Characterisation

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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)

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- 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 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.

1 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

2 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):

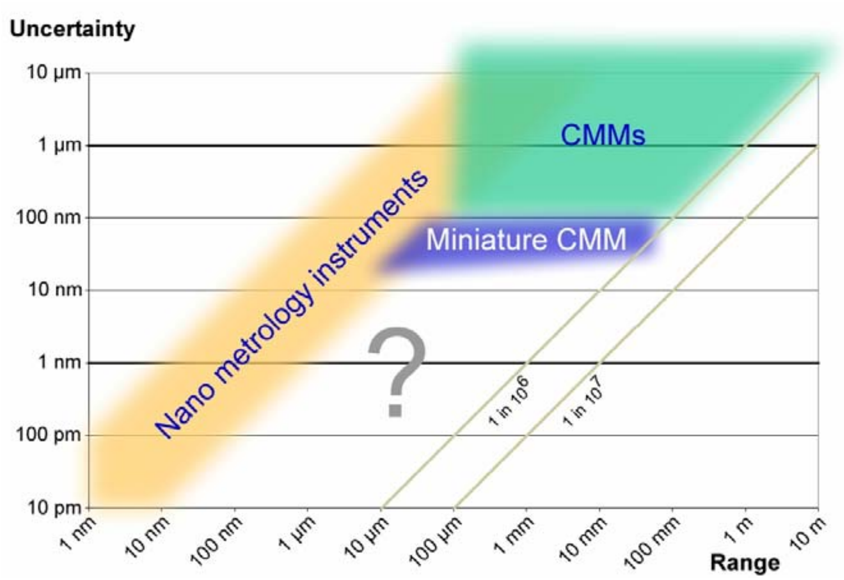


Figure –1- Positioning of nano-metrology within the footprint of conventional and miniaturized metrology methods (CMM) – Diagram courtesy of the UK's National Physical Laboratories (NPL)

The diagram (Fig-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;

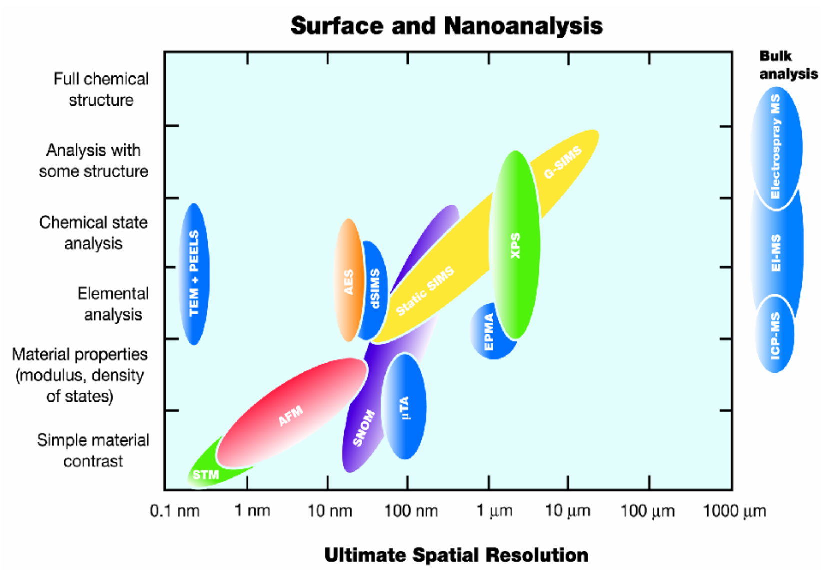


Figure –2- Metrology footprint as a function of special resolution – Diagram courtesy of the UK's National Physical Laboratories (NPL)

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

3 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:

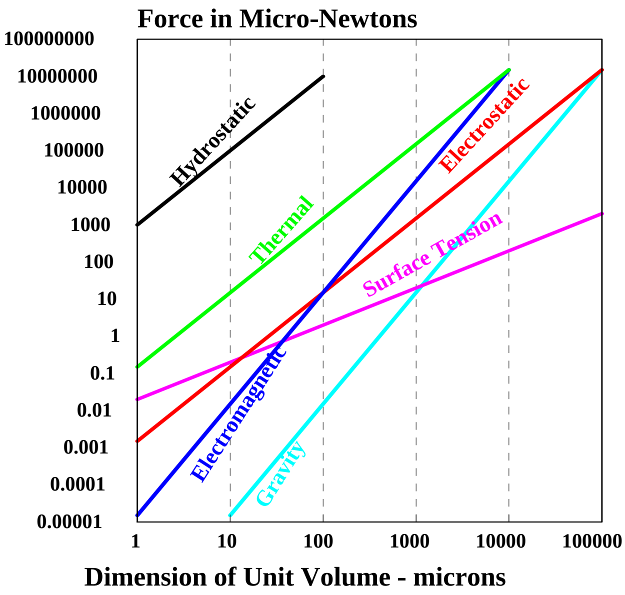


Figure –3- Force related measurements Vs volumetric dimensions – Diagram courtesy of Prof Ron Lawes (Imperial College, London)

4 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:

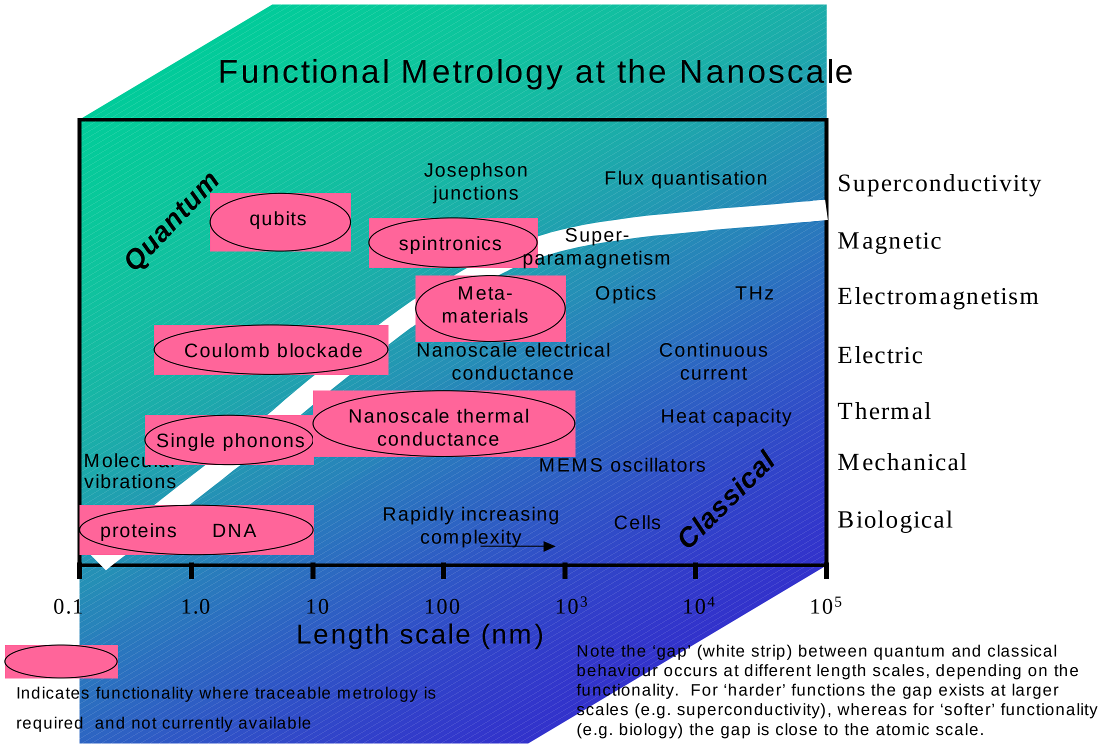


Figure –3- A diagrammatic representation of the measurable "functionalities" which relate to the nano-scale domain – Diagram courtesy of the UK's National Physical Laboratories (NPL).

5 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.

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

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

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:

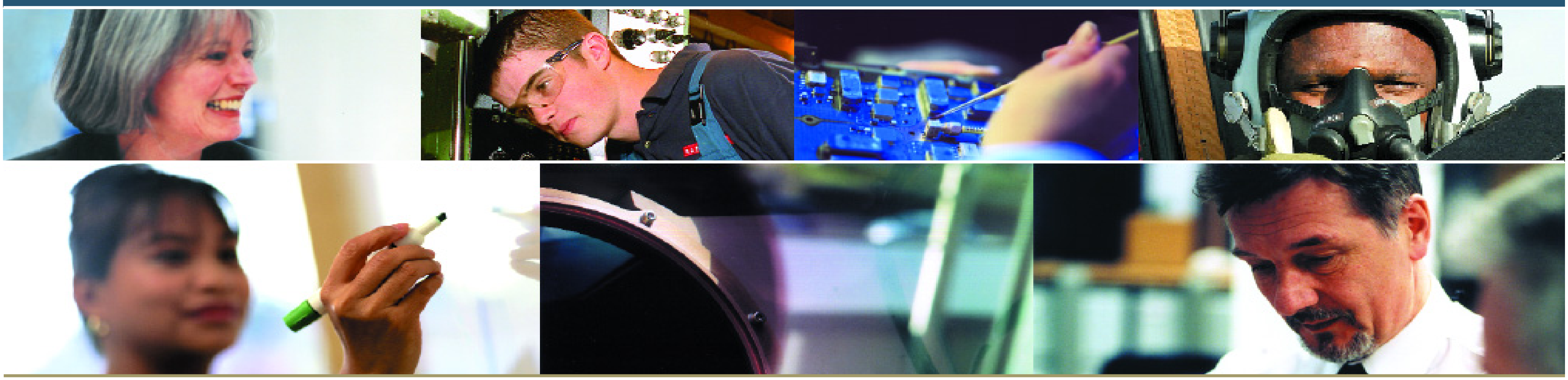
BAE Systems, NPL, Imperial College, NEXUS, enablingMNT, dstl, QinetiQ, Taylor Hobson, TNO, Oxford University, SEIC, the UK's MNT Network, INCOSE and the members of the *Nanotechnology for Autonomous Vehicles*

AVT 138/RTG-045 task group.

NATO – RTO Lecture Series AVT-129

Dr Ayman El-Fatatry

CHARACTERISATION



SEIC

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enablingMNT **dstl** **QinetiQ** **Taylor Hobson**

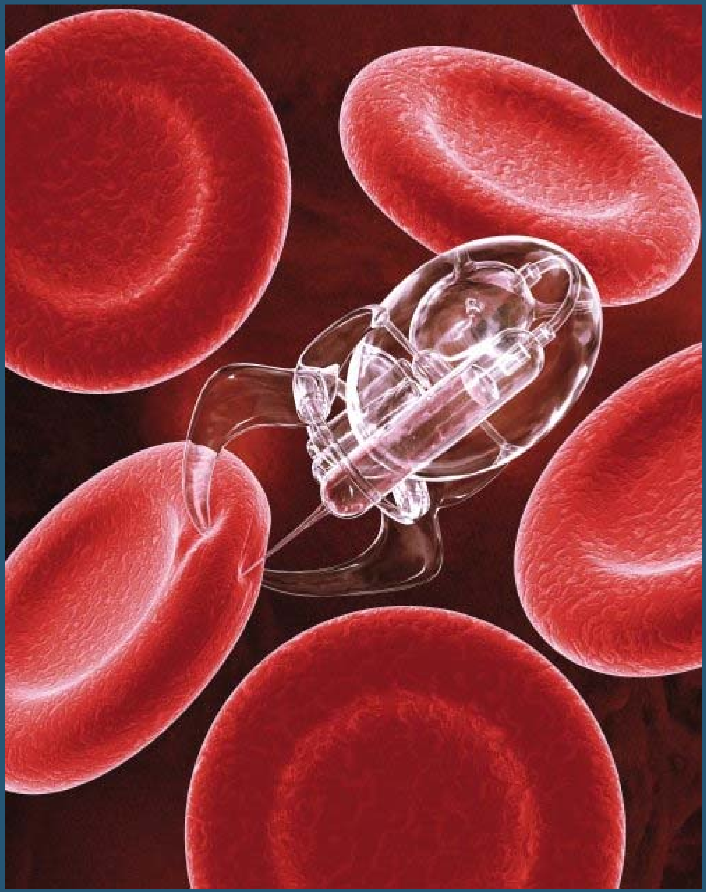
TNO **Oxford University** **the UK's MNT Network**

Nanotechnology for Autonomous Vehicles task group AVT 138/RTG-045.

Lecture Objectives

To introduce some 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.

Why is metrology of Nano-devices difficult?

