Measurement in the nanoworld

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Setting the scene

Scientific discovery and technological developments are forging ahead at an ever-faster pace. There are clear trends for scientific endeavour to lead to products that are becoming increasingly miniaturized, multi-functional, diverse and more intelligent. And, whilst there are evolutionary advances in virtually every field of technology, especially in nanotechnology, new and often unexpected scientific discoveries are being made at the boundaries between traditional disciplines. Within this exceptionally complex, and dynamic, landscape there is a crucial requirement to be able to measure what is happening in order to control, develop and understand new processes, phenomena and material properties.

To a significant extent, the foci of new discoveries and related commercial opportunities are centred on nanoscience and nanotechnology. As yet, however, the vital rôle of metrology as a way of identifying and removing scientific and technical roadblocks is largely unrecognized. Furthermore, metrology still has the stigma of the "inspection culture" rather than being seen in its true light, where the application of metrology has potential to add value by enabling processes to work more efficiently and by improving quality. To take one example, the latest mobile camera phones are appealing because they are small, through state-of-the-art electronic circuitry, and incorporate advanced displays and optics facilitated by the manufacturer's ability to measure complex components with exceptional accuracy. At the moment, critical dimensions at the very heart of mobile phones need to be known with uncertainties in the region of some tens of nanometres, but it will not be too long before the measurement requirements will become even more demanding. What is more there are many other examples where the ability of metrology to create new commercial opportunities is not well known; for example the potential for improving the critical functionality of microfluidic channels through metrology of their size, shape and surface texture.

Already, metrological instruments are available that address some of the technical research and industrial measurement requirements at the cutting edge of the nanometric range. Crucially, however, the confidence that might be placed in the measurement data collected by such

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instruments and their technical performance is somewhat problematic. Such shortcomings make improving instrument performance and developing new measuring tools to address these shortcomings critical for further technical developments in such fields as microfluidics and advanced sensor technology.

One of the key issues is that real measurement problems span a very wide range. Often measuring instruments do not exist to cover the full range of measurement values, and some measuring problems fall between the ranges of available instruments where there is no overlap. This can really only be addressed by having a very comprehensive set of measurement tools that can provide sufficient coverage for practical needs. At present, for the transition region between micro-scale and nano-scale metrology, this is far from being the case!

What issues drive the measurement requirements?

At whatever accuracy and for whatever physical quantity, traceability is the key principle of metrology, i.e. the ability to demonstrate that a measured value of a quantity and its associated uncertainty can be traced from the measurement itself right back to a national or international realisation of the unit through an unbroken chain of comparisons.

In the "traditional" metrological world, there are a number of physical quantities, mass, length, time, and so on, the measurement of which covers many orders of magnitude. Here, the challenge is to measure as accurately as makes financial sense, in order to support industrial competitiveness. Industrial users making such measurements would like to have transfer standards to enable them to access the international realization of the measurement unit in a cost-constrained and expeditious way, thereby ensuring that their industrial measuring instruments made traceable measurements.

This approach can, of course, be extended to the derived units, i.e. where more than one base SI unit[1] is involved, such as pressure and force. In some cases, measuring small forces and tiny lengths with ever-higher accuracies is important in order to support various specialized aspects of nanotechnology. Examples are: calibrating the cantilevers of atomic force microscopes for protein manipulation experiments where traceable nanonewton forces are required; and in the field of evaluating the long-term dimensional stability of ultra-stable materials traceable length measurement at the picometre level is needed. However, demonstrating traceability by means of reference artefacts becomes increasingly challenging as size diminishes towards the region of the nanoworld. In many cases it is better to utilize new approaches to develop standards with intrinsically smaller dimensions rather than strive to continuously subdivide the metre, for example.[2]

Because the development and uptake of physical standards is relatively slow, such standards require ongoing, long-term research and development in order to meet current and

[1] SI unit: belonging to the Système International d'Unités based on the metre, kilogram and second.

