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**EMERGING
TECHNOLOGY
SERIES**

1/1997

***New and Advanced
Materials***



**UNITED NATIONS
INDUSTRIAL DEVELOPMENT
ORGANIZATION**

Vienna, 1997

**EMERGING TECHNOLOGY
SERIES: NEW & ADVANCED
MATERIALS 1/1997**

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NANOTECHNOLOGY - Lead Article
Prof. P. Mckeown, UK

NANOTECHNOLOGY - OVERVIEW

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APPLICATIONS

UNIDO's *Emerging Technology Series: New & Advanced Materials* is established as a mechanism of current awareness to monitor developments in the marine industrial technology sector and to inform governments, industry and academia, primarily in developing countries.

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Dear Reader

UNIDO has long recognized that technology is at the core of competitive strategies of successful industrial firms. Access to reliable technical information can often allow manufacturers in newly industrialized countries to adopt state-of-the-art systems directly - without undertaking a painful and costly development phase. This series of UNIDO publications - the *Emerging Technology Series* - addresses a number of rapidly evolving technologies such as biotechnology, new materials and information technologies.

However, '*nanotechnology*' has implications for all the above newly evolving technologies and more. Some of the implications are practical realities today, while others are at the 'science fiction' stage. It depends on the definition of the term.

Literally 'nano' stands for one thousand millionth; in general nanotechnology refers to the manufacture of components with dimensions in the nanometric range. If the definition refers to one dimension being in this range, it is already almost 'routine'. Machine tool tolerances can now reach 1nm for surface 'flatness'. Examples are in the manufacture of aero engine turbine blades, camera lenses, telescope mirrors, silicon wafers for computer chips and hard discs. Move to two dimensions and x-ray and electron beam lithography can give features smaller than 50nm and 10nm respectively, but are not widely used at present.

Nanofabrication in three dimensions is the next goal and many international programmes are active in the industrialized world. The ultimate is molecular nanotechnology, which is about putting molecular building blocks in precise places. The implications of nanotechnology are interdisciplinary and wide-ranging across many industrial fields. Much of the potential is still far into the future, but we have to just remember that all living matter is organized on a nanoscale to realize that it is not an impossible dream.

The authors have given us an excellent insight into this emerging technology.

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1. SPECIAL ARTICLE

NANOTECHNOLOGY

Professor P. McKeown

1. Nanotechnology - what is it?

1.1 Introduction and definitions

Currently there is no single, internationally accepted, definition of nanotechnology. The journal *Nanotechnology* [1] which addresses itself exclusively to science and engineering at the nano scale, defines nanotechnology as "all those technologies and enabling technologies associated with either the top-down approach to fabricating miniature elements by sculpturing the desired structure from a microscopic piece of material as well as the bottom-up approach of building the desired structure molecule by molecule or atom by atom".

In Europe and the USA, the term 'nanotechnology' is frequently used to generally describe the science of atomic scale phenomena. However, the term 'nanoscience' is better for work such as this which is aimed at gaining a basic understanding of atomic scale phenomena and processing. The manufacturing engineering community in the field of nanotechnology simply describes it as "design and manufacture of artefacts in the range of 100 nanometres (nm) to 0.1 nm", or adopts Taniguchi's [2] description, 'integrated systems of materials processing, dimensional measurement and positional control technologies which achieve nanometre accuracies, through sub-nanometre resolution, in-process feedback and feed forward control networks'.

Perhaps a more broadly accepted working definition is "the study, development and processing of materials, devices and systems in which structure on a dimension of less than 100 nm is essential to obtain the required functional performance". This is the definition recommended by the authors of this paper; it covers nano-fabrication processes, the design, behaviour and modelling of nano structures, methods of measurement and characterisation at the nanometre scale and in particular:

- nano machining and nano fabrication techniques
- electronic device fabrication technologies
- scanning probe microscopes
- many aspects of microsystems technologies (MST)
- nano structured materials
- self organising and self assembling molecular structures
- biological and biomedical systems.

It is therefore clear that nanotechnology brings together engineering, physics, chemistry and biology. It includes materials processing through removal, accretion, surface transformation, joining and assembly right down to identification, manipulation and assembly of individual molecules. Many very high resolution techniques such as X-ray, electron beam and scanning probe microscopy etc. have been developed for the research of physical phenomena of matter at the sub nanometre and atomic scale. Through these physical techniques, analysis and even manipulation of structures at this scale have become possible. At the same time, whereas chemists have been traditionally involved in research and synthesis of small molecules, they now take a greater interest in building larger and more complex molecules. This has overlapped increasingly with research in biotechnology and thus biomedical engineering, leading overall to an increasingly interdisciplinary nature of research on the nanometre scale. It is primarily in this area that the term nanoscience is now frequently used to refer to current work where understanding molecular and atomic scale phenomena and establishing the science base at the nano scale is the current stage of achievement and where nanotechnology processes and products are the ultimate aim. Nanoscience can thus be envisaged as nanotechnology in its infancy; where working products are still far off. Examples include: supra molecular chemistry, the chemistry of nanostructured materials and mesoscopic physics e.g. where devices such as quantum wells (or dots) in which a single electron can be trapped and monitored might eventually lead to very fast, low energy quantum-computing.

1.2 What is a nanometre?

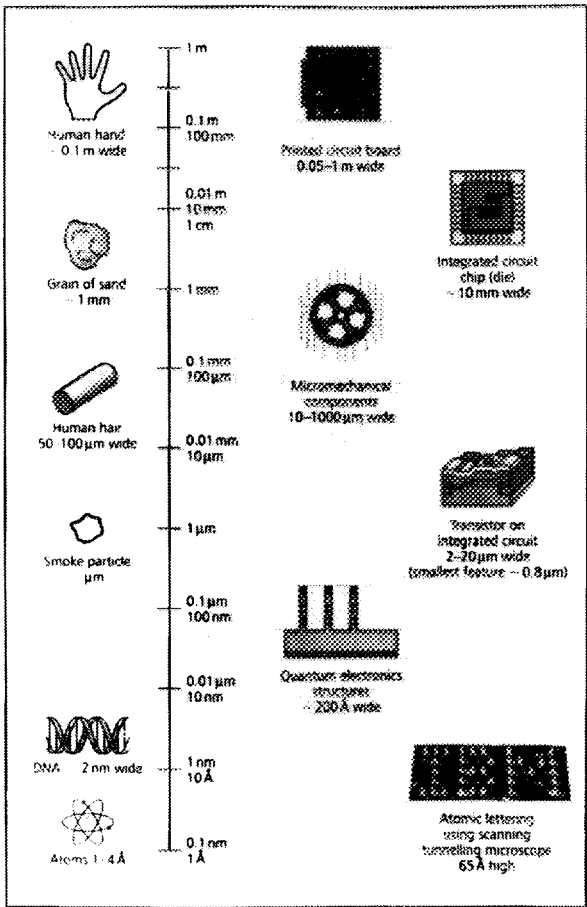
Nanotechnology, nanoscience and nanometrology all need the capability to measure and control motion to better than one nanometre in terms of displacement, size and profile. 'Nano' derives from the Greek *nanos*, a dwarf; i.e. very small.

