikes in figure 4 do not deform as the tip is scanned across them, **in practice this is not the case**.

Question 9 *Referring to figure 4, derive the above expression for the minimum resolvable distance, d and show that the above equation holds when d* $> \sqrt{2R\Delta h}$. *Use your previous calculations for the RMS vibration amplitude and the manufacturer specifications for the Contact tip radius, calculate the minimum distance, d, for 2 spikes of equal height. Investigate what happens to the minimum resolution, d when there is a difference in height of 2 nm, 5 nm, 10 nm and 20 nm.*

Note that this question is quite difficult - don't stress too much if you can't answer it.

When you have commenced scans using the SPM, further on in this and other sections, you should repeat these calculations using the experimentally determined values for the noise levels.

Clearly, one can conceive of more complicated surface topographies which would further decrease the achievable resolution of the SPM. The above exercises serve as a guide to your scanning system and of the operating environment and its achievable resolution.

Question 10 *To further complicate matters, the tip over time will degrade and change shape. Comment on how the resultant images may be affected. Discuss these implications and how you might*

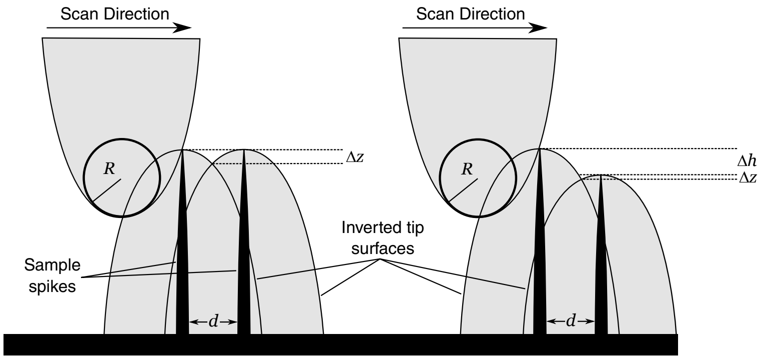

Figure 4: Two sets of spikes protruding from the sample surface. In case (a) the spikes are of equal height, while in case (b) the spikes are of a differing height, Δh. In both cases the hemispherical AFM tip is scanned across the sample spikes and traces out the path of the inverse shape of the tip.[15]

detect and monitor tip degradation.

To investigate these matters more thoroughly it is suggested to refer to the references at the end of the lab notes and go to the website of the SPM and the cantilever's manufacturer.

What is your demonstrator's experience with degraded tips? Ask them how much of a difference being ***careful*** *with your scans will make.*

2.5 Types of force microscopy

Note that throughout this experiment there are various forms of force microscopy, not only contact and non-contact, but types within these as well. You'll come across mentions of 'constant height' and 'constant force' in contact mode. Make sure you understand the differences between these, there should be good descriptions in the SMENA manual. It can be a further variable to consider when taking your scans, too.

2.5.1 Constant-force feedback system

In order to protect the cantilever and scanning head from damage during landing of the cantilever on the sample and to control the height at which the scanning is performed a feedback system is used. This feedback (FB) system maintains a constant force on the cantilever by adjusting the *absolute* height of the cantilever by extending or retracting the scanning head with the piezoelectric movers. This attempts to maintain a constant *relative* height from the tip to the sample surface. It is by monitoring the extension of the cantilever that the topology of the surface is mapped.

This is particularly important when a landing of the cantilever is made since there is a danger of crashing the tip into the sample. With the SMENA SPM the approach is performed manually with a screw adjustable back leg. Once the cantilever begins to experience a pre-set force during landing the piezoelectric scanner will retract the cantilever away from the sample to compensate.

2.5.2 Constant-height feedback system

Question 11 *Look in the SMENA manual/online and determine the exact differences between constant-force and constant-height modes. How does the cantilever behave in both modes? Draw a diagram for each.*

2.5.3 Other feedback modes

Question 12 *Select one other mode not mentioned in these notes and try to describe its operation. Again, a diagram will be useful.*

2.5.4 Sample A - Single crystal silicon chip amorphised by ion implantation (a-Si)

High energy ion implantation of silicon wafers through masks has many applications in advanced microelectronic device fabrication [1]. In some cases the implanted silicon may be rendered amorphous. Amorphous silicon (a-Si) lacks the long range order of the crystalline phase (c-Si). The operation of most devices will not tolerate crystal faults in their active volume, therefore silicon amorphised by implantation is re-crystallised by thermal annealing at moderate temperatures to recover the attractive structural and electronic properties of the crystalline phase. The incorporation of defects during annealing or incomplete re-crystallisation may compromise the use of amorphising implants in device manufacture and has therefore been studied.

