Characterisation

Dr. Ayman El-Fatatry
Systems Engineering Innovation Centre (SEIC)
BAE SYSTEMS
Holywell Park
Loughborough University
Loughborough, Leicestershire LE11 3TU
United Kingdom

a.el-fatatry@lboro.ac.uk

ABSTRACT (NANOTECHNOLOGY & CHARACTERISTATION)

This lecture will focus on the techniques currently in use or being developed for measuring nano-particles and nanotechnology-based products from all aspects including, geometries, forms and conformity as well as functional characteristics such as absorption, charge or porosity. The lecture will also aim to address the systems/response based measurements of such materials and novel nano-composites. In this context, the lecture's content will be as follows:

- *Challenges in Characterisation of nano-materials*
- *Tools and equipment for in-process assessment (Scanning Electron Microscopes / Scanning Probe Microscopes etc)*
- *Tools and equipment for product characterisation (Optical / laser diffraction etc)*
- *Techniques for functional characterisation (chemical analysis, surface assessment / mapping etc)*
- *Future directions*

1.0 INTRODUCTION

Nanotechnology based materials and devices demand a capability for the precise measurement of multi domain and inter-related parameters. In essence, in order to be able to fully characterize a material / component / subsystem or indeed a system, any or a combination of the following characterisation functions tend to be adopted:

- The measurement of the material properties (smoothness, granularity, chemical composition etc)
- The measurement of the structural properties (dimensions, density, friction, robustness etc)
- The testing the component properties (electrical, optical, thermal dissipation etc)
- The testing of the application properties (bandwidth, processing speed, resolution etc)

Clearly, the ability to acquire an in-depth knowledge of the material properties is essential for understanding and predicting device performance. Generally, the larger-sized the object is, the easier it is to rely on the bulk material properties. For small(er) devices deviations and irregularities of the material properties – from the

El-Fatatry, A. (2005) Characterisation. In *Nanotechnology Aerospace Applications* (pp. 3-1 – 3-8). Educational Notes RTO-EN-AVT-129, Paper 3. Neuilly-sur-Seine, France: RTO. Available from: http://www.rto.nato.int/abstracts.asp.

bulk domain – become more prominent and less easy to measure / differentiate. For thin films and more specifically, nanotechnology composites, the surface properties become more important and often dominant. Measurement of these properties is therefore extremely important. Currently there are a variety of methods which have been developed to characterize nano-mechanical properties (such as micro-hardness and elastic modulus, thin film adhesion, coating quality scratch). These include the use of nanoscale indenters and nano-probes.

Metrology of non electrical parameters often make use of non contact (non-invasive) methods which minimize any disturbance that is likely to affect the integrity of the device. This is particularly important for the nanotechnology domain where nano-effects and perturbations could adversely affect the performance of the ultimate device as well as the salient behaviours. Optical, non-tactile and non-intrusive techniques are ideal for such measurements. Optical microscopy, coupled with interferometery for instance is a well established analysing both the static and dynamic behaviour of nanotechnology based devices as well as for characterizing surface morphology. Other equally intricate techniques are constantly being developed to face the challenges posed by nanotechnology.

2.0 CHALLENGES OF CHARACTERISATION AND MEASUREMENT IN THE NANO-DOMAIN

- As with microsystems, there can be very complex structures (high aspect ratios and complex geometries)
- Devices tend to be composed of many different materials
- Structures are, generally, stiff at the macro-scale and very floppy at the nano-scale
- Surface physics tends to dominate
- Quantum effects may also come into play
- Biological (live!) samples may need measuring providing further / additional complications
- Probe effects cannot be ignored

3.0 MEASUREMENT AND CHARACTERISATION GAPS

The following chart maps out the field within which novel techniques for nano-metrology will be required in relation to the more conventional metrology methods (CMM).

