RSC Nanoscience & Nanotechnology

Nanocharacterisation

2nd Edition

Edited by Angus I Kirkland and Sarah J Haigh

ROYAL SOCIETY OF CHEMISTRY

Nanocharacterisation
2nd Edition

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Preface

In presenting this second edition of a collection of articles under the common theme 'nanocharacterisation', it is apparent that since the publication of the first edition, the field of nanotechnology and the necessary development of advanced characterisation tools has continued to grow.

The origins of nanotechnology could be argued to have their roots in the fourth century AD in the Lycurgus cup which is made from a glass impregnated with nanometre-sized gold and silver particles that give rise to its famous dichroic effect. Later, nanotechnology was used extensively by the medieval craftsmen who produced the stained glass which adorns many of Europe's great cathedrals; their addition of gold chloride to molten glass produced tiny, uniformly sized gold spheres ($\sim$50 nm in diameter) which impart the deep ruby-red colour that is a characteristic feature of many famous windows.

The anticipation of many of the potential benefits of nanotechnology was highlighted in a lecture 'There's plenty of room at the bottom' delivered by Richard Feynman to a meeting of the American Physical Society in 1959. Describing a process by which the ability to manipulate individual atoms and molecules might be developed, Feynman conjectured:

> What could we do with layered structures with just the right layers? What would the properties of materials be if we could really arrange the atoms the way we want them? They would be very interesting to investigate theoretically. I can't see exactly what would happen, but I can hardly doubt that when we have some *control* of the arrangement of things on a small scale we will get an enormously greater range of possible properties that substances can have, and of different things that we can do.

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In the course of his lecture, Feynman also noted that scaling issues would arise from the changing magnitude of various physical phenomena and suggested that electron microscopy would play a key role in the characterisation of these materials.

The formal term 'nanotechnology' was defined in the mid 1970s in a paper presented by Norio Taniguchi[1] in which it was defined as 'the processing of, separation, consolidation, and deformation of materials by one atom or one molecule.' Subsequently, this basic definition was explored in greater depth by Eric Drexler,[2] who vigorously promoted the technological significance of nanoscale phenomena and devices.

In the latter part of the twentieth century two significant developments had a major impact on the characterisation of nanostructures: the invention of the scanning tunnelling microscope (STM) and the development of aberration correctors for electron microscopes. The development of the STM in 1981, for which Gerd Binning and Heinrich Rohrer were awarded the Nobel Prize in 1986, followed exactly 50 years after the development of the first transmission electron microscope (TEM) in 1931 by Max Knoll and Ernst Ruska, the latter sharing (somewhat belatedly) the same Nobel Prize. STM has in turn spawned a plethora of other scanned probe microscopy techniques including atomic force microscopy (AFM), magnetic force microscopy, electrostatic force microscopy and others. The development of aberration correctors for electron microscopes in the 1990s has not only improved spatial resolution but has enabled atom by atom spectroscopic and compositional analysis and has also enabled the introduction of complex environmental cells into larger gaps in the objective lens polepiece without sacrificing resolution.

At the time of writing 'nanotechnology' embraces a large number of methods that are used to fabricate and characterise structures on a size scale below ~100 nm and 'nanocharacterisation'—the theme of this book—describes some of the broad range of techniques that underpin much of this field.

In attempting to give an overview of the different characterisation techniques now in regular use we have been necessarily selective rather than comprehensive. Hence we note that other techniques are developing which will, in time find a place in the armoury with which nanotechnologists will pursue their quest for ever-smaller and more tightly controlled structures.

In the first chapter, devoted to high-resolution (transmission) electron microscopy (HRTEM), David Smith illustrates how this (relatively venerable) technique has evolved into a widely used tool in modern nanostructure research, in part driven by the development of aberration correctors. This chapter highlights some of the problems relating to the interpretation of image contrast, as well as providing numerous examples showing how HRTEM continues to provide unique insights into the local microstructure of nanocrystalline materials.

The development of the scanning transmission electron microscope (STEM) in the 1970s by Albert Crewe and colleagues produced instruments that could use various signals generated by scanning an electron beam across a thin specimen in a controlled manner. The chapter by Stephen Pennycook and Andrew Lupini outlines this technique and demonstrates how the recent application of sophisticated aberration-correcting devices in STEM instrumentation can produce sub-Ångstrom probes that can be used for imaging, composition determination, and spectroscopy of nanostructures.

Characterisation by TEM and STEM is complemented by the various scanned probe microscopy techniques which have emerged following the initial development of the STM. These instruments rely on a variety of surface/probe interactions and delicate rastering mechanisms to obtain images with extreme depth sensitivity, thus revealing many physical and electronic properties, often with atomic resolution. The ability to interact with *individual* atoms on surfaces in a precisely controlled manner also opens up an exciting range of opportunities for nanoscale manipulation and fabrication. Martin Castell in his chapter provides an overview of many of the scanned probe techniques and explores in detail the use of these in nanoscience.

In addition to structural information, local chemical composition, bonding, and electronic states determine the properties and performance of nanostructures. A range of spectroscopic techniques are now available for probing local structures on the atomic scale within the electron microscope. Electron energy-loss spectroscopy (EELS) and energy dispersive X-ray analysis (EDX) have emerged as the most powerful and widely used. These methods probe respectively, the energy loss of electrons passing through a thin specimen and the characteristic spectra of X-rays generated by the incident electron beam. The chapter by Rik Brydson provides an overview of the physical principles involved in EELS and EDX and demonstrates how these are now being used to probe local chemistry and bonding at the atomic level.

Nanometrology of the magnetic and electrical fields in individual nanoscale objects is increasingly important for many practical applications. Instrumental developments in electron microscope have enabled electron holography, first proposed by Gabor in 1947, to be used routinely to obtain measurements of these key properties. Rafal Dunin-Borkowski and coworkers describe the theory and practice of this technique and its application to a range of important nanostructured magnetic and electronic materials.

As nanostructures become smaller, their three-dimensional shape becomes an important parameter in determining their properties. This is particularly the case for supported catalysts, where active sites may be modified by changes in the surfaces exposed. Electron tomography has been applied successfully to biological structures and it has now been extended with a resolution suitable for applications in nanoscience. Paul Midgley and Mathew Weyland in their contribution survey the instrumental, theoretical, and computational requirements of this important technique and illustrate how it can be used to gain three-dimensional information from a range of technologically important nanomaterials.

The modern SEM is capable of providing exceptional surface detail at low voltage and the more recently developed scanning helium ion microscope (HeIM) provides a new range of contrast mechanisms and spectral signals. In the final chapter David Bell and Natasha Erdman illustrate the role of these instruments in nanoscale imaging and analysis using case studies from a range of nanomaterials.

In summary, our aim in preparing the second edition of this book has been to gather together and update a selection of articles, written by internationally recognised experts, which describe some of the characterisation techniques that are currently being used in the study of nanostructured materials. It is our hope that this new edition will continue to provide a valuable resource to all involved in the characterisation of nanomaterials.

Angus Kirkland, Oxford
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2. E. Drexler, *Engines of Creation: The Coming Era of Nanotechnology and Nanosystems: Molecular Machinery, Manufacturing, and Computation*, Anchor Books, New York, 1986. (An updated version of the book, Engines of Creation 2.0, which includes more recent papers and publications, has been published as a free ebook.).

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