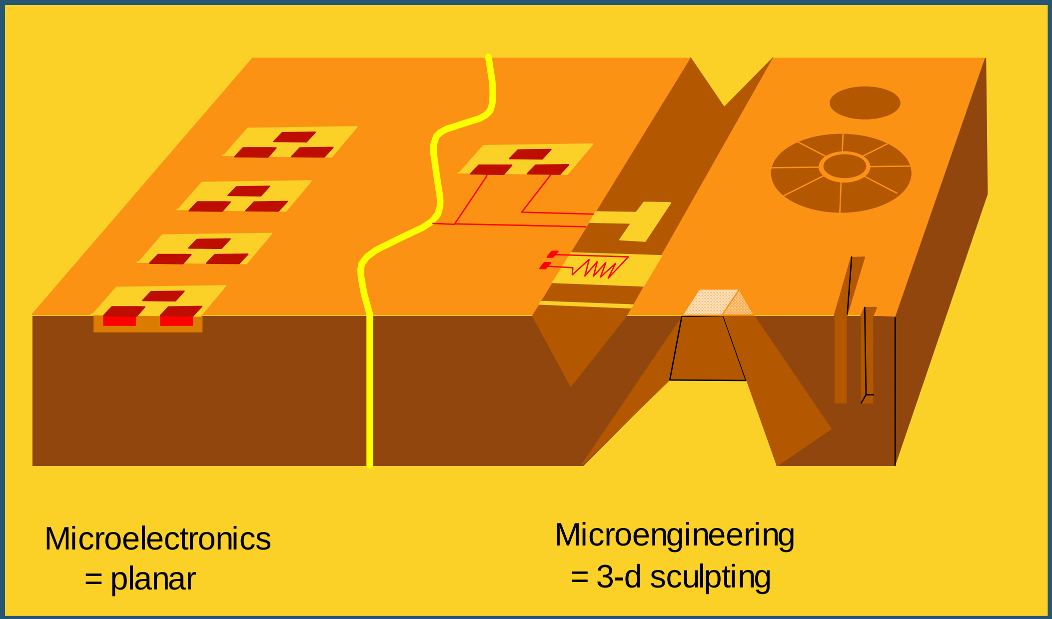


- As with microsystems, there can be very complex structures
- Can incorporate many different materials
- Structures that are stiff at the macro-scale are very flexible at the nano-scale
- Surface physics can dominate
- Quantum effects may come into play
- Biological (live!) samples may need measuring
- Probe effects cannot be ignored

Micro-Nano Systems

Technology Drivers
Shifting Towards the:

Micro
Nano
Automated
Self-assembled
Intelligent
Integrated
Interconnected
Ambient
Ubiquitous
??????

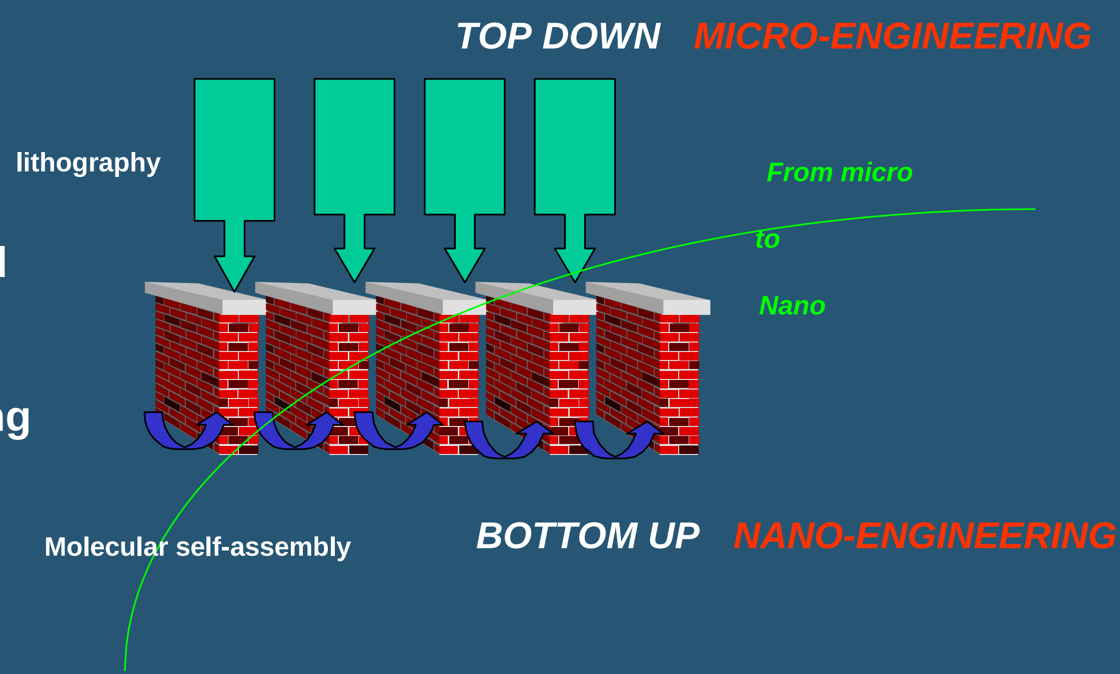


Microtechnology

Is the fabrication of millimetre-sized devices with micron and nano-sized rules by bulk deposition and patterning millions of atoms in any part.

Micro-Nano-Technology

Is the use of the properties of micro and/or nano-technology in micro and macro applications for further shrinking, integration and miniaturisation of sensing & actuating functions and for Microsystems.



Nano-Technology

A process for manipulating smallest natural structures (atoms & molecules) where Quantum mechanics rules.

Hierarchy of Systems

Increasing Complexity / Capability

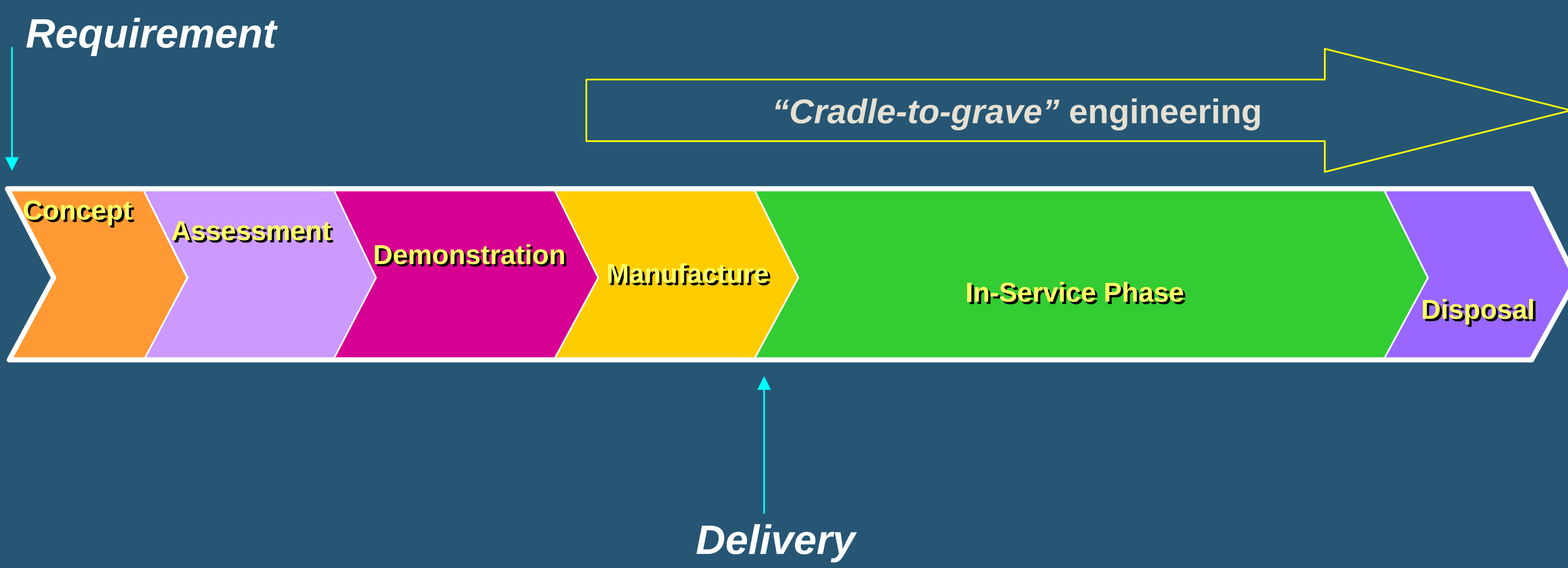
System of Systems

System

Sub-systems

Components

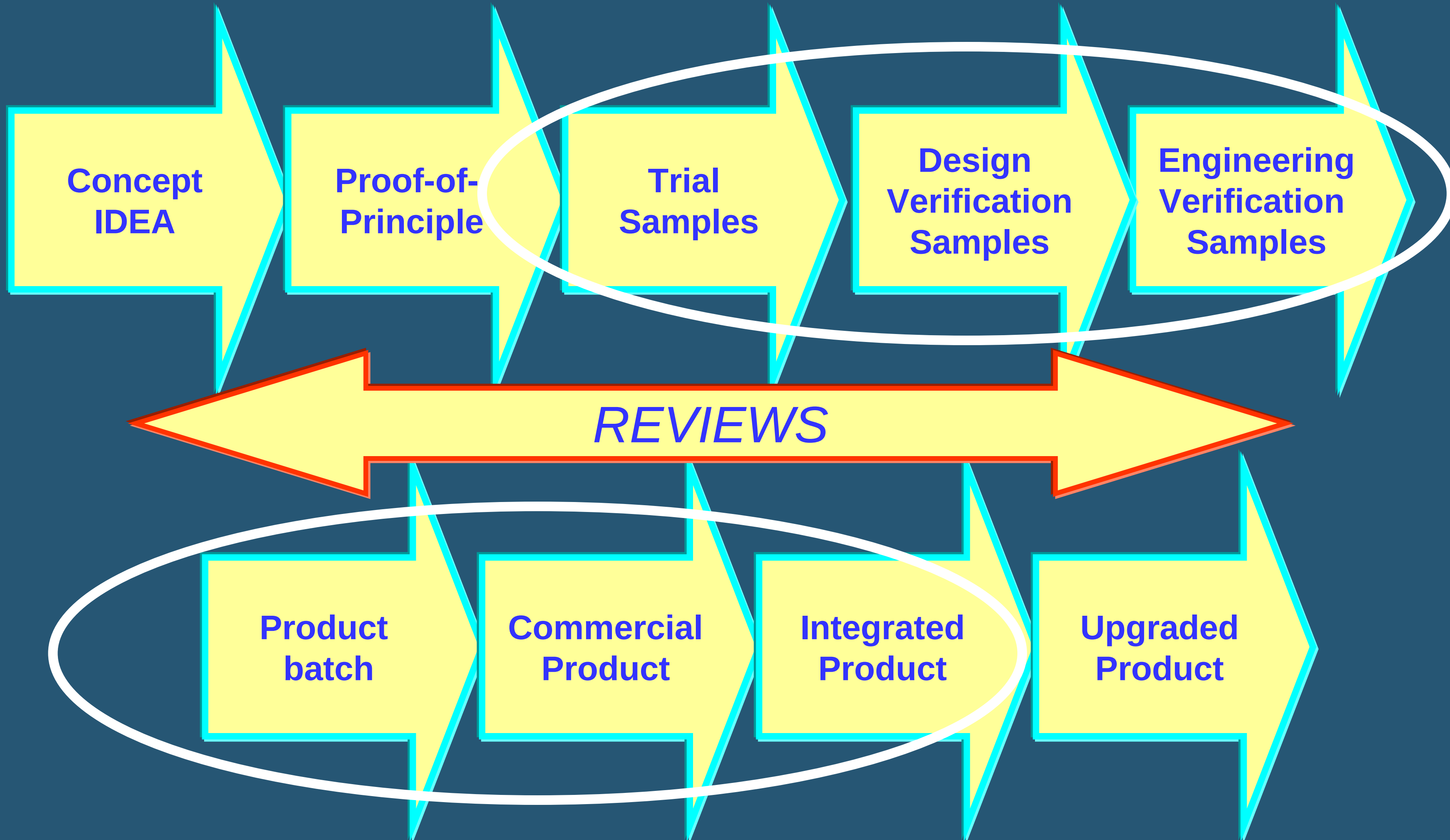
Competence of ***Systems Thinking***



Systems Engineering **is the emerging discipline that enables the integration of multiple components (technologies, people, information…) into effective and affordable products and services**

