Park NX20 AFM SOP

Prerequisites:

Prior to Park NX20 AFM training you need to discuss your project with staff to ensure your needs can be met with the tool. There are many different scanning modes available, and users must get trained on each mode they plan on using. Some of the most commonly used modes are:

1. Topography Measurement (tapping, true non-contact, or contact, can be done in air or liquid)
2. Nanomechanical measurements (force modulation microscopy, nanoindentation, lateral force microscopy)
3. Electroforce Microscopy/Kelvin Probe Force Microscopy
4. Piezoforce Microscopy
5. Conductive AFM
6. Scanning Capacitance Microscopy
7. Magnetic Force Microscopy
8. Scanning Ion-Conductance Microscopy

In addition, to become authorized you must:
• Complete training and authorization test with Nano3 staff. For further documentation see D:\SpmData\Manuals and Guides.
• Have your own AFM tips to use.
• Have your own sample to measure (must fit in an area < 6” wafer).
• Have your own tweezers for mounting tips and samples.

Nano3 is able to supply HQ:NSC15/AL BS AFM tips which are suitable for non-contact mode or tapping mode for $18 per tip. Nano3 can also supply HQ:NSC36/Cr-Au tips, which are electrically conductive and suitable for electrical measurements in contact or non-contact mode, for $36 per tip. You must have your own gel pack storage to purchase these tips. If you wish to have your own magnetic sample mounts we recommend Ted Pella 16218 (https://www.tedpella.com/AFM_html/AFM.htm#anchor842459).

Introduction to AFM

Scanning probe microscopy (SPM) refers to any technique in which a probe is tracked across a sample to measure its topography, mechanical constants, electrical properties, magnetic properties, chemistry, and other properties. The Atomic Force Microscope (AFM) is a type of SPM focusing on topographic imaging, force measurements, and manipulation. In an AFM, an atomically sharp tip at the end of a static (contact mode) or vibrating (tapping mode) cantilever is scanned across a sample. The position of the tip is controlled in the X, Y, Z directions by voltages that are applied to a piezoelectric crystal, which allow for sub-nm motion control. The cantilever properties are monitored by a laser and feedback circuit. Variations (in e.g., the topography) cause the cantilever's interaction with the sample to change. Through this feedback process, the tip tracks the sample surface resulting in a high-resolution topographic image.

Components of the system

Typical AFM systems are composed of an optical microscope, a replaceable AFM cantilever (on the end of which is a sharp tip), a method to detect the cantilevers deflection, a piezoelectric crystal positioning system (XYZ), and a feedback system. The optical microscope is to help align the cantilever coarsely to a region of interest on the sample. There are a few ways to monitor the cantilevers deflection but the most common is with a laser. The laser beam is positioned to reflect from the top surface of the cantilever. The reflected optical signal is collected in a photodiode array (often 2 or 4 quadrants) and converted to a voltage. When the cantilever deflects, the position of the reflected signal on the diode array changes. This change of position results in a change of measured voltage in the diode. The scan head is physical hardware which holds the tip and contains all the controls to tune the laser readout: laser position, cantilever positioning, and photodiode position. All of these components must be aligned correctly to ensure correct readout and optimum system performance. The piezoelectric positioning system uses an applied voltage (which deforms the piezoelectric crystal) to move and scan the cantilever with respect to the sample. The piezoelectric crystal is the reason behind the AFM's high spatial resolution - and is the most expensive part of the system! The goal of the feedback system during imaging is to change the Z position to maintain a constant tip-sample distance or setpoint as monitored by the laser. For example, when the tip is scanned across the sample and encounters a raised bump the cantilever will deflect. The deflection signal from the photodiode is fed to the controller which will tell the piezoelectric positioning system to increase Z. This process is continually occurring during image collection. Ultimately the topographic image formed in an AFM is a voltage map of the piezoelectric control system. The X-Y-Z piezoelectric voltage is calibrated to distance from the crystal properties, allowing a topographical map to be formed.

Imaging Modes

The most typical mode for gentle imaging is tapping mode. In this mode the cantilever is driven to oscillate with a fixed amplitude slightly below the resonant frequency. During imaging, the feedback circuit attempts to maintain a given amplitude setpoint. The cantilever amplitude is strongly affected by how close it is to the sample. The closer to the sample, the more constrained the vibration is and therefore the lower the amplitude of oscillation. The further away from the sample the less constrained the motion is and the higher the vibrational amplitude. Selecting a low amplitude setpoint means the tip will track the surface more intimately and has the risk of damaging delicate samples. Selecting a high amplitude setpoint means the tip may not be tracking the surface closely but has reduced risk of damaging samples. Since this mode uses a dynamic vibrational mode, other properties like the phase between the cantilever driving oscillation and the laser readout can be an indicator of how well the surface is being tracked or even a qualitative guide of sample stiffness or stickiness etc.

