Data Report for University of Illinois



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Topography analysis with atomic force microscope using AAC imaging mode

The following samples are imaged using Agilent 5500 AFM with AAC mode:

1. BFO-LSMO-STO

- 2. CCSTO
- 3. CeO2
- 4. Nano wires (Ketaki)
- 5.

AAC imaging mode:

Using a vibrating cantilever, the AFM tip is driven to oscillat near its resonance frequency (~300KHz) while engaging the sample surface. Depending on the driving power and setpoint, the very end of the sharp tip lightly "tap" the sample surface at varying force to minimize the damage and disturbance of the sample. The scanner feed back on the vibration amplitude of the oscillating to maintain such light force in order to obtain accurate high resolution image of the sample topography information. At the same time, the phase, or timing of the cantilever oscillation reveals the sample's physical stiffness information.

AAC imaging mode is the ideal technique when obtain precise topography information. After imaging processing software as well as mathematical metrology analysis tools allow user to gain rich collection of surface information, such as RMS roughness, surface texture, grain sizing statistics, grain surface coverage etc. A 3D rendering of the topography give us a very detailed lifelike view of the sample under the microscope and allow us to perform detailed measurement from the data.



BFO-LSMO-STO March 22nd sample 10x10 micron scan roughness and texture analysis









Grain size statistics

ISO 2	ISO 25178				
Height	Height Parameters				
Sq	25.8 nm Root mean square height				
Ssk	0.472		Skewness		
Sku	2.26		Kurtosis		
Sp	104	nm	Maximum peak height		
Sv	62.9	nm	Maximum pit height		
Sz	167	nm	Maximum height		
Sa	22.0	nm	Arithmetic mean height		

Cursor profile across center of image

2

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4

5

6

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8

9μm

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20 0

BFO-LSMO-STO March 22nd sample 10x10 micron scan grain analysis and statistics



BFO-LSMO-STO March 22nd sample 10x10 micron scan roughness and texture analysis









Histogram analysis (height statistics)



ISO 25178				
Height	Parameters	;		
Sq	41.0	nm	Root mean square height	
Ssk	-0.0369		Skewness	
Sku	1.94		Kurtosis	
Sp	127	nm	Maximum peak height	
Sv	111	nm	Maximum pit height	
Sz	238	nm	Maximum height	
Sa	35.5	nm	Arithmetic mean height	

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BFO-LSMO-STO March 22nd sample 10x10 micron scan grain analysis and statistics











Mean parameters on 2004 grains

Number of grains: 2004 Total area occupied by the grains: 65.7 µm² (65.7 %) Density of grains: 20.0 grains / µm2.

Area		0.0328 µm ²	+/- 0.042 μm ²
Perimeter		801 nm	+/- 484 nm
Form factor		0.507	+/- 0.150
Aspect ratio		3.82	+/- 3.14
Roundness		0.427	+/- 0.204
Compactness		0.637	+/- 0.145
Compactness	=	0.637	+/- 0.145
Orientation		87.4°	+/- 55.7°

0



For demonstration purposes only! ISO 23178 Roughtness measurement

BFO-LSMO-STO March 21st sample 5x5 micron scan grain analysis and statistics









Mean parameters on 714 grains

Number of grains: 714 Total area occupied by the grains: 19.3 µm² (77.3 %) Density of grains: 28.6 grains / µm2.



For demonstration purposes only! ISO 23178 Roughtness measurement

CCSTO sample 1x1 micron scan grain analysis and statistics



Motif segmentation analysis uses mathematical modeling to detecte individual grain size, diameter, volume and aspect ratio. Statistics model is used to obtain an overview of the sample's grain structure for easy comparison between differennt samples.



Mean parameters on 678 grains

Number of grains: 678 Total area occupied by the grains: 0.789 µm² (78.9 %) Density of grains: 678 grains / µm2.

Area	= 1163 nm ²	+/- 927 nm²
Perimeter	= 154 nm	+/- 73.3 nm
Form factor	= 0.559	+/- 0.127
Aspect ratio	= 2.54	+/- 1.42
Roundness	= 0.496	+/- 0.240
Compactness	= 0.694	+/- 0.117
Orientation	= 101°	+/- 58.6°



CeO2 film on Si sample 2 x 2 micron scan roughness and texture analysis











ISO 25178				
Height	Parameter	s		
Sq	8.71	nm	Root mean square height	
Ssk	0.0637		Skewness	
Sku	2.88		Kurtosis	
Sp	35.1	nm	Maximum peak height	
Sv	32.5	nm	Maximum pit height	
Sz	67.6	nm	Maximum height	
Sa	6.99	nm	Arithmetic mean height	

1.6

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0

0

0.2

0.4

0.6

67

CeO2 film on Si sample 2 x 2 micron scan grain analysis and statistics





Mean parameters on 2064 grains

Number of grains: 2064 Total area occupied by the grains: 2.51 µm² (62.7 %) Density of grains: 516 grains / µm2.

