Atomic Force Microscopy for **Nanostructures**:

With the emergence of nanotechnology and nanoscience the investigation and application of nanostructured materials is growing rapidly. By definition, nanostructured materials have at least one dimension that is less than 500 nanometers. The AFM is ideally suited for both visualization of nanostructured materials and for measuring the spatial dimensions of features at the surface of nanomaterials. Nanostructured materials that are measurable with an atomic force microscope include:

Nanocrystals Nanocomposites Nanograins Nanotubes Nanoceramics Nanopowders



GaAs Quantum Dots - These 30 nm diameter GaAs quantum dots are easily imaged with an AFM.

Sample Preparation for Imaging Nanostructures

There are two general types of nanostructured materials that may be scanned with an atomic force microscope. The first category, category I, are structures that are integrated or directly attached to a bulk structure. The second type, category II, are structures that must be attached to a substrate before imaging.

Category I

Category I samples can typically be imaged with an AFM without sample preparation. It is important that contamination resulting from preparation and handling be removed from the sample before scanning with the AFM. If there is residual contamination on the surface, the images of the sample will typically have streak marks.





AFM image of the nanostructure of a silicon nitride film measured in contact mode. The image size is 1.46 X 1.47 microns. Structures with only a few nanometers in horizontal dimensions are easily visualized in this AFM image.

Category II

Because an AFM scans a mechanical probe across the sample's surface, the structure being imaged must be directly attached to the surface i.e. the affinity of the nanostructure for the surface must be greater than the affinity of the nanostructure to the probe. When nanostructures do attach to the probe, the resulting images typically show reduced resolution. Also, if the nanostructure is not rigidly attached to a surface, streaking may occur.

Often nanostuctures naturally attach to surfaces. For example, nanotubes naturally directly attach to many surfaces. They can be moved on the surface but only with considerable shear forces from the probe.



Figure 3



Single walled Carbon Nanotubes produced with a CVD process and an iron catalyst.

Figure 4



Contact mode AFM image of 14 nm diameter colloidal gold spheres on a mica substrate previously coated with a poly-L-Lysine film. Figure 4A shows three spheres on the surface. Figure 4B shows an SEM image of the AFM probe used for measuring the AFM image. Figure 4C is a line profile of the sphere; the measured diameter is approximately 13 nm.

In instances where there is no "natural" substrate, it often helps to use sticky substrates. Many of the common microscopy sample mounting supplies are appropriately covered through the use of double sided conductive carbon discs, sheets, and tapes. However, the unique needs of the AFM probe require some additional accommodation to expose a flat and stable surface. Examples of the two most recently successful techniques are described below. Supplies may be purchased at several microscopy supply venders, but these were purchased from SPI Supplies, Inc., at: www.spisupplies.com.

"Tacky Dot" Slides

One of the easiest substrates for mounting nanoparticles is to use "Tacky Dot " slides which are commercially available. Each slide has an array of sticky material that can be used to bond nanomaterails to the glass slide; see figure 5.

Figure 5



This is an example of a "Tacky Dot" slide. On each slide is an array of sticky material that can bond nanomaterials to the glass slide. "Tacky Dot" sizes are available down to 15μ m in diameter.

Figure 6



This is an optical microscope view of a "Tacky Dot" in an AFM. The "Tacky Dot" is round. At the right of the image is the AFM cantilever. Note that the cantilever is not in optical focus while the "Tacky Dot" is.

Figure 7



Images 7a and 7b are of nanoparticles bonded to a "Tacky Dot". 7A is a 1.4µm X 1.4µm topographical image displayed in a 3D format. Figure 7b is a 700nm X 700nm image measured with material sensing mode. The nanoparticles are easily visualized in the image 7b. The circles in figure 7a and Figure 7b designate the same area of the sample.

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TempFix

TempFix is a mounting adhesive with unique properties that make it suitable for mounting of all types of nanomaterials that do not adhere to a substrate naturally. TempFix is a wax-like material that comes from the vendor in stick form. The technique for using TempFix as a mounting material is rather simple and fast. Simply warm a 15mm diameter metal AFM sample disk to slightly warmer than can be touched by hand, rub the end of the TempFix stick in a spiral to melt a thin and even layer across the surface and allow it to cool.

Figure 8



An optical microscope view of a powder distributed over TempFix.

Figure 9



A 40µm X 40µm area of the TempFix surface with powders embedded in the surface. The average roughness of TempFix is ~25nm with the bright peaks from nanoparticles measuring 170nm in height.

Figure 10



A 5µm X 5 µm AFM scan of the surface on top of the large single grain shown in the optical microscope image of Figure 8.