Similar to tapping mode is true non-contact mode (NCM). In this mode, the cantilever is driven to oscillate slightly above the resonant frequency. In this mode, the tip tracks the sample from a farther distance, reducing tip-sample interaction resulting in the most gentle imaging and extending the lifetime of the tip.

Another mode is contact mode. Here the cantilever is static, and during imaging the feedback circuit attempts to maintain a given deflection setpoint. Selecting a low deflection setpoint means the tip is not in close contact with the surface whereas selecting a high setpoint means the tip is in close contact with the surface (and applying higher forces). A drawback of using this mode is the high lateral force that is exerted on the sample, which makes it unsuitable for weaker samples. Contact mode is often used to measure the mechanical and electrical properties of the sample.

Proper selection of cantilever/tip plays a large part in the quality of data that can be gathered from your sample. Generally speaking, tips with stiffness (k) 3−100 N/m will work well for most tapping and NCM mode applications. For contact mode, you typically want a tip with stiffness below 5 N/m.

Scan and Feedback Parameters

There are many parameters that you can control in the software that you will learn during training. Some of the most important settings are detailed in this section. Depending on the mode of operation the AFM and feedback controller is aiming to maintain a particular setpoint as described before. It is important to select an appropriate setpoint to ensure the tip is interacting enough with the sample (rather than scanning free space).

Consider tapping mode. Prior to scanning you must perform an NCM sweep. In this process the tip is very far from the sample. The software automatically determines a driving amplitude and phase such that enough signal is gathered from the fundamental vibrational mode of the cantilever. The driving amplitude (voltage) determines the physical free oscillation amplitude (nm). If the cantilevers' motion is impeded (i.e. by the sample), the oscillation amplitude will decrease even when driving at a fixed amplitude. This is why the amplitude setpoint should be low enough so that the tip interacts with the surface strongly enough to track it.

As the tip/cantilever scans over the surface, the feedback circuits' job is to maintain that setpoint through the use of Z servo (+/-Z), proportional (P), and integral (I) gain (i.e. a PI controller). At every point in the scan, the controller is calculating how far away from the setpoint value it is. This is known as the error signal. Roughly, the feedback parameters take into account the current error (P) and the historical average of the error (I), with the Z servo gains amplifying the error signal in either the positive or negative Z directions to allow for better tracking for samples with steep variations. The reason why differential (D) is not used, is because it is highly susceptible to noise. The feedback parameters will change depending on the number of pixels, the nature of the sample being scanned, and the tip being used.

Computer and Software

PC credentials:

• Username: nano3user

• Password: ParkAFM

For offline data analysis you are free to copy the XEI software from D:\SpmData\Manuals and Guides\XEI Analysis Software. We recommend using XEI Analysis. Select 'offline' during installation. Additionally, a free, open-source SPM analysis software called Gwyddion is available from http://gwyddion.net.

Standard Operating Procedure – Tapping and Non-Contact Mode

This is the default mode. Additional training is required to user other modes.

1. Log into your FOM user account, activate Park NX20 AFM from the calendar.

2. Begin an entry in the paper log book.

3. Open SmartScan program from Desktop and wait for initiation window to close.

4. Open the acoustic enclosure and remove the scan head

• Make sure laser switch is off, flip the black paddles on either side of the scan head towards the front of the enclosure to unlock the head, and slide the scan head out to the right.

5. Place the scan head upside down on the plastic head cradle on the desk.

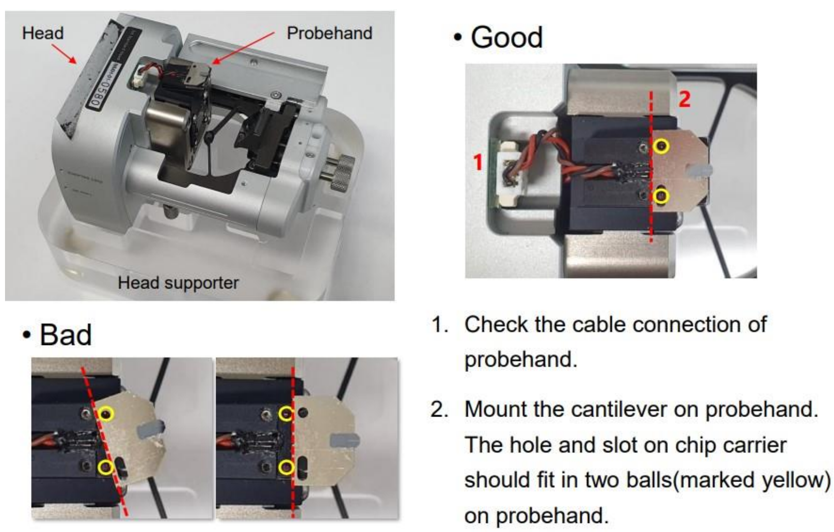

Figure 1: *Tip loading on probehand*

6. Load mounted tip onto the probehand so that the metallic balls on the probehand line up with the holes in the tip mount. See **Figure 1**.

