

UNIVERSITY OF WATERLOO
Physics 360/460 Experiment #2

ATOMIC FORCE MICROSCOPY

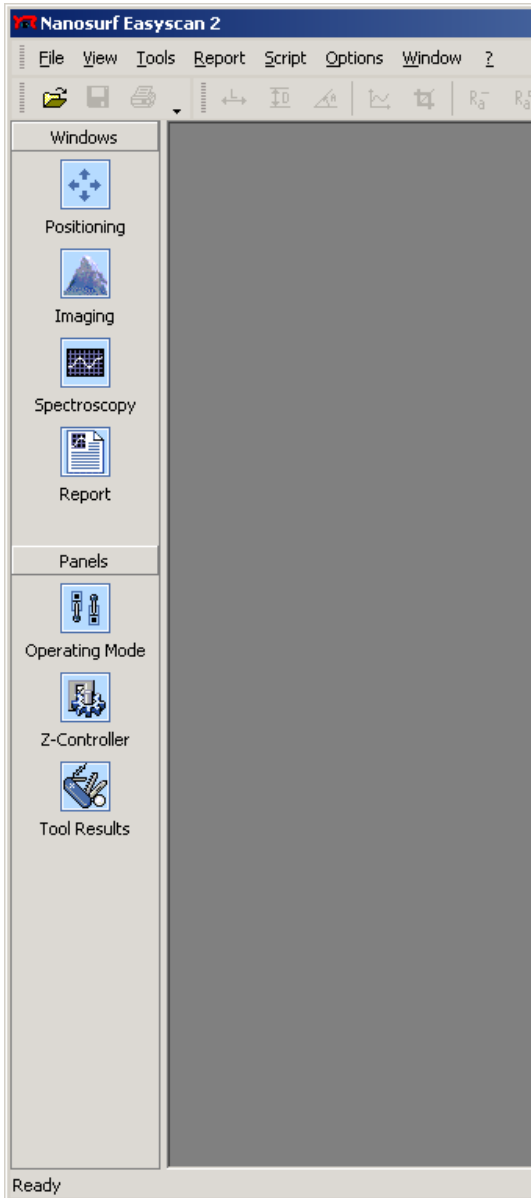
References: <http://virlab.virginia.edu/VL/home.htm>
(University of Virginia virtual lab. Click on the AFM link)

An atomic force microscope (AFM) is one member of the family of scanning probe microscopes. These microscopes do not have any optical focusing components. Instead, they use an atomically “sharp” probe tip that is scanned across the sample surface. The first example of a scanning probe microscope was the scanning tunneling microscope (STM), which was developed in the early 1980’s at the IBM research facility in Switzerland. The STM is somewhat limited however, as it can only be used on samples which are electrically conductive. The AFM is a variation of the STM and it can be used to map the surface of materials that are not electrically conductive. It has wide ranging applications in the fields of surface physics, biology and nanotechnology.

In this experiment, you will be using an Easyscan 2 AFM to make measurements of some technically relevant surfaces. The Easyscan AFM can operate in either static, or dynamic mode. In the static mode, the deflection of long probe tip (cantilever) is measured directly by a laser detection system, as the tip travels across the surface of the sample. The computer interprets the detected motion directly and produces the surface image of the sample from the movement of the tip. In the dynamic mode, a shorter probe tip (cantilever) is vibrated up and down very near the sample surface. It oscillates with a fixed amplitude at a frequency close to its resonant frequency. Repulsive forces acting on the tip by the sample will cause a decrease in the amplitude of oscillation. This motion is analyzed by the laser detection system and is used in a feedback loop to control the height of the tip above the sample. The computer maps out the surface features of the sample using the output from this feedback loop. The dynamic mode is useful for imaging rough and/or uneven surfaces without damaging the tip, or for imaging delicate samples which might be damaged by the sample – tip contact that normally occurs in static mode.

The cantilevers you will be using have been commercially prepared. The probe is typically formed by micro-machining of a piece of monolithic silicon. There are 2 types of tips to be used in this experiment. The SICONA tip is used in the static mode AFM, the ACLA tips are used in dynamic mode. These tips are pre-mounted on a cantilever that can be directly inserted into the AFM head. These tips are very expensive and are easily damaged if mishandled. They come stored in cases containing 20 or more tips, and are arranged on a bed of gel material to secure and protect them during storage and transport. See Appendix 1 for an overview of how to change the tip on the easyscan 2 AFM.