A nanometre (1 nm) is 10^{-9} metres (10^{-9} m) - one billionth of a metre, approximately 80,000 times less than the diameter of an average human hair and 10 times the diameter of a hydrogen atom. Fig 1 shows some size scales from human to atomic. The metric dimensions relevant to high precision engineering, micro engineering and nanotechnology are:

| | | | |
|----------------------------------|----|--------------|------------------------------|
| 1 millimetre (mm) | .. | 10^{-3} m | |
| 1 micrometre (μm) | .. | 10^{-6} m | .. sometimes termed 'micron' |
| 1 nanometre (nm) | .. | 10^{-9} m | |
| 1 Angström Unit (\AA) | .. | 10^{-10} m | |
| 1 picometre (pm) | .. | 10^{-12} m | |

- Note:
- a normal human eye at closest focus can just resolve 20 μm
 - visible light has a wave length between 0.4 μm (blue) and 0.65 μm (red)
 - the atomic lattice spacing in silicon is 0.54 nm
 - displacement measurement approaching picometre (0.001 nm) resolution can be achieved by X-ray interferometry and scanning tunnelling microscopy.

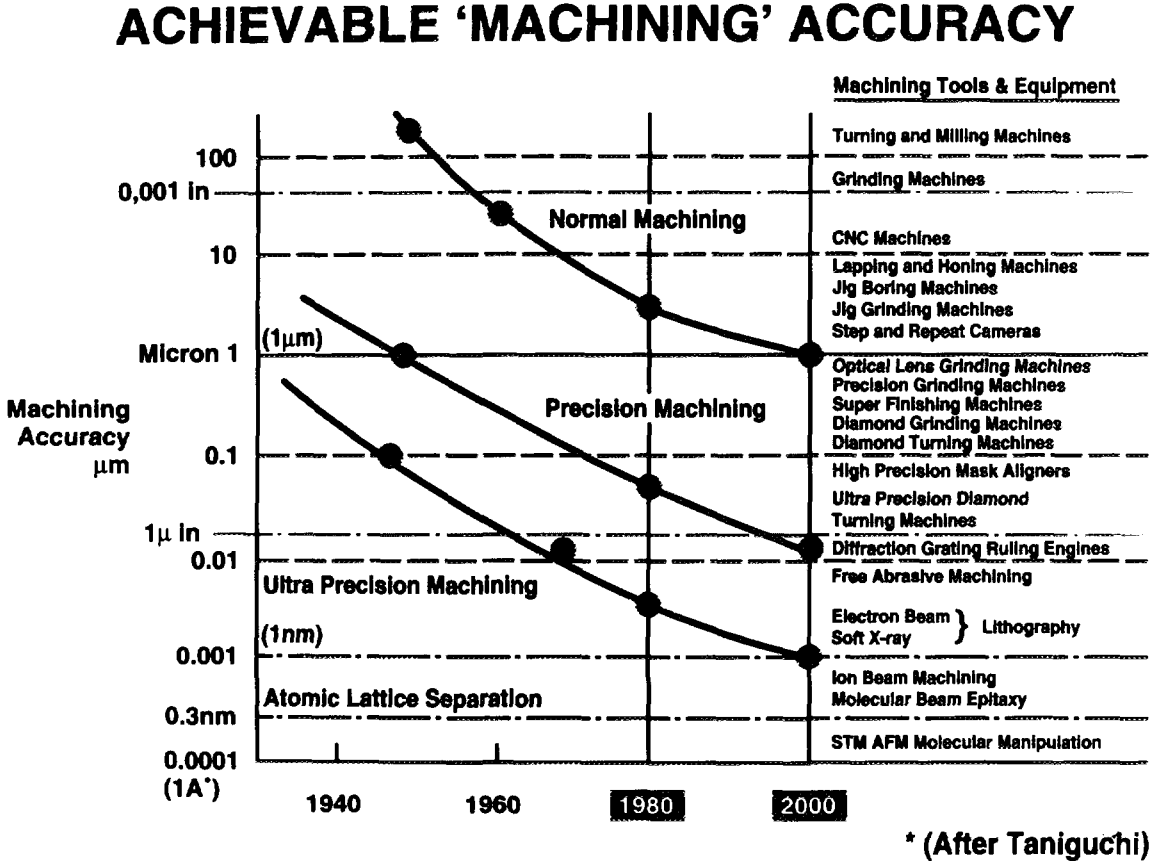
Figure 1
Some size scales from human to atomic
(Reproduced with kind permission of 'Chemistry in Britain'
and Dr. Peter Day)



1.3 The nanotechnology processes

Materials processing with nanometric resolution and control is applied to the measurement, manufacture and control of large, or 'macro' components such as optical or X-ray telescope mirrors, as well as in the fabrication of 'micro' features on very small artefacts such as integrated circuits. The inter relationships of nanotechnology and microsystems are addressed in Section 9.2; we recommend that micro and nano technologies are regarded as a continuum; they are highly interdependent.

Figure 2
The development of achievable 'machining' accuracy over the last sixty years



The ultra precision/nanotechnology processes, many of which are described in detail in this survey include:

- Single point diamond and CBN cutting (Fig. 3 shows a 1nm thick chip of copper made by diamond cutting);
- (Multi-point) fixed abrasive processes, e.g. diamond and CBN grinding, honing, belt polishing, including "ductile mode" micro-crack-free grinding of glasses and ceramics and other brittle materials (Fig. 4 shows the principle of ductile-mode cutting of materials);
- Free abrasive (erosion) processes, e.g. lapping, polishing, float polishing (mechano-chemical, chemo-mechanical processes);
- Chemical (corrosion) processes, e.g. etch-machining (perhaps as part of photo or electro-lithography);
- Biological processes, e.g. chemolithotrophic bacteria etch-processing of inorganic materials;

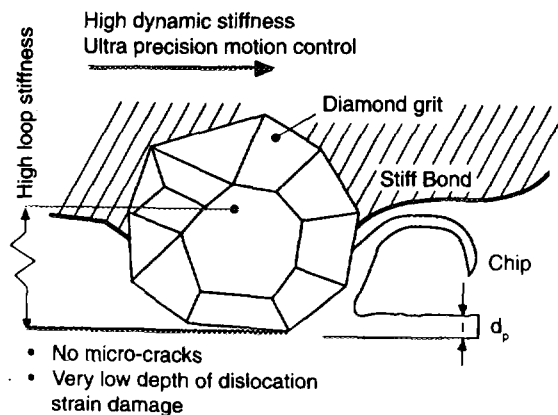
Figure 3

Scanning electric micrograph of 'chip' of electroplated copper cut by a sharp diamond on an ultra precision machine tool. The underformed chip thickness is about 1 nm. The resulting surface smoothness is about 1 nm R_a - a super smooth, very high reflectivity surface is produced directly on aluminium, copper, electroless nickel etc. without the need for subsequent polishing (Courtesy of Professor J. Ikawa, Osaka University)



Figure 4

The mechanism of 'ductile' or 'shear mode' CNC grinding of brittle materials; depth of cut d_p must be less than the critical transition depth at which brittle fracture occurs. This demands high precision, high stiffness machine tools and achieves very high surface smoothness with virtually no sub-surface microcracks



- Energy beam processes (removal, accretion and surface transformation processes), including:
 - photon^x (laser) beam: micro-cutting, drilling, transformation-hardening
 - electron beam^x: lithography, welding, micro and nano-drilling, (EBM) (Fig. 5 shows the principle behind electron beam machining while Fig. 6 shows text written with 4nm diameter e-beam cut holes)

Figure 5
The principle of electron beam machining (EDM); high energy electrons are focused electromagnetically onto the target in a vacuum; nanometre patterning accuracy can be achieved