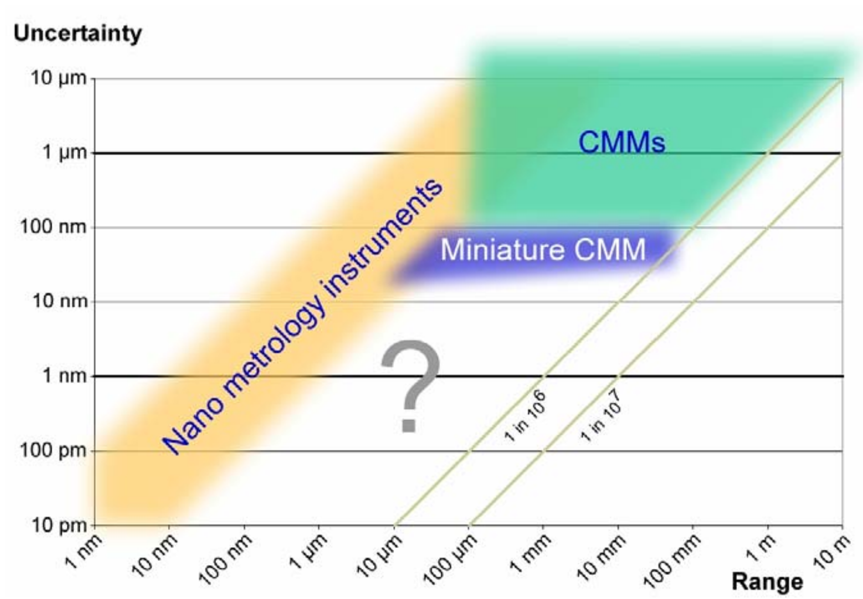

Figure 1: Positioning of Nano-Metrology within the Footprint of Conventional and Miniaturized Metrology Methods (CMM) – Diagram courtesy of the UK's National Physical Laboratories (NPL).

The diagram (Figure 1) illustrates the distinct gap in capabilities, particularly with regards to the characterisation of 3D structures. The requirement can also be described in terms of the characteristics to be measured, as illustrated in the following diagram.

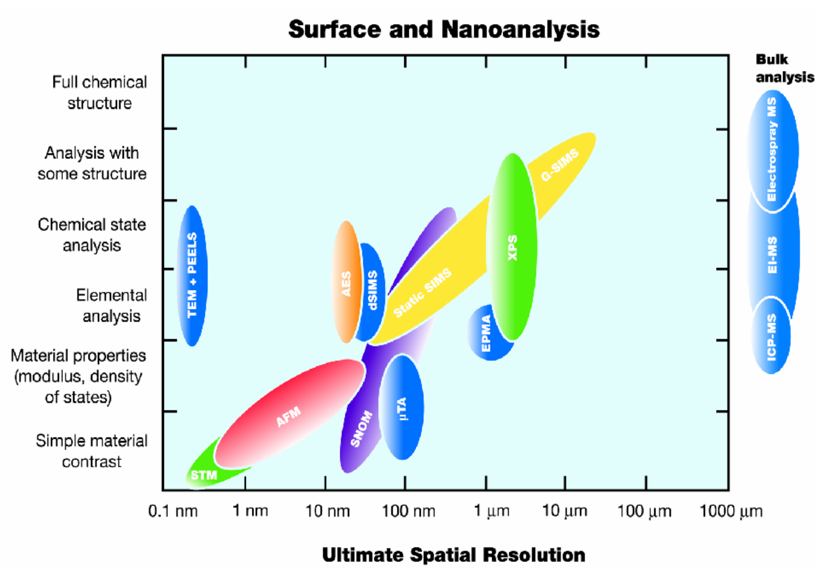

Figure 2: Metrology Footprint as a Function of Special Resolution – Diagram courtesy of the UK's National Physical Laboratories (NPL).

This diagram (Figure 2) identifies a gap at around the 1nm and below region where techniques are yet to be developed, Standardised or invented.