The Easyscan 2 software controls the most of the microscope functions. The main window is illustrated below. Along the left edge of the main window are the controls to bring up the operating windows or control panels.



- Select the “Positioning” icon, and the positioning window will open. It will take a minute for the video camera to initialize, after which you will observe a magnified view of the cantilever tip. You can switch between either the Top or Side view of the tip using the buttons on the lower left of the window. There are also controls for adjusting the tip illumination, and the brightness and contrast of the image.
- The “Imaging” icon will open the window where the actual image from the AFM will appear. This window will open automatically if you’ve initiated an automatic tip approach from the positioning window.
- Select the “Operating Mode” icon and the operating mode panel will appear. This panel allows you to set the type of cantilever you have mounted, and the type of AFM mode (static or dynamic) that you intend to be using.
- The “Tool results” panel will open when one of the many measurement tools are used on a completed scan. The controls that are displayed in this panel will vary depending on what tool is currently active in the imaging window.
- The Z-controller panel, and the Spectroscopy and Report windows are advanced functions of the AFM software and will not be used in this experiment.

In this first section you will use the static mode to image the surface of a 10 μ m calibration grid. This will acquaint you with the basics of how to obtain and analyze an AFM image.

First, remove the scan head from its storage container, and plug in the two connectors from the Easyscan control module. Use the screwdriver to secure the small screws on the

multi-pin connector. Make sure the connection to the video module (round plug) is properly oriented before you insert it and tighten it down.

Using the technique outlined in Appendix 1 of this experiment, load a SICONA tip into the scan head. Once this has been done, place the scan head in position on the sample stage (see Figure 1), and turn on the Easyscan controller.

The sample holder is a small disk, approximately 5 cm in diameter. It has a flat surface, and a magnet in the base to secure it to the small stage that supports the scan head. Remove the calibration grid sample from its container and use some double-sided tape to secure it to a position that is roughly in the center of the sample holder. Place the sample holder on the scan head stage, and connect the small grounding wire from the holder to the jack at the right rear corner of the scan head.

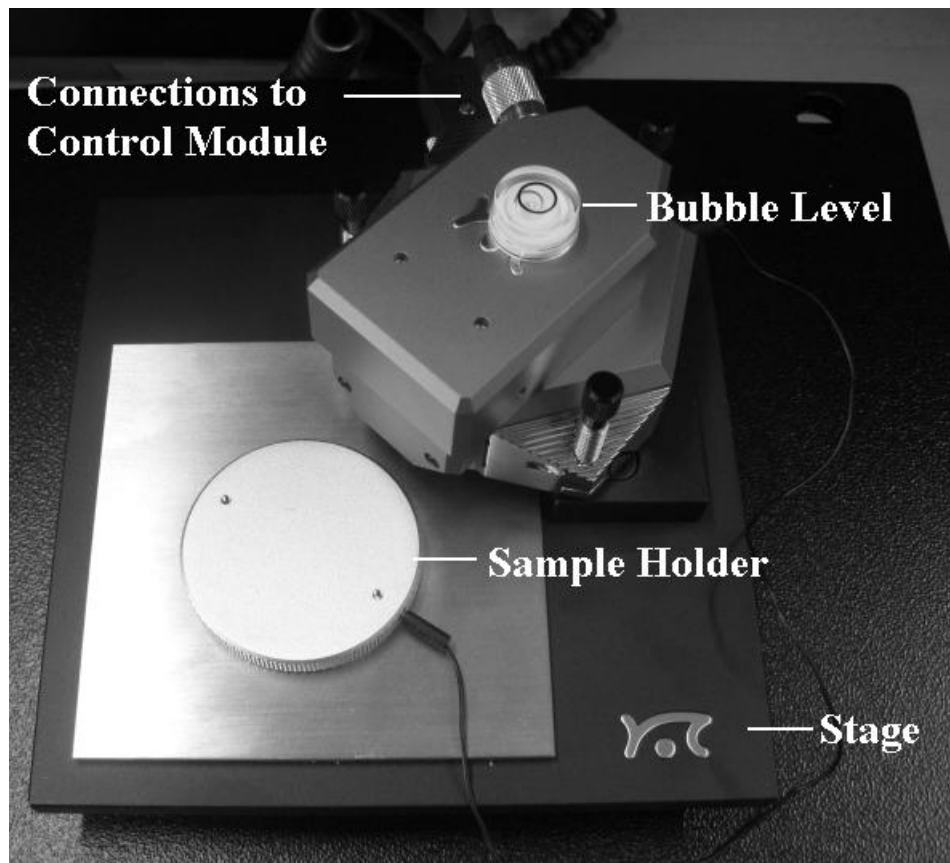


Figure 1: The scan head and the sample holder

Slide the sample holder towards the scan head. Make sure there is adequate clearance between the surface of the sample and the cantilever tip hanging down below the scan head. If there is insufficient space to slide the sample into place without contacting the tip, use the 3 leveling jacks to raise (turn clockwise) the height of the scan head until there is enough space for the sample to slide in without contact.

