Characterization of nanoparticles

To understand the potential of nanoparticles, a deeper knowledge of their synthesis and applications is needed. Characterization is done by using a variety of different techniques, mainly drawn from materials science.

Traditional characterization of a particle

Traditional approach to characterizing a particle (not necessarily nanoparticles) includes:

- Molecular structure or composition using chemical characteristics of the materials used to synthesis nanoparticles.
- Melting point determined to make a decisional understanding for the synthesized particles purity.
- Boiling point
- Vapor pressure: is an indication of a liquid's evaporation rate. It relates to the tendency of particles to escape from the liquid (or a solid). A substance with a high vapor pressure at normal temperatures is often referred to as **volatile**. The vapor pressure of any substance increases non-linearly with temperature. The atmospheric pressure boiling point of a liquid (also known as the normal boiling point) is the temperature at which the vapor pressure equals the ambient atmospheric pressure. With any incremental increase in that temperature, the vapor pressure becomes sufficient to overcome atmospheric pressure and lift the liquid to form vapor bubbles inside the bulk of the substance. Bubble formation deeper in the liquid requires a higher pressure, and therefore higher temperature, because the fluid pressure increases above the atmospheric pressure as the depth increases.
- pH : effecting the solubility characters of nanoparticles as well as the chemical behavior specially when conjugated to biomolecules.
- Solubility: one of the main features of nanoparticles intended to used in biological experiments. Nanoparticles can be soluble in different types of solutions with hydrophobic and hydrophilic properties such as organic solvent or watery solutions (buffers). For most of nanoparticles intended to be used in biological applications,

the synthesized nanoparticles should be water soluble. That's mean they should be able to form a hydrogen bonds with water molecule or should behave as emulation or colloidal in water environment.

Nanoparticle characterization

Nanoparticle characterization parameters include:

- Surface area and porosity
- Solubility
- Particle size distribution
- Aggregation
- Hydrated surface analysis
- Wettability
- Adsorption potential
- Shape and size of interactive surface

Understanding nanoparticle characterization parameters

There are several techniques used to understand these characterization parameters in nanoparticles. They include:

- atomic force microscopy (AFM)
- electron microscopy including TEM and SEM
- dynamic light scattering (DLS)
- x-ray photoelectron spectroscopy (XPS)
- powder X-ray diffraction (XRD)
- Fourier transform infrared spectroscopy (FTIR)
- matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF)
- ultraviolet-visible spectrosc
- nuclear magnetic resonance (NMR)
- Nanoparticle tracking analysis (NTA) for tracking of the Brownian motion

Characterization technologies for manufactured Nanoparticles

Developments of measurement and calibration technologies are in practice for particle size, distribution and concentration of manufactured nanoparticles from 1nm to 100nm using practical methods.

Technologies statistically analyse the shape and size of manufactured nanoparticles such as carbon nanotubes, fullerenes and titinium oxides in tissue samples from electron microscope images. In addition, some methods are used to filter capture efficiency of manufactured nanoparticles in air and some calibration technologies have been developed to eliminate errors due to the shape and material of manufactured Nanoparticles.

Preparation and publication of a manual is important to standardize all of the technologies for characterization of Nanoparticles.

Atomic Force Microscopy

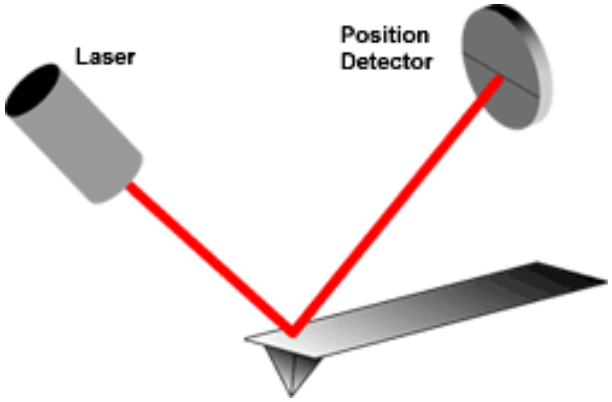
The Atomic Force Microscope was developed to overcome a basic drawback with STM - that it can only image conducting or semiconducting surfaces. The AFM, however, has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, and biological samples.

Binnig, Quate, and Gerber invented the Atomic Force MIcroscope in 1985. Their original AFM consisted of a diamond shard attached to a strip of gold foil. The diamond tip contacted the surface directly, with the interatomic van der Waals forces providing the interaction mechanism. Detection of the cantilever's (Projecting horizontal beam fixed at one end only) vertical movement was done with a second tip placed above the cantilever.

AFM probe deflection

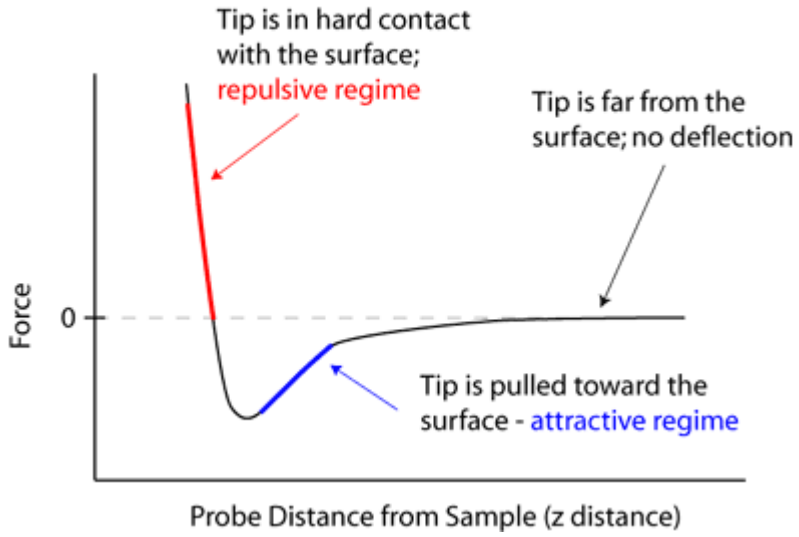
Today, most AFMs use a laser beam deflection system, introduced by Meyer and Amer, where a laser is reflected from the back of the reflective AFM lever and onto a position-sensitive detector. AFM tips and cantilevers are microfabricated from Si or Si_3N_4. Typical tip radius is from a few to 10s of nm.