4.0 TECHNIQUES FOR CHARACTERISATION AND METROLOGY

Metrology at the nano-scale tends to be based on one of the following, relatively well-established, methods:

- Scanning Probe Microscopes (including atomic-force microscopy / AFM)
- Optical interferometers (including x-ray interferometery XRI)
- Force-based microscopy / analysis

Force-based microscopy relies on the use of probes to indent surfaces has been established as a viable technique to assess surface / material characteristics. Such force-based metrology needs, however, to be refined for nanoscale materials and measurements. The following table provides an indication of the scaling-down associated with probe indentation and associated metrology.

Force	Material	Technique
KN	Bulk	Indentation
N	Thin Films	Micro-indentation
Nano-N +	Surface zone	Nano-indentation
Nano-N	Surface layer	Modulus mapping
Pico-N	Single molecules (sub-layer)	Molecular pulling

This table can also be represented, diagrammatically as is shown in figure 3 below:

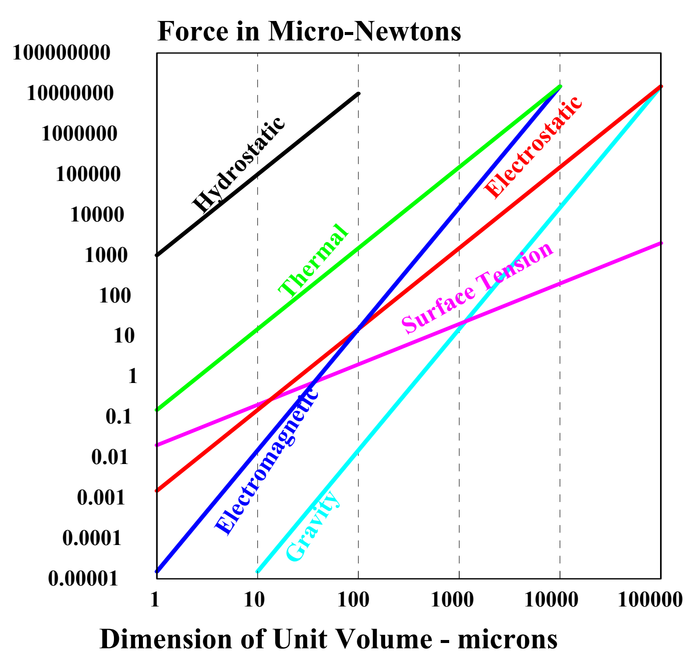

Figure 3: Force Related Measurements vs Volumetric Dimensions – Diagram courtesy of Prof Ron Lawes (Imperial College, London).

5.0 FUNCTIONAL METROLOGY

These techniques encompass methods that rely on the functional / behavioral aspects associated with the nanomaterials used or incorporated. The following diagram provides an illustrated perspective on the typical functionalities that can be measured and, therefore, directly related to the material composition and its specific characteristics.