Turn on the computer and start the Easyscan software. Open the Operating Mode panel and use the dropdown menus to select SICONA as the mounted cantilever, and Static Force as the operating mode. Close the operating mode panel, and open the Positioning window. It will take a moment for the video camera to become active. You should then observe the image of the cantilever on the left side of the window.

- Slide your sample under the cantilever tip. You will likely see a blurry image of the sample moving around in the background in the video window. Look back at the actual sample holder on the stage, and position the sample holder so that the cantilever tip is roughly in the center of the sample.
- Manual coarse approach: Place the round bubble level on a flat portion on top of the scan head. Use the three leveling screws to lower (turn counter-clockwise) the AFM head towards the sample. Turn each screw a bit at a time, so the head remains roughly level (bubble in the center circle of the level). Use the Side View in the positioning window and watch the video display. As the tip gets close to the sample, the shadow of the cantilever on the sample surface will begin to show. At this point stop and make small adjustments to ensure the scan head is level, but be careful to avoid “crashing” the tip onto the surface of the sample.
- Switch to Top view, and the surface of the sample should now be in focus just below the cantilever tip. Carefully position (slide) the sample holder on the stage so that the tip end resides in the middle of an area of the sample which appears to be relatively flat and free from surface contaminants. Switch back to Side view.
- Now proceed with another “manual” approach using the controls in the approach window. Press the “Advance” button so that the tip moves closer to the surface. Do this in short bursts, so as to avoid crashing the tip down on the surface of the sample. When the image of the tip and its shadow appear to be almost touching, it’s time to let the automatic approach take over.
- Press the Approach button, and the AFM software will perform the automatic final approach. When the tip has made contact with the surface of the sample, a dialog box will pop up. Simply accept the message by clicking the OK button.
- Scanning begins automatically. The Imaging window will have opened, and a surface scan will start to take place.

The default values for the Imaging Area and Imaging Options will be suitable. Click on the “Finish” button on the top toolbar. This will ensure the scanning will stop when the full area (as defined in the Imaging Area settings) has been imaged. It will take several minutes for the AFM to scan the surface of the calibration grid. On the main toolbar under chart type select: Colour Map. On the Data Filter select: Line fit. Once the scan is complete, you should see an array of raised square surfaces (light colour) on a lower (darker colour) background. If you do not see this, seek the assistance of the instructor. To save an image of this scan press the Photo button in the upper right corner of the imaging window. A new window will open with a title Image#. On the main Easyscan toolbar select File > Export and choose: Save document to... and a dialog box will open. Save your results to a Windows bitmap file for inclusion with your lab report. This will be the standard way of saving data throughout the experiment. Any time an image window opens this (Export) technique can be used.

There are various measurement tools available to you to further analyze the image. Look on the main window toolbar, and click on the ↔ (measure length) icon. Your mouse cursor will change to an image of a small fountain pen tip when you hover over the sample image (colour map). Use this tool to draw a line across several of the raised squares. Specifics about the line length etc. show up in the Tool Results panel. Another useful tool is the “Create Cross Section” tool. Use this tool to draw a line across several squares of the calibration grid. In the Tool Results panel, click on the “Cut out line” button. This will open a new window showing a line graph of the cross section defined by the line drawn on the colour map. This is a quick way of measuring height and /or spacing of objects found on a surface scan. On the main toolbar select 3D view as the chart type, and Raw data (or Mean fit) to see a 3-D map of the calibration grid.