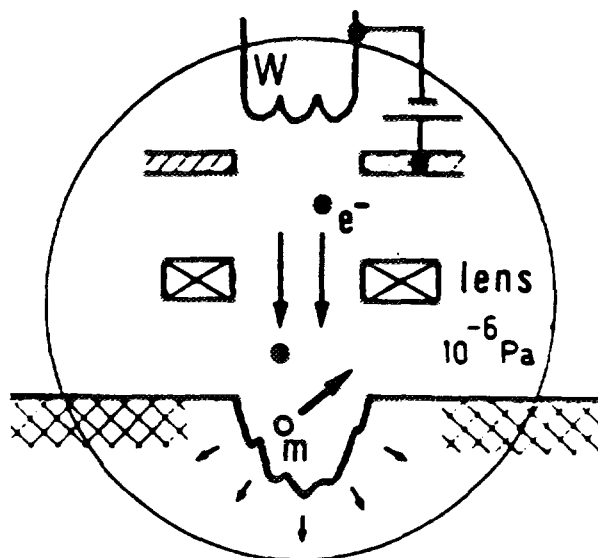
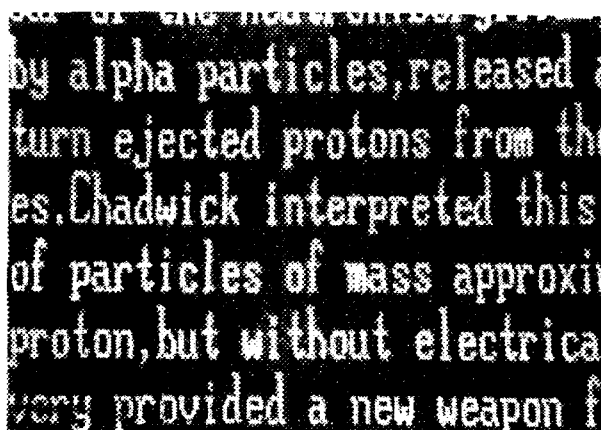


Figure 6
An example of high speed raster scan EDM: 'writing the Encyclopaedia Britannica on the head of a pin'. The line width of each letter is two electron beam drilled holes; each hole is 4 nm diameter; the substrate is silicon fluoride. (Courtesy of Professor C. Humphreys, Cambridge University)



- electro-discharge^x (current) micro-machining (EDM)
- electrochemical (current) machining (ECM)
- LIGA deep etching using X-rays or excimer laser beams for lithography; then electroplating and moulding of micro components

- inert ion beam^x machining (erosion) (focused (FIBM) and broad beam with mask) (Fig. 7 shows the principle behind ion beam machining while Fig. 8 shows the use of a gallium ion beam in repairing a Si chip, in comparison with laser cutting.)
- reactive ion beam etching (RIE)

Figure 7
The principles of ion beam machining; electrically accelerated inorganic ions such as argon are projected at the target surface in a high vacuum, providing 'atomic-bit' machining capability

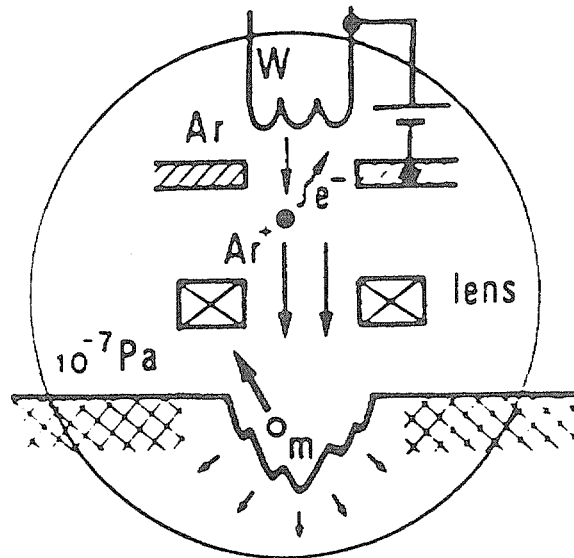
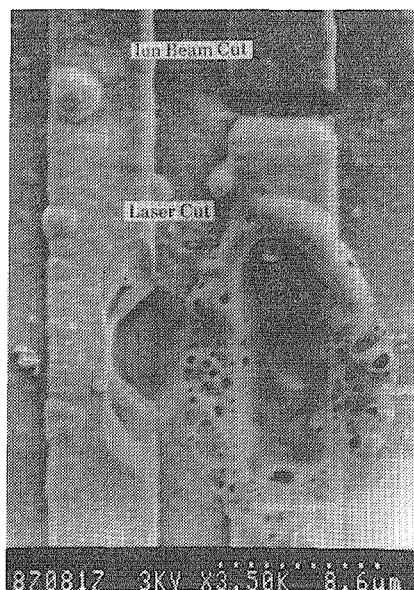


Figure 8
Relative control accuracy in repairing an LSI chip line of 4 μm width by focused ion beam (FIB) machining and a low power micro-focused laser. Today's excimer lasers can achieve finer focus and control



- thin film techniques (Langmuir Bodgett) (LBTF)
- molecular beam epitaxy (accretion) (MBE)
- manufacture of nano phase powders and nanocomposites, liposome based structures etc.

- scanning probe microscopy (STM, AFM) for process engineering, enabling molecular manipulation, assembly and modification (Fig. 9 shows the principle of operation of an STM, Fig. 10 shows an STM surface scan of a graphite crystal showing individual carbon atoms and Fig. 11 shows how individual atoms can be manipulated on a surface by using STM).
- x indicates the process is essentially electro thermal; for "atomic-bit" processing, high density energy in the range of 10^4 to 10^6 Joules/cm³ is necessary.

Figure 9
Schematic of a scanning tunnelling microscope probe, showing interaction of the electron clouds of the nearest atoms of the probe and object. The probe is moved in the X, Y directions and servo controlled in the Z direction with picometre resolution - to produce a Y scan trace of the surface atoms (topography) of the object

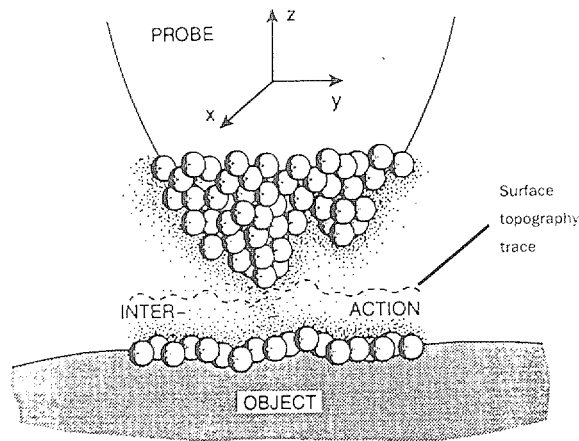


Figure 10
The screen image of an STM surface scan showing atoms of carbon; this whole image is approx. 1 nm x 1 nm.

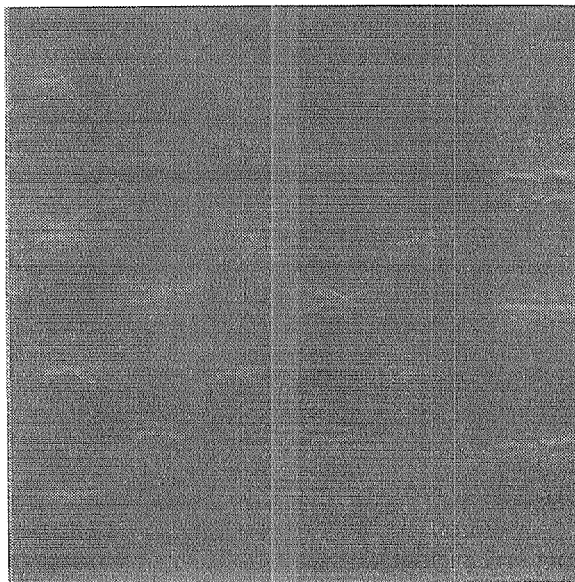
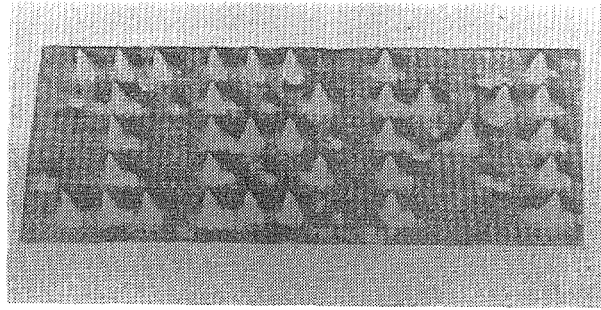


Figure 11
SEM image of 35 Xenon atoms on nickel substrate - IBM



1.4 The scope of this paper

The aim and scope of this paper is to explain what nanotechnology is, briefly trace its origins and recent historical development and then to describe the current state-of-the-art, in both the production of engineered artefacts and microbiological/biomedical fields. Ultra precision macro and micro machines are essential for success in nanotechnology for macro and micro components etc. Thus the paper also addresses the principles of design and control for ultra precision machines and instruments and gives some examples.

