APPENDIX C

AFM Standards and References

Introduction

Standards and references are required for proper operation of an AFM and to verify optimal operating conditions and calibration of the instrument. Standards are used to assure that the absolute measurements are correct, while references assure that the instrument is giving consistent results.

For establishing calibrated topography measurements, the AFM scanner must be certified with calibration standards having pre-established dimensions in the X, Y and Z axis. References can be used for establishing that an AFM mode is operating correctly and for establishing the proper operation of the AFM.

This guide serves as a reference for finding standards and references that are helpful for making meaningful measurements with an AFM. When possible several vendors for a particular type of standard are listed.

Z Calibration

Calibration standards/references are needed to calibrate SPM in the vertical axis. For calibrations greater than 10 nm step height, standards or references are typically fabricated by etching patterns in a quartz substrate. Another source for references is etched silicon, or silicon dioxide coated with a uniform layer of metal. When calibrating the instrument for Z height measurements below 10 nm, nano-spheres, atomic terraces of silicon or HOPG may be used as a reference specimen.

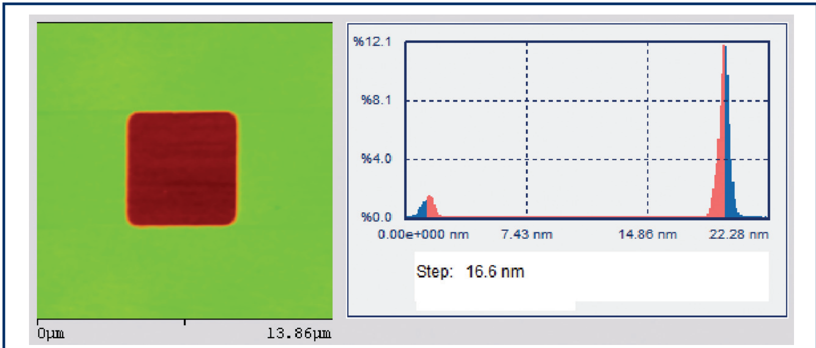


FIGURE C-1 This AFM image is of a single etched pattern of an AFM standard. The histogram at the right show the height of the feature is 16.62 nm.

Company	Standard / Reference
VLSI Standards www.vlsi.com	Standards Z=18nm, 44nm, 100nm, 180nm Pitch=1.8µm, 3µm, 5µm ,10µm, 20µm References: Z=18nm, 44nm, 100nm, 180nm Pitch=3µm,10µm
MicroMasch www.spmtips.com	Z=20÷25nm, 90÷104nm, 496÷503nm, 1000nm, 15000nm Pitch=3µm,10µm HOPG all grades
SPI Supplies www.2spi.com	Pitch=300nm, 700nm Z~100nm
Ted Pella www.tedpella.com	Pitch=0.463µm Z~31nm
Pacific Nanotechnology www.pacificnanotech.com	Pitch=20µm, 10µm, 5µm, 3µm Z~75nm
Electron Microscopy Science www.emsdiasum.com	Pitch=150nm, 300nm, 700nm Z~100nm
NTT-AT www.ntt-at.com	AtomicStep=3.1nm

XY Linearity / Calibration / Orthogonality

Calibration of the AFM in the XY axis so that meaningful dimension measurements can be made requires establishing the linearity and orthoganality of the AFM scanner. This is typically done with etched

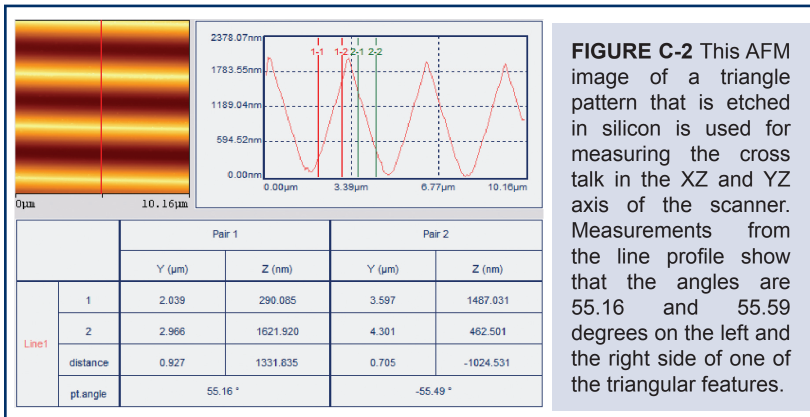
patterns in quartz or silicon. Often the specimen that is used for calibrating the Z axis can be used for the XY axis as well.