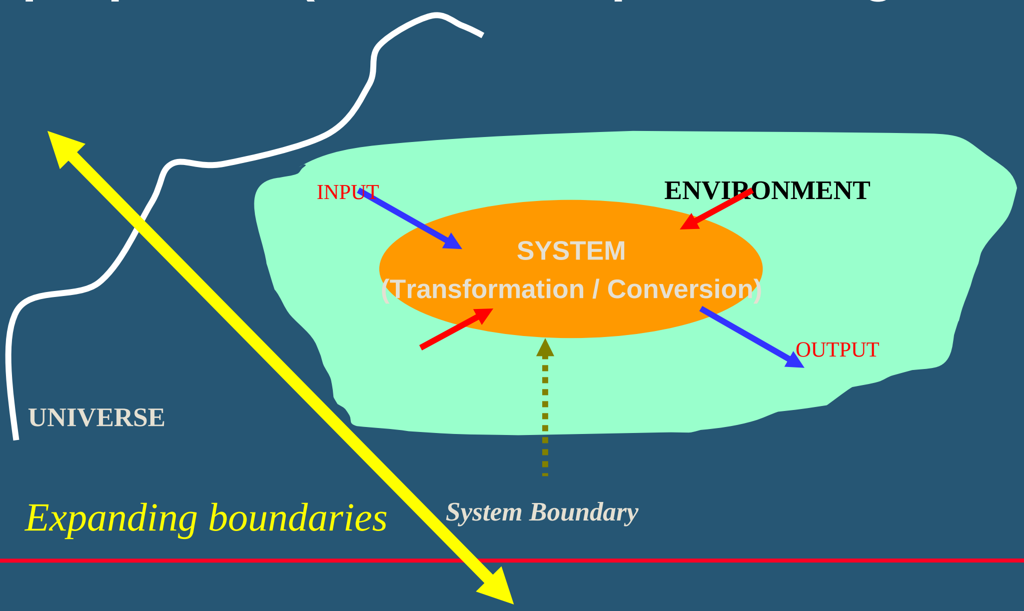
Metrology & the Commercialisation Processes

www.enablingMNT.com

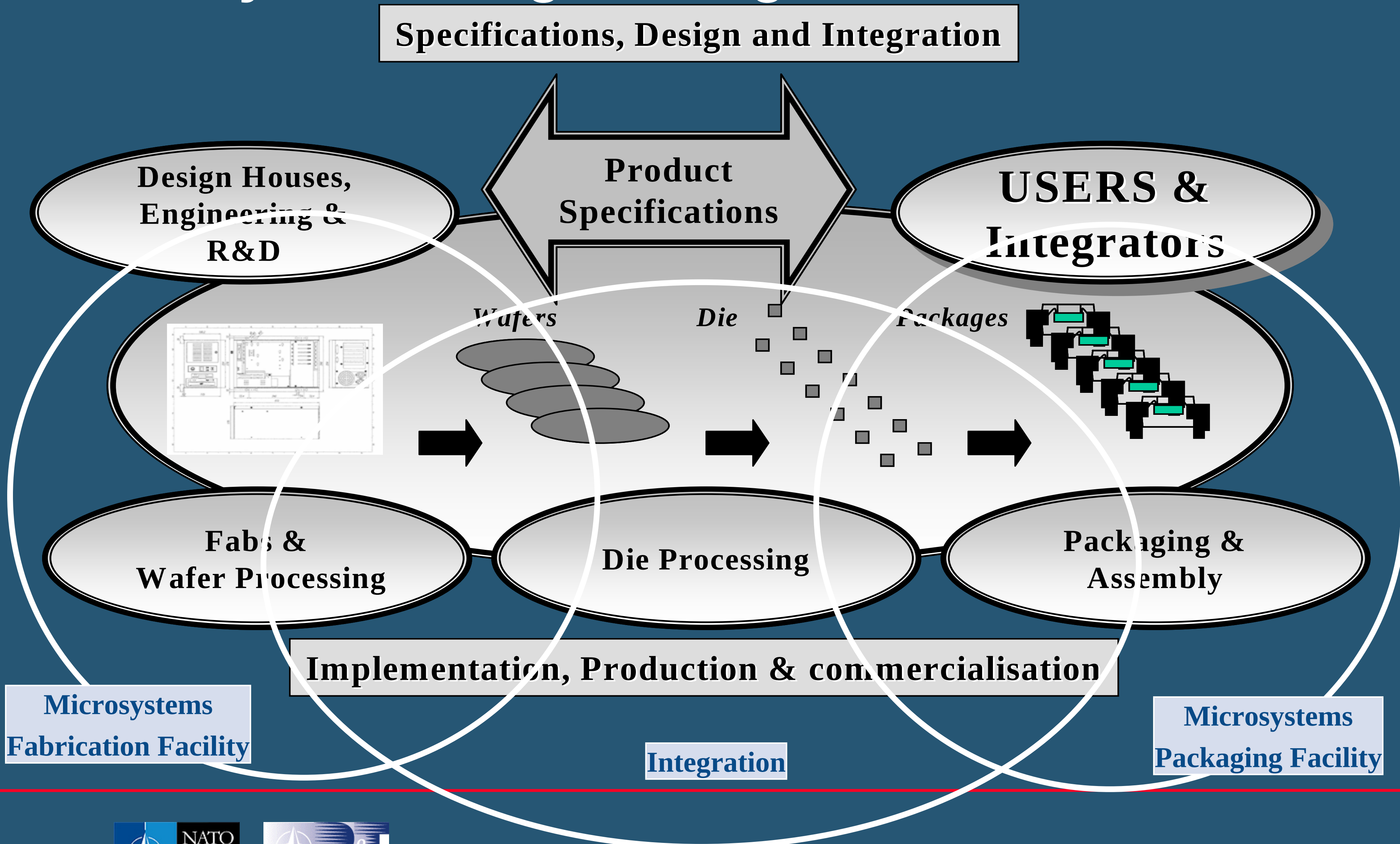


Metrology & the Environment / Boundary

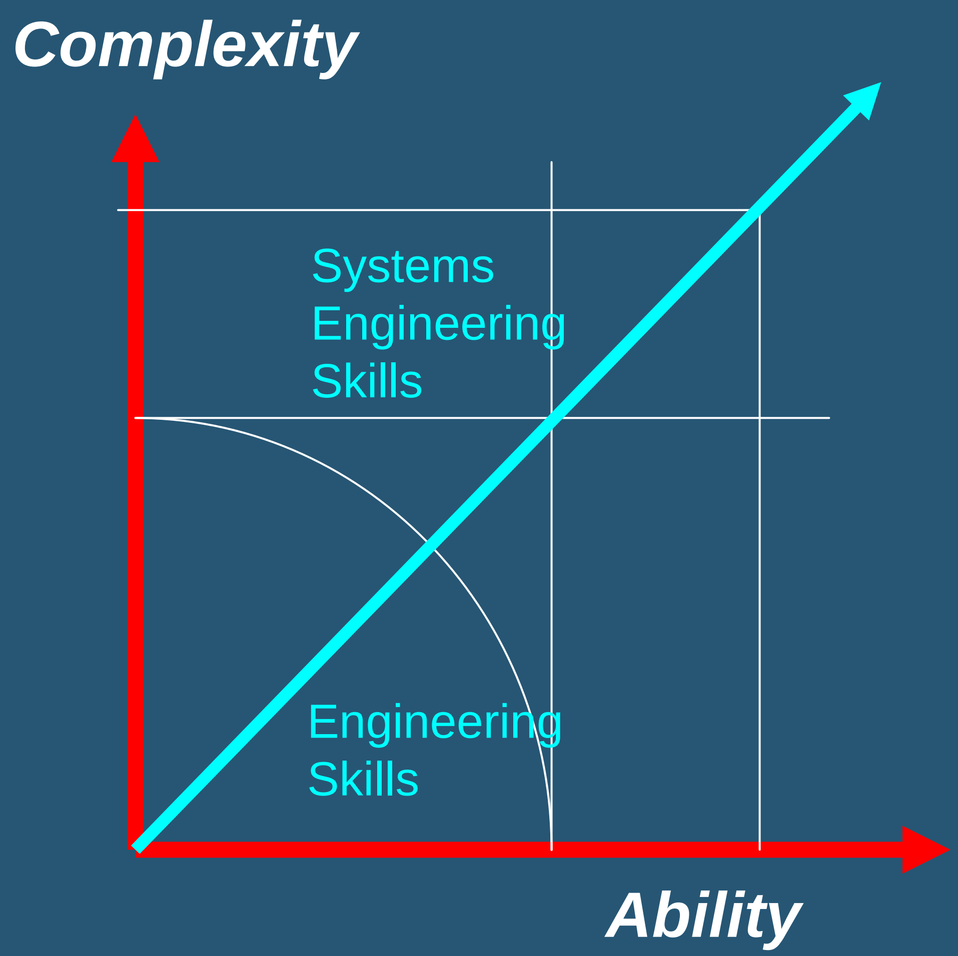
- ✓ **Measurement of the material properties (smoothness, granularity, chemical composition etc)**
- ✓ **Measurement of the structural properties (dimensions, density, friction, robustness etc)**
- ✓ **Measurement of the component properties (electrical, optical, thermal dissipation etc)**
- ✓ **Measurement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



Systems Engineering for commercialisation



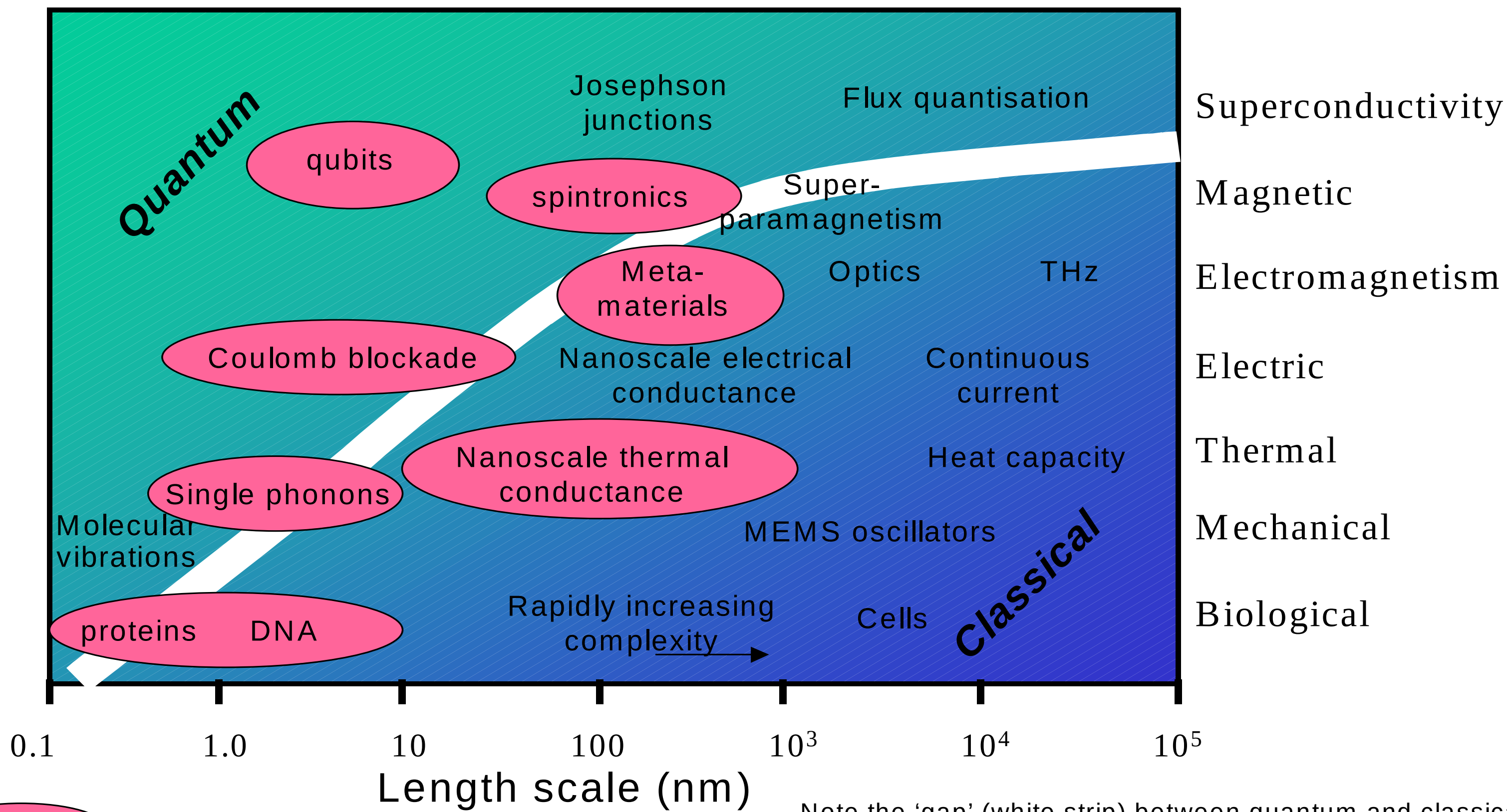
Complexity Vs Ability



Moving the envelope Outwards

Systems Engineering across *the* Metrology Gap

Functional Metrology at the Nanoscale



Indicates functionality where traceable metrology is required and not currently available

Note the ‘gap’ (white strip) between quantum and classical behaviour occurs at different length scales, depending on the functionality. For ‘harder’ functions the gap exists at larger scales (e.g. superconductivity), whereas for ‘softer’ functionality (e.g. biology) the gap is close to the atomic scale.