2. Nanotechnology - its origins and historical background

2.1 Precision Engineering

Manufacturing with higher precision is a development which has been gathering momentum over the last 200 years and accelerating over the last 25 years in terms of research, development, and application to product innovation. It has been driven by demands for much higher performance of products, higher reliability, longer life and miniaturisation. This development is widely known as precision engineering and today is generally understood as manufacturing to tolerances smaller than one part in 10^4 or perhaps 1 part in 10^5 .

The historical roots of precision engineering could be said to be horology, the development of chronometers and watches and, of course optics, e.g. the manufacture of mirrors and lenses for telescopes and microscopes. Major contributions were made to the development of high precision machine tools and instruments in the late 1800s and early 1900s by ruling engines for the manufacture of scales, reticules and spectrographic diffraction gratings. Today, ultra precision machine tools under computer control using single point or multi point diamond grinding wheels can position the tool relative to the workpiece to a resolution and positioning accuracy in the order of 1 nm. However, it will be noted from Fig. 2 that achievable "machining" accuracy includes the use of not only cutting tools and abrasive techniques but also energy beam processes such as ion beam and electron beam machining, plus scanning probe systems for surface measurement and molecular manipulation (pick-and-place).

So ultra precision manufacture has progressed through micrometre (μm) accuracy capability to enter the nanometre scale (nano scale) regime. However, this 'top-down' development has been joined by the prospects of 'bottom-up' nanotechnology such as the possibility of mobilising nano scale molecular structures to act as machines to guide and activate the synthesis of larger molecules, i.e. "molecular nanotechnology" with its promise of "building with atoms".

2.2 Taniguchi

The term nanotechnology was first introduced by Professor Norio Taniguchi formerly of Tokyo Science University, in 1974 at the International Conference on Production Engineering in Tokyo, sponsored by the International Institution for Production Engineering Research (CIRP) and the Japan Society for Precision Engineering (JSPE) [3]. Taniguchi used the word to describe ultra fine machining - the processing of a material to nano scale precision - work that he started in 1940 by studying the mechanisms of machining of hard and brittle materials such as quartz crystals, silicon and alumina ceramics, primarily by ultra-sonic machining. Subsequently he was in the forefront of research in using energy beam processes (EBM, FIB, RIE etc.) for fabrication of such materials to nanometric accuracies.

2.3 Feynmann

Although Taniguchi was the first to coin the term nanotechnology in 1974, it can be argued that the concept of nanotechnology was first enunciated by the American physicist Dr Richard Feynman (who became a Nobel Laureate in 1965) in his visionary lecture given to the annual meeting of the American Physical Society at the

California Institute of Technology, Pasadena, California in December 1959. He entitled his talk "There's plenty of room at the bottom", [4]. At the outset he asked, "Why cannot we write the entire 24 volumes of the Encyclopaedia Britannica on the head of pin?". He pointed out that if you magnify the head of the pin by 25 thousand times, the area would then be equal to the area of all the pages of the Encyclopaedia Britannica, and went on to point out that all that was necessary was to reduce the size of all the writing in the encyclopaedia by 25 thousand times. He argued that the scanning electron microscope could be improved in resolution and stability to be able to "see" atoms and went on to predict the ability to arrange atoms the way we want them, within the bounds of chemical stability, to build tiny structures leading to molecular or atomic synthesis of materials. On all counts his predictions have been remarkably accurate, (see Fig. 6 for an example of electron beam writing). He did not use the term nanotechnology as such but accurately described its potential for extreme miniaturisation and the self organising and self assembly of molecules. It is on this concept of "building with molecules" that the work of another American, K.E. Drexler has been developed.

2.4 Drexler

K. Eric Drexler was a student at MIT. He has become well known for "molecular nanotechnology", the concept of an all embracing manufacturing technology based on bottom-up molecular manufacture, popularised in his book "Engines of Creation" [5] first published in 1986. He postulated the possibility of mobilising nano scale molecular structures to act in a machine-like manner to guide and activate the synthesis of larger molecules. His ideas include billions of robotic type machines called "assemblers" which form the basis of a molecular manufacturing technology capable of building anything atom by atom and molecule by molecule.

Drexler's ideas gained greater publicity with the first designed protein produced at Du Pont in 1987 and then in 1990 the highly publicised manipulation of 35 xenon atoms by Eigler and Schweizer (Fig. 11), at IBM Research Division, Almaden, California using scanning tunnelling microscopy. The major research objectives of Drexler and his colleagues and followers in molecular manufacturing are based on designing a feasible assembler, modelling it computationally, and then building it. In 1995, Krummenacker and Lewis [6] implied that it may take 20 years of rigorous large scale research before such an advanced manufacturing technique using highly reactive molecules could become a reality. In the meantime many scientists and engineers remain somewhat sceptical about Drexler's ideas for molecular manufacture and referring to Drexler as a "futurolgist", and warn of the dangers in fuelling unrealistic expectations. However, the Foresight conferences on nanotechnology sponsored by Drexler and his colleagues have continued to grow in size and influence within the United States. Perhaps the best definition of Drexler's molecular nanotechnology is "the projected ability to use potential control of chemical reactions to building complex materials and devices (including molecular machinery) resulting in precise control of the structure of matter at the molecular level". The positive aspects of Drexler's publicity for nanotechnology include the encouragement of highly interdisciplinary research in the field and a much wider public awareness.

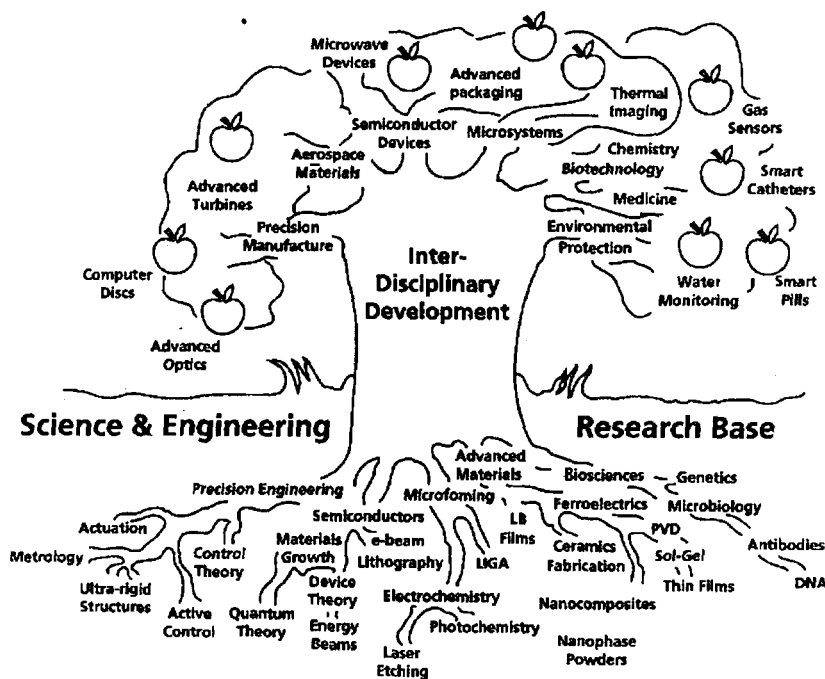
3. Nanotechnology - why is it important?

