Nanoparticle Visualization with an AFM

Paul West, Natasha Starostina
Pacific Nanotechnology, Tustin, CA
pwest@pacificnanotech.com

Introduction

It is often said that: "*...a picture is worth a thousand words...*" This can be certainly true for images from an Atomic Force Microscope.

Although we often know that nanoparticles exist and we have a mental picture of what they look like, it is often helpful to be able to "see" the particles to verify that they are morphologically what we imagine they are. When the particles are much less than a micron in diameter, they cannot be directly visualized with an optical microscope. At best, sub micron particles appear as "diffraction spots" in an optical microscope. It is possible to visualize nanoparticles with SEM or TEM. With these electronic beam methods, the particles may appear as 3-D objects when in fact the images are 2-D. It is not possible to directly measure the depth of the particles using SEM/TEM.

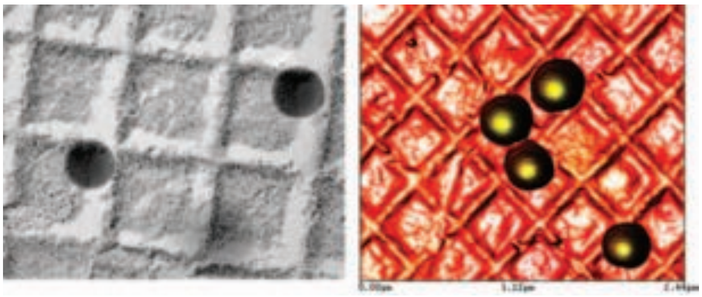

Figure 1: Comparison between TEM (Left) and AFM (Right) images of 261nm latex spheres mounted on a TEM grid. Spacing of the grid is 463 nm.

With the AFM, it is possible to directly visualize nanoparticles with sizes ranging from a nanometer up to 10,000 nanometers. Sample preparation for nanoparticle characterization with the AFM is relatively simple. The ideal substrate will have a clean, flat surface. The nanoparticles are dispersed on the substrate with the nanoparticles having a greater affinity for the substrate than for the AFM probe.

There are many properties of nanoparticles that can best be studied by direct visualization of an AFM image. Examples of such properties include shape, size, dispersion on the substrate and surface texture.

Shape:

When nanoparticles are fabricated, they may be homogeneous or non-homogeneous in phase. Figure 2 shows nanoparticles of a material that were found to have both amorphous and crystalline phases. This is clearly visualized in the image.

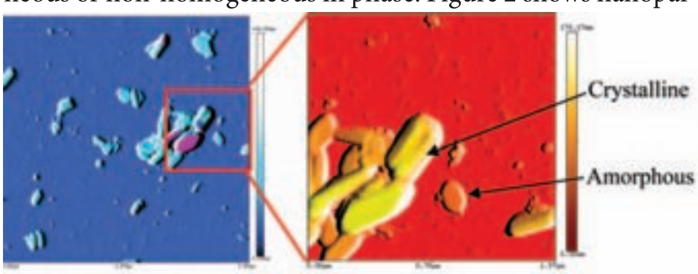

Figure 2: AFM image of crystalline and amorphous phase nanoparticles.

Dispersion

When nanoparticles are dispersed on a surface, they may precipitate into large organized regions, or they may be found as single particles on the surface. An AFM image quickly helps to visualize the level of nanoparticle dispersion. Figure 3 shows images of nanoparticles dispersed on a surface. Some of the nanoparticles agglomerate while others form an organized monolayer.

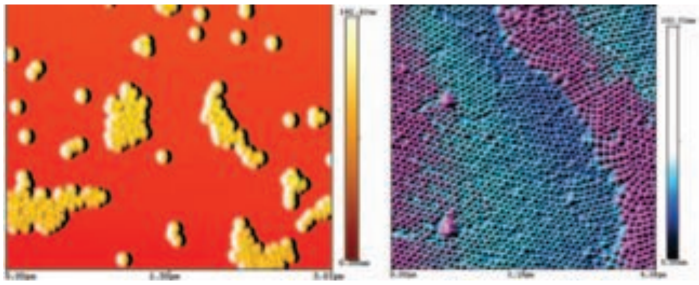

Figure 3: AFM images of 100 nm polymer spheres illustrating agglomerations (Left) and monolayers (Right).