Area	$= 1216 \text{ nm}^2$	+/- 1140 nm²
Perimeter	= 170 nm	+/- 87 1 nm
Form factor	= 0.495	+/- 0.150
Aspect ratio	= 3.66	+/- 2.76
Roundness	= 0.417	+/- 0.205
Compactness	= 0.632	+/- 0.134
Orientation	= 97.2°	+/- 65.7°



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For demonstration purposes only! ISO 23178 Roughtness measurement



Grain coverage detection



Mean parameters on 547 grains

Number of grains: 547 Total area occupied by the grains: 0.802 µm² (80.2 %) Density of grains: 547 grains / µm2.

Area Perimeter Form factor Aspect ratio Roundness Compactness	= 1467 nm ² = 181 nm = 0.500 = 3.30 = 0.459 = 0.663	+/- 1286 nm ² +/- 86.2 nm +/- 0.147 +/- 3.11 +/- 0.190 +/- 0.139
Compactness	= 0.663	+/- 0.139
Orientation	= 89.0°	+/- 59.6°





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For demonstration purposes only! ISO 23178 Roughtness measurement

NbSrTiO film sample 5x5 micron scan grain analysis









Mean parameters on 2750 grains

Number of grains: 2750 Total area occupied by the grains: 15.3 µm² (61.2 %) Density of grains: 110 grains / µm2.

Area	=	5564 nm²	+/- 6249 nm²
Perimeter	=	322 nm	+/- 139 nm
Form factor	=	0.615	+/- 0.126
Aspect ratio	=	2.41	+/- 1.18
Roundness	=	0.485	+/- 0.121
Compactness	=	0.691	+/- 0.0913
Orientation	=	107°	+/- 57.2°

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NbSrTiO film sample 1.5 x 1.5 micron scan roughness and texture analysis









Histogram analysis (height statistics)



ISO 25178				
Height	Paramete	ers		
Sq	0.683	nm	Root mean square height	
Ssk	2.77		Skewness	
Sku	23.6		Kurtosis	
Sp	7.17	nm	Maximum peak height	
Sv	1.60	nm	Maximum pit height	
Sz	8.77	nm	Maximum height	
Sa	0.481	nm	Arithmetic mean height	

NbSrTiO film sample 1.5x1.5 micron scan grain analysis

1.2

1.4 µm

500

+/- 2744 nm²

+/- 168 nm

+/- 0.170 +/- 2.84

+/- 0.368

+/- 0.164

+/- 58.3°

600 nm





"Tom's sample" 2x2 micron scan grain analysis and statistics







Mean parameters on 1194 grains

Number of grains: 1194 Total area occupied by the grains: 2.85 µm² (71.2 %) Density of grains: 299 grains / µm2.

Area Perimeter Form factor	= 2385 nm ² = 234 nm = 0.463 = 4.22	+/- 4033 nm ² +/- 204 nm +/- 0.165
Aspect ratio	= 4.32	+/- 3.66
Roundness	= 0.381	+/- 0.186
Compactness	= 0.599	+/- 0.151
Orientation	= 87.8°	+/- 59.6°



t "Tom's sample" 2x2 micron scan grain analysis and statistics







All furrows are displayed.

Maximum depth of furrows : 17.5 nm Mean depth of furrows : 6.70 nm Mean density of furrows : 287232 cm/cm2



For demonstration purposes only!





Mean parameters on 476 grains

Number of grains: 476 Total area occupied by the grains: 0.791 µm² (79.1 %) Density of grains: 476 grains / µm2.

Nano wire sample 4x4 micron topography image





left: topography image 2D up: amplitude image





left: topography image 3D up: phase image below: cursor profile across the image center



Nano wire sample 20 x 20 micron topography image





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For demonstration purposes only!

21 µm

Force indentation with atomic force microscope to measure sample stiffness

The following sample is studies with force indentation to measure the sample stiffness:

1. nanofibers

AFM force indentation:

Using force indentation mode, a stiff AFM probe is pushed into the sample with higher force to allow the apex of the tip to indent the sample. By analying the vertical load vs the depth of the penatration, the stiffness can be calculated.