3.1 The opportunities of the "Nanometre Age"

Nanotechnology is a group of generic technologies that are becoming crucially important to many industrial fields and offering great promise of massive improvements to standards of living throughout the world. It is also a new way of thinking about possible solutions to problems currently obstructing new developments that can enhance the welfare of mankind. The Nanotechnology Tree (Fig. 12) shows in very general terms the wealth creating opportunities for the international business community which are arising from the science and engineering research base in micro systems technologies, nano science and nanotechnology. The main driving forces in this broad field from micro to nano systems are:

- new products that can work only on a very small scale or by virtue of ultra precision tolerances
- higher systems performance
- miniaturisation, motivated by "smaller, faster, cheaper"
- higher reliability, and
- lower cost.

Figure 12
The Nanotechnology Tree of Commercial Opportunity



3.2 Nanostructured materials

Nanotechnology is already giving rise to a wide range of new and greatly improved materials for engineering, electronic, opto electronic, chemical, microbiological and biomedical applications. The term nano-structured materials usually refers to solids most of those in common use today being microstructured. Materials often behave very differently when nano-structured. The much finer grain size can produce denser materials with greatly improved mechanical properties perhaps three times better than the microstructured version. Thus aerospace and defence will benefit from much higher performance, e.g. light weight, high strength nano-composite materials and even ceramics. Stronger, improved life hip prostheses is just one of many benefits offered in the biomedical field.

Crystalline catalysts can exhibit better performance when in nano-structured form which also can improve conductivity useful for a variety of sensors/microsensors and energy conversion and storage applications. Nanoparticles and nanopowders which can be produced in suspensions (colloids), sol-gel and aerosols etc. offer a wide range of new and improved products. Smaller particles have larger active surfaces per unit of mass; this improves their chemical activity such as greater solubility in water. Stronger ceramics, more uniform and durable surfaces on porcelain and better inks for inkjet printing, are some benefits that derive from nanoparticle technologies. Buckminsterfullerene in the form of carbon C_{60} "buckyballs" may prove to be a very effective nanoparticle dry lubricant in engineering applications. The "nanotube" version can perform as a mould for making nano-wires in suitable metals such as gold for electronic connectors and may be fabricated to form "molecular sieves" for faster and more selective filtration.

3.3 Thin films

Thin films or mono layers usually from 1 nm to 5 nm in thickness can be only one molecule or even one atom thick in some cases. They can be organic or inorganic and provide a wide range of excellent properties such as being chemically active in a useful way and being dense and hard for wear resistance. Monolayers, deposited on semi conducting substrates that emit electrons when sunlight falls on them are the basis of solar energy cells that can be expected to exhibit improved efficiency in the future. Thin layers can be deposited successively in different materials forming multi layers having for example, specific magnetic properties useful in magnetic recording with high packing densities; very many other useful properties are offered from high erosion resistance in hostile environments to the focusing of X-rays.

3.4 Nanofabrication

Nanofabrication covers a range of manufacturing processes that produce patterns and/or layers of material to form micro or nano-structures. The semiconductor/microelectronics industry has led the development and application of the photo- and electron beam lithography techniques which are the main basis for continuing miniaturisation in

large scale production in the future. Ultra large scale integration (ULSI) chips that are smaller, faster, cheaper and with more memory will bring further massive improvements to the performance of micro processors, computers etc. and onwards to telecommunications, domestic, automotive and medical products and services. (Section 8 below, amplifies this topic.) However, microsystems technology (MST) products with micro mechanical features such as sensors, or arrays of sensors and actuators fully integrated into the same (silicon) chip, are already burgeoning; applications will expand in navigational, automotive, biomedical and pharmaceutical industries, e.g. the concept of the "laboratory on a chip" that may lead to entirely new ways to design and build pharmaceutical and industrial chemical plants with greatly improved quality control and efficiency (see sections 9 and 11 below).

3.5 Ultra precision machining

Ultra precision engineering and machining such as cutting, grinding and super finishing have been developed over the last thirty years to provide nano-precision surfaces on macro-components such as:

- ultra precision spindles - rolling element and hydro-dynamic gas or liquid bearings for next generation, higher performance magnetic memory disk file systems, high definition large scale projection television and video cassette recorders
- mirrors and lenses for optical systems operating primarily in the visible, ultra violet and X-ray wavelengths; further advances in space communications, optical and X-ray astronomy, biomedical engineering etc. will be boosted by increasing accuracy and reducing the cost of the specialised machines and processes some examples of which are described in section 6.3 below.

3.6 Bio-medical applications of nanotechnology

This is a field that will, increasingly, be a major beneficiary of MST and nanotechnology developments; examples include:

- minimally invasive surgery, which is starting to be helped by remotely operated surgical instruments and diagnostic tools, e.g. micro-catheters down to 100 μm diameter incorporating optical fibres for delivery and retrieval of light images for high resolution cameras; nano-scale sensors for measuring blood chemistry. Incorporation of tip-mounted micro-turbine rotary cutters for arterial plaque removal is entirely feasible: cutters of this general type will also be useful for ophthalmic surgery.
- accurate and efficient drug targeting and delivery is made possible by nano-particle technology. Particles with specific surface topographies and selective reactive molecular coatings that already demonstrate a remarkable ability to arrive at targeted sites within mammalian bodies in enormously larger concentrations than normal, are being designed and tested. In effect, these medicinal 'bullets' coated with antibodies resist attack by the body's own defence cells and yet can lock on to the target cell such as a tumour, thereby avoiding the release of toxic drugs on to healthy tissue. Commercial availability of a range of such nano-particle drug delivery systems may emerge in the next 5 years (see section 11.4 below).
- many other biomedical applications currently the subject of R & D in Europe, Japan and the USA include:
 - replacement of damaged nerves by artificial equivalents
 - restoration of hearing or sight in some cases of damage or disease
 - improved adhesion growth of living tissue cells on to prosthetic implants by micro and nano surface patterning of implant materials, i.e. making more bio-compatible materials and surfaces.

3.7 Molecular nanotechnology

Molecular nanotechnology with the prospect of 'self assembly' in which atoms, molecules, molecular aggregates organise and arrange themselves into ordered functioning entities without human intervention, such as organic monolayers, presents fascinating challenges and opportunities.

Steps have already been taken towards the "interfacing of molecules" by chemical activation of the probe and substrate that is to be built up or modified. Eventually and well into the future, self organising and self-assembly molecular systems that mimic the self assembly of molecules in biology, could well become a reality. Thus in what Rohrer refers to as the forthcoming 'Nanometre Age' [7] the massive, parallel operation of "bottom-up assembly" and self organisation of molecules will to some extent replace the "top down" miniaturisation philosophy of today's MST/MEMS and engineering nanotechnology. This chemistry/biochemical approach to nanotechnology is being led by Drexler and followers (see section 2.4 above; and section 11 below).

He wrote: "Assemblers will be able to make virtually anything from common materials, without labour, replacing smoking factories with systems as clean as forests. They will transform technology and the economy at their roots, opening up a new world of possibilities" [5]. Many regard this as highly futuristic and bordering on science fiction.