[2] G.N. Peggs and A. Yacoot, "A review of recent work in sub-nanometre displacement measurement using optical and X-ray interferometry". *Phil. Trans. R. Soc. Lond.* **A 360** (2002) 953–968.

future requirements from researchers and industrial producers. But, there is now a rapidly increasingly number of additional practical factors to be taken into account. These arise from anticipated needs, such as:

· non-contact measurements, where the surface, or feature, under test is not touched, and hence not damaged, by the measuring instrument;

· dynamic measurements, where the measurand is varying either periodically, or steadily changing, or changing in a stepwise way; and

· measurement in hostile environments, where extremes of a different physical quantity might disrupt the essential measurement.

Even for relatively simple measurements in the macroworld these additional requirements add considerable complexity to the design and implementation of a metrology process. As an example of the many factors involved in a complex measurement, the requirement to be able to characterize magnetic properties of a surface is an important driver for the next generation of computer memories. But the additional requirement to have excellent (submicrometre) lateral spatial resolution tends to make such measurement techniques very demanding, and this is exacerbated when considering very thin multifunctional films, which need exceptional (nanometric) vertical spatial resolution.

For the nanoworld, all the complexities discussed above still apply. However, in addition there can be several extra complications associated with: variations in material properties with physical scale; multimaterial devices as might be found in biological applications; and transitions of material characteristics when changing from a classical to a quantum physics régime.

How to deal with metrology at the extremes

Although very difficult, it is perhaps possible to differentiate between various nanometrology issues that arise from nanofabrication, nanoassembly and nanoproduction, and those arising from determining the composition and the properties of component parts, (nanocharacterization).

In the case of nanofabrication, the key issues concern the ability to make structures that are only one, or a few, atoms wide, either by a process involving self-assembly techniques, or by ultraprecise nanomanipulation. Arguably, such techniques can be based on quantum metrology, i.e. by counting atomic layers or individual atoms and hence essentially eliminating the problems of determining linearity. Such quantum approaches hold great promise, but unfortunately many practical measurements are in the transitional region between the conventional and the quantum régimes, making neither approach particularly convenient.

Nanomanufacture—nanofacture—can be thought of a direct analogue to conventional manufacture but using advanced techniques, such as femtosecond laser machining, to create structures on the submicrometre to nanometre scale. So far, the only inspection tools for nanofacture are those that have their genesis in the flatlands of the semiconductor industry and are, as yet, unable to measure the truly three-dimensional structures that will be absolutely essential for future developments where much closer integration of components will be vital.

Therefore, the real challenges for the future of nanofacture are developing the ability to perfect, inspect, and control the production process to make 3-dimensional measurements at extreme levels of fractional accuracy, so that tiny features on small structures can be replicated in large numbers in the same way as the semiconductor industry developed with 2.5-dimensional structures on wafers.[3] This will then enable the high levels of integration of multifunctional, multimaterial components needed for the next generations of advanced sensor systems.

At the level of the materials and components from which complex nanostructures are made, the real metrological challenges arise mostly from requirements to make measurements of material properties and composition such as magnetism, force, hardness, chemical composition and so on, on tiny structures such as nanowires, carbon nanotubes and ultrathin films.

Irrespective of whether the focus of the metrology is a complex device or a structural or functional element of the whole, two universal requirements for successful metrology at the nanometric scales seem to apply: good spatial (lateral) resolution must be achieved whatever the parameter to be measured, and valid measurement data in the presence of many potentially perturbing influences must be collected.

Finally, it has to be remembered that measurements made, for example, with nanometric vertical and horizontal resolutions or by hyperspectral systems collect tremendous amounts of data. Such data, which is often multidimensional, requires terabytes[4] of storage space. This intense data production is already, to some extent, being handled, and visualized, using neural networks, multivariate analysis and so on, but even so, issues, associated with the amount and complexity of data are likely to become a significant problem in the relatively near future, not only for nanometrology, but also for other emerging technologies where vast amounts of data are collected. Innovative data management is, however, moving to provide a series of new opportunities for creating more reliable, and secure, measurements through self-correcting and self-calibrating systems and networks.

The nanometrology challenge

Many, if not all, the issues described previously require a paradigm shift in thinking to develop the new generations of instruments needed for nanometrology. In the nanoworld the requirements for specifying the measurement data required are relatively easy to define, but incredibly difficult to deliver with the degree of reliability and levels of confidence expected from metrology at more conventional size régimes.