In this sample multi-energy ion implantation was performed through a grid like mask with micron sized holes rendering the implanted regions amorphous. The a-Si is constrained in five directions by the surrounding crystalline silicon but not in the sixth (the surface). Due to a-Si having a lower density than c-Si and its ability to plastically deform it expands out of the surface giving rise to 'hillocks' that can be analysed with the AFM.

It is instructive to use a surface topology that is periodic and relatively featureless, hence the single crystal Si sample with amorphous hillocks. The main reason is that interpretation of images and the determination of any observed artifacts due to the variation of operating parameters etc can be more easily identified.

The experiment in the main is very open ended, and it is up to you as the experimenter to make sense of all these effects and observations. How can one be sure that images acquired from the SPM are accurate? To what degree can you rely on the measurements by the SPM of a surface feature? Does varying the scanning parameters affect not only the quality of the images but the observed dimensions? What is the appropriate mode, spring constant etc for getting an image? What are the important scanning parameters?

Systematic and methodical thought processes are the key here, a haphazard exploration and discussion will be obvious in your report. When discussing your results it is suggested that if you have nothing concrete to say about an image then leave it out, this experiment will generate lots of often meaningless, pretty and ugly pictures so it is how you differentiate between them that will make the difference. You want to be short and to the point with reference to images and highlighting a particular effect or feature. Make it very clear what scanning parameters were used.

How you present the data is very important here, if you are comparing and contrasting between the two scan modes then **tables are a good idea**. Summarising your data and findings will help you to assess what it is you can be confident you have determined. Secondly, consolidate regularly and write up as you go because this experiment cannot be performed to an adequate degree if there is a lack of re-evaluation every few hours.

2.5.5 Setting up the laser and photodiode for contact mode imaging

Ask your demonstrator to introduce you to the components of the SMENA.

Question 13 *Use your own words to describe the SMENA system and the path the light takes from the laser to the cantilever and to the photodiode. A simple diagram relating to what you see in the microscope will be useful.*

Question 14 *Which of the cantilevers present would be most suitable for contact mode imaging and why?*

The following outlines the method for taking a scan. Be advised that the software might have changed slightly and the directions might be a bit off-track or more obtuse than detailed here. Any mention of the 'advanced' tab should be the button with the set of squares to the top right of the screen.

1. Check with your demonstrator and **ask them to install the cantilever** in the SMENA scanning head.
2. Slot sample A in the base. **Make sure the back leg of the SMENA is fully extended before replacing the SMENA head on the base**.
3. To start the SMENA control program double-click the shortcut on the Windows desktop labled 'SMENA'.
4. Above the main window is a set of buttons. Place the mouse over the [operation] button and then click on the [Scanning...] button that pops up. Alternatively click on the folder in the Scan section of the left-hand toolbar.
5. This brings up the scanning window. Click on the [SPM] tab if not already selected. Do not be intimidated by all the controls present. Firstly in the top left hand corner is a schematic of the cantilever, sample, laser and photodiode.
6. Note that the sample is electrically grounded. The lines indicate electrical connections. Clicking on any of the squares around the lines will re-connect the line to that square. Leave the sample grounded.
7. Turn on the laser by clicking on the 5V power supply above the laser. Click on the multisection photodiode to bring up a monitor for its output.
8. The top two displays in the monitor show the difference in the detected light in opposing sections of the photodiode in nA. [VER DFL] indicates the signal as a result of the vertical deflection of the cantilever and [LAT DFL] refers to the signal due to the lateral deflection or twisting of the cantilever. [LASER] refers to the total detected intensity of the reflected laser spot.
9. Use the microscope and lamp provided to look at the cantilever and laser spot through the mirror hole (see figure 5) . Adjust the laser spot with the two left hand knobs on the SMENA head (1

and 2 in figure 6) until it appears on the tip of the cantilever. Further adjust the laser position until the laser signal is maximised, the signal should be greater than 20. If you can't get a signal larger than this you may need to adjust the photodiode position (see below). Check that the laser really is on the cantilever.

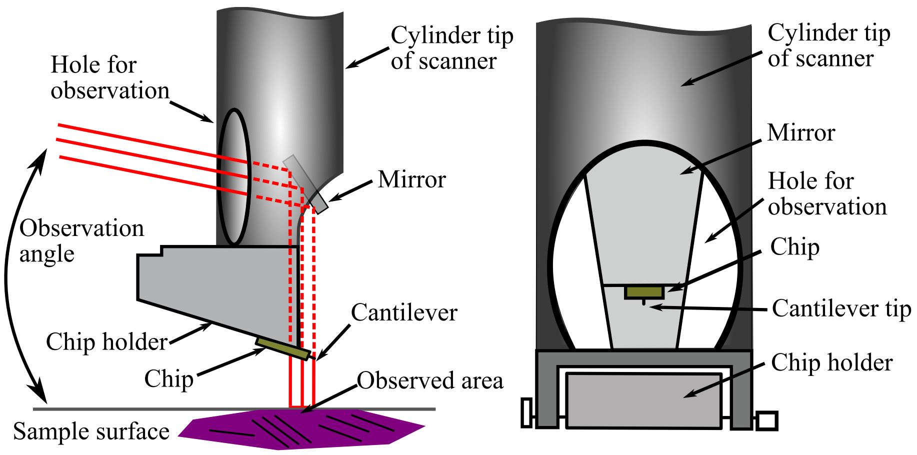

Figure 5: The mirror that allows the cantilever to be seen.