• If using unmounted probes, first mount them using the probe mounter and the clip-type chip carriers

• Flip open the probe mounter and place the clip-type chip carrier so that it rests on the small peg. See **Figure 2**

• Flip the probe mounter closed and tighten the screw until the clip lifts up.

• Slide the unmounted tip under the lifted clip until it hits a stop. Be careful as it is easy to go over the stop if the tip is angled.

• Unscrew the screw, flip the probe mounter open, and remove the chip carrier.

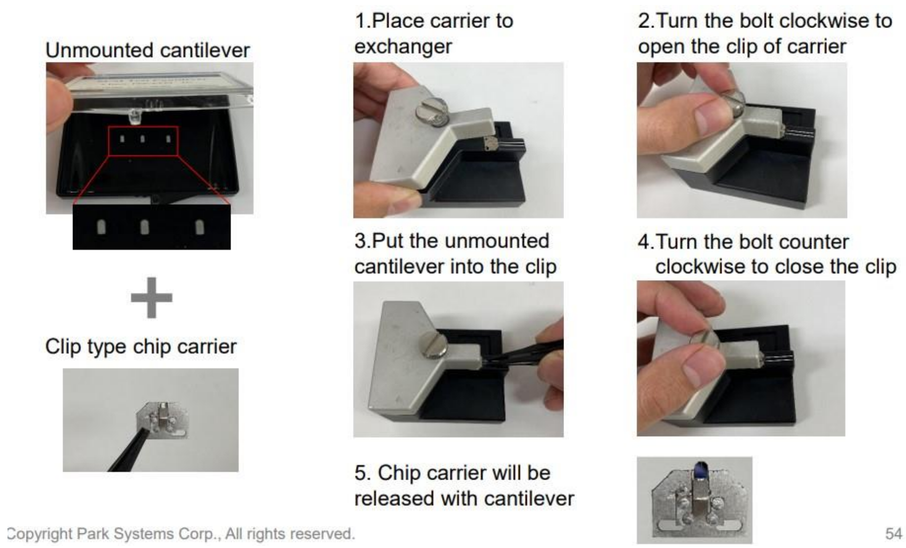

Figure 2: *Loading unmounted tips on clip-type chip carrier*

7. Place scan head back into tool, lock into place by pushing black paddles, and turn the laser switch ON

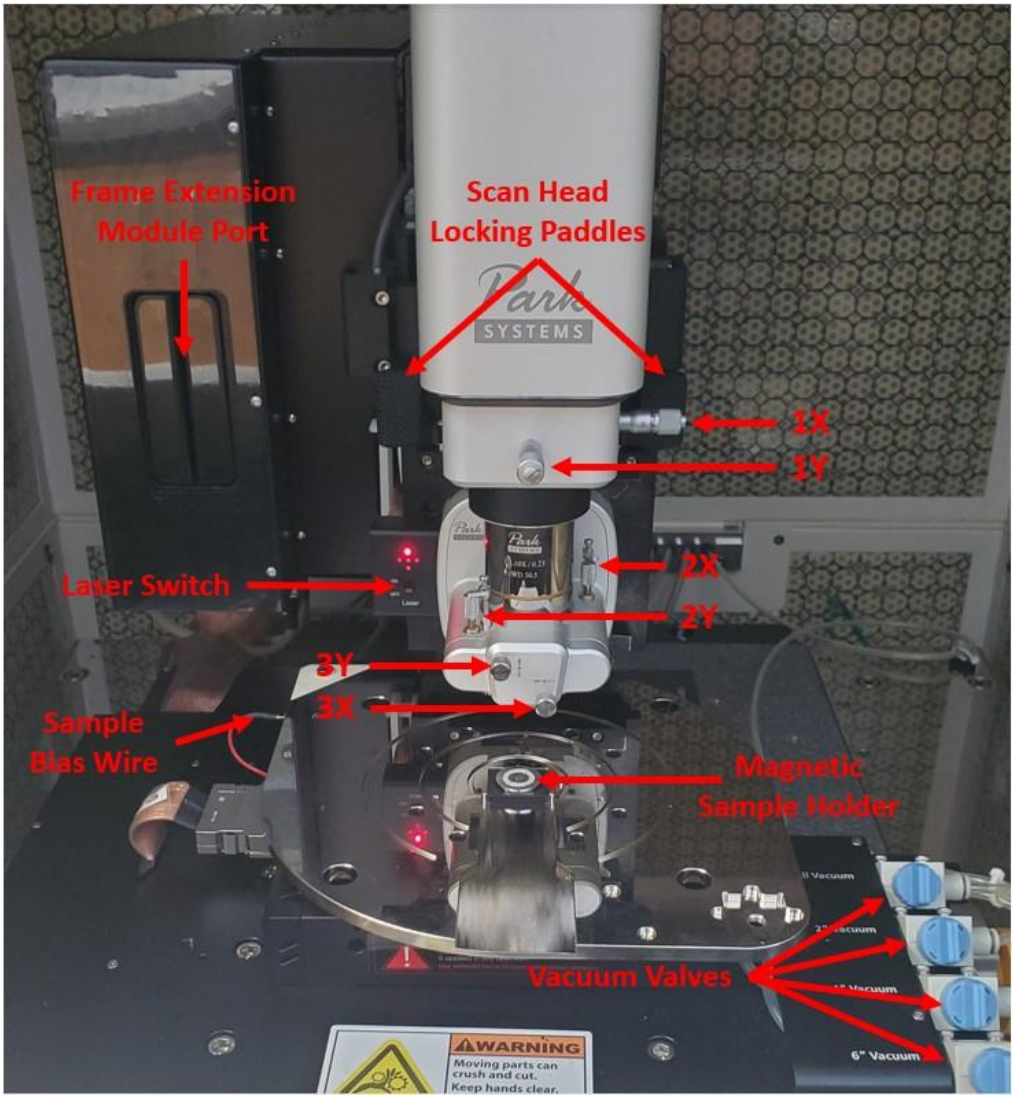

Figure 3: *Controls in the enclosure*

8. Use the top pair of knobs on tool to center the cantilever in camera view. See Knobs **1X** and **1Y** in **Figure 3**. You can expand the camera view using the Expand Button (**Button 6.1** in **Figure 6**). You should line up the end of the tip with the cross displayed on the screen.

9. Use the pair of knobs on the top part of the scan head to move the laser so that it is centered over the end of the cantilever. See **Knobs 2X** and **2Y** in **Figure 3**.