Company	Standard / Reference
VLSI Standards www.vlsi.com	Standards Z=18nm, 44nm, 100nm, 180nm Pitch=1.8µm, 3µm, 5µm ,10µm, 20µm References: Z=18nm, 44nm, 100nm, 180nm Pitch=3µm,10µm
MicroMasch www.spmtips.com	Pitch=3µm, 10µm Z~1µm, 2µm
Pacific Nanotechnology www.pacificnanotech.com	Pitch=20µm, 10µm, 5µm, 3µm Z~75nm

XZ, YZ Orthogonality

An AFM may be calibrated in XY and calibrated in Z and may not be useful for making angular measurements. This is because the XZ and YZ axis may not be orthogonal. With the orthogonality references, this problem can be avoided. This reference is fabricated by making a 1-D array, or line, of triangles in a silicon wafer.

Company	Standard / Reference
MicroMasch www.spmtips.com	Pitch=3.0µm ±5nm Triangle edge curvature<10nm Top angle=70deg



		Pair 1		Pair 2	
		Y (µm)	Z (nm)	Y (µm)	Z (nm)
Line1	1	2.039	290.085	3.597	1487.031
	2	2.966	1621.920	4.301	462.501
	distance	0.927	1331.835	0.705	-1024.531
	pt.angle	55.16 °		-55.49 °	

FIGURE C-2 This AFM image of a triangle pattern that is etched in silicon is used for measuring the cross talk in the XZ and YZ axis of the scanner. Measurements from the line profile show that the angles are 55.16 and 55.59 degrees on the left and the right side of one of the triangular features.

Flatness / Roughness

An AFM is capable of measuring surface roughness of samples at the nanometer scale. However, it is important to establish that the surface roughness measured with the AFM is not the noise floor of the instrument. Also, when measuring flat samples, it is important to know the “bow” associated with the scanner. These references are useful for establishing the performance of the instrument with respect to noise floor and bow.

Company	Standard / Reference
NT-MDT www.ntmdt.com	Sa=0.06nm
SPI Supplies www.2spi.com	Atomic corrugations on HOPG

Particles

SPM height calibration can be done on nanospheres of uniform size; it also verifies SPM performance on samples with weak adhesion to the surface. Often it is helpful to include nanoparticles with a sample to establish the sizes of features in the imager relative to the nanoparticles.

Company	Standard / Reference
Duke Scientific Co. www.dukescientific.com	20<D<900nm Traceable and certified polymer spheres come in dry powder and aqueous suspensions in a great variety of sizes.
TedPella Inc. www.tedpella.com	D=5nm D=15nm D=30nm AFM gold standard kit consists of three sizes of gold colloids in aqueous suspensions.
American Dye Source Inc. www.adsdyes.com EvidentTechnology Inc. www.evidenttech.com	All colors of q-dots. Quantum dots of any size come in great variety of kits

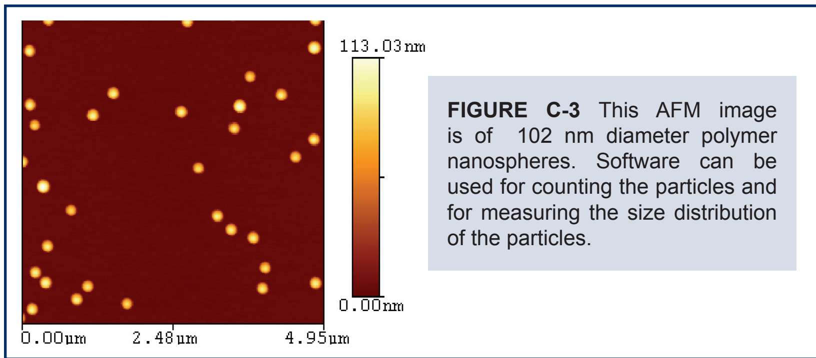


FIGURE C-3 This AFM image is of 102 nm diameter polymer nanospheres. Software can be used for counting the particles and for measuring the size distribution of the particles.

