Reference: TN009

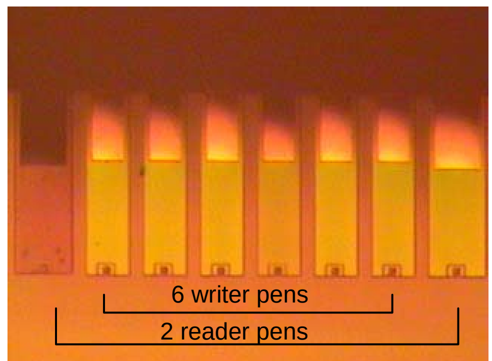
NSCRIPTOR™ Active Pen™ Array

By Jason Haaheim and Emma Tevaarwerk, Ph.D.

1.0 Introduction: Active Pens

Dip Pen Nanolithography® (DPN®) is the process of writing nanoscale patterns of molecular "ink" onto a sample substrate via a coated SPM tip. Often DPN patterns are simply written and imaged with a single inked tip, contaminating the pattern even as it is imaged. This contamination can lead to reduced phase contrast with continued imaging, and eventual pattern overwriting. Furthermore, selective inking and individual cantilever actuation becomes even more important for the patterning of biological inks, where cross contamination can lead to non-specific binding or interfere with later florescence characterization. Finally, as the combined patterning[1] of biological materials, small functional molecules, and inorganic nanoparticles becomes more necessary to produce functional nanostructures, such tools will become pivotal to nanotechnology research and development.

NanoInk's Active Pen™ Arrays are specifically engineered for flexible and multiplexed nanopatterning. Using thermal bimorph technology, individual cantilevers can be actuated to enable multi-ink writing without any cross-contamination or unintended surface patterning. Additionally, "reader" tips can be left clean to image surface patterns, or address specific surface structures without unintentional inking. NanoInk's Active Pens are used in conjunction with NanoInk Universal InkWells, which enable selective inking of individual pens without cross-contamination. A picture of an Active Pen array dipping into a NanoInk MicroWell is shown to the right.



2nd Generation Active Pens – T1 Array with short heat spreader, Pen1 extended (top view)

NanoInk's NSCRIPTOR DPN System is a fully-integrated hardware and software system that is optimized for the DPN process. Active Pen Arrays are a revolutionary technology designed to take DPN patterning to the next level of productivity and versatility. Active Pens use thermal bimorph technology to individually approach and retract pens from the surface via computer control, and their control is seamlessly integrated into NanoInk's InkCAD™ control software.

With Active Pens you can:

- Use up to 6 different inks simultaneously
- Avoid changing individual pens during experiments
- Semi-automatically calibrate the tip-to-tip alignment between adjacent pens in order to use multiple pens to write into the same area with sub-100 nm registration.
- Write patterns with an inked pen and immediately inspect the area with an un-inked pen, preventing smearing, and contamination of DPN pattern features.
- Image a surface area using a clean tip, then write into that area with a DPN pen.
- Write nanoscale structures in one patterning job that consists of multiple functional ink materials, using coated Active Pens (i.e., instead of having to switch single pens for different inks).

For a complete description of Active Pens and InkWells, see the accompanying datasheets. In the following technology note we will show you how to use Active Pens for your experiments. Specifically, we will lead you through both technical discussions to facilitate your understanding of the technology, as well as step by step instructions to help you execute your experiments on the NSCRIPTOR.

<u>NSCRIPTOR Active Pen Arrays: Table of Contents</u>

2.0 Setting Up Active Pens

2.1 Preparation and Setup

First you will need to prepare you system for Active Pen use, as it is not normally configured for this. Before beginning, go carefully through the checklist below:

a) Attach the Active Pens cable from the back of the controller to the workstation computer. Reboot the server, controller, and InkCAD, in that order. This tells the InkCAD software that the new capability is present. This should only need to be done the first time Active Pens are used, but can be a good check in case the PC is not communicating with the pen array.

b) Attach the provided digital multimeter (DMM) to the Z(s) [MON 7] channel 7 of the signal access module; this provides a measurement of the z-piezo extension. The sensor for the z-piezo ranges from ~7.5 V when fully extended to roughly ~2.2 V when fully retracted. For optimum imaging and lithography, the z-piezo should be centered within its dynamic range. Monitoring Z(s) will enable the user to maintain this condition by adjusting the setpoint accordingly.

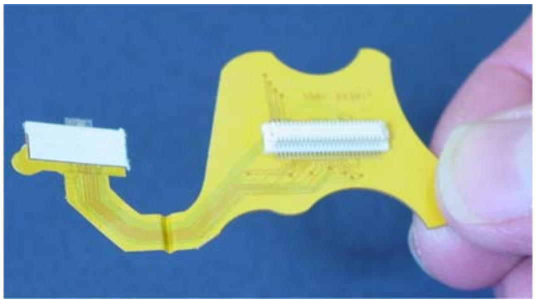
c) Remove any neutral density optical filters from in front of the photodetector. Since these cantilevers are not coated with gold, there is no need to reduce the reflected laser signal. But typical laser sum intensities reflected from the bare (mostly transparent) silicon nitride still measure between 1.5 and 2.0 V.

d) Prepare inks, and ink dispensing apparatus, and tweezers.

e) Prepare inkwells and substrates and position them carefully on the sample puck, adjacent to each other. Position them close enough so that you will be able to navigate between the two using the stage motors.

2.2 Installing Active Pens

The Active Pens should look similar to the picture below, with a flex circuit running between the electrical connector and the MEMS array itself, and a small tab near the probe array used to guide the pens into place on the scan head.



a) To mount the Active Pens, first perform a tip exchange and eject the scanner.

b) Hold the small tab at the side of the Active Pens between thumb and forefinger, making sure to gently ease the array onto the "L" bracket on the underside of the scan head. Avoid letting the magnets snap the array into place, as this may damage the cantilevers.

c) Once the array is seated on the scan head, insert the flex circuit connector into the socket on the underside of the scan board and press firmly to ensure a good connection. Check for proper alignment of the pens, tapping to the right and down to check for the ridge. Reinsert the scanner into its cassette and click "restore" to return the focus motors and z-motors to their proper positions.

d) Focus on the cantilevers, adjusting the x and y position of the lens as necessary. Zoom in fully on the cantilevers and bring them into sharp focus.

e) Open the "Pen Settings" dialog and select the appropriate probe array (T1, T2, or T3). Select the "control" tab to access the cantilever controls. Verify that each of the pens is actuating in response to applied current; click the "extend all" button in the Pen Control window, seen below.