However before this, we can expect to see the emergence of experimental and then fully functional "micro-factories", i.e. very small machines grouped within table-top dimensions for the manufacture of very small industrial products, such as micro-valves, pumps, electro magnetic/electro static motors and optical devices. Substantial space and energy savings together with reduced environmental pollution are the objectives of the Japanese NEDO, Micromachine Technology R & D Project for which an investment of US\$ 200m is being made in the 10 year programme launched in 1991.

3.8 The global market for nanotechnology - brief summary

There can be no doubt that many new products will arise from today's nanoscience and nanotechnology R&D work in academia, research institutes and industry. Many existing macro products will be replaced by MST and nanotechnology products, produced by new nanotechnology based manufacturing facilities. Many estimates have been made for the size of the global market for "nanotechnology" products but it is impossible to reconcile and aggregate them accurately. Predictions include:

- a 1995 VDI report for the German Government estimates that by the year 2000 it will exceed US\$ 250 bn [8].
- the market for MST/MEMS alone will exceed US\$ 14 bn by 2000
- the market for 0.1 μm (100 nm) DRAMS will exceed US\$100 bn also by 2000.

The following sections go into greater detail on the technologies, applications and prospective markets.

Nanotechnology is a major new technological force that will have substantial socio-economic effects throughout the world. Many benefits in standards of living and quality of life can be confidently expected.

4. Ultra-precision machines and instruments

4.1 Nano-precision processing machines

In order to use most of the nanotechnology manufacturing processes listed in section 1.3 above, ultra-precision machines and instruments are needed to control the three dimensional (3D) spatial relationship of the "tool" to the workpiece to accuracies in the order of 1 nm, 0.1 nm or even less. The tools can be:

- solid tools for cutting, abrasive or chemico-mechanical action
- energy beam tools
- scanning probe tools such as STM, AFM, magnetic, thermal or chemical-reactive probes etc.

Ultra-precision machine systems fall into three main classifications:

- large, computer numerical control (CNC) macro-machines for measuring, shaping or forming conventional macro-sized component parts; today, this can mean working to nano tolerances on macro-components
- instruments for metrological applications to macro and micro-components
- very small 'Feynman' machines ranging in size from a few millimetres down to micrometre dimensions.

In each case, there are eleven main principles and techniques that must be used in design and build to achieve under computer control, a highly deterministic performance as a sound basis for nano-precision capability.

4.2 Error reduction - the machine designer's guide to high precision

The Eleven Principles and Techniques for the design and manufacture of high precision machines and their control systems are now set out and briefly addressed. The 'Designer's Guide to High Precision' [9] [10] are essential steps in the process of 'error reduction' of machines, and apply to conventional as well as ultra-precision machines:

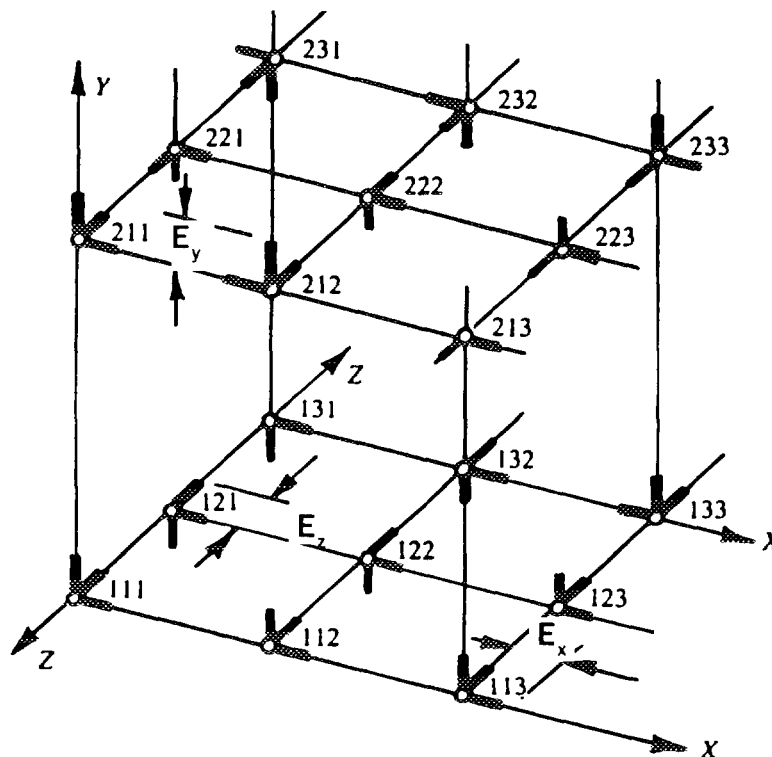
- (i) **MACHINE STRUCTURE** - the size of the loop between tool and workpiece should be as small as possible; this structural loop should be as resistant as possible to all disturbing agents; highly symmetrical structures can best achieve this; the structure should be made of materials that ensure long term secular stability, high static and dynamic stiffness (to resist internally and externally generated vibrational forces) and high damping (to rapidly attenuate any vibrational motions that do occur). Thus the structure must be as stiff or rigid as possible in its own right, mounted kinematically on three support 'feet' only and thus be geometrically unaffected by any distortional movements of the foundation on which it is mounted. It must exhibit the highest thermal stability i.e. be as geometrically insensitive as possible to internal or external temperature variations, for example, through the use of low expansion materials. Where temperature gradients exist, symmetry is very important. Seismic and acoustic isolation is essential for ultra precision machines. Other disturbing agents can include magnetic fields, humidity, atmospheric pressure and light e.g. radiation heating.
- (ii) **KINEMATIC and SEMI KINEMATIC DESIGN** (first comprehensively enunciated by James Clerk Maxwell circa 1876) - machine elements should be designed as 'rigid' bodies and connected to mating elements with the minimum number of constraints needed for fixed location (6 constraints) or the required degrees of freedom (5 constraints for a linear or rotary motion); kinematic design practice offers high precision at lowest cost.
- (iii) **ABBE alignment PRINCIPLE (or options)** - (enunciated by Ernst Abbe, Jena Germany circa 1890) - the line of action of the displacement measuring system must be co-linear with the displacement to be measured; the two options are that either the motion of an element such as a carriage must be free of all angular motion such as 'pitch' or 'yaw' or these angular motions must be measured in order to calculate and compensate for the consequences of the 'Abbe offset'.
- (iv) **"DIRECT" DISPLACEMENT TRANSDUCERS** - the use of displacement measuring systems that are totally independent of disturbing agents such as drive mechanisms, e.g. lead screws; examples are scales and laser interferometers.
- (v) **METROLOGY FRAMES** - a means of isolating the displacement measuring systems from machine structural distortion caused by motion of machine carriage elements, by mounting them on an independent, high stiffness framework geometrically related to the workpiece support datum.