Surface Texture

Surface tension forces cause many nanoparticle surfaces to be very smooth. However, certain processes create nanoparticles with substantial texture. For example, the rough texture could be facets of a crystalline phase. To make meaningful surface texture measurements, the AFM probe has to have a diameter smaller than the features being measured. Typically the diameter of the probe is 20 nm so it is difficult to study surface texture on nanoparticles that are less than 100 nm in diameter.

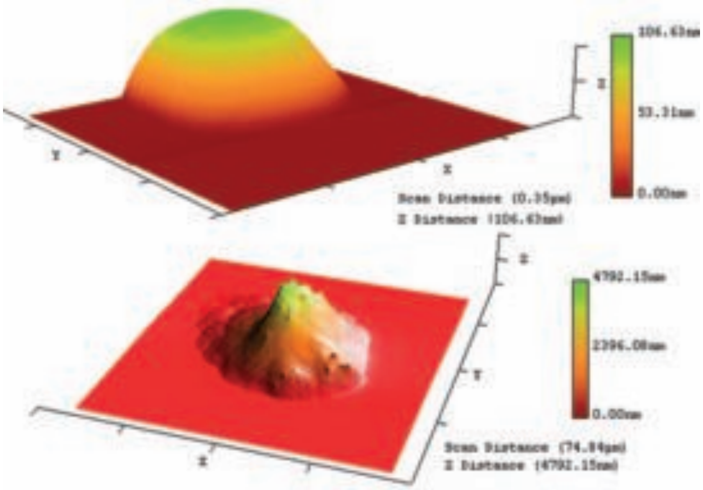

Figure 4: Surface morphology: smooth versus rough particle. On the top is a 3-D image of a 100nm polymer sphere; the bottom image shows a milk powder nanoparticle, ~5 micron.

Relative sizes

Relative sizes of nanoparticles can be characterized quantitatively. Figure 5 compares AFM images of nanoparticles having different diameters. The ratio of the numbers of particles at a given size is immediately apparent by AFM visualization and study of the image.

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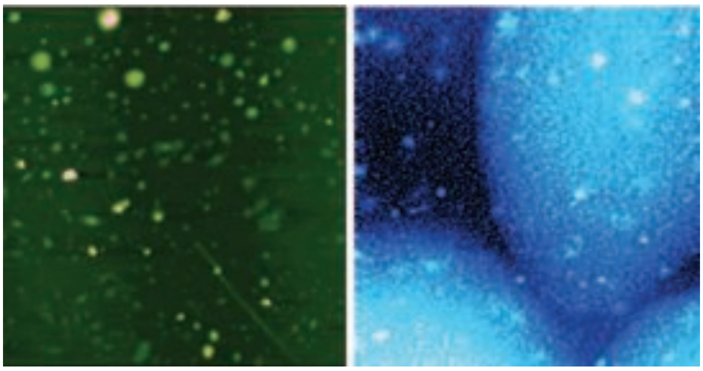


Figure 5: (Left) 2.64 micron image of powder nanoparticles. (Right) 78 micron image of inkjet particles on glossy photo paper.

Display Formats

AFM images are initially stored as a three dimensional array of numbers. There is a *Z* height value for each of the *X* and *Y* data points measured in the image. With visualization software, images may be displayed in a number of different formats and color schemes. Often it is helpful to visualize nanoparticle images with a variety of different display formats.

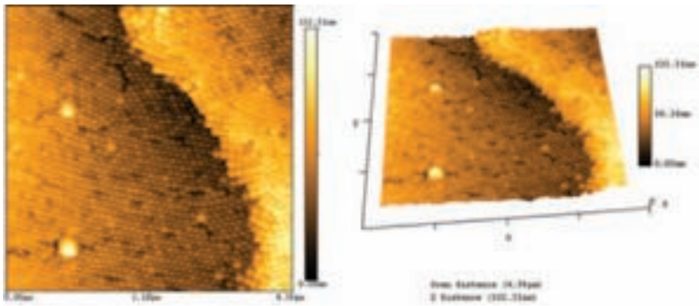


Figure 6: (Left) two dimensional and (Right) three dimensional images of 100 nm nanoparticles in monolayers on a surface.