• If you can see the purple glow from the laser, but not the laser spot, then the laser is hitting the sample plate. Turn **Knob 2Y** clockwise to move the laser up until you see the laser spot on the cantilever chip, then move it over the tip.

• If you cannot see the purple glow from the laser, then the laser is likely on the cantilever chip. Move around the camera with **Knobs 1X** and **1Y** to try to find the laser, then move it towards the tip.

• IMPORTANT: you generally will not need to make large adjustments to the laser position. You should not need to turn **Knobs 2X** and **2Y** more than 1 full revolution. **If you cannot see the laser spot, do not blindly turn the laser positioning dials!**

• Position the tip close to the end of the cantilever without decreasing the intensity. You should see an increase in laser intensity when the laser is moved over the cantilever. See **Figure 4.**

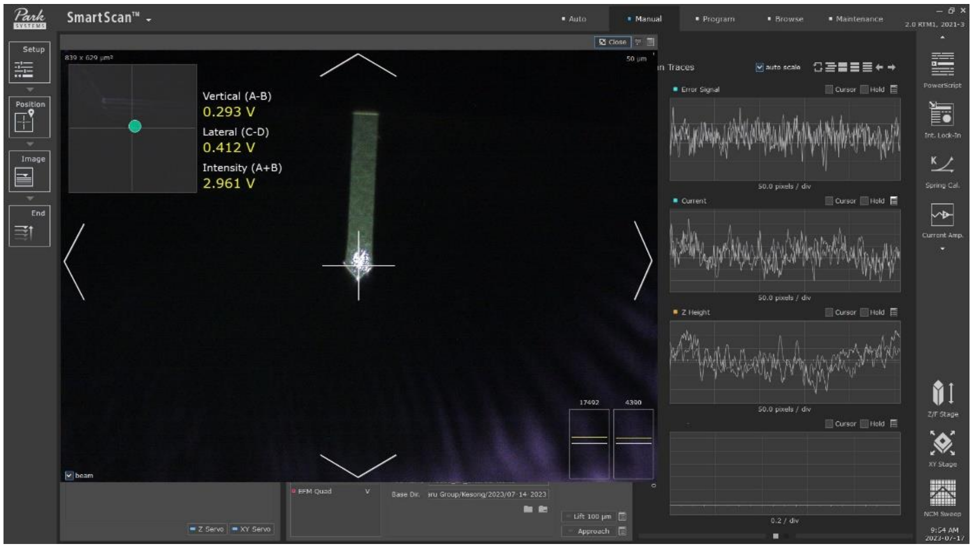

Figure 4: *Laser positioning on cantilever*

10. Use the bottom pair of knobs (Knobs **3X** and **3Y** in **Figure 3**) to center the laser beam in the detector

• Look at the graph in the corner of the camera screen and adjust the knobs to bring the dot into the center. If the dot does not move when you turn the knob, look at the intensity. If the intensity is decreasing, then you are turning the knob the wrong direction. See **Figure 5**.