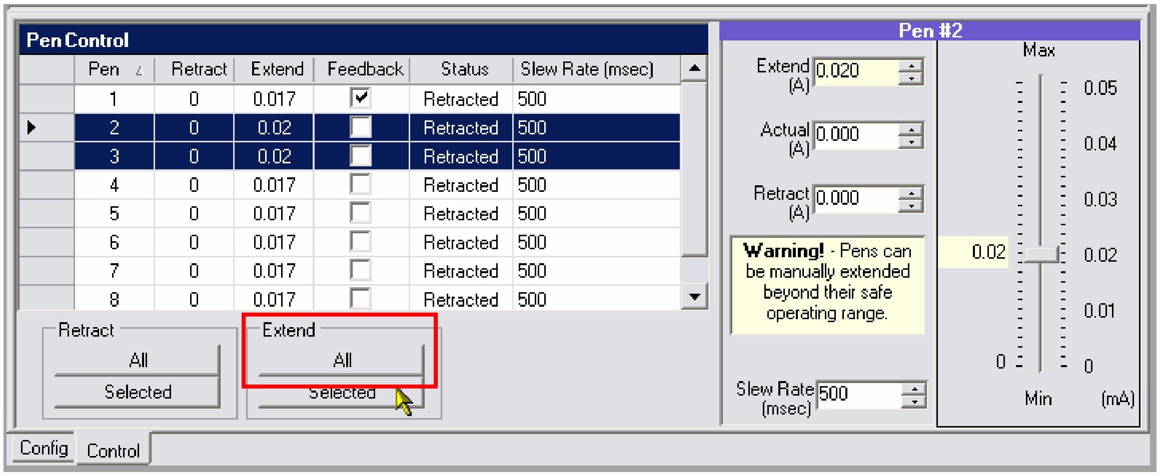
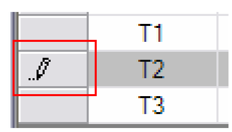


Figure 3

f) Notice the cantilevers' change in appearance as they are actuated; this is due to the change in light reflected from the cantilever surface. (Note: NanoInk does not guarantee that all of the cantilevers in a given array will be functional. While we are attempting to achieve 100% yield, at present NanoInk considers the following an acceptable product: at least one reader and its immediately adjacent writer functioning, and no more than 2 faulty cantilevers per array. Additionally, cantilevers may actuate slightly different amounts toward the surface in response to the same current.)

Note which pens are actuating for future reference; this data is initially supplied with the Active Pen shipment. To change the current delivered to a cantilever, first change the number, click the small pencil icon in the left margin (shown right) of the panel to "write out" the data, and then click "extend selected" to re-extend the pen. Otherwise, the value will not be changed.



g) Retract all of the cantilevers, insert the sample puck, and approach the sample surface. Once near the surface, level the pens using the Z2 and Z3 motors (as described in the Tips and Tricks Document). Adjust the relative positions of Z2 and Z3 until all of the cantilevers contact the surface uniformly.

Note: To ease the process of pen leveling, NanoInk recommends going through the "Calibration Leveling" procedure

2.3 Laser and Photodetector Alignment

2.3.1 Background: The Differences Between Passive and Active Pen Alignment

Normally, any cantilever that you would use for either contact mode or AC mode AFM would have a fixed angle with respect to the z-axis. That is, it would be angled so that the laser spot would reflect directly into the region of the detector, as seen in Fig. 3 below.   This is carefully determined by the cantilever fabrication process, as well as the angle of the scan head with respect to the photodetector, and the angle of the tip mount with respect to the scanner. Most of the time this geometry is well enough optimized that the photodetector can be aligned to the cantilever-reflected laser with relative ease.

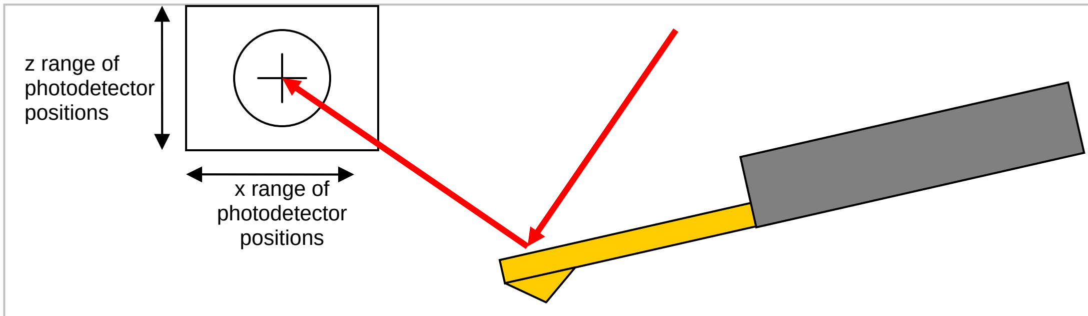


Figure 3 - Typical geometry for “static” contact mode passive cantilevers

However, as shown in Fig. 4, Active Pens offer the user the ability to actuate cantilevers up and down, so that the user can write with only one pen at a time. Higher currents correspond to greater bending and in turn a different z position, as illustrated in the picture below. This flexibility adds an additional variable when aligning to the cantilever-reflected laser. As a result, Active Pens laser and photodetector alignment is more complex than with passive pens, and will require practice.