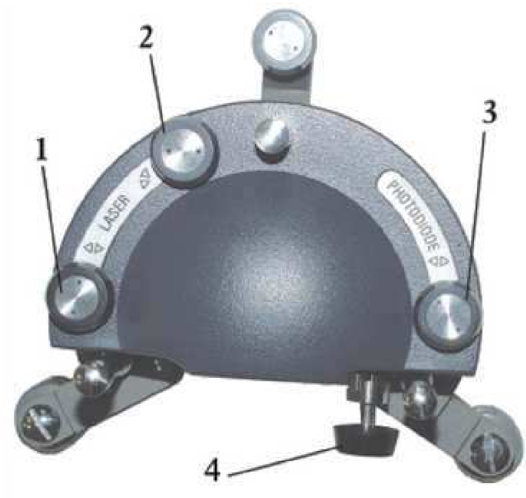

Figure 6: Control knobs on the SMENA.

10. Since the cantilever should still be far from the surface it should not be bent or flexed in anyway. Therefore, adjust the photodiode position (3 and 4 in figure 6) so that the signals for deflection are both zero. If you can't get the signal to zero you are probably at the opposite end of range from the correct position.

11. N.B. You can exit any open window by pressing <Esc> or by clicking the right mouse button anywhere on the screen.

Question 15 *What do all of the numbers on the laser alignment screen mean? You are told to reach specific values for each - do you think all of these are important or could one be left to drift?*

2.5.6 Positioning of the cantilever over the sample

The scanning head of the SMENA has a maximum X-Y travel of about $50\times50\,\mu m^2$ so it is necessary to roughly position the cantilever over the region that you are interested in. This is acheived by slightly lowering the tip with the back leg screw to 2 or 3 mm from the sample surface and adjusting the sample position with the two micrometers on the SMENA base. N.B. The Y position is best adjusted by looking at the tip from the side and the X from the front at low angles by looking at the chip that the cantilever is mounted on.

Question 16 *Draw an image of what you see on the sample with your eyes. How accurately do you think you can place the sample relative to the* $50\times50\,\mu m^2$ *area of implantation?*

Make sure you know where the cantilever actually is before proceeding. If the cantilever is overhanging the sample when it is lowered the feedback wont kick in and the scanning head will crash into the sample. This can damage both the scanning head and the sample.

2.5.7 Landing the cantilever in contact mode

To begin landing the cantilever in contact mode **open the [scanning] window under the [SPM] tag** to adjust the feedback. The feedback control system appears in the top right of the window.

Click on the [FB In] button and change the mode to [DFL] or deflection mode. This is so that the feedback operates from the deflection of the cantilever.

The **[Sign] should positive be [+]**.

Set the [Spoint] to 1.5 (nA). This means that the feedback system will attempt to keep the probe at a deflection corresponding to 1.5 in the [VER DFL] on the photodiode.

Set the [FBGain] to about 1.5.

Underneath the feedback controls are the controls for the probe. **Change the connection from [Probe] to [Off]** to disable the oscillation of the cantilever.

Close the circuit labelled [I] to start the FB system. Since the cantilever is so far away from the sample the FB system will extend the cantilever as far as possible (3 μm). You can see this from the green bar on the top toolbar.

To successfully land the cantilever you will need to monitor the feedback, which indicates how much feedback is required to maintain a constant force and therefore provides information on the forces experienced by the cantilever.

We can monitor the feedback with the upper multi-function display (MFD). The display mode is indicated by a button on the left of the MFD toolbar, it should have automatically switched over to feedback [FB] mode. **Press [Go] in the MFD toolbar** to start monitoring FB. You can shift the axis scale by left-clicking over it and moving the mouse or zoom in and out by pressing the shift-key while left-clicking over it and moving the mouse. **Change the y-axis range to [0,2] nA and the x-axis range to [0,8] seconds**.

Manually lower the scanning head so that it is visually about 1 mm above the surface of the sample.

Continue to slowly lower the scanning head over the sample while keeping a close eye on the FB. A 10^o turn of the back screw moves the tip about 3.5 μm. **You will notice a jump in the feedback when the cantilever comes close to the sample, stop immediately**. Adjust the back leg screw until the green bar reaches half-way, i.e. the piezo is extended by half of it's maximum. If you go too far and the piezo has completely retracted simply raise the tip by screwing the back leg **clockwise**. Congratulations you've just landed a cantilever within a couple of microns from the sample.