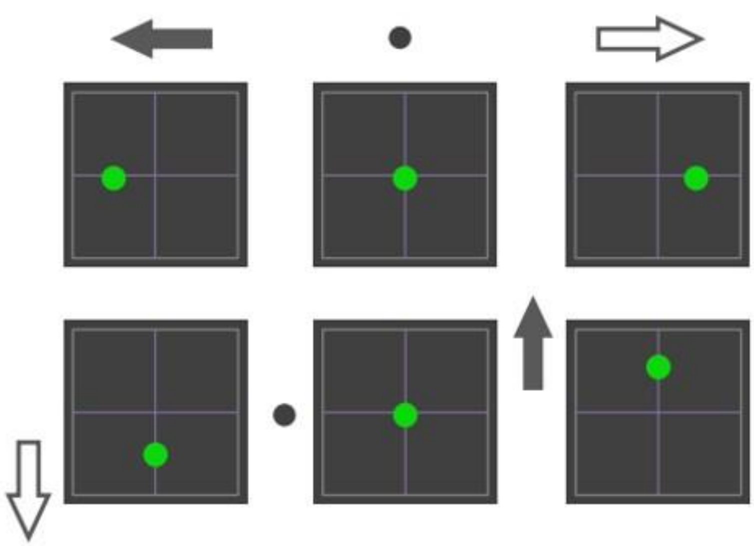

Figure 5: *Detector positioning*

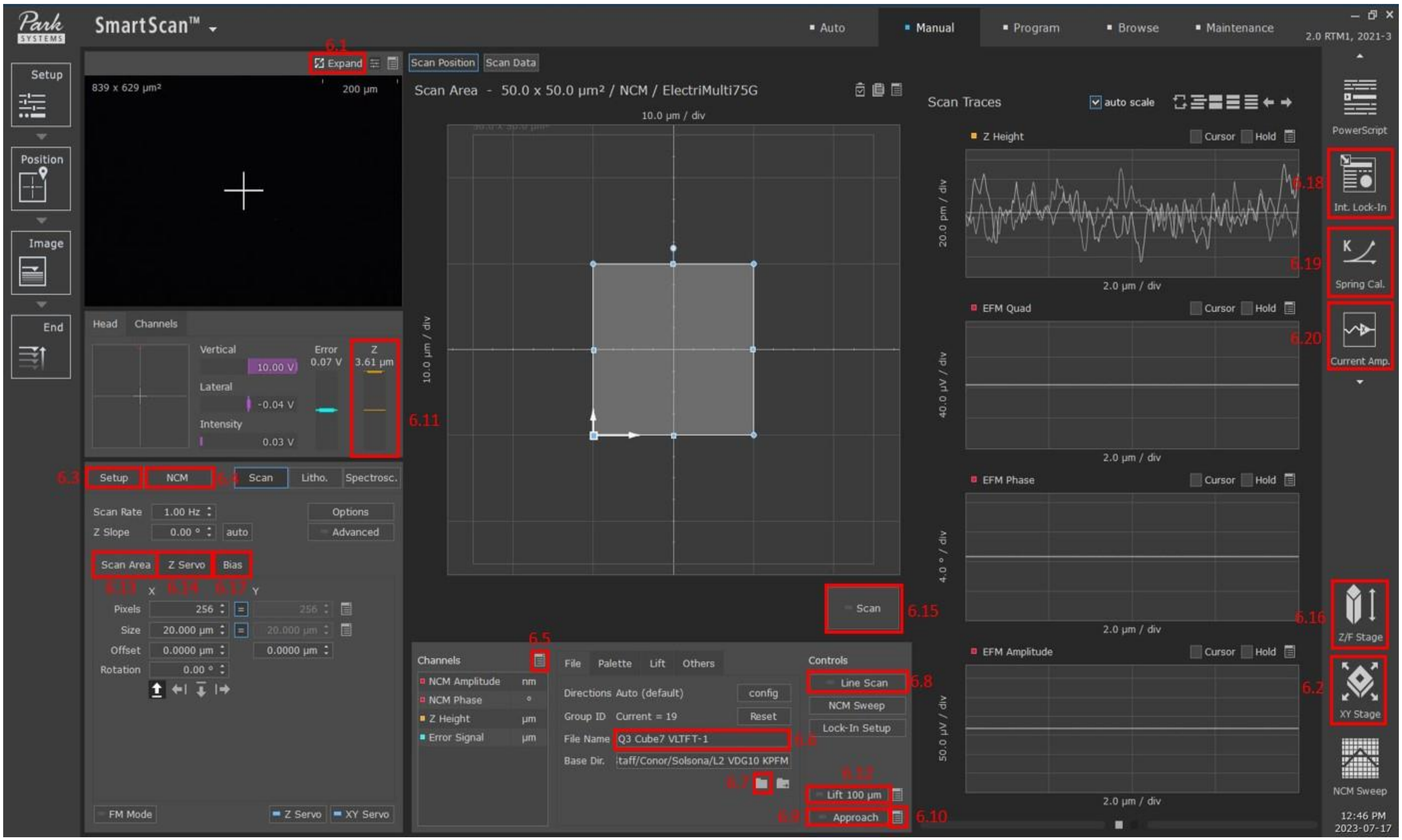

Figure 6: *Main Control Page*

11. Load sample. Use the XY Stage control (**Button 6.2** in **Figure 6**) to move the stage towards the enclosure door.

• On the XY Stage graph, click and hold in the direction you want to move the stage (up on the graph corresponds to moving the stage towards the door of the enclosure). Clicking farther from the center will move the stage faster.

• If using the vacuum chuck, unscrew magnetic sample holder, turn on vacuum pump under desk, and turn the appropriate valve on the right side of the chuck. See **Figure 3**

• Use the XY Stage control to move the area of interest on your sample underneath the tip. You can use the light from the microscope to get a rough idea of where the tip will land on the sample.

12. Close the acoustic enclosure and latch shut. All further operation will be done through the software.

13. Press the Setup Button (**Button 6.3** in **Figure 6**) and select the cantilever you are using from the library. If the cantilever you are using is not listed, contact Nano3 Staff to help you add it.

• Make sure the Line Scan is turned off when changing the cantilever type. You can turn off the Line Scan with **Button 6.8** in **Figure 6**

14. Press **Button 6.4** in **Figure 6** to select your desired scanning mode.

• If you select a non-contact type mode (NCM, Tapping, EFM, etc.) the NCM Sweep window will open and automatically start the NCM Sweep. The NCM sweep is successful if it finds a peak near the tip's listed resonant frequency, and the Q Factor listed is above 100.