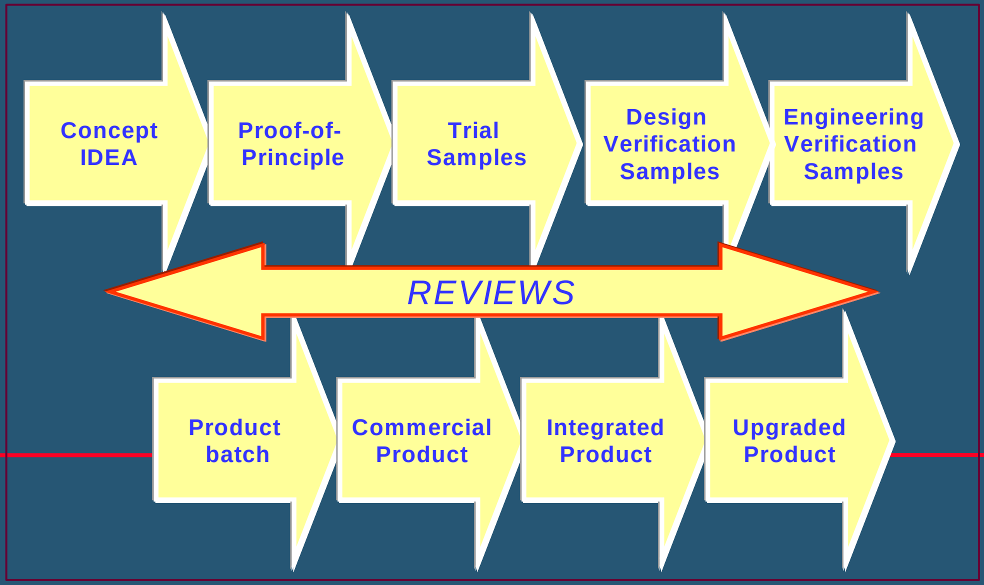
From the Traditional towards a Systems Engineering Approach

-Traditional

- Focused on components
- Functional Teams
- Company driven decisions
- Design & build to tolerances
- Test end product
- React to emergent behaviours
- React to customer problems

-Systems Engineering Approach

- Considering the wider system
- Multi-functional teams
- Customer driven decisions
- Design & build to target values
- Optimise product and process design
- Verify & Validate product and process design



Challenges in measuring micro & nano –based (sub)systems

- **The complexity of the structures (high aspect ratios and complex geometries)**
- **Devices tend to encompass many different materials**
- **Structures are, generally, stiff at the macro-scale and very soft 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**

Aspects of measurement and characterisation

Structural 1

- 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

Aspects of measurement and characterisation

Structural 2

- 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

Aspects of measurement and characterisation

Structural 3

- 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

Aspects of measurement and characterisation

Structural 4

- 2D and 3D surface texture measurement - optical surface characterisation capability
- flatness and spherical dimensions / uniformity
- TEM sample preparation, high-resolution imaging and EDX analysis
- Surface morphology
- depth profiling with sub-ppm sensitivity and a few-nm depth resolution

Aspects of measurement and characterisation

Chemical 1

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

Aspects of measurement and characterisation

Functional 1

- magnetic
- capacitive
- electrical
- force modulation
- operation within gaseous and high temperature environments

Aspects of measurement and characterisation

Functional 2

- 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)

Aspects of measurement and characterisation

Functional 3

- 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)

Measurement Techniques

-Visible

-Optical Microscopy

-Electron beam Microscopy

-Tactile

-Probe (scanned-proximity probe microscopes)

-Functional Effect

-Chemical

-Thermal

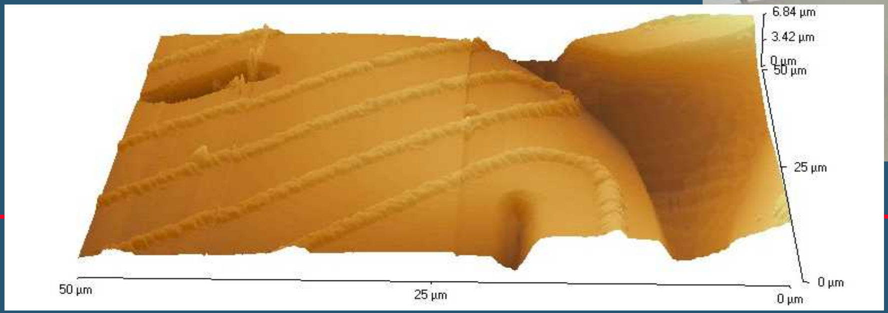
-Magnetic

-Optical

Commercial kits for characterisation and metrology

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

Optical Microscopy
Optical Microscopes
Nomalski contrast, frame storage and image analysis.
Electron Microscopy (SEM)
secondary and backscattered imaging, Frame store, image processing and EDX Analysis.
Atomic Force Microscopy (AFM)
Surface Analysis
Auger, XPS and SIMS



Measurement Techniques

-Visible

-Optical Microscopy

-Electron beam Microscopy

-Tactile

-Probe

-Functional Effect

-Chemical

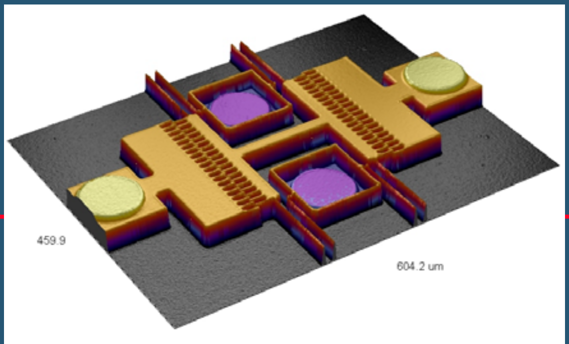
-Thermal

-Magnetic

-Optical

Current metrology tools – Optical instruments

- -Scanning White Light Interferometry
- -Confocal microscopy
- -Micro-mirror devices
- -Holographic interferometry
- -Many others…
- -Give 3D information, but commercial devices have leap-froged standards development
- -Fast but can be expensive
- -Very versatile, but interpretation of results can be difficult or misleading, e.g. phase and edge effects, *etc.*



Thin Film Measurement

The need for measuring a broad array of single and multi-layer film characteristics including, metallic, semiconductor, amorphous, crystalline, and dielectric materials to determine:

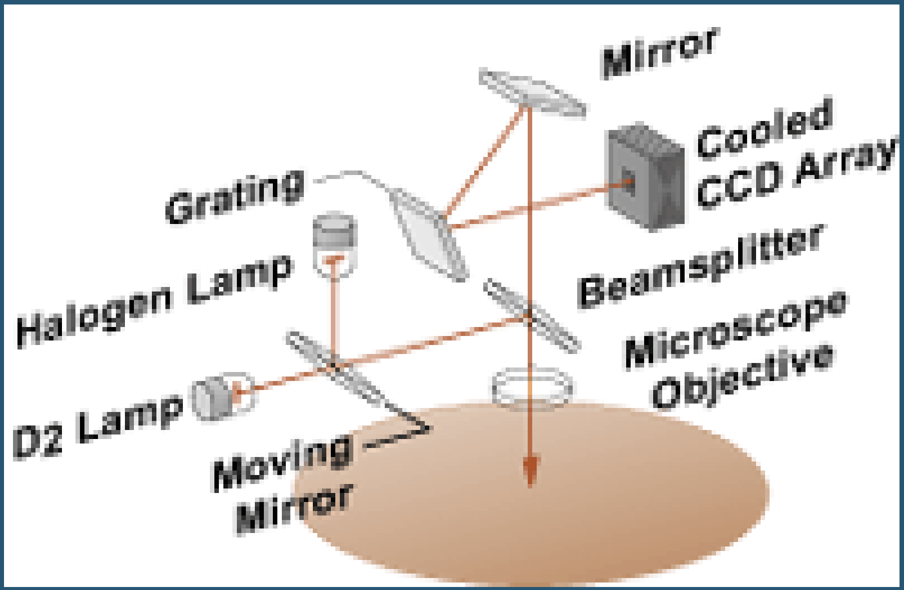
- “Multiple” layer thicknesses (from <1Å to 250 microns)
- Indices of refraction [n(l)] (both TE and TM components of index)
- Extinction (absorption) coefficients [k(l)]
- Birefringence (l)
- Energy band gap [Eg]
- Surface roughness and damage
- Porosity, composition, and crystallinity (EMA model)
- Film properties versus temperature
- Wafer curvature and film stress

NanoSpec Optical Interference thin film measurement

Max film thickness = 50 microns
Min film thickness = 10 nm
Min spot size = 5 microns
Stages manually operated

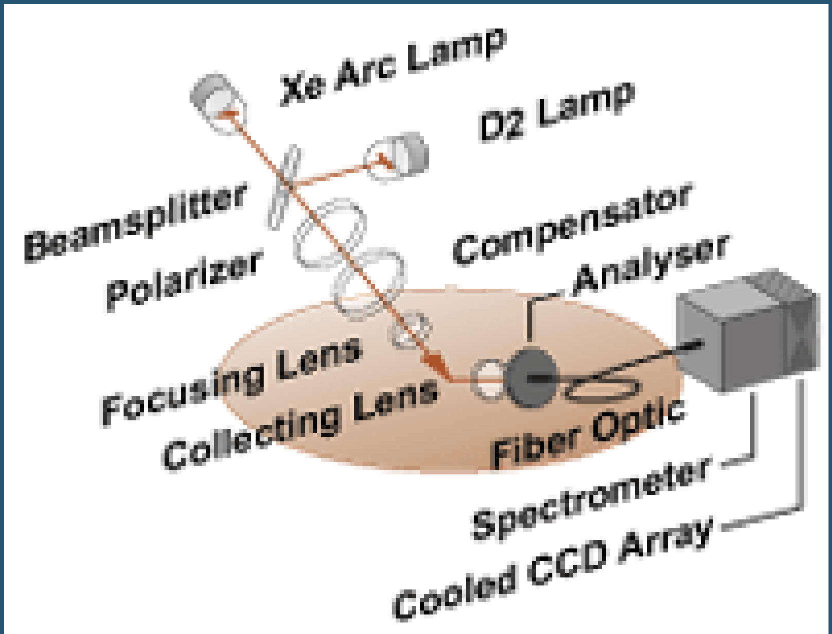
SPECTROSCOPIC REFLECTOMETRY

Spectroscopic reflectometry for film thickness metrology measures either the ratio of the intensity of the reflected beam of light relative to either the intensity of the incident beam (absolute reflectance) or of the beam reflected from a known sample (relative reflectance).



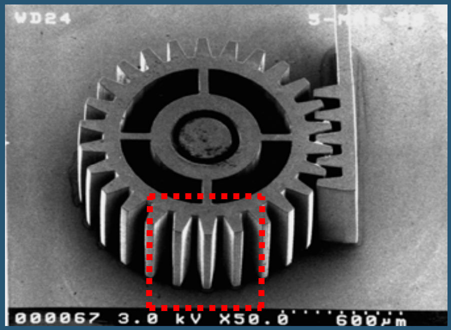
SPECTROSCOPIC ELLIPSOMETRY

Thin film ellipsometry is a non-destructive optical technique that measures the relative phase change in a beam of reflected polarized light. Because the technique is based on phase change, it is very sensitive to the properties of the thin films used in the semiconductor industry.



Confocal Microscopy and Digital Volumetric Imaging

-Accurate digital 3D Representation
-Large Volume of data
-Micron resolution (at least)



<u>Confocal Microscopy</u>
generates 3D spatial data
uses xy laser scan (spiral Nipkow disc)
12 X 512 pixels (160-1600 um)
2 X 2 stitching of images
z axis scan (3000 frames/ 350 um)
only images surface
Test is NON-DESTRUCTIVE

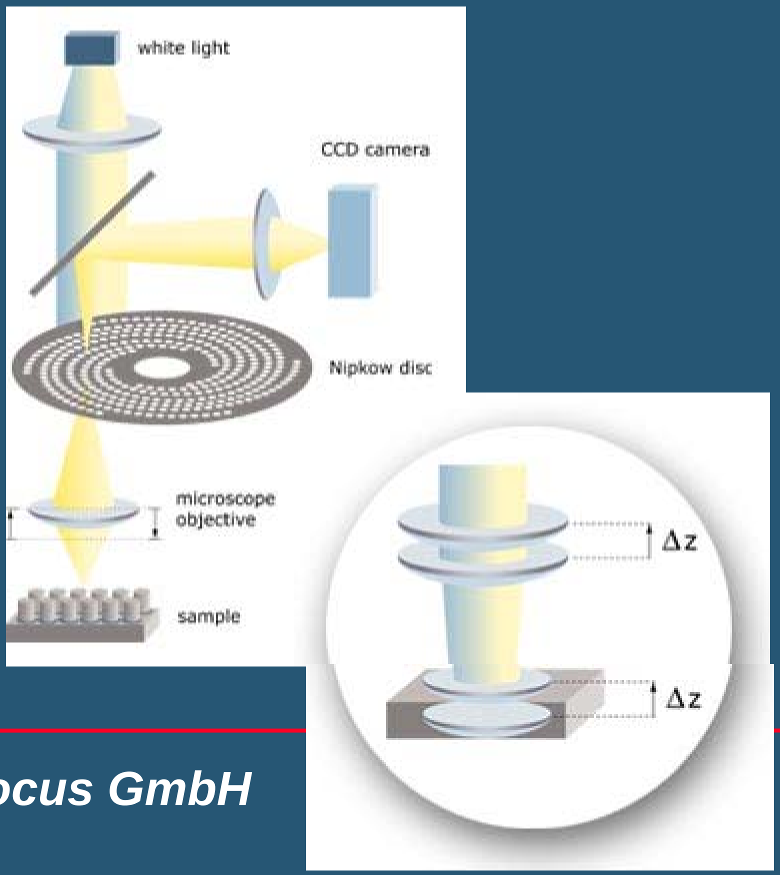
Nanofocus GmbH

<u>Digital Volumetric Imaging (DVI)</u>
covers test piece in polymer
repeated sectioning by diamond knife
reflected image @ 2000 X 2000 pixels
resolution to 0.25 microns-
Test is DESTRUCTIVE

Resolution Sciences Corp.