Question 17 *Why does the cantilever suddenly 'drop' onto the surface when it is near? (Hint: Re-examine your graph for the Van de Waal force.)*

Next you will need to adjust the feedback gain in the [scanning]/[spm] window. The feedback gain controls the speed and rate at which the system attempts to compensate for changes in the force experienced by the cantilever. If it is too small then it will not change fast enough to compensate for small features on a sample and the cantilever may crash into a tall structure or provide false data for structures. If it is too large however it will overcompensate and make the cantilever oscillate. Monitor the FB signal while you increase the FB Gain. Note down the value at which the FB begins to oscillate and then set the FB gain to be half that value.

Place the bell-jar cover over the SMENA head. **ALWAYS USE TWO HANDS WHEN MOVING THIS BELL JAR.**

2.5.8 Scanning Sample A

1. The scanning parameters that you will need to set can be found in the [Scanning] window under the [Scan] tab. The Signal should be on **Height mode** and the Dir. can be left as is.
2. Begin with a low resolution large area scan by setting the number of points to scan ([NX] x [NY]) at 256 $\times$ 256 and set the step size as large as possible.
3. Next you'll want to adjust the scan velocity to about 100 μm/s (the units can be changed by clicking and holding the units tab).
4. Put the Subtract mode on [1st slope] if you so desire. This will subtract the average slope of the sample off the image to account for any long range tilt in the sample. This can be set after the scan as well.
5. Start the scan by pressing [RUN]. Avoid bumping tables or making any other unnecessary vibrations anywhere in the immediate vicinity.
6. The bottom MFD will show you the 2D profile of the last line scanned.
7. During scanning you can normalise the contrast by pressing the space bar. This will maximise the contrast for what has already been scanned.
8. You can also readjust various parameters during the scan using the arrow keys or cancel a scan at any time by pressing <Esc>.
9. You may notice that there are 'shadows' in your scans. This is an indication that your feedback gain may be too small. You may also notice that after a large surface feature that there are spots after the scan, this is an indication that the cantilever is oscillating (out of phase with the

oscillation we put on it) as a result of too large a feedback. Adjust accordingly and note this down in your log book.

10. Once the scan is completed you can re-adjust the contrast by use of the magnifying glass over color gradient button in the left-hand toolbar. After clicking on this you can select a region on the scan that you want to normalise the contrast over.

11. If you didn't choose to subtract the average slope off the sample then you can do this now with the [Sub] button in the left-hand toolbar.

12. By clicking and holding the triangle on the XYZ button on the left hand toolbar you can change this to a XY button and use this to obtain a profile of the sample along a line that you specify by clicking two points on the image. There are several other features you may want to explore.

13. If you can't see the 'hillocks' you may need to retract the cantilever and move it to another part of the sample then land it again.

    Question 18 *Try out the various curve subtractions. What can you glean from this information about the surrounding environment, or maybe the equipment itself?*

    Question 19 *What is the smallest resolvable feature that you can see? That is, look for the smallest speck of dust. What are its dimensions?*

    Question 20 *What are the dimensions of the hillocks? What is the separation between hillocks?*

    Question 21 *Amorphous silicon is 1.8 % less dense than crystalline silicon. The a-Si exists in the hillock and a region directly underneath the hillock. If the a-Si in the hillock could completely fit into the region taken up by the a-Si below the hillock if it was all c-Si how deep must this region below the hillock be? As a side note, this depth of a-Si is equivalent to the end of range depth of implanted ions.*

    Question 22 *Estimate the volume of a hillock. Include errors.*

    Question 23 *Rather than surface features, how can you tell what is an artifact due to noise, or insufficient feedback? What is the noise level in Z?*

14. If you can't see the 'hillocks' you may need to retract the cantilever and move it to another part of the sample then land it again.

15. Zoom into an area and perform a higher resolution scan by clicking on the [AREA] button and then the [Relative] button. Readjust the velocity to give a scan rate of 1 Hz.

16. Note that previous images can be accessed from the left triangle on the bottom of the main program window.

17. Save the settings and images you take by saving a data set as a .MDT file that can be re-opened at a later stage. To save click on the [FILE] button and then the [Save...] button. Images can also be separately saved by selecting [FILE] [Export] [SPM Window].

18. Once you've finished scanning retract the probe from the surface by **turning off the feedback** and then **screwing the back leg screw clockwise**. If this is incorrectly done the cantilever and scanning head can be damaged.

19. Turn off the laser.

Summarise briefly your findings before considering how these might compare to non-contact scans. Do you expect similar results, or will there be different factors to alter?