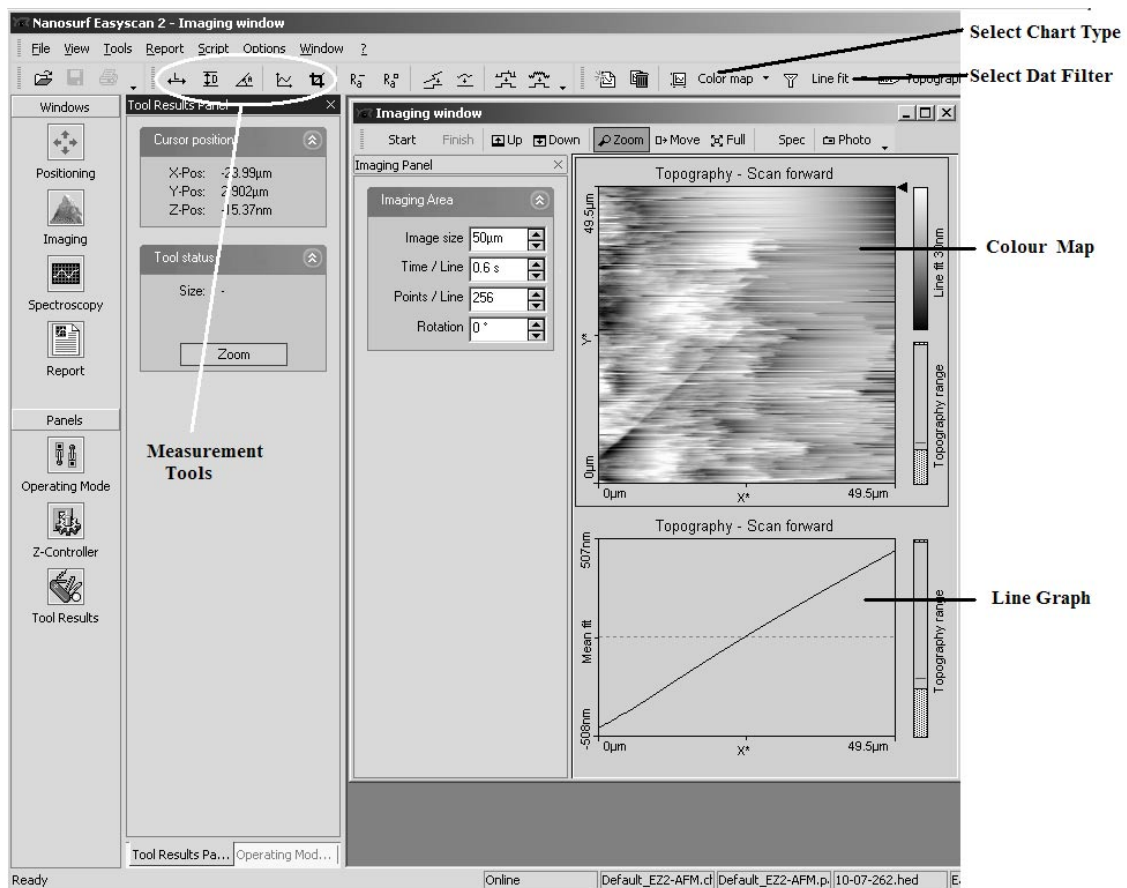


Figure 2: Important areas of the Easyscan interface

Switch back to the Colour map. In the imaging window, locate and then click on the Zoom tool. Use this tool to outline any area which encompasses only one of the raised sections of the calibration grid. Click on the Zoom button in the Tool Results panel. In the Imaging window, press the Start button and the microscope will now automatically begin rescanning this zoomed area. Make sure the Finish button is selected (blue) so that the image acquisition stops after the area has been scanned. Decrease the time/line to 0.5 sec to make the scan happen a bit quicker. Switch the Data Filter (on the main toolbar) to

Derived data. When the scan is completed, use the Measure Distance tool to get an accurate measure of the size of the square on the calibration grid. Measure both the dimensions of the base of the square and the size of its upper surface.

You will now investigate another sample. To switch samples, proceed as follows: Press the Withdraw button in the Positioning Window. Then use the Retract button to move the cantilever tip well away from the surface of the sample. Use the three leveling screw jacks to raise (turn clockwise) the scan head further away from the sample. One or two turns of each leg screw should be sufficient. Slide the sample holder out from under the scan head, and carefully remove the calibration grid sample from the holder and place it back in its storage case.