Bio-Molecules

Often it is necessary to verify SPM image quality on soft samples or samples with weak adhesion to the surface. It is important that this type of reference have a long-life, be stable and be indestructible. Before measuring images of "unknown" biological molecules it is often helpful to practice on this "known" sample.

Company	Standard / Reference
NTI-Europe www.ntinstruments.com	Z=2nm Length~1009nm

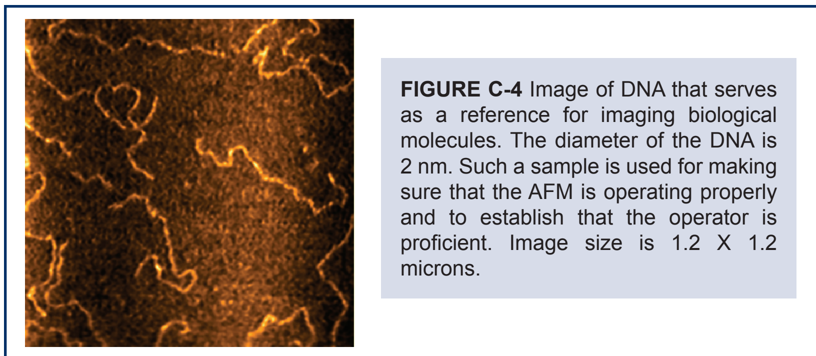


FIGURE C-4 Image of DNA that serves as a reference for imaging biological molecules. The diameter of the DNA is 2 nm. Such a sample is used for making sure that the AFM is operating properly and to establish that the operator is proficient. Image size is 1.2 X 1.2 microns.

Friction Mode

LFM reference comes as 1-D array of triangular steps having precise linear and angular dimensions. Establishing quantitative LFM data requires standardizing the AFM scanner output in the vertical and horizontal axis.

Company	Standard / Reference
MicroMasch www.spmtips.com	Pitch=3.0µm ±5nm Edge curvature<10nm Top angle=70deg

Vibrating Phase Mode

In vibrating mode, the AFM can measure changes in chemical composition of polymer samples. A reference of polymer material that has hard and soft regions is used for this purpose.

Company	Standard / Reference
Electron Microscopy Science www.emsdiasum.com	As small as 10nm

Tip Visualization

Tip Visualization helps visualize the geometry of the scanning probe without the costly need of SEM. Tip radius sample exhibits features sharper than an AFM tip. First-order approximation of the tip apex can be obtained.

Company	Standard / Reference
Aurora NanoDevice Inc. www.aurorand.com	Z~30nm X,Y~10nm

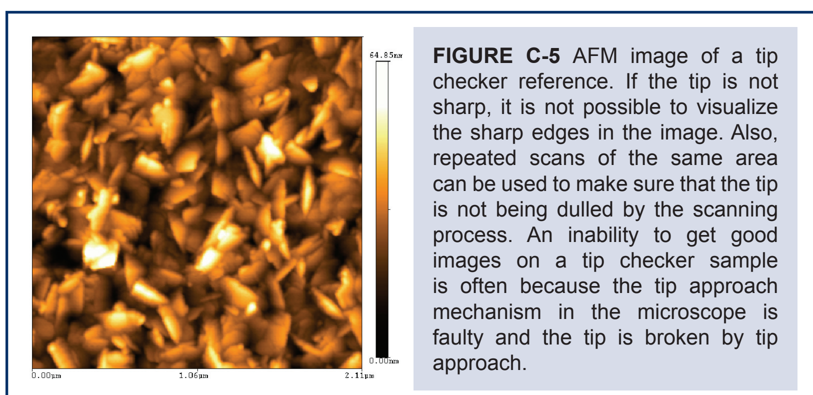


FIGURE C-5 AFM image of a tip checker reference. If the tip is not sharp, it is not possible to visualize the sharp edges in the image. Also, repeated scans of the same area can be used to make sure that the tip is not being dulled by the scanning process. An inability to get good images on a tip checker sample is often because the tip approach mechanism in the microscope is faulty and the tip is broken by tip approach.

Conclusion

Making consistent, or even quantitative, measurements with an AFM requires diligence and the appropriate standard or reference. This partial list of standards/references can be used for making consistent or quantitative measurements.