3 Non-Contact Mode

In the last section we saw that to obtain maximum sensitivity for contact mode imaging we need to bring the cantilever tip close to the sample. In practice however this greatly increases the risk of bringing the tip too close and ‘crashing it’. Furthermore even if the tip doesn’t get damaged the surface of certain materials can be irreversibly scratched while imaging. So with contact mode there is a compromise between image resolution and practical considerations. Non-contact mode (NC-mode) however enables us to image a sample from a greater distance and yet maintain good resolution.

Non-contact mode is also known as AC-mode as the cantilever is now attached to a metal that can be oscillated courtesy of a bimorph ceramic. This bimorph oscillator consists of a sandwich of two piezo electric polycrystals.

Question 24 *Re-examine the plot for the Van der Waal force and indicate the region in which the non-contact mode operates.*

Question 25 *Considering non-contact mode shouldn’t damage the cantilever while maintaining good resolution, what are its disadvantages over contact mode? i.e.: Is contact mode still used in SPM analysis? Why?*

The cantilever has a frequency dependent oscillation amplitude (see figure 7) with a resonant frequency given by:

$$\omega_o = \sqrt{\frac{k}{m}} \tag{5}$$

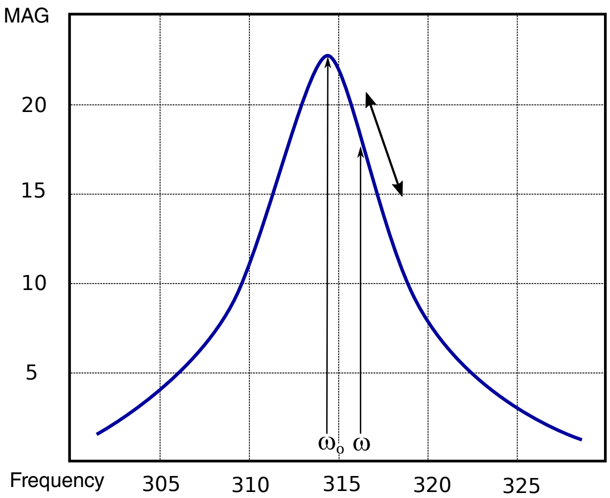

Figure 7: The frequency response of a NC-mode cantilever.

Most thermal and mechanical vibrations are low frequency so if we use a high oscillation frequency we can avoid this type of noise. From equation 5 we need a large spring constant for the resonant frequency to be large.

To utilise this oscillation to image the sample though we consider the spring constant to change in the presence of a force gradient, dF_z/dz. This occurs according to:

$$k' = k - \frac{dF_z}{dz} \tag{6}$$

Substituting this into equation 5 we obtain:

$$\omega' = \omega_o \sqrt{(1 - \frac{1}{k}\frac{dF_z}{dz})} \tag{7}$$

For small force gradients we have:

$$\omega' \approx \omega_o (1 - \frac{1}{2k}\frac{dF_z}{dz}) \tag{8}$$

This gives a total shift of:

$$\Delta\omega \approx -\frac{1}{2k}\frac{dF_z}{dz} \tag{9}$$

So in this way we can extract the force from the response of the cantilever and hence map the topology of the sample.

Note that we still need a small spring constant to have a large detectable change in frequency, meaning that there is a trade off between resolution and the avoidance noise.

Question 26 *Consider a surface shaped like a step function and another shaped like a triangular saw tooth wave. How would you expect the resonant frequency to change as we scan across it? Sketch the response on a change in frequency verses displacement graph as well as a frequency verses displacement graph. What do you notice?*

Question 27 *In practice the AFM operates by measuring the change in amplitude rather than directly measuring the change in frequency (this is magnitude slope detection). Re-examining figure 7, at what oscillation frequency would you expect to give the best resolution?*

3.1 Setting Up the Laser and Photodiode for Non-Contact Mode Imaging

Question 28 *Which cantilever is most suitable for non-contact mode AFM?*

Check with your demonstrator and ask them to install it for you. Make sure the back leg of the SMENA is fully extended before replacing the SMENA head on the base. You will need to access a larger set of functions now so instead of opening the scanning window select the button in the top tool bar with a box on it.

Set up the laser and photodiode as for contact mode.

3.2 Setting Up the Cantilever Oscillation

Change the switch for the line that connects the cantilever substrate to the electronics from [OFF] to the [Probe] position. This will allow the cantilever to be oscillated.

You will need to set up the Lock-In amplifier to successfully detect the out of phase changes in the cantilever. i.e. Since the cantilever is being oscillated we use a Lock-In amplifier to monitor any other changes to it that are not in phase with this oscillation.

Switch the lock in to Hi freq mode since the cantilever resonance should be greater than 50 kHz. Also switch the imput to the DFL position and the harmonic [Har] value to 1.