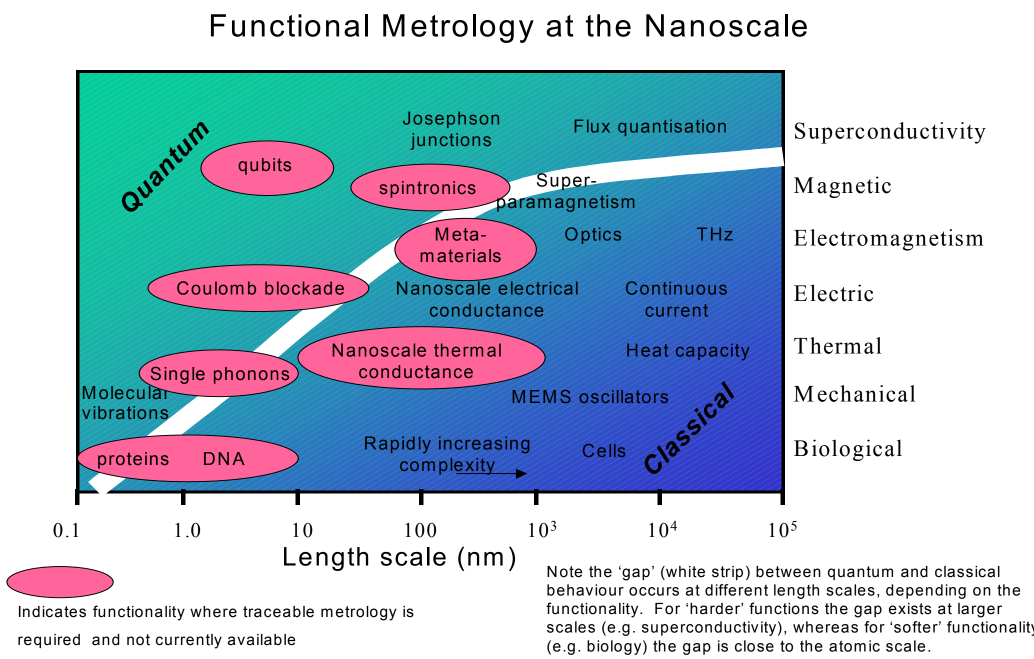

Figure 4: A Diagrammatic Representation of the Measurable "Functionalities" which Relate to the Nano-Scale Domain – Diagram courtesy of the UK's National Physical Laboratories (NPL).

6.0 ASPECTS OF MEASUREMENT AND CHARACTERISATION

Many aspects of nanotechnology based materials and (sub) systems need to be characterized and measured. Most, to date, relate to the developmental and fabrication processes in terms of repeatability, yield and tolerances. Some measurements relate to the end-functionality and some to the enabling capability. The following is by no means a comprehensive list but provides a wide enough range of the spectrum of measurements currently undertaken by researchers and developers in the field.

6.1 Structural

- characterization of surfaces
- particulates
- surface wear
- displacement properties

- Nano-particle sizing
- Mechanical and friction properties of thin polymer films
- defects inspection
- residual stress measurement for micro and nano materials
- critical dimensions of microfluidic devices and high aspect ratio structures
- high accuracy measurements over large surface areas to measure complex forms and structures
- micro-roughness, recessed structure and complex surfaces such as super polished surfaces
- analysis of, super finished-machined components, soft materials and recessed surfaces.
- precision optical surface form measurement
- modelling of coatings from nanometres to microns thick
- strain and deformation measurement of surfaces and wafers in 3d
- nano-indentation
- particle sizing and zeta potential measurement (for particle sizes from 1 nm to 1 mm)
- displacement of surface features with sub-pm resolution
- stylus profilometry – traceable periodic feature measurement
- 2D and 3D surface texture measurement – optical surface characterisation capability
- flatness and sphericity
- TEM sample preparation, high-resolution imaging and EDX analysis
- Surface morphology
- depth profiling with sup-ppm sensitivity and a few-nm depth resolution

6.2 Chemical

- electrochemical activity
- thermo-mechanical properties
- properties of nanocrystals, nanocomposites, nanotubes
- chemical analysis of surfaces
- deposition of biological particles and films at variable temperatures
- biocompatibility

6.3 Functional

- magnetic
- capacitive
- electrical

- force modulation
- operation within gaseous and high temperature environments
- Depth dependence of polarisation of ferroelectric thin films
- piezolectric, magnetic properties
- SEM based functional measurement
- mechanical bench testing in an SEM
- mechanical properties of thin films in air (modulus, wear)
- ferroelectric polarisation measurements (thermal properties)
- thermal and rheological properties of thin films, nano-rheology ("fluid" flow of polymer thin films)
- dielectric impedance for ceramics, and nanoporous membranes over a wide frequency range
- low force measurement
- AFM spring constant measurement
- Lifetime measurements (accelerated tests)

ACKNOWLEDGMENTS

The following organisations are to be gratefully acknowledged for the information and data upon which these lecture notes and the presentation has been based:

BAE Systems, NPL, Imperial College, NEXUS, enablingMNT, Dstl, QinetiQ, Taylor Hobson, TNO, Oxford University, SEIC, the UK's MNT Network, INCOSE and the members of the *Nanotechnology for Autonomous Vehicles*

AVT 138/RTG-045 Task Group.