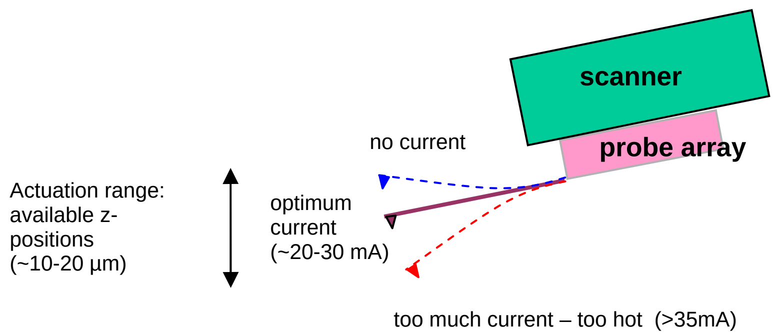


Figure 4 – Active Pen cantilever actuation, available z-positions

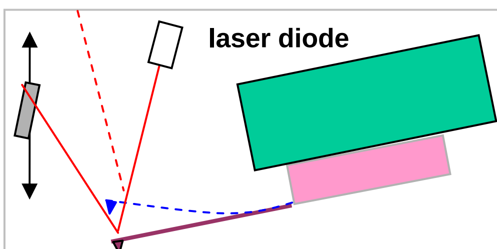


Figure 5 – Actuation and laser position

Without any current (and therefore without actuation), a laser spot reflected off of the back of the cantilever may strike above the photodetector, making alignment impossible (illustrated in Fig. 5).

Because small changes in cantilever angle are mechanically amplified over very large ranges of laser sweep, the full range of the x and z positions of the photodetector may not be sufficient to bring the detector into alignment with the laser spot. This concept is illustrated in Figs. 5 and 6, wherein there are two *ranges* of motion that must overlap in order to achieve alignment. These ranges have been aligned through the use of very precisely angled magnetic wedges glued to the flex circuit immediately atop the probe array.

A good general rule for laser and photodetector alignment is this: it is desirable to keep the current through the cantilever as low as possible to keep the tip cool, while still maintaining enough deflection to differentiate its z-position from its neighbors. This is because the current heats up the cantilever, and this heating may effect ink deposition.

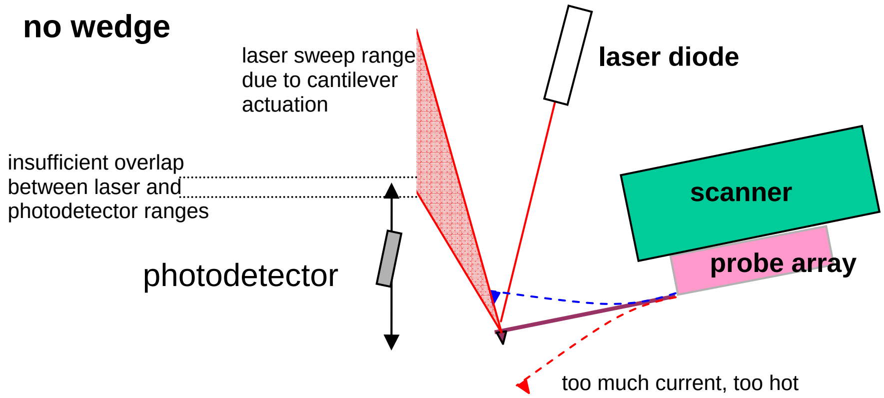


Figure 5 - With insufficient current to the pen, the laser spot is out of the range of the x and z photodetector position adjustment knobs, and the photodetector cannot be aligned.

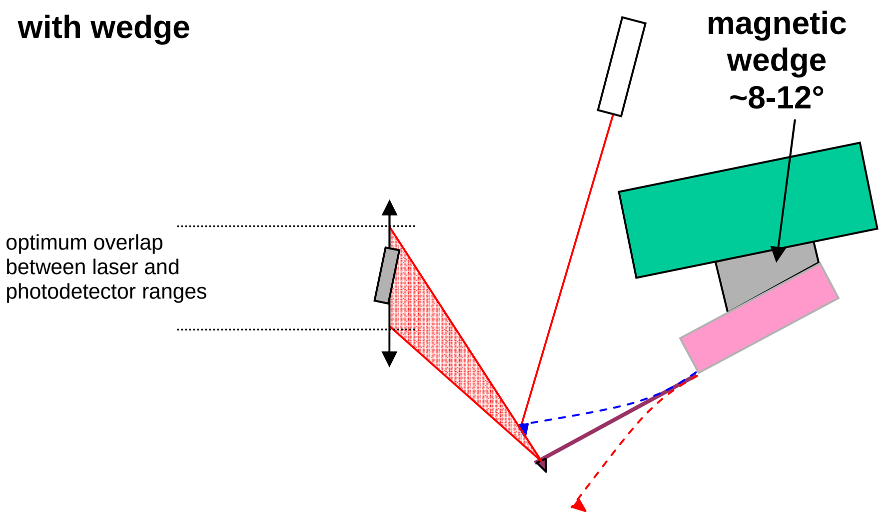


Figure 6 - With more current the pens are bent down more, bringing the reflected laser position into the range of the x and z photodetector position adjustment knobs, and the photodetector can be aligned.

2.3.2 Alignment Procedure

Prior to alignment, be sure to remove any neutral density optical filters in front of the photodetector.

a) Raise the array 500 µm above the substrate. This will make it easier to identify the laser spot for alignment.

b) Turn off the system light and adjust the laser into the vicinity of the desired reader pen (#1 or #8). Zoom out with the camera if necessary.

c) Turn the light back on, zoom in on the array.

d) Align the laser just above the tip "pit." (Fig. 7.) Since the bare silicon nitride cantilever is mostly transparent, it can be difficult to identify precisely where the spot is. However, the pit