A stiff AFM cantilever with not sharpened apex is ideal for accurate measurement of the sample stiffness. In such experiment, a 50 N/m, 350 KHz cantilever suite the dual purpose of indentation and AAC mode imaging. Ideally, a non-sharpened tip is suggested for better estimation of the tip shape, which is important in accurate calculation of the sample stiffness. However, the unsharpened the tip also would compromise the topographic resolution a little.

Mathenatical model used here to calculate the Youngs Modules is programmed into the "plug in" feature of the PicoView software. In order for accurate mearuement of the nanofiber stiffness, an "volume" indentation is carried to measure an matrix of points, Youngs Modules is automatically calculated with the "plug in", and displayed as an low pixel image. Youngs Modules of the nanofiber can be read out of pixel on top of the nanofiber.



Force indentation analysis of nanofiber on aluminum foil



left: An 32 x 32 array of indentation is performed and the Youngs Modulus are calculated on each measurement point. An stiffness map is generated. The nanofiber appear to have lower stiffness compare to the substrate aluminum.

Magnetic Forc Microscopy imaging of nanofiber containing magnetic nanoparticles, mixed with free magnetic nanoparticles

The following sample is studies with magnetic force microscopy (MFM):

1. magnetic nanoparticle contained inside nanofibers, mixed with free magnetic nanoparticles.

Magnetic Force Microscopy

Using a magnetically coated AFM probe, local magnetic field can be imaged. The AFM probe first scan the sample with regular AAC mode to obtain topography image, on second scan, the AFM probe is lifted 50 away from the surface, and hover over the sample surface without touching the sample, the magnetic field would shift the probe oscillation phase depends on the strength and direction of the field. Mapping out such phase shift would reveal the local magnetic field domains.

The magnetic nanoparticles and fiber do not generate strong enough magnetic field, therefore, a uniform magnetic field is placed beneath the sample. The existance of the magnetic nanoparticle cause the disturbance of magnetic field, which is imaged with this MFM image mode.



Magnetic Forc Microscopy imaging of nanofiber containing magnetic nanoparticles, mixed with free magnetic nanoparticles



Magnetic Sample Modulation imaging of nanofiber containing magnetic nanoparticles, mixed with free magnetic nanoparticles

The following sample is studies with magnetic sample modulation (MSM):

1. magnetic nanoparticle contained inside nanofibers, mixed with free magnetic nanoparticles.

Magnetic Sample Modulation (MSM)

To image magnetic nanoparticles, instead of applying a uniform constant magnetic field, we use an alternating magnetic field to modulate the sample. The particle and fiber would respond to the AC magnetic field, and oscillating within its mechanical confine. We then use a contact mode non-magnetic AFM probe to scan the sample, and the oscillating particle and fiber would push to vibrate the probe. Compare to the MFM, where the tip would scan the sample at an separated distance, therefore compromise spatial resolution, the MSM directly measure and image the magnetic response, instead of the magnetic field variations. MSM gives much higher resolution and reveal much more contrast when working with magnetic nano materials.



Magnetic Sample Modulation imaging of nanofiber containing magnetic nanoparticles, mixed with free magnetic nanoparticles



Topography image with contact mode



simultaneous MSM image. Since we use a lock-in amplifier to detect the sample oscillation, the MSM signal not only shows the amplitude of the sample particle and fiber modulation, it also gives phase information. Depends on the mechanical environment, the sample particle and fiber would have its own different natural resonance frequency. when driven at different frequency, which would result in difference in phase shift of the vibration. Here the darker color shows us that there is around 180 degree in phase shift in the sample vibration compare to the driving signal.

Kelvin Force Microscope imaging to study sample electrical property

The following sample is studies with Kelvin Force Microscopy:

1. conductive nanowires

Kelvin Force Microscope:

There are many techniques to measure electrical property. When a sample can be easily grounded, one could use contact mode conductive AFM to measure the conductivity. However, when one need to measure the electrical property of isolated nanowires, one need to choose an AC bias tip techniques. Here we present KFM imaging of electrical property of nanowire array.





Phase ω1 Lock-in #2 mponent nplitude ω2 AC DRIVE KFM Servo Tip Bias Sample Bias Sample Sample Bias DAC

Kelvin Force Microscope imaging to study sample electrical property



Pico Image Expert 6.2.6561