Traceable nanomaterial metrology with scanning probe microscopy

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The need for the accurate and reliable characterisation of nanomaterials is growing as their application in a broad range of sectors increases. The use of these materials in consumer products, their use and handling in the workplace, and the management of their eventual environmental fate are likely to become subject to standards and regulations. These will need to be underpinned with a metrological infrastructure to enable nanometre scale measurements that are traceable to national standards within the International System of Units (SI).

The National Measurement Institute Australia (NMIA) has established a scanning probe microscopy (SPM) metrology laboratory to link dimensional measurements made at the nanometre scale with the SI metre [1] which at NMIA is realised with an optical frequency comb [2]. The laboratory is equipped with a metrological scanning probe microscope (mSPM), which is being developed to combine the resolution of dimensional measurements achievable with SPM with the accuracy achievable using laser interferometry. Conventional SPMs can readily achieve nanometre scale precision, and by incorporating laser interferometry into an mSPM, traceability to the SI metre can be achieved [3].

The mSPM will provide traceable measurements with a combined uncertainty of the dimensional measurements ≤1 nm in a working measurement volume of 100 µm × 100 µm × 25 µm. Design of the mSPM (Figure 1) has followed a number of principles aimed at minimising the magnitude of the contribution to the measurement uncertainty from alignment errors (particularly Abbé errors) [4]; deformations of the mechanical structures (for example due to thermal expansion); motion errors of the translation stage; form errors of the interferometer mirrors; nonlinearities of the interferometers; and fluctuations in the refractive index of air. The mSPM operates in frequency modulation dynamic non-contact atomic force microscopy (AFM) mode using a tip mounted on a two-tined oscillating quartz tuning fork [5].

Due to its complexity, it is anticipated that routine imaging and measurement with the mSPM will be somewhat impractical; therefore it will be used primarily to calibrate SPM transfer standard artifacts, for example step height and pitch length. The SPM laboratory is equipped with a companion commercial AFM which, by means of calibration with these top-level SPM transfer standard artifacts, becomes the next element in the traceability hierarchy. This companion instrument will be used for calibration of clients’ transfer standard artifacts and to measure and characterise properties of nanomaterials such as nanoparticles. Measurement of nanoparticle reference materials in this AFM will enable the validation and/or calibration of other instruments for nanoparticle dimensional measurements such as dynamic light scattering and differential centrifugal sedimentation, thereby providing traceability for these measurements.

Accurate dimensional measurement of nanoparticle reference materials with AFM is challenging. The particles need to be attached to a flat substrate (Figure 2); the number density needs to be such that a sufficient number of individual particles on the substrate can be imaged to derive a statistically representative measurement; and broadening of the apparent lateral dimensions of particle images due to tip convolution means that particle diameter can only be reliably derived from the particle height requiring the assumption that the particle is spherical [6].

An AFM image is a map of constant force between tip and sample which only approximates the true topography of the sample. To accurately measure the height of a particle on a substrate, it is necessary to identify and investigate the interactions of the tip-particle-substrate system, including those arising from the capillary action of water films that may be present as well as surface functionalisation [7][8]. We present results from a force spectroscopy study of some nanoparticle reference material/substrate systems used in AFM nanoparticle metrology.
References


Figure 1. Photograph of the mSPM under construction at NMIA. The laser interferometers surrounding the central metrological frame enable traceable dimensional measurements at the nanoscale.

Figure 2. 3D rendered AFM image of nominally 15 nm diameter gold nanoparticle reference material attached to a cleaved mica substrate. The particle diameter is derived from the height difference between the apex of the particle image and the substrate.