Obtain the CD sample from its storage case, and mount it on the sample holder using some double-sided tape. Make sure the opaque side of the CD sample is down. With the CD sample mounted, slide the sample holder under the scan head and position the holder so the tip is approximately centered on the sample. Select the Imaging window and set the imaging parameters as follows:

- Area: $\sim 15\mu\text{m}$
- Time/Line: 0.7 s
- Points/line: 256
- Rotation: 0°

Then select the Positioning window, and use the technique outlined previously to establish tip-surface contact. Once you have obtained a scan of the CD surface you should see the CD “pits” arranged in tracks on the sample surface. Use the measurement tools to obtain some data on the various pit sizes (lengths), and obtain a value for the average spacing between the “tracks” of the CD. Make sure you save an image of the CD scan to include with your report. When you are done, withdraw and raise the cantilever tip as before, raise the scan head, remove the CD sample and return it to its storage case.

You will now investigate the surface of a very fine diffraction grating that has been covered with a coating of gold. This surface is much more irregular than the previous ones studied, so it’s an ideal candidate for use with the Dynamic mode of the AFM. This will require a change of the cantilever used. Refer to Appendix 1 and change out the SICONA tip with a new ACLA one. This tip is shorter and stiffer than the SICONA tip. In the software remember to open the operating mode panel and change the mounted cantilever selection to ACLA and the operating mode to Dynamic AFM.

Mount the gold-coated diffraction grating on the sample holder and slide it under the scan head. This sample is slightly thicker than the previous 2 samples so take care in making sure it slides below the tip and not making contact with the tip as you slide it in place. Once you have completed your manual approach use the Top View in the positioning window to look for an area on the surface that appears to be free of defects (scratches dust etc,) and move the sample stage so the tip is positioned roughly in the center of this area. Once again, use the approach technique outlined previously to

establish the tip-sample “contact”. It is suggested that you use the following settings in the imaging window:

- Area: $\sim 15\mu\text{m}$
- Time/Line: 0.7 s
- Points/line: 256
- Rotation: 0°

When this scan is complete, save an image of it and then look for a smaller area where you can see a relatively uniform distribution of the grating lines. Use the zoom tool to select this area (typical area $\sim 5\mu\text{m}$) and Start the zoomed in scan. Use the cross section tool to measure the grating spacing, and look for the appearance of the “pillow shaped” grains of gold on the grating surface. Use the measure tool to get an estimate of their “typical” size. Remember to save the images from this analysis, and include them in you report.

When you are through, switch off the Easyscan controller, close the software application, and disconnect the cables from the scan head. Carefully remove the ALCA tip and return it to the package, making sure you place it tip up (“T” piece visible) on the gel bedding. Return the scan head to it’s storage container, making sure you’ve removed and stored the drop stop and cantilever insertion tool first.

Appendix 1: Changing tips on the Easyscan 2 AFM

- Remove the cantilever insertion tool (short stainless steel post) from its storage position inside the drop stop.
- Turn off the Easyscan controller, using the switch located near the power cord.
- Turn the scan head over (upside down) and place the drop stop over the front end of the scan head. The chamfered edges of the stop face up towards you. The stop is designed to keep things from falling down into the opening at the front of the scan head while attempting removal or insertion of cantilever tips.

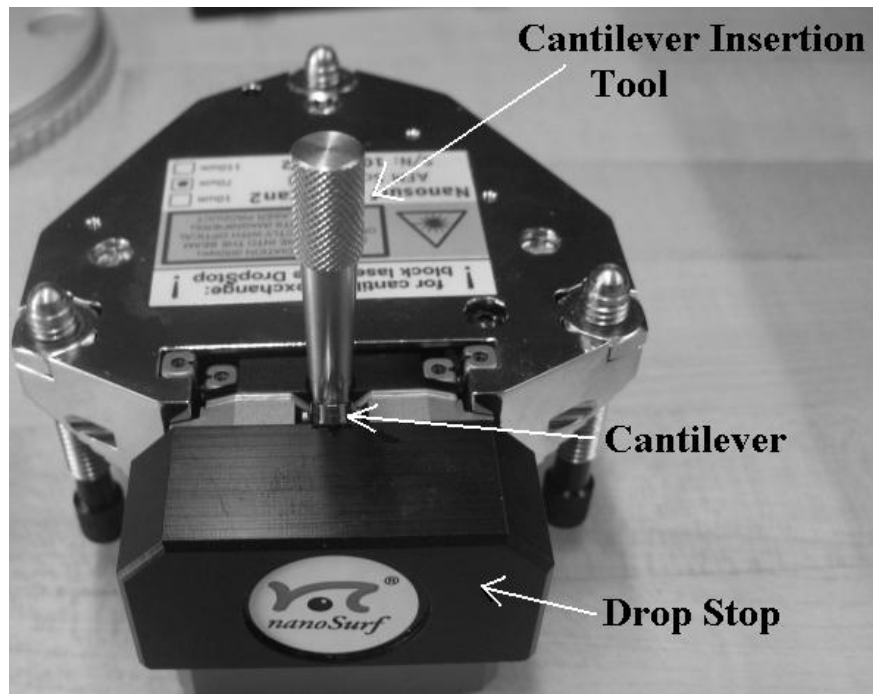


Figure 3: Preparing for tip removal/insertion.