- (vi) **GUIDEWAY and ROTARY BEARINGS** - low limiting-friction, high 'averaging effect' bearings to provide high accuracy motion with low thermal effects and high damping; fluid film bearings such as hydrostatic air, oil or water are frequently used for macro-motions. Flexures in the form of monolithic linear or rotary spring mechanisms can be optimal for micro-motions.
- (vii) **DRIVES** to carriages should be positioned to operate through 'axes-of-reaction' to ensure translation without rotational effects - or to achieve pure rotation with lowest radial, axial and 'tilt' error motions.
- (viii) **THERMAL STABILITY** - it is essential to eliminate or minimise thermal inputs, external or internal, thus minimising thermal distortions and 'thermal drift' of tool to the workpiece datum; it is achievable by use of low expansion materials, temperature controlled air or liquid passed over and/or through the machine or by servo error compensation, etc.
- (ix) **SERVO DRIVES and CONTROL**
 - servo drives - high dynamic stiffness, high bandwidth, fast response; high speed dynamic position loop compensation to achieve zero following errors i.e. between actual and commanded position
 - CNC - high speed simultaneous control of all axes; 2D/3D systematic error compensation, refractive index compensation for laser interferometers and thermal drift error compensation are all essential capabilities for ultra precision control systems.
- (x) **ERROR BUDGETTING** - essential at schematic design stage to ensure achievement of specified accuracy capability in an optimised manner:
 - Geometrical : angular, e.g. pitch, yaw (Abbe) and roll, straightness and orthogonality error motions
 - Thermal : loop expansions and distortions
- (xi) **ERROR COMPENSATION** - CNC software error compensation - of systematic, quasi-static 2D or 3D error map values established by calibration; dynamic error compensation by use of fast tool servo drives; thermal error compensation by servo adjustment etc.

4.3 Error compensation through the control system

The eleven principles and techniques, are essential steps in error reduction of machines. However, powerful CNC systems incorporating the latest VLSI processors offer software error compensation capabilities (xi above) for high precision machines. A few, world-leading manufacturers now use the technique of software error compensation whereby the systematic errors in X, Y and Z at perhaps 64000 points in the 3D workzone of the machine can be greatly reduced and brought close to zero. Fig. 13 shows a 3D error map for a multi axis machine. The X, Y, Z systematic errors result from the inevitable geometric and deformation errors of machine structures and from guideways and displacement measuring systems etc. The intrinsic errors of the machine can usually be reduced by at least a factor of 10 by software error compensation.

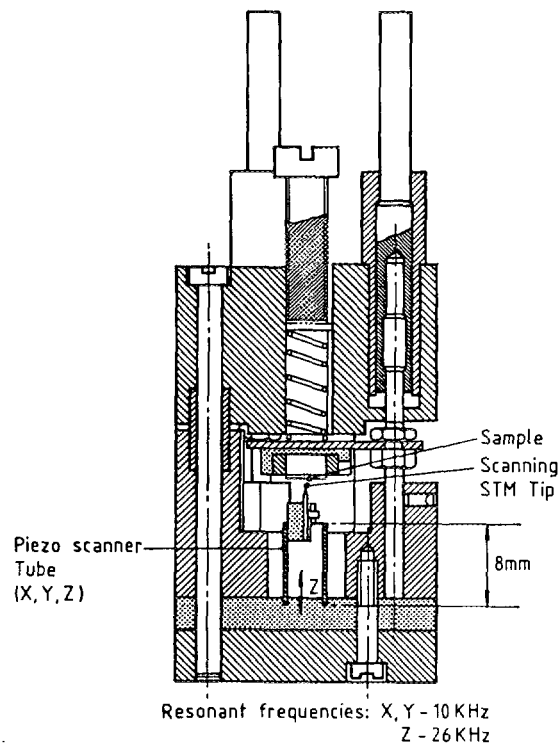
Figure 13
A 3D error map for a precision machine showing the uncorrected systematic errors in x, y and z directions that can be reduced by software error compensation



4.4 The importance of dynamic stiffness

The dynamic loop stiffness of the machine is of utmost importance in achieving high accuracy, whether the 'tool' is one that generates intermittent disturbing forces such as a grinding wheel or is a virtually 'non-contacting' measuring probe such as used in a scanning tunnelling microscope (STM). As size reduces so the resonant frequency of the structure increases; its dynamic stiffness goes up and it can resist dynamic disturbing forces such as seismically and acoustically transmitted vibrations. Thermal expansion and thus thermally induced distortions of a structure also scale directly with size. So in these terms, the accuracy capability of a machine or instrument improves as it is made smaller. Thus micro-miniature machines typically of millimetre or even micrometre dimensions have excellent ultra precision potential. The first STM instruments could not achieve atomic resolution primarily because they were built essentially as macro-structures i.e. with low loop-stiffness between probe and the workpiece, so that seismic and acoustic vibrations caused them to oscillate with amplitudes much greater than atomic dimensions. Fig. 14 shows the small, compact but simple design of an early experimental STM that has very high resonant frequencies (26 KHz in the vertical Z direction). It can achieve atomic resolution with very little vibration isolation. Micro-miniature scanning probe devices can be fabricated onto silicon in a similar manner to VLSI circuits. These "nano-fingertips" can be used for micro or nano surface patterning either directly or by lithography (see section 7 below). In the future we can expect to see miniaturised nano-tools with thousands of tips which can recognise, extract and deposit molecules and atoms to build nano- devices such as sensors, actuators, complete nano-robots and other Feynman machines. Machines and instruments with nanometric resolution and accuracy are built today. Some examples on the macro scale are briefly described in sections 6.3 below.

Figure 14
STM Design for High Dynamic Stiffness



5. Nanometrology

For many products, the dimensional geometry of discrete components is crucial in determining quality of performance. Therefore, metrology, the science of measurement, the ability to measure with appropriate accuracy becomes important. Accuracy is defined, in the Oxford English Dictionary as "exact conformity with a standard or with truth". In metrology terms, accuracy is understood, according to the International Vocabulary of General Terms in Metrology, to be "the closeness of the agreement between the result of measurement and the (conventional) true value of the measurand" (measured quantity) [11]. In addition it is stated that the use of the term 'precision' for accuracy should be avoided; precision is better understood as repeatability.

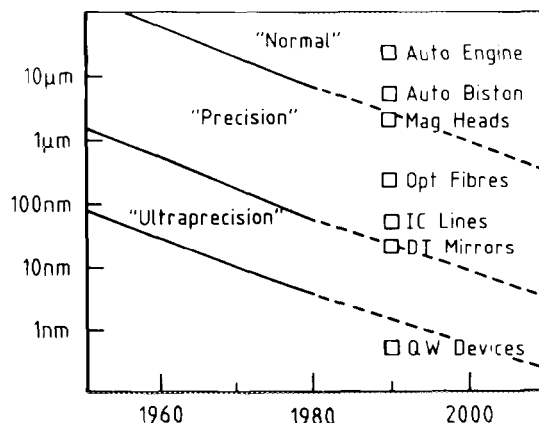
Nanometrology can be described as the science of measuring the dimensions of objects, or features on objects, to uncertainties in the order of 1 nm or better. The limit to accuracy is set by uncertainty of measurement. In practice it is the same thing. It is the size, shape and surface finish of parts, which ultimately determines functional performance, wear and fatigue life, ease of assembly etc.

Numerous measuring techniques are used to measure the dimensional characteristics of machined parts such as length, profile, eccentricity, surface roughness, etc.

5.1 Trends in metrological needs

The long term manufacturing trend for many strategic products, from automobiles to microchips, is for tolerances to be decreasing by up to a factor of three every ten years, as shown in Fig 15. According to Swyt [12] "between 1980 and 2000 state-of-the-art tolerances will decrease from $7.5\ \mu\text{m}$ to $1\ \mu\text{m}$ in the most demanding 'normal' machine tool based production, from $0.075\ \mu\text{m}$ to $0.01\ \mu\text{m}$ in the most demanding 'precision' production, e.g. diamond turning/grinding and from $0.005\ \mu\text{m}$ ($5\ \text{nm}$) to $<0.001\ \mu\text{m}$ ($<1\ \text{nm}$) in the most demanding atom-, electron-, or x-ray machining based production". An issue which has to be addressed concerning this trend is the requirement to measure more accurately, and to reference and thus trace, these measurements to international (ISO) standards. Production control measurements are required to have an accuracy between 4 times and 10 times better than specified component tolerances, and, it is commonly accepted that reference standards should be between 4 and 10 times more accurate than the production control tolerances. Thus reference standards should be 16 to 100 times better than component manufacturing tolerances. As component tolerances reduce to sub μm values, then national standards laboratories face increasing challenges to satisfy industrial metrology demands e.g. see section 8.6, where true nano metrology is required for next generation ULSI circuits.