You will need to determine the resonant frequency of the cantilever now. On either side of where [$< F <$] is marked are the minimum and maximum frequencies that you must set to scan through. Adjust this to be between 0 and 500 kHz. Set the oscillation amplitude to 0.2 V and the phase to 0. Press [F-SCAN] to begin the scan. The lower MFD will display the mag. of the cantilever as a function of oscillation frequency. Repeat for smaller frequency intervals to get an accurate value for the resonant frequency. Set the resonant frequency by clicking on the [kHz] button.

Adjust the gain of the lock-in so that the resonant magnitude of the cantilever is 10-15 nA.

3.3 Landing the Cantilever in Non-Contact Mode

The feedback should be on [MAG] mode since we will use the change in magnitude of the cantilever oscillation to detect the force.

Question 29 *How is this possible if we only said that forces changes the frequency of the cantilever oscillation?*

Next set the set point [SPoint] to be half the oscillation magnitude of the cantilever. The oscillation magnitude of the cantilever will be indicated by the red outlined box just under the [FB In] box. If not then press [F-SCAN] again. The feedback system will attempt to maintain the cantilever at a height where the oscillation magnitude is at the [SPoint].

Question 30 *If we said that the greatest resolution can be obtained by setting the frequency off resonance why have we chosen to oscillate the cantilever on resonance?*

Close the circuit labelled [I] to start the FB system and proceed to lower the cantilever with the bag leg screw as for contact mode landing.

Next you will need to adjust the feedback gain in the [scanning]/[spm] window. The feedback gain controls the speed and rate at which the system attempts to compensate for changes in the force experienced by the cantilever. If it is too small then it will not change fast enough to compensate for small features on a sample and the cantilever may crash into a tall structure or provide false data for structures. If it is too large however it will overcompensate and make the cantilever oscillate. Monitor the FB signal while you increase the FB Gain. Note down the value at which the FB begins to oscillate and then set the FB gain to be about 0.5 that value.

3.4 Scanning Sample-A

Scan the sample as per contact mode to image the hillocks. If you can't see them you may need to retract the cantilever and move it to another part of the sample then land it again.

By this stage you should be investigating the differences between contact mode and non-contact mode. Do they give different measurements for scans of the same region? How do the noise levels differ? How are they sensitive to differing frequency ranges? Do the two scan modes respond differently to varying the scanning parameters? What are the important scan parameters? etcetera.

Take your time to think about what it is you want to explore, and how you want to compare and contrast between the two modes. It is how you answer and investigate these questions and more that you will be assessed on.

You are by all means encouraged to bring in samples of interest but please discuss this with your demonstrator beforehand.

3.5 Various Calculations for the Non-Contact Cantilever

3.5.1 Calculating the Quality Factor of the Cantilever

The Quality Factor determines the amplitude of vibration of the cantilever. Mechanical oscillators with a high Q value will have low friction, i.e. the fractional energy lost per period for a damped oscillator will be very small. Lightly damped oscillators will therefore have a large Q while the heavily damped oscillators will have the converse. You may have noticed that the quality factor of the cantilever is high, and you will have observed that it has a narrow resonance width. If the resonance is sharp a mechanical system will only respond when the driving frequency is equal to the resonant frequency, i.e. a high Q means a high frequency selectivity.

Question 31 *Experimentally determine the quality factor, Q of the non-contact cantilever:*

$$Q = \frac{\omega_o}{\Delta\omega} \tag{10}$$

where $\Delta\omega$ *represents the full bandwidth, at* $1/\sqrt{2}$ *of the maximum amplitude.*

It is necessary to point out that the curve in figure 7 is a steady-state curve, and only after a sufficient time will the vibration amplitude settle on a new steady-state value, ω'. The following equation can be used to calculate the response of the oscillating mechanical system in terms of a time-constant, τ which will limit the available bandwidth.

$$\tau = \frac{2Q}{\omega_o} \tag{11}$$

The following equation can be used to calculate the minimum detectable force gradient and therefore estimate the sensitivity of the non-contact mode:

$$\frac{dF_z}{dz} = \sqrt{\frac{2\,k\,k_B\,TB}{\omega_o Q < \Delta z_{osc}^2 >}} \tag{12}$$

where $< \Delta z_{osc}^2 >$ is the mean-square amplitude of the driven cantilever vibration, B is the measurement bandwidth, Q is the quality factor of the cantilever resonance, and $k_B T$ is the thermal energy at room temperature [16].

Clearly, the quality factor, Q restricts the bandwidth of the mechanical system. Equation 11 indicates that the behaviour of the system is such that a low Q will enable a faster response, allowing the cantilever to quickly respond to a sudden change in height across a sample, thereby allowing higher scanning velocities. The trade off is a low Q means that the minimum detectable force gradient increases causing the system sensitivity to be reduced. The converse is true with a high Q, where the sensitivity is greater at the expense of a reduced response by the cantilever.