Conclusions

AFM is ideally suited for the visualization of both nano and micro- size particles. Single particles, clusters and layers together with their surface texture can be imaged directly and non-intrusively. Flexible data display options such as choice of color and shading enable a better understanding of nanoparticles on surfaces ■

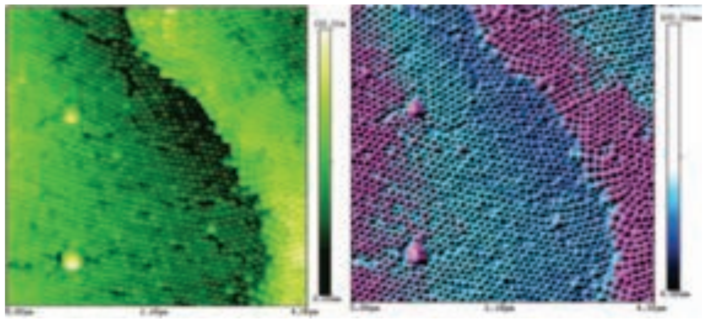


Figure 7: Images of 100 nm nanoparticles showing different color palettes. (Left) Green color palette, (Right) purple blue light color palette with shading..

SIDEBAR

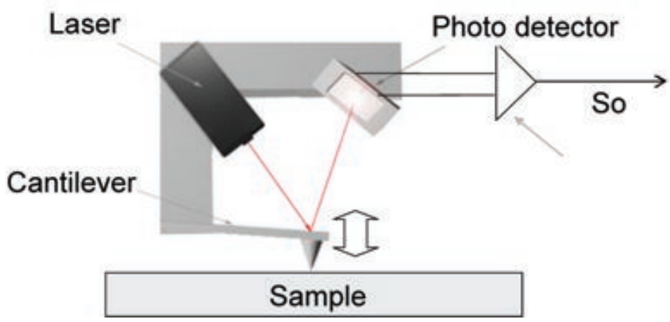
Atomic Force Microscopy – AFM

AFM is the most common of the SPM techniques and is now routinely used for metrology and surface characterization of a multitude of materials.

Invented in 1986 by Binnig, Quate and Gerber, AFM has developed beyond a tool to produce high resolution topographic information to one which is applied to many scientific disciplines from semiconductors to the life sciences. While most measurements are made in air, systems are available enabling the user to study samples in different environments – from liquids to gases, from ambient to high vacuum.

Like all other SPM techniques, AFM uses a sharp probe (usually made from silicon or silicon nitride) moving over the surface of a sample in a raster scan. The tip is at the end of a cantilever which bends in response to the change of force between the tip and the sample.

Most AFMs use an optical lever technique to detect flexure of the cantilever. A laser is focused onto the back of the cantilever and is reflected onto a four quadrant photo detector. This enables topography to be measured by the up-down movement of the cantilever while frictional or lateral forces may be measured by following the twisting of the cantilever (see diagram).



There are many different modes of AFM. The topographic modes use the force interaction between tip and sample. In contact mode, the tip is working in the repulsive region very close to the surface when the cantilever is pushed away from the surface. In the various non-contact modes, the cantilever is oscillated at its resonant frequency and is attracted to the surface. Change of amplitude or shift in frequency may be monitored to measure the force interactions.

Specific properties may also be measured using AFM. For example, the tip may be coated with an appropriate metal to enable properties such as magnetic or electrostatic field changes emanating form the sample surface. Again, vibrating techniques are used yielding simultaneous spatial topographic and materials properties.

For a complete list of modes, visit Pacific Nanotechnology's AFM tutorial at http://www.pacificnanotech.com/afm-tutorial.html.

Paul West, Pacific Nanotechnology

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