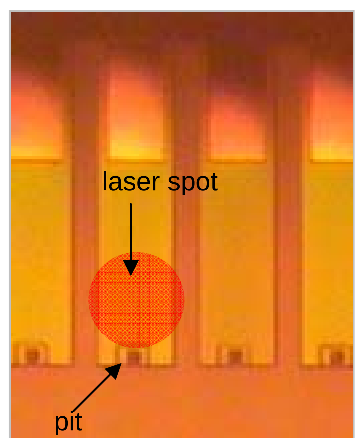


Figure 8

inherently scatters the laser light into the optics, and the laser spot will appear very bright when it is directly on the spot. Use this to align the laser to the pit, and then move slightly up on the cantilever. Typical laser sum values – even for these transparent cantilevers – are ~ 1.5 – 2.0 V. By monitoring the laser sum, it is possible to "blindly" determine when the laser is on or off the cantilever.

e) Set the reader pen to extend at 20 mA. This provides a stable baseline position for photodetector alignment. Click "extend" and note the cantilever's color change.

f) Align the photodetector as per the normal procedure, keeping the red dot toward the bottom of the grey rectangle. It may be necessary to explore the limits of the photodetector x and z ranges. For more information on this alignment procedure, see Appendix B.

g) If step (f) is not possible despite an exploration of the full ranges of the photodetector, you may need to increase the current to bend the cantilever more and bring it down into the grey region on the

2.4 Surface Approach, Establishing Good Feedback, Imaging With Active Pens

2.4.1 Background: The Differences Between Passive and Active Surface Approach

With normal passive cantilevers, the cantilever will only begin to deflect as it touches the surface. This cantilever deflection is used to determine whether the tip is in contact with the surface or not. However, since Active Pens actuate via a thermal bimorph, they can deflect for one of two reasons: (1) they are physically touching the surface, or (2) an additional thermal source or sink changes their temperature profile.

Substrates, especially gold, act as a significant radiative and convective heat sink. As an actuated cantilever approaches the surface, its heat will bleed down to the substrate. As it loses heat, it will begin to curl back upward. So during an approach, the laser will begin to deflect first because of a change in the cantilever's thermal profile, and then finally because it is in contact with the surface. This is illustrated in Fig. 9 below. This is a long range heat loss effect, and can occur anywhere from ~ 2 – 30 µm from the surface. (Similar effects have been observed during UHV Kelvin probe experiments.) Anything closer than 2 µm will usually imply the formation of the water meniscus, and thus contact.

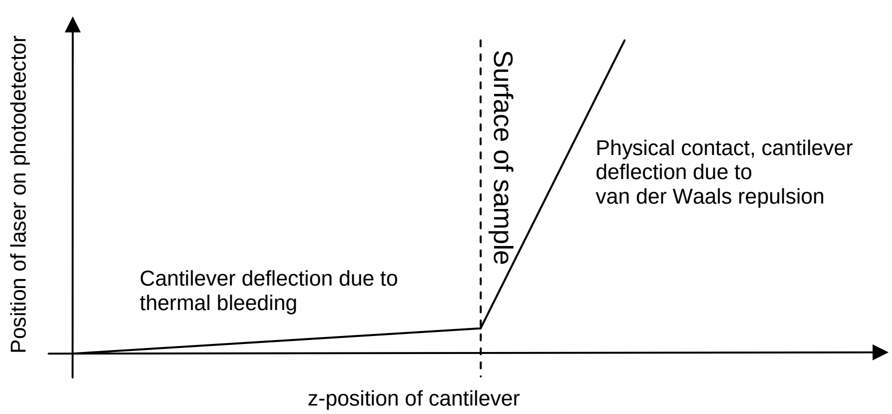


Figure 9 – laser deflection vs. cantilever z-position, showing thermal loss to the substrate

This initial bending due to "thermal bleeding" can sometimes cause the feedback loop to think that the tip is in contact with the surface when in reality it is not. As a result, there are some occasions when one may approach, go into feedback, and initiate a scan, but the tip will not actually be in contact with the surface. In order to bring it into contact, it is necessary to extend the z-piezo via increasing the setpoint.

2.4.2 Active Pens Approach Procedure

2.4.2.1 Selecting A Feedback Pen

Due to an idiosyncrasy in InkCAD (v3.2.17) internal updating routines, it is necessary to perform a convoluted series of steps to set the feedback pen.

Make sure the correct pen (in pen settings) is selected for feedback!

1) Select the correct pen array in the pen settings dialog (T1, T2, or T3).
2) Select the control tab.
3) Check the box for the feedback pen, and then click the small pencil icon in the left margin of the panel to “write out” the data.
4) Close InkFinder.
5) Close the pen settings dialog.
6) Reopen the pen settings dialog.
7) Reopen InkFinder.
8) The correct feedback pen should now be shown in "align pens."

This sequence of steps must be followed exactly. Otherwise the correct feedback pen will not be displayed in the “Align Pens” tab, and this will lead to incorrect scanner behavior, placement, and imaging in the wrong locations. The following workaround is necessary, and the exact order of these steps must be followed.

Additionally, if a user is capturing images (video or SPM), or performing alignment routines, these specifically reference the feedback pen at that time, and are thereafter pegged to that pen for alignment purposes. This reference is used to place video and SPM images with respect to one another in the stage map, allowing the user to navigate more easily. Changing feedback pens midstream will result in incorrect alignments, positioning, and imaging/lithography locations. The same holds if one opens an image database that was saved referencing a different feedback pen.

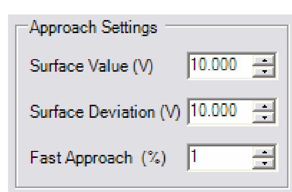
2.4.2.2 Approaching the Surface

a) Turn off the laser and manually approach the surface using the Z-All motor command. Get to within ~50 µm of the surface and then start moving in smaller increments (~5 µm) until a *slight* deflection of the extended reader pen is optically evident.