Beam deflection system, using a laser and photodector to measure the beam position.



Measuring forces

Because the atomic force microscope relies on the forces between the tip and sample, knowing these forces is important for proper imaging. The force is not measured directly, but calculated by measuring the deflection of the lever, and knowing the stiffness of the cantilever. Hook's law gives F = -kz, where F is the force, k is the stiffness of the lever, and z is the distance the lever is bent.



AFM Modes of operation

Because of AFM's versatility, it has been applied to a large number of research topics. The Atomic Force Microscope has also gone through many modifications for specific application requirements.

Contact Mode

The first and foremost mode of operation, contact mode is widely used. As the tip is raster-scanned across the surface, it is deflected as it moves over the surface corrugation. In constant force mode, the tip is constantly adjusted to maintain a constant deflection, and therefore constant height above the surface. It is this adjustment that is displayed as data. However, the ability to track the surface in this manner is limited by the feedback circuit. Sometimes the tip is allowed to scan without this adjustment, and one measures only the deflection. This is useful for small, high-speed atomic resolution scans, and is known as variable-deflection mode.

Because the tip is in hard contact with the surface, the stiffness of the lever needs to be less that the effective spring constant holding atoms together, which is on the order of 1 - 10 nN/nm. Most contact mode levers have a spring constant of < 1N/m.

Lateral Force Microscopy

LFM measures frictional forces on a surface. By measuring the "twist" of the cantilever, rather than merely its deflection, one can qualitatively determine areas of higher and lower friction.

Noncontact mode

Noncontact mode belongs to a family of AC modes, which refers to the use of an oscillating *Having periodic vibrations* cantilever. A stiff cantilever is oscillated in the attractive regime, meaning that the tip is quite close to the sample, but not touching it (hence, "noncontact"). The forces between the tip and sample are quite low, on the order of pN (10^{-12} N). The detection scheme is based on measuring changes to the resonant frequency or amplitude of the cantilever.

Dynamic Force / Intermittant-contact / “tapping mode” AFM

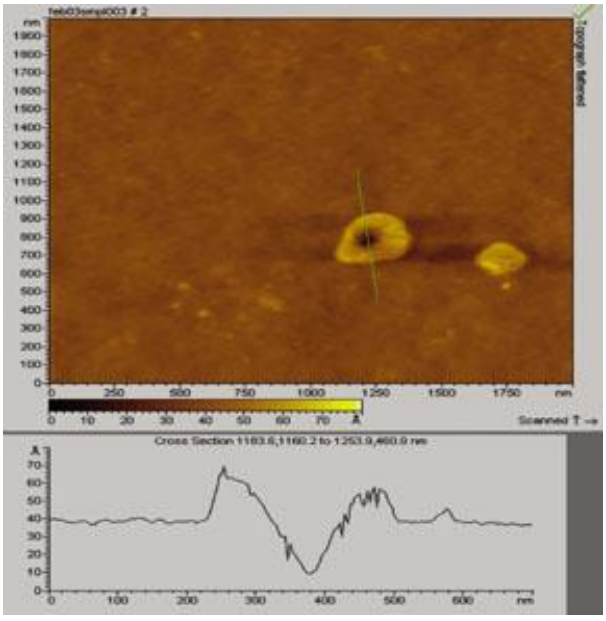
Commonly referred to as “tapping mode” it is also referred to as intermittent-contact or the more general term Dynamic Force Mode (DFM).

A stiff cantilever is oscillated closer to the sample than in noncontact mode. Part of the oscillation extends into the repulsive regime, so the tip intermittently touches or “taps” the surface. Very stiff cantilevers are typically used, as tips can get “stuck” in the water contamination layer.

The advantage of tapping the surface is improved lateral resolution on soft samples. Lateral forces such as drag, common in contact mode, are virtually eliminated. For poorly adsorbed specimens on a substrate surface the advantage is clearly seen.

Force Modulation

Force modulation refers to a method used to probe properties of materials through sample/tip interactions. The tip (or sample) is oscillated at a high frequency and pushed into the repulsive regime. The slope of the force-distance curve is measured which is correlated to the sample's elasticity. The data can be acquired along with topography, which allows comparison of both height and material properties.



Phase Imaging

In Phase mode imaging, the phase shift of the oscillating cantilever relative to the driving

signal is measured. This phase shift can be correlated with specific material properties that effect the tip/sample interaction. The phase shift can be used to differentiate areas on a sample with such differing properties as friction, adhesion, and viscoelasticity. The techniques is used simultaneously with DFM mode, so topography can be measured as well.

Examples of atomic force microscope systems

The Nanosurf AFM systems are designed to be easy to use, and ideal for those just getting started with AFM. There are several models for a very wide range of applications. Here you can find a list of various atomic force microscopes.