15. Press **Button 6.5** in **Figure 6** to open the Channel Config window.

• Select the desired channels for your measurement. You can filter the channels by scan mode by selecting the dropdown menu that says "Show All" (**Button 3-2**) and selecting the scan mode you are using.

• Press "Add" to add the available channels on the right side to the selected channels on the left side. (See **Figure 7**)

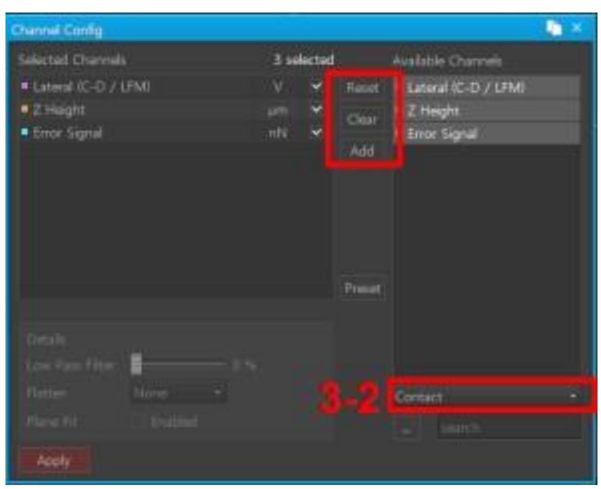

Figure 7: *Channel Configuration Window*

16. Give your scan a filename in **Box 6.6** in **Figure 6** and select the folder to save the scans in.

• All scans must be saved in a folder in **D:/SpmData** as this is the folder connected to the data computer. You can press **Button 6.7** in **Figure 6** to open Windows File Explorer to create and select the folder you want to save your scans in.

17. Manually approach towards the sample until you can focus on the sample

• Expand the Camera view (**Button 6.1** in **Figure 6**) and move the microscope focus down as far as it will go (1350µm) using the Focus stage adjustment (**Button 8.1** in **Figure 8**). Click and hold below the white line to move the focus down. The farther from the center line you click, the faster it will move.

• Move the Z Stage down (**Button 8.2** in **Figure 8**) in the same way as the Focus stage until you start to see your sample come into focus. Be careful as you get close to the sample, as if you approach too far, you can crash the tip and potentially damage the tool and your sample.