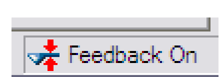
b) Withdraw from the surface 20 µm.

c) Turn the laser back on, and double-check the photodetector alignment. The red dot will likely have moved up, so reposition it to the bottom of the grey rectangle.

d) Under “SPM Setup” → “Settings,” set the approach setting values seen at right:



e) Click “Approach.” The system should successfully go into feedback. If not, repeat a) through e). (Because of the thermal bleeding effect, it’s possible for the system to abort the approach attempt, or go into false feedback.) If the feedback loop turns on, as indicated by the icon at right, continue to f).



f) The *quality* of feedback now depends on whether the tip is truly in contact with the surface. Initiating a 1 µm scan, 1 Hz, at (-30, 30) is an easy way to verify this. If surface features are readily apparent, the system has established proper feedback and the approach routine is done. If the image is ambiguous or noisy, the tip is likely not in contact. Proceed to g).

g) Ensure that the digital multimeter (DMM) is connected to the Z(s) channel 7 of the signal access module; this provides a measurement of the z-piezo extension. The sensor for the z-piezo ranges from ~7.5 V when fully extended to roughly ~2.2 V when fully retracted. For optimum imaging and lithography, the z-piezo should be centered within its dynamic range. Monitoring Z(s) will enable the user to maintain this condition by adjusting the setpoint accordingly.

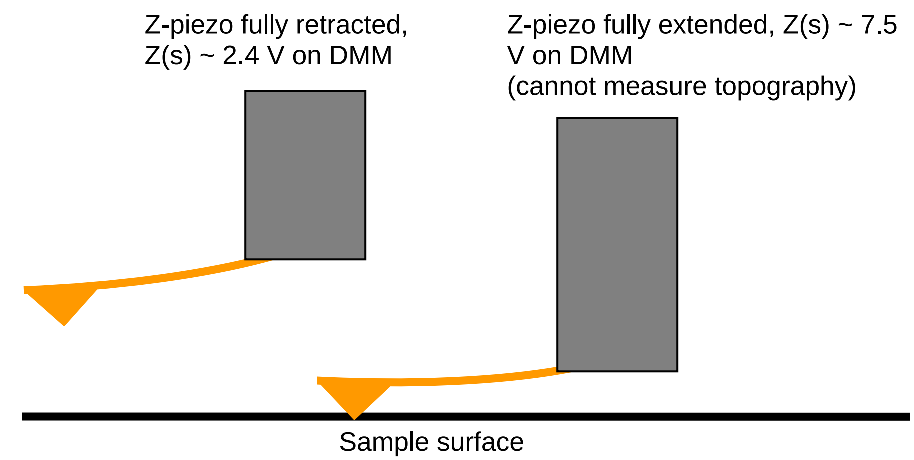


Figure 10 – z-piezo extension as measured by the Z(s) signal.

h) While still observing an ambiguous or noisy image in a continuous scan, monitor the Z(s) [MON 7] reading and begin to slowly increase the setpoint. The Z(s) reading should increase accordingly.
The z-piezo is a piezoceramic material which is attached to the cantilevers and controls their vertical position. You can control the extension of the z-piezo by applying additional setpoint. Depending on the pen, you may need to apply varying setpoints.
The goal is to observe surface features (and thus establish good contact) prior to maxing out the Z-piezo extension (measured by hitting the max value of the Z(s) signal). It is important to keep the Z-piezo near the middle of its working range. If the Z-piezo becomes fully extended, it can only sit at its maximum position and has no ability to move up or down to respond to surface features (Fig. 10); the topography channel will then go blank (no data to report).

i) If features are observed, proceed to j). If not, repeat a) – h).

j) Carefully note the cantilever current and system setpoint necessary to achieve contact with the surface. These parameters are vital to maintaining surface contact with Active Pens, and subsequent imaging or lithography.

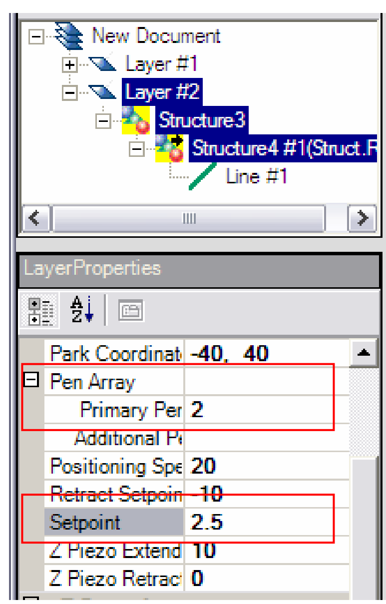


Figure 11 – layer design properties showing the given layer set to pattern with pen 2 with a setpoint of 2.5 V.

For imaging: it will be necessary to use the same extension current and setpoint throughout the rest of the experiment (i.e., the same session of being in feedback).
For lithography: it will be necessary to adjust the extension current of the other writer pens, ***and*** use the noted setpoint in the layer properties of the InkCAD design. See Fig. 11.

Note #1: Different pens within an array may require different setpoints to bring them into contact. For example, see the following example necessary currents and setpoints required to get particular pens to establish "good" contact.

Table 1

Pen number	1	2	3	4	5	6	7	8
Current to get it in photodetector range	0.04	0.05	0.04	0.05	0.04	Dead	0.04	0.04
Setpoint needed to obtain physical contact	3 V	0 V	0 V	0 V	0 V	Dead	0 V	3 V

Note #2: Only one pen can be extended during imaging – this is the selected feedback pen. It is not possible to have another pen actuated while imaging with the feedback pen. There are only two instances when the system will allow more than one pen to be actuated at once (while in feedback):
1) tip to tip alignment – all pens selected for alignment will actuate
2) lithography – the feedback pen stays in feedback the whole time, and as needed subsequent pens will extend and retract to pattern their designated layer

As soon as a scan is initiated, all pens besides the feedback pen will be automatically retracted.

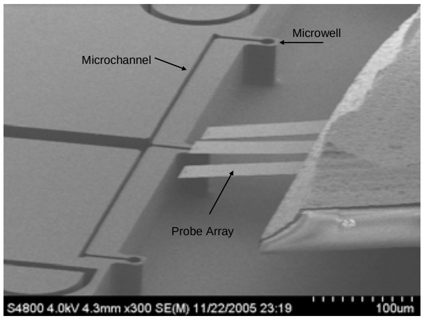
Note #3: The recommended operational current ranges for Gen2 Active Pens are 20 – 30 °C. Currents in this range should ensure tips that are cool enough to pattern a wide variety of substances.