Confocal 3D Microscopy

In the confocal microscope all structures out of focus are suppressed at image formation. This is obtained by an arrangement of diaphragms which, at optically conjugated points of the path of rays, act as a point light source and as a point detector respectively. Rays from out-of-focus are suppressed by the detection pinhole.



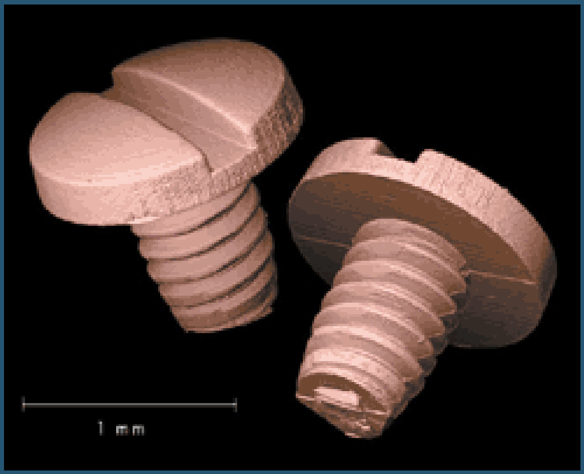
Nanofocus GmbH

NATO OTAN
R&T ORGANIZATION

Digital Volumetric Imaging (DVI)

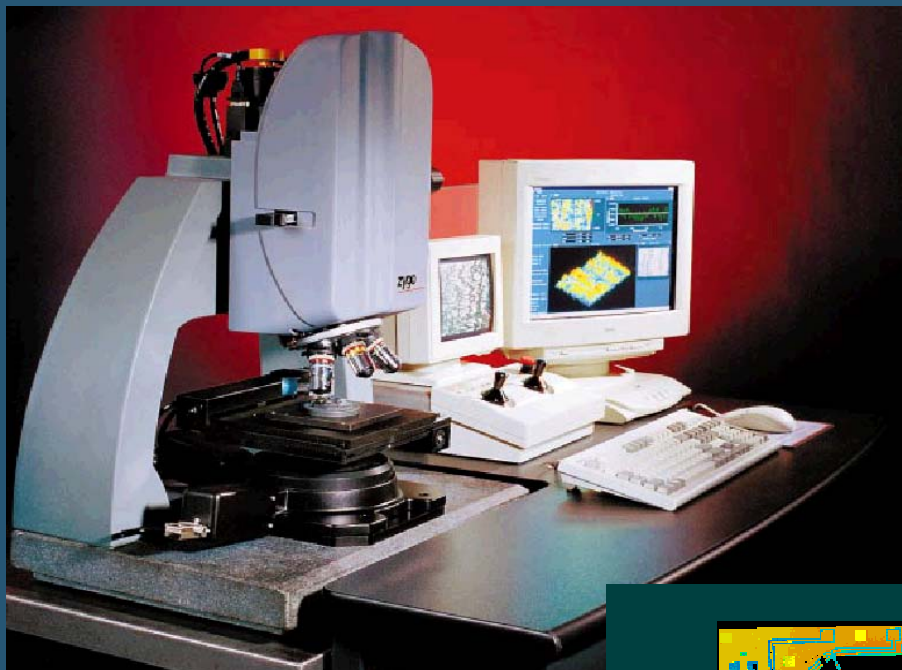
DVI is a digital microscopy technique that permits the generation of high-fidelity three-dimensional images of tissue and other materials. Developed for medical applications but now finding use in MNT.

Software tool driven



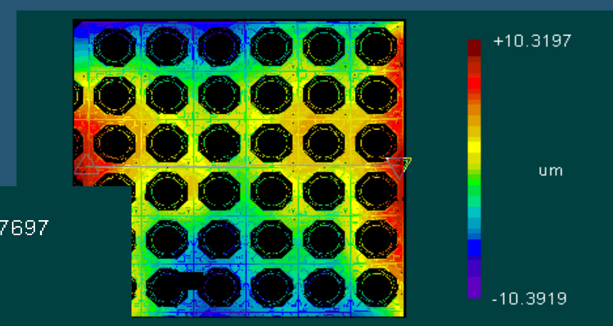
Resolution Sciences Corp.

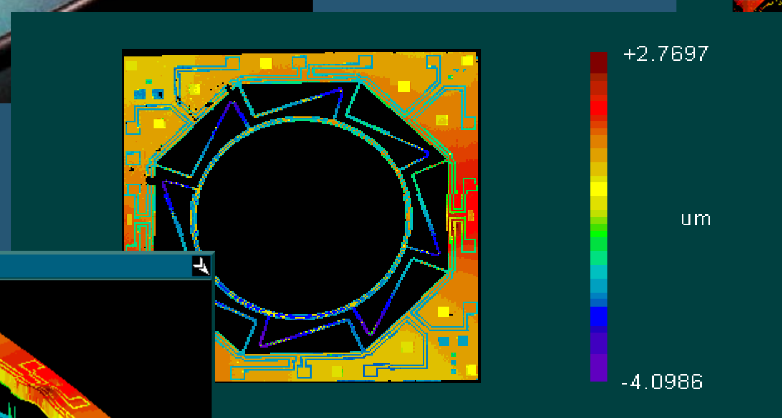
Optical profilometry



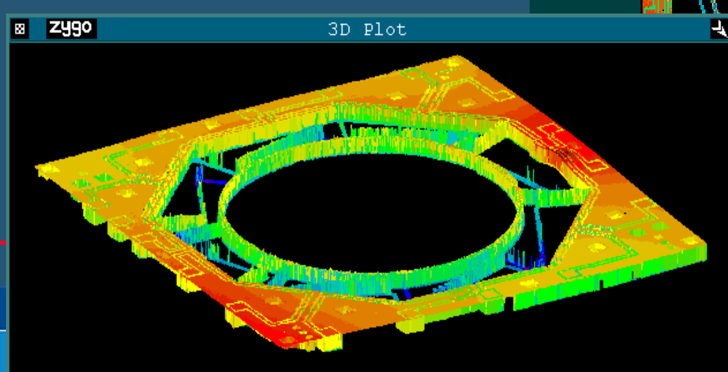
2005
New equipment has dynamic capability for analysing vibrating structures

Across-wafer profiles

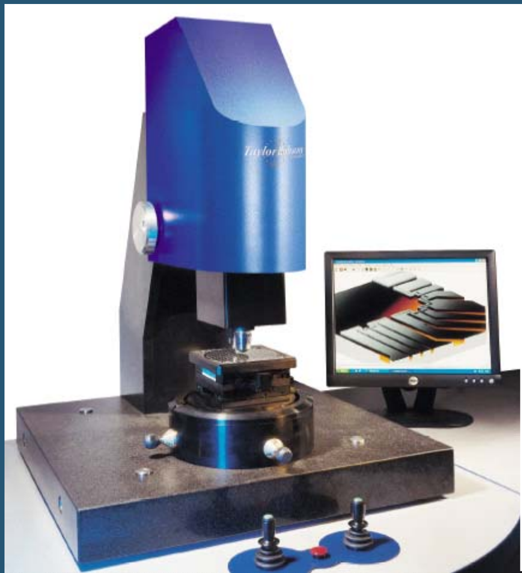


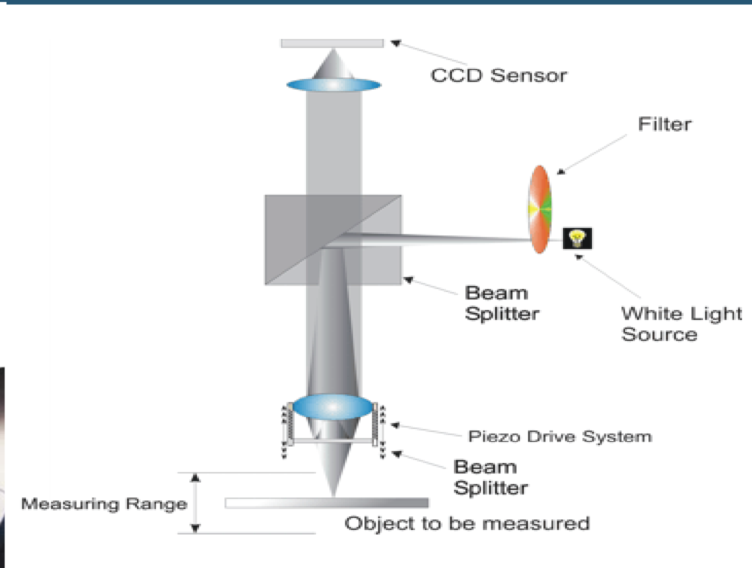


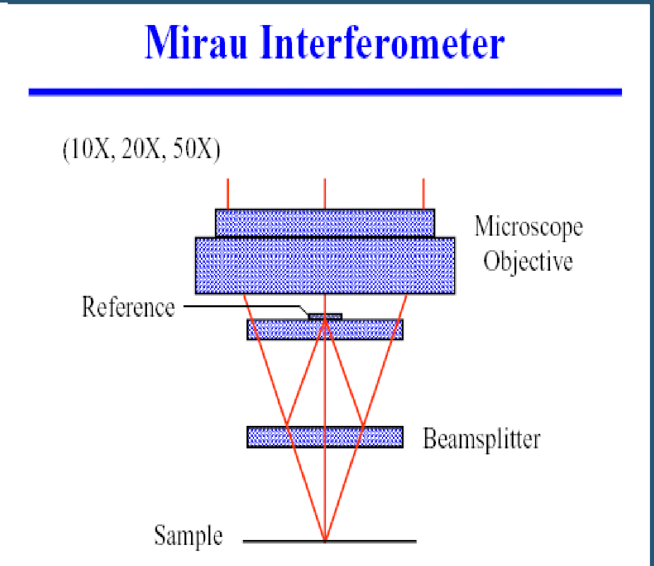
Gyro profiles



New Metrology







- **The TalySurf CCI is a new type of measurement interferometer, called a Coherence Correlation Interferometer (patented by Taylor Hobson) which provides both high resolution and high sensitivity to returning light**
- **This method combines the surface imaging quality of a high powered microscope with the accurate measuring capability of a surface profiler**
- **As a results this instrument measures 3D surfaces, step height, roughness and micro dimensional measurements over small areas (7.2mm square or smaller)**
- **Ideal for both rough and smooth surfaces. Typical 0.01nm vertical resolution, lateral resolution from 0.4µm* and up to 10mm vertical range* (*objective lens dependant)**

Particle measurement

Physical & Surface properties

- **Materials**
- **Devices**
- **Catalysts**
- **Pharma / biotechnology (bioactive agents)**

Small-angle x-ray scattering systems (SAXS)
Ultra-small-angle x-ray scattering (USAXS)
Small angle neutron scattering systems (SANS).

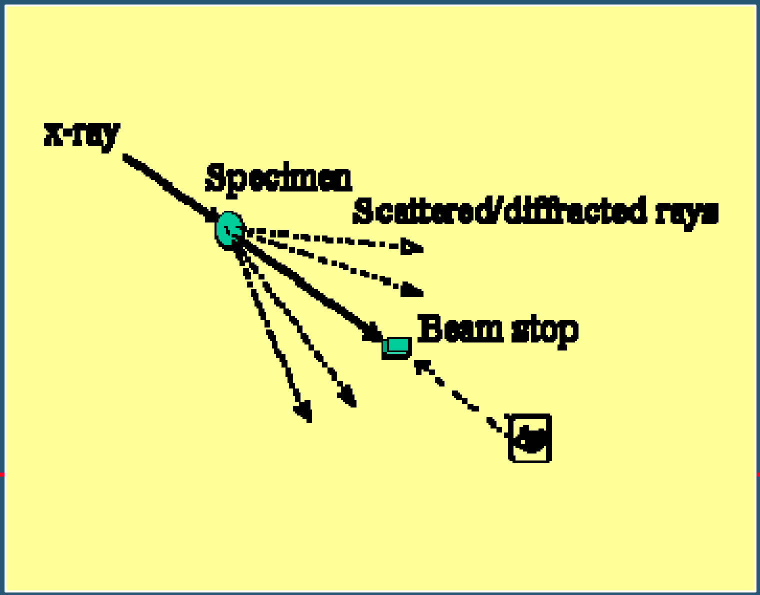
Ultra-small-angle x-ray scattering (USAXS)

Ultra-small-angle X-ray scattering (USAXS) provides quantitative, statistically significant, volume-averaged information on microstructures ranging in size from a few nm to over 1 µm. The technique provides direct images of the scattering objects and their spatial arrangement.