• Tip: Most samples will come into focus with the Focus stage at its lowest position of 1350um and the Z stage around 10,000µm. Usually, you can approach quickly until the Z stage is at 15,000um, and then slow the approach until the sample is in focus.

18. Once sample is in focus, use the XY stage controls in the Camera View (**Buttons 8.3** in **Figure 8**) to move to your desired scan location. If you aligned the cantilever with the cross in Step 10, then you can use the cross as an indicator of where the tip will land.

• You can adjust the movement speed by using the mouse scroll wheel while hovering over **Buttons 8.3**. 100% is the fastest movement speed and 1% is the slowest.

19. Once you have selected the spot to scan, move the Focus Stage back up to the cantilever (**Button 8.1**).

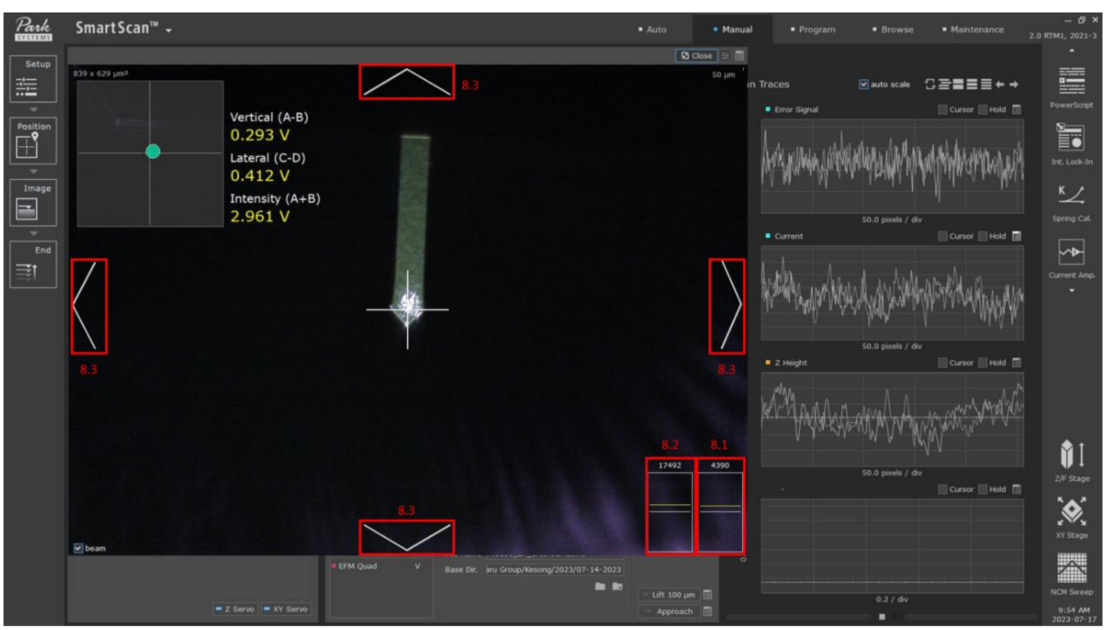

***Figure 8**: Expanded camera view*

20. Close the expanded Camera View and start the automatic approach with **Button 6.9** in **Figure 6**.

• You can change the approach method by pressing **Button 6.10** in **Figure 6**. “Quick and Safe” is the recommended approach method for scans in air.

• The approach window will open automatically and close when the approach is complete.

• If the approach is taking longer than 2 minutes, cancel the approach, double check your laser alignment, and rerun the NCM sweep if applicable, then try approaching again.

• If the approach was successful, you should be able to see the sample and cantilever both in focus in the camera view and the Z-Scanner Bar (**6.11** in **Figure 6**) should be half orange. If it is fully orange, it means you had a false approach. Lift the tip using **Button 6.12** in **Figure 6** and try approaching again.

21. Select the Scan Area Tab (**Button 6.13** in **Figure 6**). Input your desired scan size (maximum 50µmx50µm), desired number of pixels, rotation, and select your scan direction. Make sure to confirm changes by pressing Enter after each change.

22. Select the Z Servo tab (**Button 6.14** in **Figure 6**) and turn on the Line Scan (**Button 6.8**) if it is not already activated. This will cause the tip to start scanning back and forth on the same line to make sure the tip is tracking the surface correctly.

• The Z Height channel on the right side will show two lines, one of the topography in the forward direction, one in the backwards direction. Ideally, these curves should be perfectly matched, but it might require some adjustments to Set Point, Z Gains, and Scan Rate to get them matched.