3.0 Aligning, Inking, and Calibrating Active Pens

3.1 Background: Selective Ink Delivery Mechanisms

Since every ink behaves slightly differently, you will need to determine an appropriate formulation. When exploring new inks for InkWell delivery, it is a good idea to perform several tests on the ink prior to attempting to write with it. First, drop a small amount onto a glass slide and see how long it takes the ink to evaporate. Inks that are too volatile and evaporate within less than a minute will be difficult to deliver using Inkwells, and you may need to change your formulation. Before attempting to deposit inks with active pens, we recommend that you practice with passive pens.

Extensive information on InkWells can be found in the InkWells datasheet. Briefly, the inkwell reservoirs supply inks to the microwells by capillary wicking in micron-sized conduits, or "microchannels." The microchannels run from individual reservoirs (at the periphery of the die) to the center of the die, where they feed the microwells. The tip is then dipped into the microwell, as shown below. We recommend that you dip "cold"—that is, use the stage z-motors to lower the tips into the microwells rather than actuating the cantilever by driving current. This is because the additional heat from actuating the cantilever may cause have an undesirable effect on the inking process.



It is important not to deliver too much ink, as this will cause the ink to spill out over the sides of its well and contaminate the “cliff” area and possible other reservoirs. To help prevent the ink from wicking outside of its intended reservoir, the regions surrounding NanoInk microwells are coated with a hydrophobic coating. However, keep in mind that this hydrophobic coating is only helpful if the ink solvent is hydrophilic; this prevention mechanism will not be as helpful for inks in hydrophobic solvents. Finally, we recommend that you practice inking passive pens before inking active pens.

3.2 Selective Ink Delivery Procedure

1) Micropipette Practice: You will need a micropipette to deliver the ink to the reservoirs. If you are unfamiliar with micropipettes, practice several times with water to try and dispense the correct amount of liquid. Touching only the top of the liquid ink to prevent picking up unwanted droplets, suck between 0.10 and 0.25 μL into a fresh pipette tip. Practice inking the inkwells on an old inkwell several times, finding just the right amount so that your ink does not run over the sides, and inspect under an optical microscope. There should be no traces of ink in the cliff region of the InkWell. If there is, we recommend that you do not use the InkWell, as you may contaminate adjacent tips during inking.

2) Prepare the Inking and Writing Areas: Once you are satisfied with your ability to deliver ink to the inkwells, place the inkwell on the sample puck. Keep in mind that the entire cantilever chip will come down with the cantilevers, and it is therefore desirable to face the InkWell cliff away from the writing substrate.

3) Before pipetting the ink into the microwells, navigate to the wells you wish to deposit in, noting which letters (A-F) you have aligned to. Perform a video capture of the location where you wish to write and the location of the InkWell reservoirs where you will ink. This will allow you to navigate help to move quickly and ink the tips before the ink evaporates from the reservoir. In addition, make a small mark on the base of the large removable sample puck and the portion that remains in the microscope. This will allow you to maintain a rough rotational alignment of the puck when you remove and replace it.

4) Position the cantilevers over the InkWells and practice lowering the cantilevers into the microwells until you are comfortable. You will need to the x, y and z motors to lower the tip into the microwell. Because the motors work on a screw mechanism, they precess as they are lowered down in z. That is, there will be some x and y motion coupled to the z motion. Thus, you will need to constantly adjust x and y as you lower z. In addition, because of the accuracy of the stage, use a minimum step size of 3-5 μm; smaller steps may cause the stage not to move and it will lose track of where it is.

5) Retract the tips using Z-all 2000 microns. Remove the sample puck, and wearing gloves, dispense the previously optimized amount of ink (between 10-30 μL) into the microwell of choice. Make notes of which ink is dispensed into which microwell for future reference. Carefully reinsert the puck and rotate to align the mark you made before at the base of the puck and the permanent stage. Click on the microwell position to relocate the first microwell.
6) Slowly lower the cantilever into the microwell, moving in 5 micron increments in x, y and z as necessary. Lower the tip until the capillary force from the liquid draws the tip down. Finally, retract the cantilevers in one large z-step of ~50-100 microns; this big retract helps prevent cross-contamination to other cantilevers.
7) Wait until the liquid evaporates. You may find that you need to “re-dip” the tip to coat it with more ink—you will have to explore this and optimize the number of dips for each ink.
8) Repeat as needed for other tips in other inkwells.
9) Withdraw ~1000 microns and navigate to your substrate for writing. Bring the system into feedback using the procedure described previously.

Note: For enhanced ink delivery – and to further reduce chances of ink wicking and cross contamination – use the procedure for “Fast Dipping,” see Appendix B. Keep in mind, however, that this will introduce additional heat to the inking process.

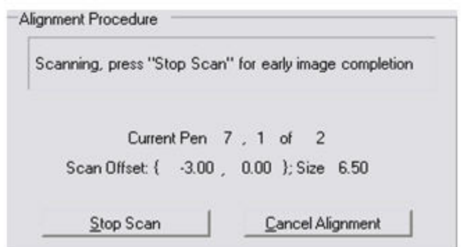
3.3 Aligning Active Pens

Now that you have inked each pen, you will need to perform a “tip to tip alignment”—that is, you will align each tip spacing with respect to the feedback pen precisely. To do this, you will go down a write an **X** alignment mark with each pen you wish to write with. You will then image this mark with the feedback pen and precisely align to it by clicking at the center of the **X**. This will help you to determine your available writing area as well. Keep in mind that the scanner can only move a set amount about the center of the pen array; thus, the region of the sample reachable will on the number and spacing of the pens selected.

Use the following procedure to align a pen array. Before starting the procedure, you need to open the Instrument window, select the pen array type you want to use, and select and ink the appropriate pens.