Frequently measurement solutions do exist, but they require access to equipment at exceptional levels of sophistication, such as particle accelerators, and might also need a veritable armoury of expensive ancilliary analytical equipment for the measurement of additional physical parameters that might influence the actual measurements. What is more the data

[3] Wafers: thin (approximately 10–100 μm) slices of silicon cut from large (diameters ranging from 80 to 300 mm) silicon single crystals.

[4] Terabytes: tera is the prefix indicating 10 to the power 12, or approximately 2 to the power 40; one byte corresponds to 8 bits of data, which usually represents 1 character.

collection may well be far too slow, the measurement process might risk damaging or destroying the surface under test, and the instrument might need to operate in an environment hostile to the component, such as ultrahigh vacuum.

Just taking the apparently simple requirement to measure composition and location of nanoparticles or nano-objects generally, note that: one might need a transmission electron microscope to measure the precise shape of the nanoparticles, a synchrotron to perform X-ray tomography, or electron holography techniques to establish internal atomic structures, and finally picometric resolution interferometry to determine the relative positions of particles. At present only a few of these measurement processes are remotely practical within realistic budgets and timescales. Thus one has to rein back on the state-of-the-art approaches to simultaneously addressing all aspects of the problem and optioneer.[5]

Thus, in order to meet the need to provide affordable solutions, the challenge is to measure in such a way as to achieve multiparameter results so that, for example, chemical, spatial and mechanical data can be collected simultaneously. This might be achieved by the close integration of measurement techniques in a multitasking scanning probe microscope with functionalized tips capable of providing spectroscopic data with nanometric resolution.

The eternally ingenious and resourceful metrologist

With such daunting challenges for metrology in the nanoworld, it is hard to believe that much progress will be made towards economic solutions delivering the much needed information about the effectiveness of a nanomanufacturing process, or the composition and mechanical properties of a new type of multilayer thin film. However, if the lessons of recent times are any indicator, one may have every expectation of spectacular developments in the field of metrology in the next few years.

A good example upon which to base optimism for the future development of metrology is the continuing applicability of Moore's Laws, which are essentially global statements of a trend, or a technical requirement, whereby computing power increases exponentially with time, but the cost of production facilities rises less quickly, resulting in no increase in the cost of production of a square centimetre of computer chip, even though far more electronic components are embedded in it.[6] In the early phases of microprocessor development, microprocessor speed was a direct consequence of the dimensional accuracy of the critical features of the transistors on the chip and the relative positions of one to another. Metrology tools were often based around optical microscopes operating in the visual part of the spectrum. Their performance was determined by the characteristics of the objective lens and the illuminating radiation.[7] By using relatively conventional microscope systems with water immersion objectives, 193 nm

[5] Optimize the possible options with the aim of establishing a practical measurement approach.

[6] *Chips with everything*, Evidence to the House of Lords Select Committee on Science and Technology, HL paper 13-II, 2002.

[7] Rayleigh's criterion is $d = k_1\lambda/N$, where d is the minimum resolvable feature, k_1 is a constant, λ is the wavelength of the illuminating radiation and N is the numerical aperture of the objective lens.

illumination, and employing various optical tricks, it is possible to get down to spatial resolution, for the wafer steppers, in the region of 70 nm—a feat considered highly improbable until a few years ago. But such seemingly matter of fact statements belie the massive investment in the infrastructure and technical developments needed to support sophisticated sources capable of working at ever shorter wavelengths, coupled with the investment in the technologies required to make the optics perform within exceptionally strict limits of aberration (wavefront errors and lens alignment requirements in the region of 1 nm) and with exceptionally low levels of light scattering (implying surface texture in the region of $R_a = 0.2$ nm). Moreover, such optical systems are embedded into highly elaborate fabrication complexes and have to perform faultlessly over long periods of continuous operation. Thanks to these technological achievements, undoubtedly Moore's Law is safe for a few years yet.

Conclusions

Nanometrology and nanoscale instrumentation is one of the "Grand Challenges" for the future.[8] Despite some negative perceptions in the past, metrology already has, and will continue to have, a remarkable track record in facilitating real advances in science and technology. Moreover, metrologists will certainly continue to be inspired by current and future opportunities to push back the barriers to allow fresh horizons to unfold in this uniquely fascinating field of scientific endeavour.

[8] See http://www.ostp.gov/NTSC/html/NTSC.Home.html