• If Forward and Backward curves show very small topography (1-2 Angstrom) and they are not matched at all, then the tip is not tracking the surface. Decrease the Set Point value if using a NCM mode (target a smaller oscillation amplitude), or increase the Set Point value if using a Contact mode (target a larger cantilever deflection)

• Generally, increasing gain will improve the matching of forward and backward curves, but will also increase the amount of noise. See **Figure 9** to visualize the effect of Setpoint, Z Gain, and Scan Rate on the scan quality.

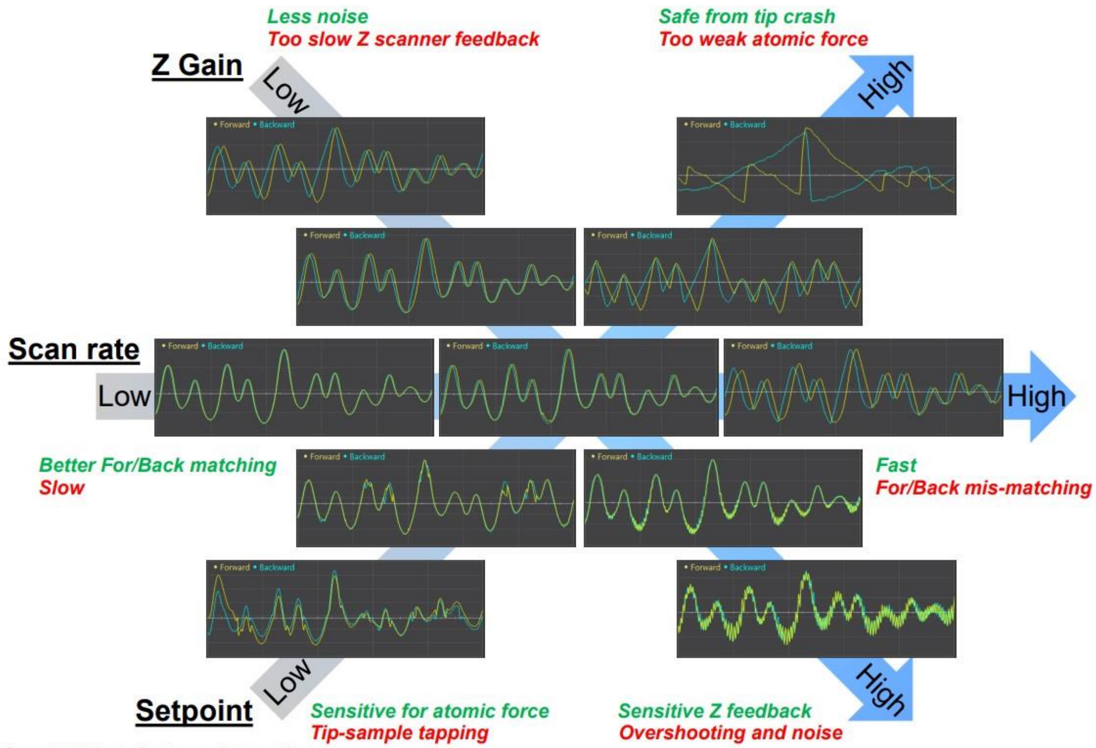

Figure 9: *Effect of Gain and Setpoint on topography reading*

23. When the Z Height scan is looking good, press the Scan button (**Button 6.15** in **Figure 6**) and the tip will begin the raster motion and scan your sample. During the scan, monitor the Z Height to make sure the forward and backwards scans remain well matched. Adjust gains and setpoints as needed during the scan but be aware that large adjustments can cause artifacts in the scan.

24. The Scan will be saved to your selected folder once the scan is complete or the Cancel button is pressed.

• If you want to scan a different area within the 50µmx50µm area you approached, you can move the tip by repositioning the square on the Scan Area or input the Offset manually in the Scan Area tab.

• If you want to scan outside of the 50µmx50µm area you approached, then you need to Lift (**Button 6.12**), move the XY Stage, and then reapproach following the same procedure as before.

25. When done scanning, lift the tip to its maximum position either by using the Z stage or by pressing the “Lift Z All” button in the Z/F Stage Control (**6.16** in **Figure 6**). Once the stage has stopped moving, turn off the Laser switch, unlock the scan head with the black paddles, and remove the scan head.

26. Remove your cantilever from the scan head, and if using the clip-type chip carrier, then remove your tip from the carrier

• IMPORTANT: Do not leave the probe mounter screw tightened on the chip carrier. If left like this, it will bend the chip carrier and damage it!

27. Remove your sample, then put the scan head back into the tool and lock it into place.

28. Close the acoustic enclosure, turn off the vacuum pump (if used), and close the SmartScan software.

29. Data can be accessed from the computer behind the AFM. Do not put any USB drives directly into the tool computer. Note the D: drive is not permanent storage and data may be removed. Collect all important files ASAP.