Question 32 *Comment on the calculated value for the time-constant and how it may be used to determine an appropriate setting for the scanning velocity of the cantilever. In reference to equation 12 suggest further ways to improve the sensitivity of the system in the non-contact mode.*

3.5.2 Calculating the Spring Constant of the Cantilever

The following formula calculates the spring constant, k, of a cantilever and it is assumed that the bending is small enough to stay within the range of elasticity so that Hooke's Law is still applicable:

$$k = \frac{3EI}{l^3} \tag{13}$$

where E, I and l represent Young's Modulus, the area of moment of inertia, and the length of the cantilever respectively.

For the non-contact rectangular cantilever the area moment of inertia, I, is:

$$I = \frac{wt^3}{12} \tag{14}$$

where the variables w and t represent the width and thickness of the cantilever respectively.

Question 33 *Use the manufacturer's specifications for the dimensions of the cantilever, and the value of 169 GPa for the Young's modulus of Si to estimate the spring constant of the non-contact cantilever. Compare it to the Manufacturer's specifications, and comment on any discrepancies.*

3.5.3 Calculating the Effective Mass of the Cantilever

Realistically, the cantilever consists of a rectangular lever with a distributed mass, m_d and a tip on the end of the lever with a concentrated mass, m_c. This means that equation 5 can be rewritten as:

$$\omega_o = \sqrt{\frac{k}{m_{eff}}} \tag{15}$$

where m_{eff} represents the effective mass of the cantilever, and is equal to the sum of the distributed mass of the rectangular lever and of the concentrated mass of the tip. Thus to make the SPM insensitive to external vibrations it would be desirable to minimise the effective mass of the cantilever.

The Classical solution to a vibrating lever gives the fundamental frequency of vibration of a solid rectangular cantilever as:

$$\omega_o = \sqrt{\frac{Ewt^3}{4l^3(m_c + 0.24wtl\rho)}} \tag{16}$$

where the term in the brackets represent the effective mass of the cantilever, ρ represents the density of the cantilever material. If you were to assume that the mass of the lever is much greater than the mass of the tip then equation 16 becomes:

$$\omega_o \simeq \frac{t}{l^2}\sqrt{\frac{E}{\rho}} \tag{17}$$

Question 34 *Use equation 17 to verify your experimentally determined value for the resonant frequency of the non-contact cantilever. Calculate both the effective mass of the cantilever and of the tip (ρ(Si) = 2.33 gcm^{-3}, E(Si $< 100 >$) = 169 GPa).*

Question 35 *Given that tip degradation will have an impact on the scanned images, for the non-contact mode suggest a simple way to monitor any changes that may occur to the cantilever and tip.*

Typically the frequency range of the vibrations from a building lie somewhere between 10 to 100 Hz, and naturally these will be coupled in some form to the cantilever and have an affect on its oscillation and natural resonance. The frequency range of human speech is approximately from 100 to 8000 Hz and it too will have a noticeable affect on the cantilever's natural resonance. (In the non-contact mode try "talking" to the SPM, you should observe an significant increase in the noise level and a change in the cantilever oscillations.) To reasonably suppress the excitation of the cantilever oscillation the resonant frequency should be at least an order of magnitude greater than the highest frequencies of the vibrations that are problematic.

Question 36 *Using your estimated value for the spring constant of the cantilever from the previous section, calculate separately the upper limits of the effective mass of the vibrations due to the building and to human speech. Then estimate the desired upper limit for each case of the cantilevers effective mass. How does this compare to your estimate of m_{eff} of the cantilever? Make comments where any discrepancy may exist.*

4 Magnetic Force Microscopy

Please note the majority of your time here will be spent trying to get the conditions right for a magnetic scan. It is very important that you read the notes carefully and double-check all of the settings on the Nova software, a single button on the wrong setting will mean absolutely no magnetic signal.

Additionally, magnetising the tip can be troublesome. Your demonstrator should have some ideas about different methods.

4.1 The Magnetic Force

In MFM we utilise a cantilever with a tip coated with a magnetic material that has a permanent magnetic moment (μ). For a sufficiently sharp tip (nm radius) we can treat it as a dipole at a point. Now if we consider the potential that the dipole is in when in an external magnetic field we have:

$$U = -\vec{\mu}.\vec{B} \tag{18}$$

Question 37 *For the case of a general magnetic field ($\vec{B}$) and a vertical dipole (i.e. aligned in $\hat{z}$) determine an expression for the force on the dipole (i.e. the tip). You may need to use the following vector identity:*

$$\bigtriangledown(\vec{A}.\vec{B}) = (\vec{A}.\bigtriangledown)\vec{B} \; + \; (\vec{B}.\bigtriangledown)\vec{A} \; + \; \vec{A} \times (\bigtriangledown \times \vec{B}) \; + \; \vec{B} \times (\bigtriangledown \times \vec{A}) \tag{19}$$