- Place the cantilever insertion tool in to the hole just behind the cantilever holder spring. This causes the spring to lift up and release the existing cantilever tip.
- Use the tweezers to carefully remove the existing tip (if applicable), and place it back in an appropriate storage case. Should you drop the cantilever tip into the scan head, seek the assistance of the instructor immediately. Do not attempt to recover the tip yourself.
- Use the tweezers to obtain a new tip from the storage case. The tip end of the cantilever is identified by the “T” piece visible on the cantilever base assembly. Pick up a tip with the tip end facing you, and carefully place it into the cantilever holder. Use the small magnifier and the incandescent work light to assist you in viewing as you place the tip in the holder.

- Position the rectangular end of the cantilever to sit perfectly aligned in the cantilever alignment chip, with the front (tapered) portion (tip end) protruding out over the drop stop. (See Figure 3)

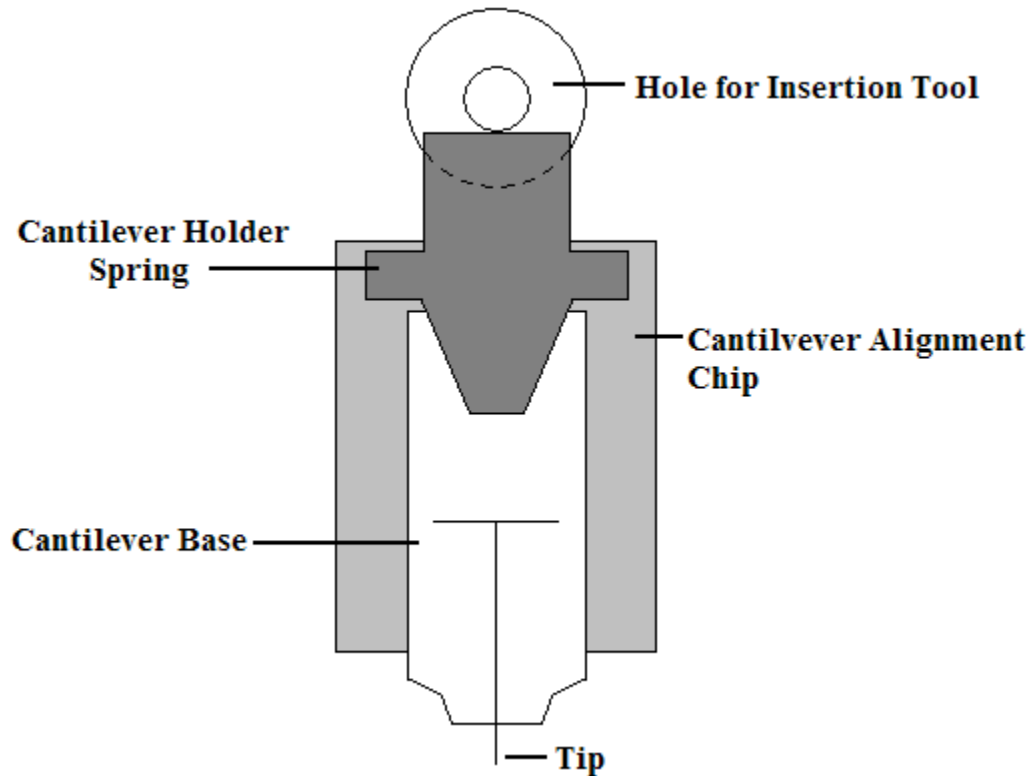


Figure 4: Close up view of a cantilever inserted into the alignment chip.

- Remove the cantilever insertion tool to allow the spring back down to hold the tip in place. Make sure the tip stays aligned while doing this.
- Turn the scan head over and remove the drop stop.
- Return the insertion tool back into its storage position inside the drop stop.
- Place the scan head back onto its mounts on the sample stage.