Whereas small-angle X-ray scattering (SAXS) is used to study the structure of polymers on a nanometre scale, ultra small-angle X-ray scattering (USAXS) extends the observable length scale towards structures ranging in the order of magnitude of hundred nanometres up to micrometres.

Ultra-small-angle x-ray scattering (USAXS)

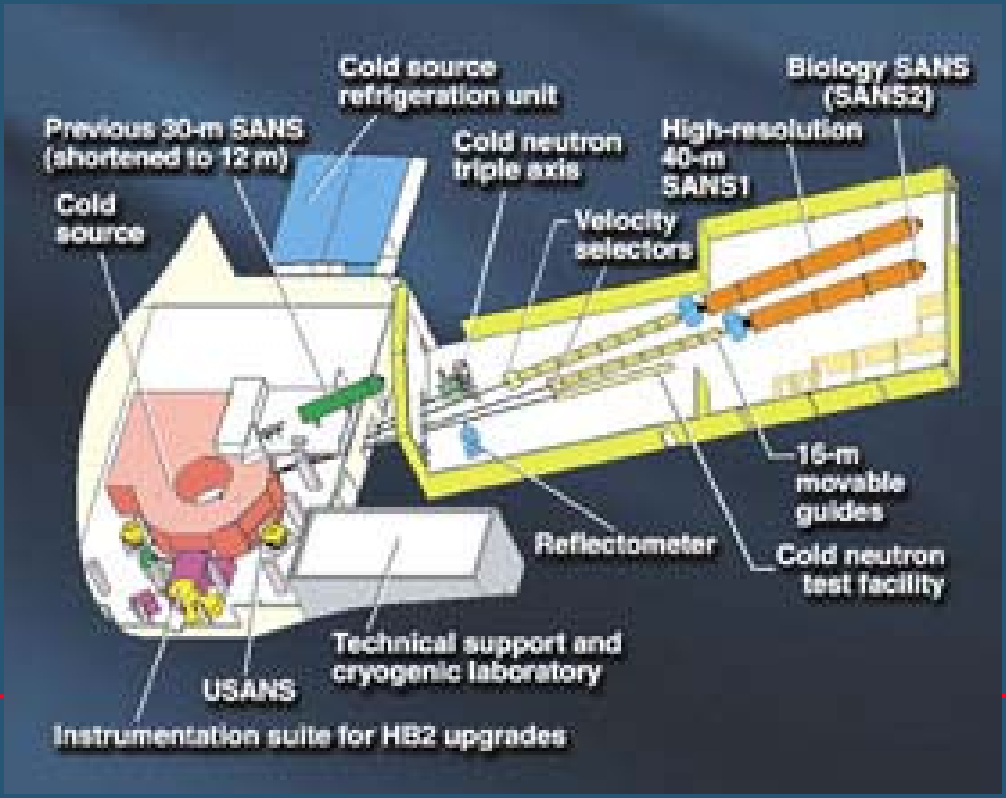
It is performed by focusing a low divergence x-ray beam onto a sample and observing a coherent scattering pattern that arises from electron density inhomogeneities within the sample. Since the dimensions typically analyzed are much larger than the wavelength of the typical x-ray used (1.54Å, for Cu), dimensions from tens to thousands of angstroms can be analyzed within a narrow angular scattering range. This angular range or pattern is analyzed using the inverse relationship between particle size and scattering angle to distinguish characteristic shape and size features within a given sample.



Small Angle Neutron Scattering (SANS)

Neutrons can be used to study nanoparticles themselves and also to examine protective coatings that are placed on the nanoparticles to prevent oxidation. Neutrons can also be used to study dispersants that disperse nanoparticles in a solvent or host medium.

Oak Ridge National Laboratory. ORNL is a multiprogram science and technology laboratory managed for the U.S. Department of Energy by UT-Battelle, LLC.



Measurement Techniques

-Visible

-Optical Microscopy

-Electron beam Microscopy

-Tactile

-Probe

-Functional Effect

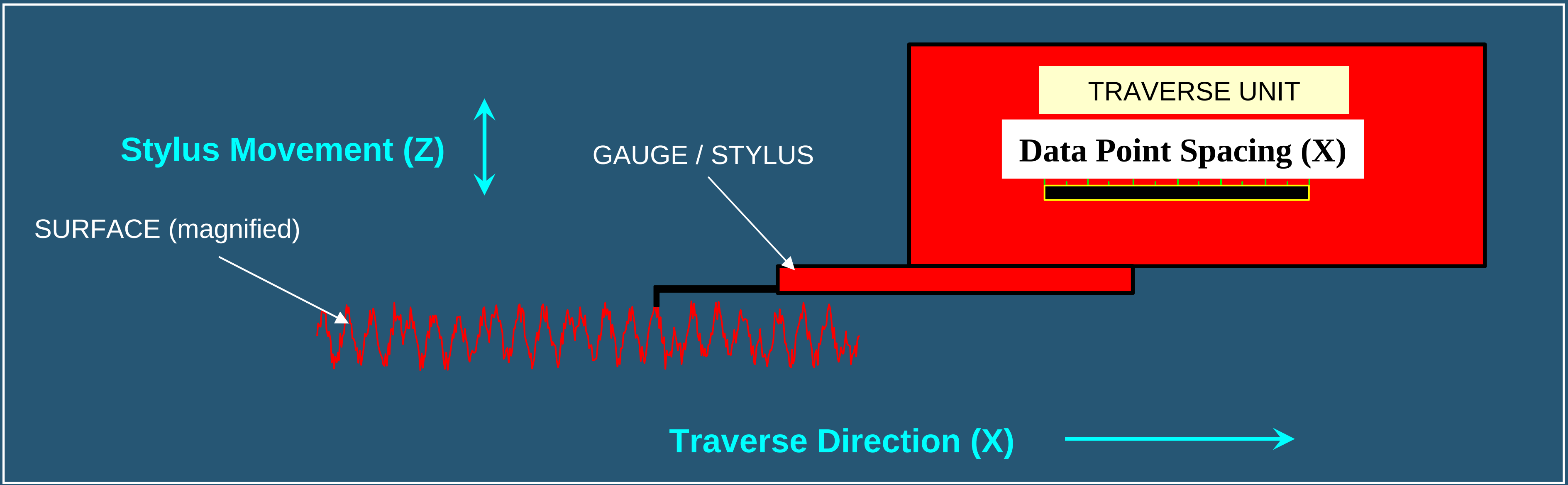
-Chemical

-Thermal

-Magnetic

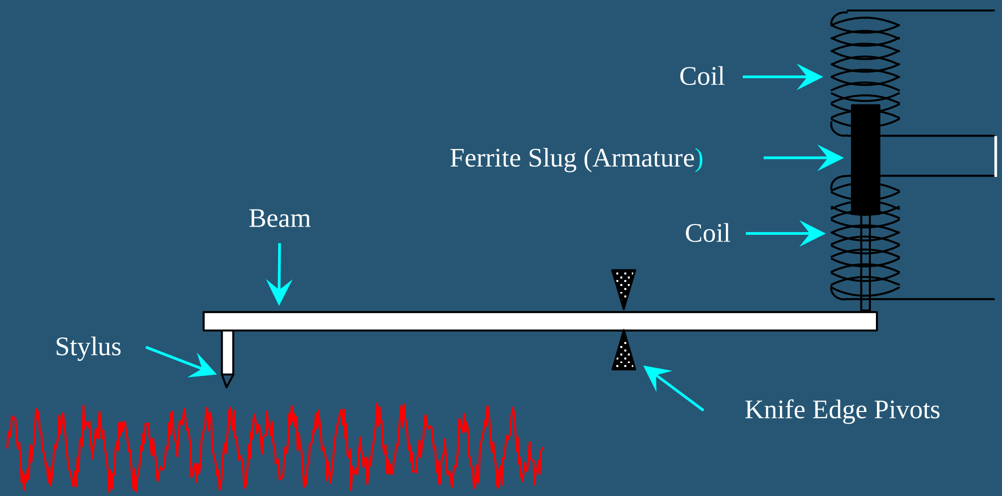
-Optical

Stylus contact instrument



- Stylus is dragged across surface (~0.5 - 1mm/s) by traverse unit.
- X datum provided by either a skid or straight datum
- Gauge transducer converts Z (surface) movement to an electrical signal.
- Z data sampled in X - spatial (grating) or timed from motor drive.
- Resulting digital data processed via software for user analysis.
- Z data range and resolution dependant on type of gauge transducer
- X spacing usually ~0.25 - 1um. Grating is more accurate than timing.

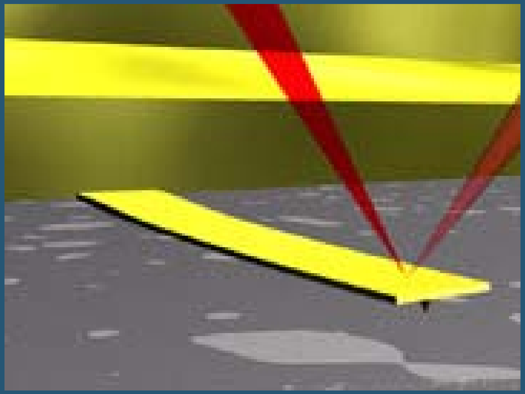
Inductive

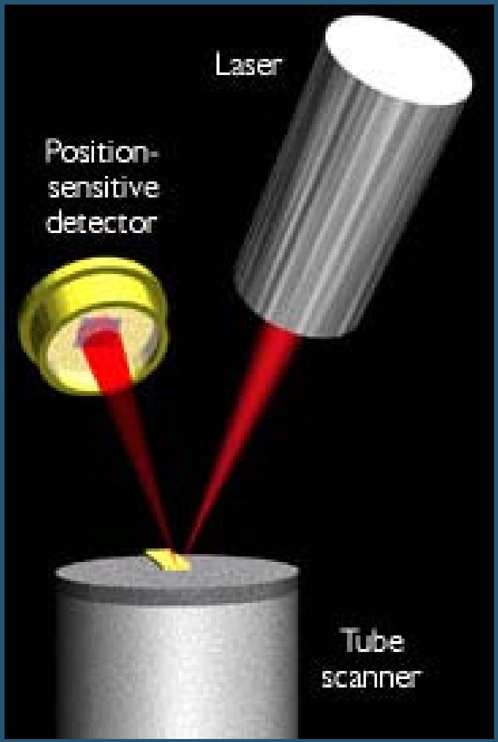


- The inductive gauge is traced across the component to be measured
- Changes in the surface deflect the stylus in a vertical direction (Z input)
- This deflection moves a slug in between two coils which alters the inductance in the circuit
- The change in inductance is monitored by the system as stylus movement (Z output)
- The gauge is calibrated for gain using a standard Ra roughness patch.

Atomic Force Microscopy (AFM)

An AFM operates by measuring attractive or repulsive forces between a tip and a sample. It measures topography using a force probe.





Transmission Electron Microscopy

Electron Microscopes use a beam of highly energetic electrons to examine objects on a very fine scale. This examination can yield the following information:

Topography

The surface features, its texture; direct relation between these features and materials properties (hardness, reflectivity...etc.)

Morphology

The shape and size of the particles making up the object; direct relation between these structures and materials properties (ductility, strength, reactivity...etc.)

Composition

The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties (melting point, reactivity, hardness...etc.)

Crystallographic Information

How the atoms are arranged in the object; direct relation between these arrangements and materials properties (conductivity, electrical properties, strength...etc.)

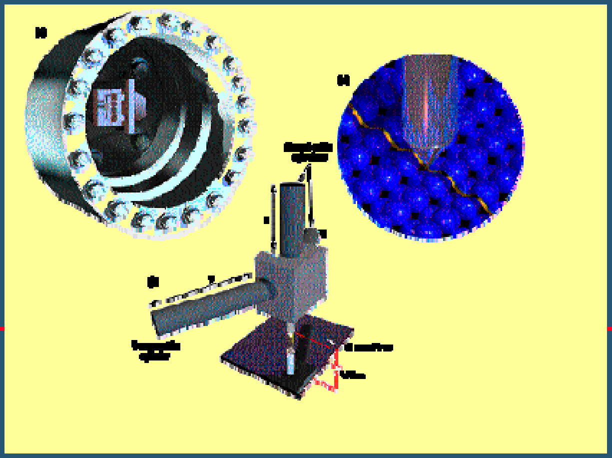
.

STM Vs AFM

The scanning tunnelling microscope (STM) provides a picture of the atomic arrangement of a surface by sensing corrugations in the electron density of the surface that arise from the positions of surface atoms.