Show that for the case where the magnetic field is also aligned in $\hat{z}$ we have:

$$\vec{F} = \mu_z \frac{\partial B_z}{\partial z} \hat{z} \tag{20}$$

Question 38 *Can a magnetic cantilever with a vertical dipole also image a sample that has magnetic regions horizontally aligned in the surface of the sample? Hint: Think about the magnetic field lines around a bar magnet.*

4.2 Basic Magnetic Force Microscopy

The fundamentals for contact mode MFM are identical to that of contact mode AFM except that there is an additional force that the cantilever responds to. However, if the cantilever responds to both the magnetic and Van de Waals force now how can the surface topology (which is what the Van de Waals force gives us) be separated with the magnetic topology of the sample? One method is to scan the sample with a non-magnetic tip to determine the surface topology as well as scanning with a magnetic tip so that the surface topology can be subtracted from the gross magnetic topology image. However this is impractical because when the cantilevers are changed over there may not be an accurate way to return to the same scan region as before. A much better method is to utilise the fact that the Van de Waals force is a strong but short range force and the magnetic force is a weaker but longer range force. So by scanning the sample at a short distance the cantilever should be mostly affected by the Van de Waals force and at a greater distance the magnetic force.

The technique that you will use will be constant height non-contact mode MFM. This means that you scan the sample in the exact same way as with NC-AFM except at a greater distance. In order to obtain a better image we scan at both small and large distances so as to seperate the magnetic component from the topological component. This is acheived by taking the difference between the two images or the ‘out of phase’ component.

4.3 Sample B - Hard Disk Drive (HDD) platter

Hard Disk Drives actually comprise of several ‘platters’ for data storage. These platters are typically made from an aluminium alloy [6] that is coated with a ferromagnetic thin film [5]. The platters need to be very smooth since the read/write heads fly over the platters at a height of tens of nanometers [4] and speeds of around 300 km/h [7].

Data bits are stored on circular tracks on both sides of the platters and there are often a thousand or more tracks on each side of a platter [8]. The number of tracks that a drive can store is characterised by the number of cylinders it has. Where a cylinder is characterised by a track position that cuts through all the platters of the HDD, this is used because the read/write heads for each side of the platters are joined together so that tracks on in cylinder can be read at the same time.

4.4 Setting up and landing for MFM

Keep the NC mode cantilever in place as it has a cobalt coating that is magnetic. However you will need to align the tip magnetisation in the vertical direction. **Remove the SMENA head and place upside down on the bench top. Take a bar magnet and bring one end within 1 or 2 mm of the cantilever tip and remove again always moving in a vertical motion. Repeat this movement roughly 15 times.**

Ask your demonstrator to assist in this, there are a few different methods.

Replace the SMENA head and set up the laser and photodiode as per NC mode.

Land the Cantilever as per NC mode and set up other parameters as per NC mode.

4.5 Scanning Sample B

The SMENA control software allows simultaneous imaging in AFM and MFM mode by making two passes during a scan. To set this up:

1. click open the [Scanning] window and **click on the [2nd pass] button** at the bottom. Then **click on the [=1st] button** to copy over the same parameters.
2. select the [==>] button and **click on the [ΔZ] button and enter 9000**. This specifies how far it should move the tip away from the AFM scan height.
3. Open the [SPM] tag while in 2nd pass mode and **turn off the feedback** by opening the circuit.

4. Open the [SCAN] tag and **set the [Signal] for [B:] as [Phase1]** then **set the direction [Dir.] as [=2=>]. Set the scan size to its maximum, number of points to 256x256** and **select an appropriate velocity**.
5. Close the [SCANNING] window and **press the double triangle button in the lower left of the main window**. This will allow you to see both the topography as measured with NC-AFM and the magnetic image measured.
6. Press RUN to scan.

Don't worry if you can't see anything in the phase1 image window, it may just be that the contrast needs to be adjusted. If you still see mostly the topography in the phase1 image this must be because the Van de Waals force is still dominant. You will need to further increase the height of scanning [ΔZ], try by 500. If you only see a homogeneous image you may be scanning a region that simply doesn't have any bits on it or it may be that you need to reduce the 2nd pass scan height.

Question 39 *How rough is the mirror smooth HDD platter?*

Question 40 *Can you tell if you are looking at bits on a track or at bits on separate tracks? Hint: Compare the MFM image to the topological image and think about how the surface would be polished.*

Question 41 *What is the size and separation of the bits in a track and the separation of the tracks? If multiple tracks can't be seen assume the smallest possible number.*

Question 42 *What is the capacity of the disk if it has a radius from 2 cm to 4.5 cm? In what year would you estimate this disk was manufactured?*

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