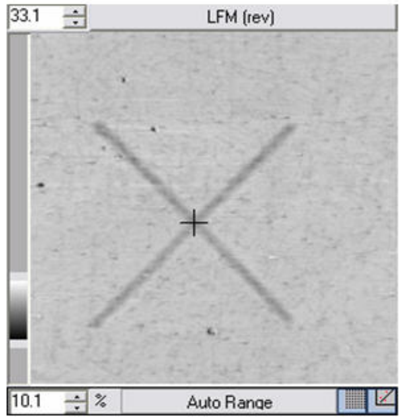
1. Go to the Align Pens tab and click on the Start Alignment button. For active pen arrays, InkCAD extends each selected pen in turn. The system draws alignment marks with the selected pens, displaying this status message:

2. When it finishes drawing all the alignment marks one after the other, InkCAD immediately begins performing an SPM scan for one of them:



   On the left side of the Instrument window, the SPM Images tab opens, displaying the standard set of contact-mode scan images. These images have the controls described in the Chapter 6 section on SPM scanning – you can enable or disable the auto range feature, open an image in its own window, right-drag over a region to start a new scan, modify the color range, etc. You can also modify the scanning parameters in the SPM Controls tab (on the right side of the Instrument window).

3. When the scan is done, you should be able to see the tip-to-tip alignment mark in one or more of the images. Specify the location of the alignment mark by clicking once at the intersection of the two lines (the mouse pointer changes to a "+" sign):



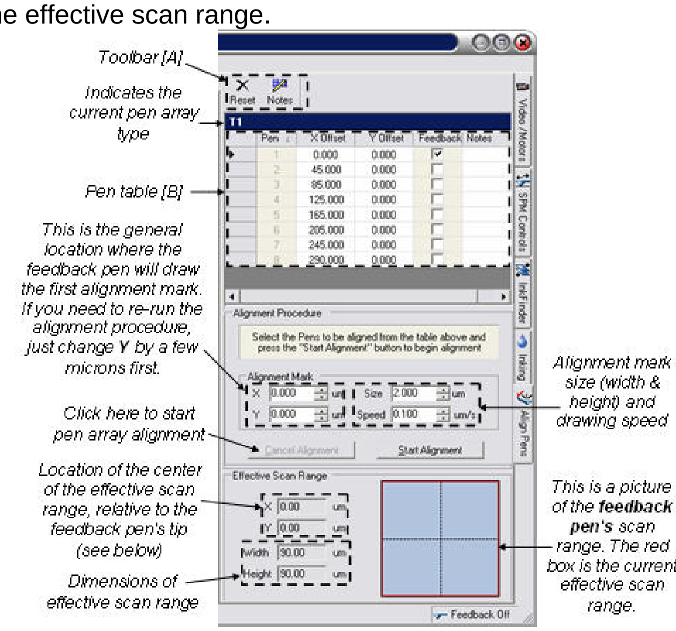
Then click on the Next Pen or Done button (depending on whether you're working with the alignment mark for the last pen) in the Align Pens tab. InkCAD calculates the alignment offsets for the pen and starts scanning the next mark (if there is one).

(If an alignment mark does not appear in the scans, you can adjust the scan parameters and repeat the scan. To scan a smaller area, right-drag over a region in one of the scan images, then right-click inside the box and edit the scan parameters as needed. To scan a larger area, go to the SPM Controls tab on the right side of the window. Adjust the scan parameters as needed and click on the Scan button.)

4. If this is not the last writer pen to align, return to step 2 above. Otherwise, the array alignment process is done.

About the Align Pens tab

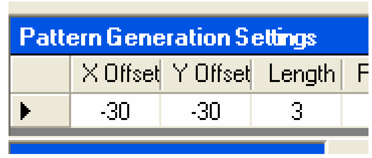
You use the Instrument window's Align Pens tab to perform a pen array alignment and estimate the size of the effective scan range.



3.4 InkCal for Each Tip – Calibrating Ink Diffusion for Active Pens

You will need to perform an InkCal for each of the inks that you are writing with. In addition, slight differences in current in the writing pen current will change its temperature, and this will likely strongly affect ink diffusion rates[2]. We therefore strongly recommend that you calibrate each tip, regardless of whether or not the same ink is used. InkCal can be performed exactly as it would for a normal passive pen, with a few minor changes.

1) Make sure that the system is in feedback. Then, in InkCAD, specify a layer for each pen you will be writing with. When calibrating for each layer you will need to specify the layer—this is how you will select each pen in InkCal.
2) Open InkCal, and specify which pattern you wish to write.
3) First make sure that where you are attempting to write the pattern is within the range that can be reached by the scanner. Otherwise, you may write your pattern but never be able to image it, or vice versa. Change the location in the x and y offsets accordingly, in the portion of the screen to the left.

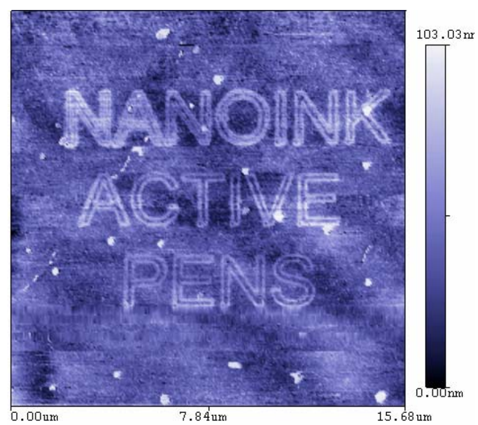


4) Specify the extension current in the pen settings dialog.
5) Specify the pen to write, and the setpoint at which to write it. See step h in “Approaching the surface.”
6) Execute the InkCalibration, and click on scan as you normally would to image the pattern. Measure feature sizes and perform curve fit to the diffusion. For a more complete description of InkCal, see Chapter 4 of the Users Guide.
7) Repeat as necessary for each pen. Save the InkCalibration for each pen with a unique name. You will need to recall this information when executing patterns with each tip.

4.0 Using Active Pens – Multi-Layer, Multi-Ink Lithography

Once you have performed tip to tip calibrations and InkCals for each tip, you are ready to write your patterns. Many of the features of the NSCRIPTOR which you will use in writing multiple inks with Active Pens are similar to the multi-layer writing you may have done with passive multi-pen arrays. Please refer to the DPN User Guide Chapter 8, as needed.