AFM has, it is believed, surpassed the resolution of STM. With AFM one not only see the atoms added to the surface, but also the rest of the atoms in the lower layer. With STM this is not possible, because the system probes electron clouds of the surface atoms. AFM allows us to do spectroscopy at specific sites. When we vary the interaction, we can do chemical analyses at different sites, of non-conducting surfaces. This opens up a window of opportunity for surface science

A finely sharpened tungsten wire is positioned within 2nm of the specimen by a piezoelectric transducer. At this small separation, through the principles of quantum mechanics, electrons "tunnel" through the gap. If a small voltage (bias) is applied between the tip and the sample, then a net current of electrons (the "tunnelling current") flows through the vacuum gap in the direction of the bias. As the tip scans the surface, corrugations in the electron density at the surface of the sample cause corresponding variations in the tunnelling current, thereby, producing a 2D map of the electron density at the surface.



Optical Profilometry: A comparison of Advantages

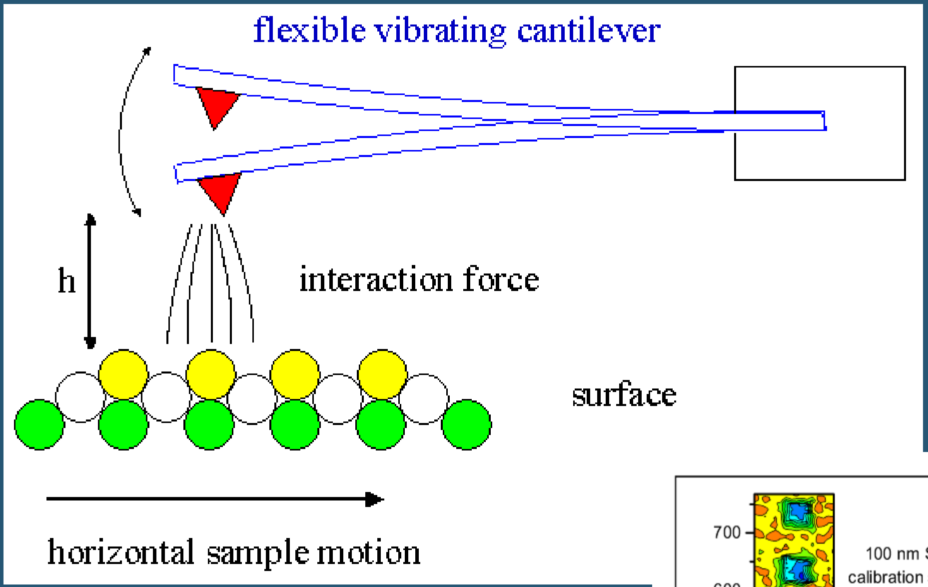
White light chromatic aberration	White light Interferometery & Phase shift Technology	Laser Triangulation	Stylus Contact	AFM
Good Lateral Resolution down to 1.7micron.	Very good Lateral resolution down to 0.3microns	Good for metrology work	Lowest price Technique for standard systems	Best lateral resolutions for detail imaging
Works on high aspect ratio surfaces and angular surfaces. This allows to test all types of materials including paper, textile, sand paper	Uses a CCD camera to take roughness measurement in a few seconds.	Somewhat faster than Chromatic Aberration Techniques	Depending on design can test steep angular surfaces	Can reveal surface structure in details
Works on materials of any colours and reflectivity.	Stitching technique allows to cover large surfaces	Lower price technique compare with chromatic aberration because of its limit		
Quick, up to 4000points per second.	Ideal for many microelectronic applications			
Wide range from nm to 24mm combine with good lateral resolution allows both metrology and surface roughness to be measured.	Excellent Z resolution down to 0.1nm			

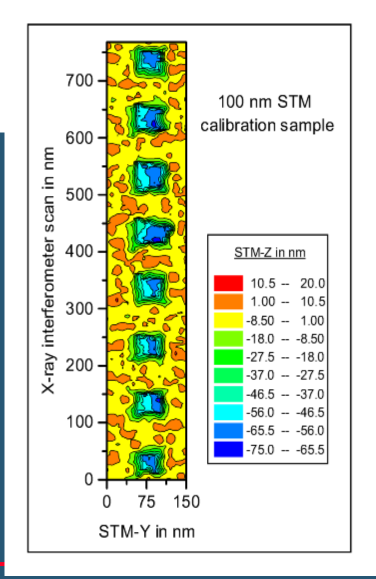
Optical Profilometry: A comparison of Disadvantages

White light chromatic aberration	White light Interferometery & Phase shift Technology	Laser Triangulation	Stylus Contact	AFM
Some applications require higher speed to measure quick roughness on surfaces provided by White light Interferometery .	Difficulty on high angular surfaces or surface where line of interference pattern are difficult to see. Chromatic Aberration Technique works better on these surfaces.	Because of the shadow effect caused by incident light not being perpendicular to the surface, it is impossible to measure high aspect ratio surfaces , also part of the information will be missing if the surface is rough.	Tip size often larger than 10microns but can be down to 1microns in higher priced system	Slow compare to white light systems
Higher resolution in Z direction might be needed. Phase shift technology goes down to 0.1nm	More limited than White light chromatic aberration in the Z direction. Stitching allows a few mm.	Limited use compare with Chromatic Aberration on high angular surfaces	Depending on size of probe and load applied, deformation may occur on softer materials such as polymers or plastics	Requires more knowledge for use.
		Lateral resolution limited to 30microns in most systems and sometime down to 10microns. This with also a poor Z resolution limits the use for roughness measurements	Normally 2D instruments	Relatively small surfaces can be tested (100microns by 100microns)
		With standard wavelength it is impossible to measure dark blue surfaces or black surfaces	Slow especially if 3D imaging is performed.	

Current metrology tools – **Scanning probe microscopes – have limitations**

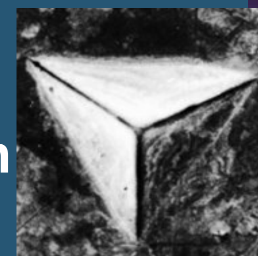
- Most common for MST is the AFM
- Limited range, but high resolution
- Recent comparisons show large discrepancies – "…*each AFM has its own nanometre.*"*
- NPL developing reference standards using combined x-ray and optical interferometers (COXI)
- NPL developing traceable ultra-low force standards



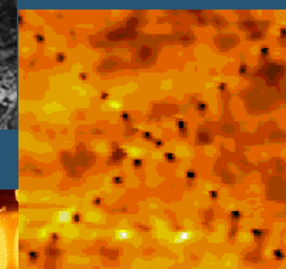
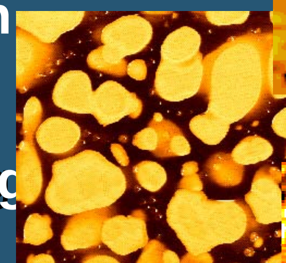
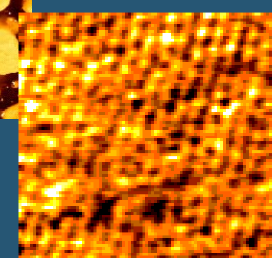
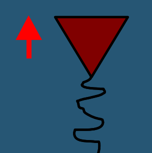


Force Measurement scale

Force	Material	Technique
kN+	Bulk	Indentation
N	Thin Films	Microindentation
nN +	Surface zone	Nanoindentation
nN	Surface layer	Modulus mapping
pN	Single Molecules	Molecular pulling

Indents

SAM

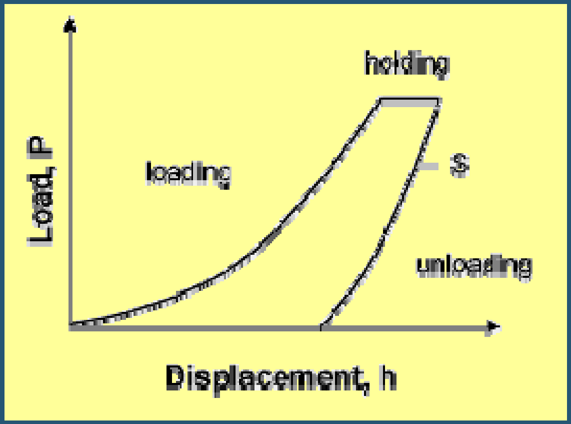
Probe indentation and associated metrology

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

Nano-indentation as a measurement technique

Mechanical properties

- **Thin films**
- **Small volumes**
- **Standards and standard refernce materials**



The most commonly applied means of testing the mechanical properties of material where a hard tip, typically a diamond, is pressed into the sample with a known load.

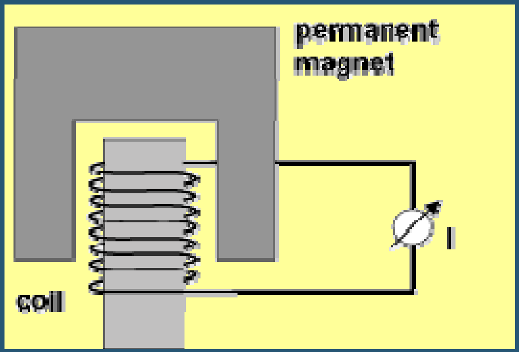
After some time, the load is removed. The area of the residual indentation in the sample is measured and the hardness, H, is defined as the maximum load, P, divided by the residual indentation area, Ar , or

$$H = P/Ar$$

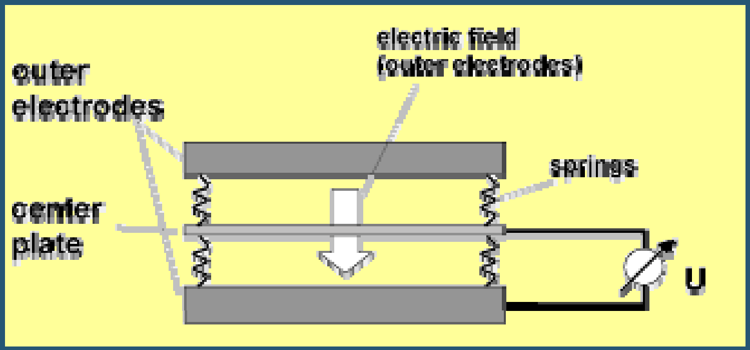
Nano-indentation as a measurement technique

Nanoindentation refers to depth-sensing indentation testing in the submicrometer range using equipment that can produce nano indentations while recording load and displacement with very high accuracy and precision

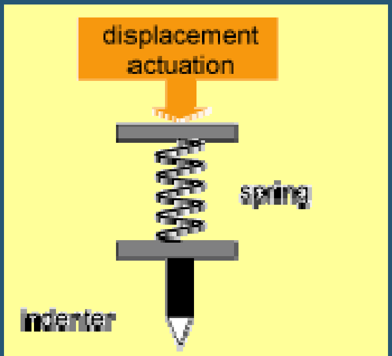
The equipment is also able to provide analysis models by which the load displacement data can be interpreted to obtain hardness, modulus, and other mechanical properties.