4.1 Multi-Layer Design & Execution



Multi-layer design for Active Pens is accomplished much as it is for passive pens. Keep in mind that you will need to specify in the later properties the pen, its InkCal ink diffusion coefficients, and the setpoint necessary to bring it into contact with the surface, as described in section 3.3 of this document. For a complete description of multi-layer design and InkCAD, see the DPN User’s Guide Chapters 3 and 5.

Be sure to specify the currents at which you would each pen you are writing with to actuate in the Pen Settings Dialog. Input the actuation current and setpoint you measured in first aligning and bringing the pens into feedback. Again, be careful to specify the pattern within the scanner ranges for the pens selected. Execution of the multi-layer pattern you have created is as simple as selecting the layers you wish to write and selecting go.

Once you have written the pattern, you can scan the area by right clicking to select the pattern in InkCAD and clicking on scan. The NSCRIPTOR will go and image the area with the un-inked reader pen. An example of a pattern written with an inked pen and read with an un-inked pen is shown in the above figure.

4.2 Imaging the Results Non-Destructively

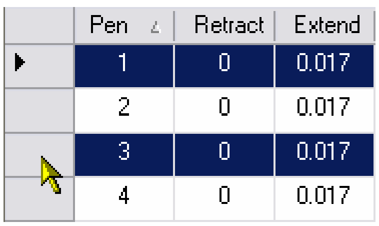
If you wish to image the results non-destructively, with AC AFM, you will need to perform a “layer to layer” alignment so that you are able to return to the same location after you have switched tips. This process is described in pages 45 and 46 of the "Getting Started" Users Guide. AC AFM is easy to perform on the NSCRIPTOR and is described in detail in Appendix C of the DPN User's Guide.

5.0 Appendices

Appendix A: Software Peculiarities

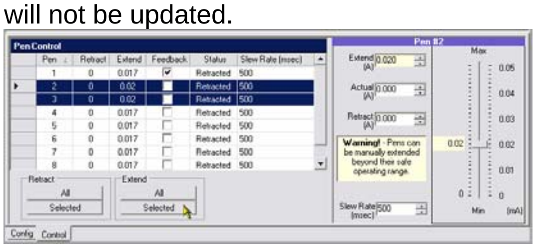
<u>To change a parameter value:</u> Hit enter after changing a value. Listen for gong sound. If you don't hit enter, the value will not change, and you may become confused.

<u>To select a different Active Pen:</u> Click on the left side to create a dark gray/blue highlight, as seen below.



	Pen	Retract	Extend
▶	1	0	0.017
	2	0	0.017
	3	0	0.017
	4	0	0.017

<u>To change the current on an Active Pen,</u> you will need to hit the extend button again, or the new value will not be updated.



<u>To change the designated feedback pen:</u> If the system is in feedback, take it out of feedback, as you will not be allowed to change feedback pens while in feedback. As above, click on the left side to create a dark gray highlight, and change which box is checked under the feedback column. Load a fresh database into the instrument window, otherwise the program will default to the old feedback pen when you reopen it. Close the pen settings dialog and the instrument dialog, and then reopen them. You should now see a different feedback pen selected. This peculiarity of the system has to do with the alignment programs, which require a single reference point.

<u>To align a laser after just closing and reopening the instrument window,</u> you will need to first move the focus or Z-position motors. This is because the laser spot will initially appear to be dead, as the instrument has not updated the appropriate value to the user interface. Moving the focus motors reminds the controller to communicate the new values to the user interface. This is a software glitch that we are in the process of fixing.

Appendix B: <u>Active Pens – "FastDip" Protocol</u>

1. Mount an active inkwell (with "cliffs") near your sample on the sample puck.
2. Use the metal plate sample puck holder to keep the puck steady.
3. Use a micropipette, and draw 0.1 µL of Ink/Solvent.
4. Deposit this by hand into one of the inkwell reservoirs.
5. Make sure the Inkwell substrate is aligned such that the cantilevers are lengthwise perpendicular to the edge of the cliff. Using z-motors, get within ~ 50 µm of the inkwell substrate surface.

6. Navigate so that the tips are in line with the microwells.
7. Move +Y, so that the tips no longer go into the microwells, but hit the surrounding surface first. Use X adjustments if necessary.
8. Use the z-motors to bring the tips within ~5 µm of the surface.
9. Use small z-motor steps to move the tips down until you see a small amount of deflection. Back up 1 µm (+Z). (Cantilevers are still unactuated)
10. Use the X-Y stage to position the desired tip above the desired microwell. There should be no wicking, or tip/ink interaction yet.
11. Go to the Inking tab, and select Active Pen fast dip. Select the pen #. Make sure that the Pen Settings dialog has an "extend" value of 28 mA for the desired pen.

Click "FastDip." Your pen is now inked.

Appendix C: Cantilever Specifications

Table 1: Cantilever Specifications

Probe Type	Writer *k* (N/m)	Writer width (µm)	Writer length (µm)	Writer-Writer pitch (µm)	Writer-Writer gap (µm)	Reader *k* (N/m)	Reader width (µm)	Reader length (µm)	Reader-Writer pitch (µm)	Reader-Writer gap (µm)
T1	0.18	30	150	40	10		40	150	45	10
T2	0.15	26	150	30	4		40	150	37	4
T3	0.11	18	150	23	5		30	150	29	5

Heat spreader distance from tip ~ 80 µm
[T1 arrays are available for ale. T2 and T3 arrays are in development.]

For more information including pricing, please contact NanoInk Sales Department at sales@nanoink.net or 1-847-679-NANO.

Cited References

1. Hong, S.H., J. Zhu, and C.A. Mirkin, *Multiple ink nanolithography: Toward a multiple-pen nano-plotter.* Science, 1999. **286**(5439): p. 523-525.
2. Rozhok, S., R. Piner, and C.A. Mirkin, *Dip-pen nanolithography: What controls ink transport?* Journal of Physical Chemistry B, 2003. **107**(3): p. 751-757.