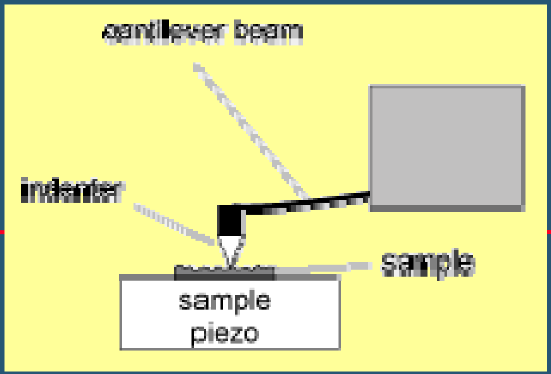
Electromagnetic actuation



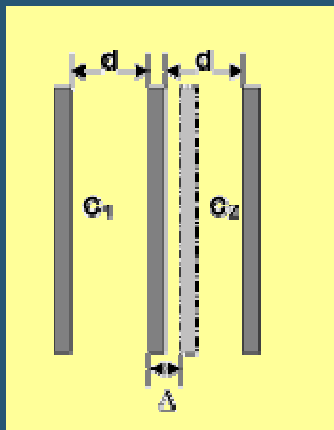
Electrostatic actuation



Spring / cantilever actuation

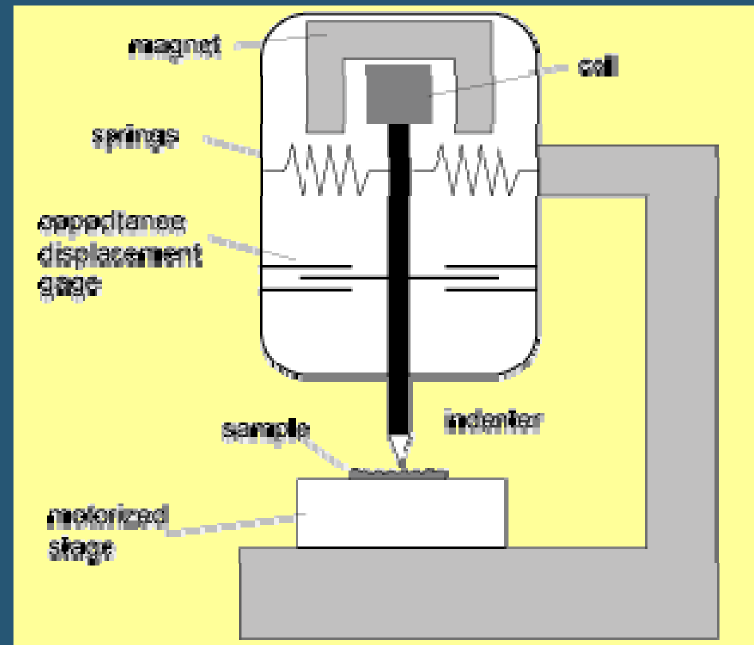


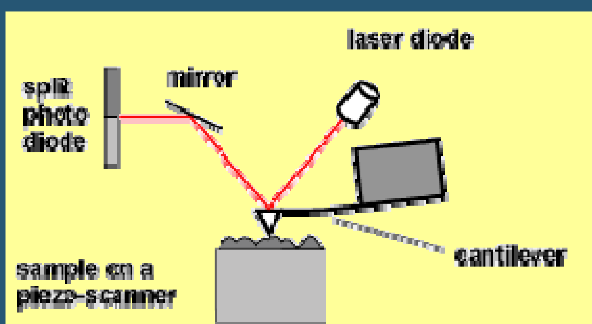
Nano-indentation instrumentation



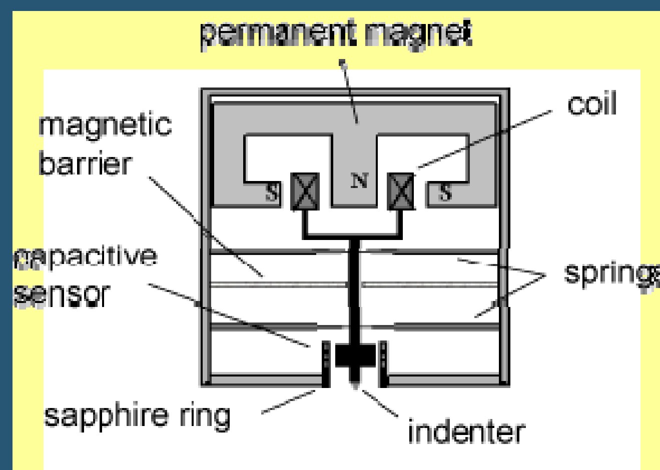
Capacitive displacement

Nano Indenter





Optical beam angle displacement



Nano
Hardness
Tester

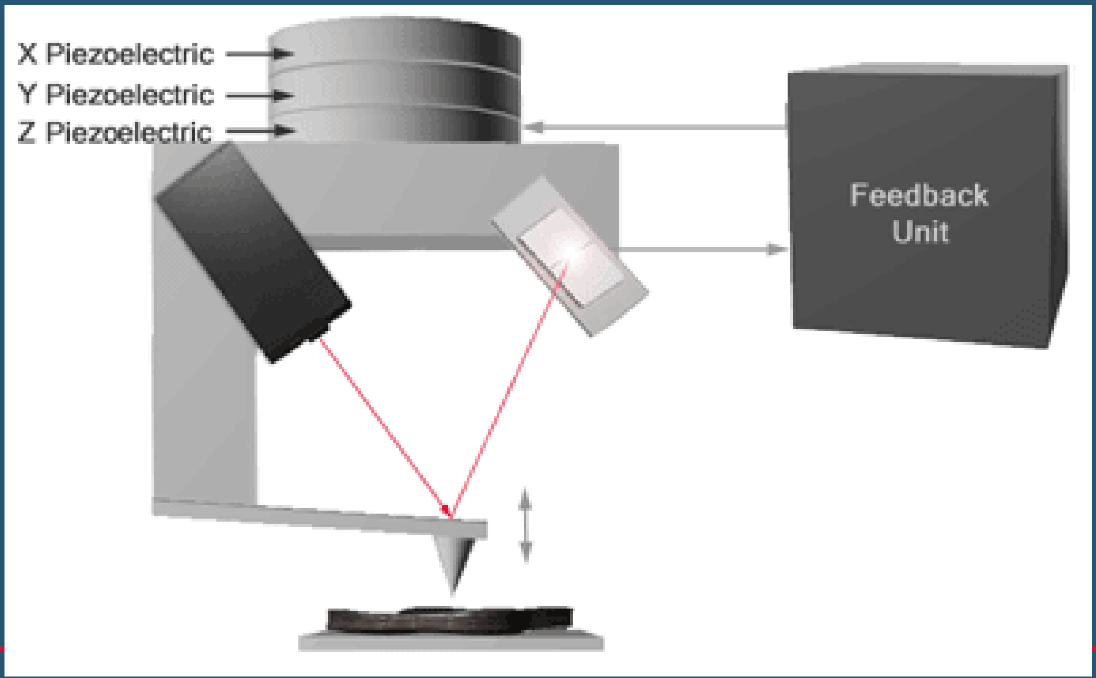
Nanotribology & surface properties

Tribology is the methodical study of adhesion, friction, lubrication, and wear.

Controlling surface textures / surface properties
Reliability & durability
Lateral forces (nN)
Scanning Probe Microscopes (SPM) ; Atomic Force Microscopes (AFM)

Nanotribology & surface properties

In an AFM a constant force is maintained between the probe and sample while the probe is raster scanned across the surface. By monitoring the motion of the probe as it is scanned across the surface, a three dimensional image of the surface is constructed.



Pacific Nanotechnology, Inc.

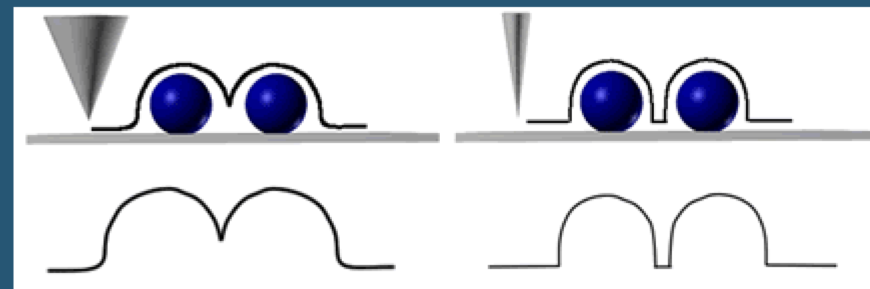
Nanotribology & surface properties

AFM Issues;

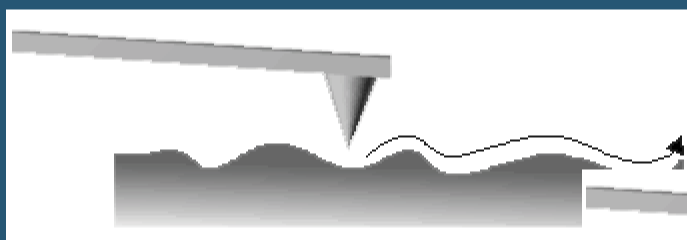
Surface contamination

Electrostatic forces

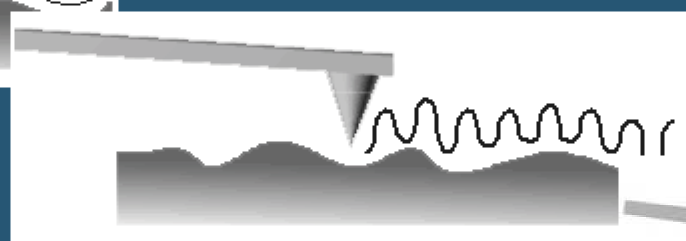
Surface Material Properties



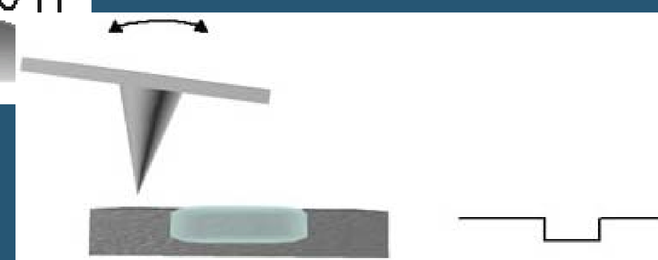
Resolution



Deflection Mode



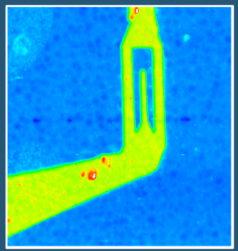
Vibration Mode

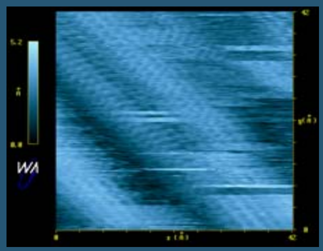


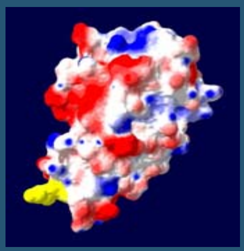
Torsion Mode

Role of low force standards in nano-bio

- Nanoscience needs traceable measurements of sub-*nano-Newton* forces.
- This is particularly applicable to single bio-molecule studies.
- Optical radiation pressure seems to be ideally suited..
- Work in liquids remains to be developed, will require positive feedback response enhancement.







Measurement Techniques

-Visible

-Optical Microscopy

-Electron beam Microscopy

-Tactile

-Probe

-Functional Effect

-Chemical

-Thermal

-Magnetic

-Optical

Surface chemistry & structure measurement

Surface bound molecules

- **Self assembled monolayers**
- **Molecular templates**
- **Lubricants**
- **Nanotubes**

Near-Edge X-ray Absorption Fine Structure Spectroscopy (NEXAFS)

Measures (non-destructively) chemical bond concentration, Orientation etc

Surface chemistry & structure measurement

Near-Edge X-ray Absorption Fine Structure Spectroscopy (NEXAFS)

Measures (non-destructively) chemical bond concentration, Orientation etc

Tunable photons from a synchrotron radiation beamline are used to interrogate the likelyhood of absorption** (close to an absorption edge and about the first 30eV above the actual edge) **in a compound as a function of photon energy. The spectrum thus obtained reflects the chemical composition and the bonding environment of the compound. Quantitative compositional analysis of materials can be performed with a few per cent accuracy if spectra of relevant model compounds are utilized.

Today, the term NEXAFS is typically used for soft x-ray absorption spectra and XANES for hard x-ray spectra.

Effects-based Nanometrology techniques

Brillouin light scattering (BLS) provides a way of characterizing acoustic waves (phonons) or spin waves (magnons) by measuring the direction and shift in frequency of inelastically scattered light. Used for measuring the interactions of spin waves in ferromagnetic films, elastic constants of nonporous dielectrics and propagation of acoustic waves in nano-patterned polymers. (1 GHz to 1 THz).

Electron Backscattered Diffraction (EBSD) is a technique which allows crystallographic information to be obtained from samples in the scanning electron microscope (SEM). With EBSD, a stationary electron beam strikes a tilted crystalline sample and the diffracted electrons form a pattern on a fluorescent screen. Used to assess strain, stress, crystalline defects and texture.

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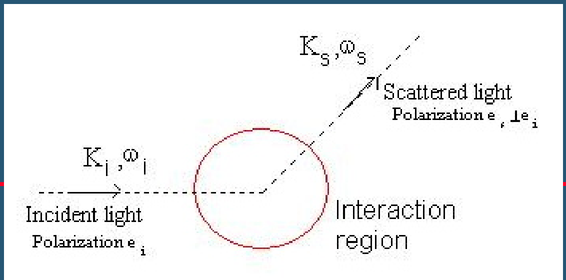

Thermo-chemistry & interfacial interactions measurement

In-line processing & kinetic modelling

- **Electronic devices**
- **Complex materials mixtures (metals, ceramics, semiconductors)**
- **Temperatures > 900oC**
- **Transition from micro-scale (bulk dominated) to nano-scale (surface dominated)**

A concept for modelling chemical reactions, by studying the generic and the individual elementary reactions taking place.

(Nano) Metrology and the future challenge

- Improved techniques to measure and characterise nanoscale processes and structures
- Development of reliable and repeatable metrological standards at the nanoscale
- Identifying standard reference materials for calibration and reproducibility of nanoscale measurements
- Development of standardised instruments with nanoscale resolution
- Development of analytical instrumentation with enhanced sensitivity in the nanoscale.
- Development of quantitative models for interpretation of data, images and results in general
- Development of new analytical approaches to characterise novel materials (e.g. soft materials, biological systems)
- Development of instrumentation with nanometre-position accuracy / resolution.
- Development if new fundamental standards (e.g. quantised electron devices)

Further Information

For market intelligence and industry reviews:

www.enablingmnt.com

For advanced applied technology and research:

http://www.baesystems.com/ocs/sharedservices/atc/index.htm

For systems engineering solutions:

http://www.seic-loughborough.com

SEIC

For metrology & characterisation:

http://www.lboro.ac.uk/research/cemmnt/

For networking:

http://www.nexus-mems.com

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