Figure 15
Tolerance trends in manufacturing with product examples
(ref. D.A. Swyt - NIST, USA)

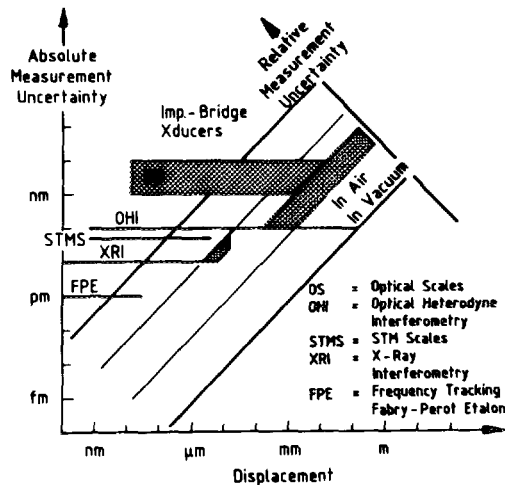


5.2 Length and displacement measurement

In most measuring instruments or machines, size/length is measured by displacement of an appropriate fiducial sensor over the component. In nanometrology, instruments and machines have to incorporate linear displacement transducers with resolutions and accuracy in the order of $1\ \text{nm}$.

Fig 16 indicates the measurement accuracy limits for different physical principles [11]; it plots absolute measuring uncertainty i.e. accuracy against length of displacement for perhaps the most important linear displacement transducers in the field of nanotechnology, namely laser interferometry in the forms of Optical Heterodyne Interferometry (OHI) and Frequency Tracking Fabry Perot Etalon (FPE) together with X-ray Interferometry (XRI) all of which are capable of sub-nanometre resolution and accuracy. However FPE and XRI are limited in range to a maximum of $400\ \mu\text{m}$ so far and are thus very restricted in nanometrology applications. However, it should be noted that the accuracy of all these systems is influenced and limited by other factors, such as linearity of output signals, robustness, stability, sensitivity against changes in working environment etc. All of these must be taken into account when specifying a measuring system for a measuring or production machine.

Figure 16
Nanometrology: limits for different measurement principles
(ref. C Teague - NIST, USA)



Temperature variations, in particular, can have a major negative effect on repeatability and measuring accuracy of metrology systems. Manufacturing engineers are often unaware of the magnitude of errors caused by changes in the thermal environment; it is imperative that they become familiar with ways to minimise and to compensate for the whole range of thermally induced errors [10].

Laser interferometers i.e. OHI and optical scales (OS) are widely used for linear displacement measurements in ultra precision machines and instruments.

Modern optical grating scale systems with a 1 nm resolution and a range of 100 mm are currently available; this range is expected to increase to at least 500 mm soon. Current, commercially available, laser interferometer systems can be obtained with a 0.6 nm resolution and a range of several metres.

Thus the current relative accuracy applications of optical scales and laser interferometers can be said to be: Vacuum laser interferometry (OHI) can give the highest resolution and accuracy for large displacement measurement e.g. 0.1 nm over 1 m (Fig. 16).

- For accuracy requirements between 10 nm and 100 nm there is little to choose between scales and interferometers assuming they are calibrated properly.

Scales are usually the preferred choice for accuracies between 0.1 μm and 10 μm . Their cost advantage usually makes them the appropriate choice for application to machine tools and coordinate measuring machines in the sub micrometre rather than the nanometric accuracy bandwidth.

For short ranges i.e. from a few micrometres to a millimetre or so, and if the range to resolution ratio is less than 2000 and the linearity is less than 0.1%, analogue transducers can be considered. These include linear variable displacement transformers (LVDT's), capacitance gauges, eddy current probes, etc., which are available with nanometric resolution. XRI and FPE techniques are expensive techniques to use in short range nanometrology applications but they can give near picometre resolution.

5.3 Surfaces - metrological characterisation

Knowledge of surface topography at the nanometre and increasingly at the atomic scale is important in understanding the functional performance of that surface. In addition, other surface properties such as chemical, magnetic, hardness, stress levels etc. are important for the same purpose. There are essentially two main methods of measuring surface topography i.e. surface form/profile, waviness and roughness; they are stylus techniques and optical interferometry.

In the conventional stylus method a diamond with a tip radius of typically 1 μm can achieve a profile height resolution of 1 nm and approaching 0.2 nm, based on a very high resolution LVDT. The scanning probe microscopes such as STM and AFM described in section 7 below, can achieve atomic resolution (Fig. 10).

The AFM is now being applied widely to the study of inorganic and organic surfaces e.g. the epitaxial growth of silicon layers for microelectronic applications and the study of biological molecules adsorbed on to surfaces.

Albert A. Michelson (1852-1931) developed the first optical interferometer system by exploiting a knowledge of optics, and how interference fringes are formed by light waves interfering with each other. He developed this concept of interference to measure displacement. Fig. 17 shows, schematically, the principal components of a modern optical interferometer system, namely an Optical Heterodyne Interferometer (OHI). Developments in modern electronics enable the use of optical interferometry for nano surface profile and roughness measurements. A commercially available system uses a piezoelectric transducer (PZT) to move a reference mirror, which results in

the generation of a number of fringe patterns each time a measurement is taken. The movement of the PZT shifts the fringe pattern which is recorded by a detector and transferred to the system's computer.

Figure 17
Principal optical components and operating principle of an optical heterodyne interferometer (ref. A. H. Slocum)

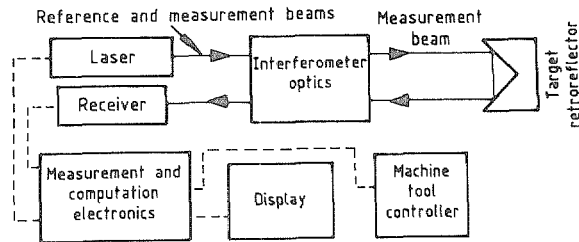
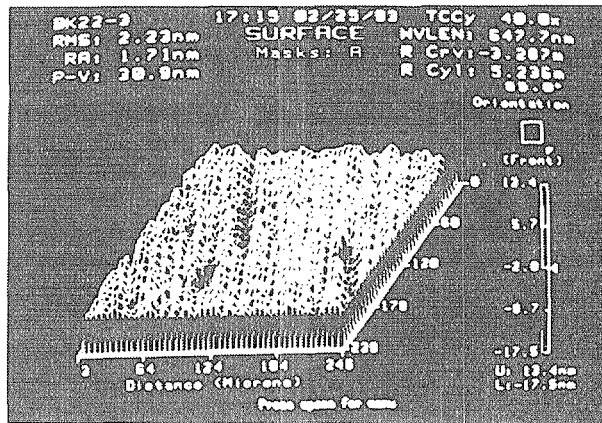


Fig. 18 shows an interferometric plot of a sub nanometre surface finish, measured with a commercially available system, namely 0.8 nm Ra obtained by ultra precision single point diamond turning of high purity optical germanium used for lenses in infra red night vision systems.

Optical techniques are limited by the amount of light which is reflected, or scattered off the test surface and returned through the microscope objective. However, their performance can be improved by the use of multiple-wavelength techniques, which enable the measurement of steep slopes, or steps up to a height of 15 µm in height.

Figure 18
Interferometric plot of a sub-nanometre surface finish produced on optical germanium

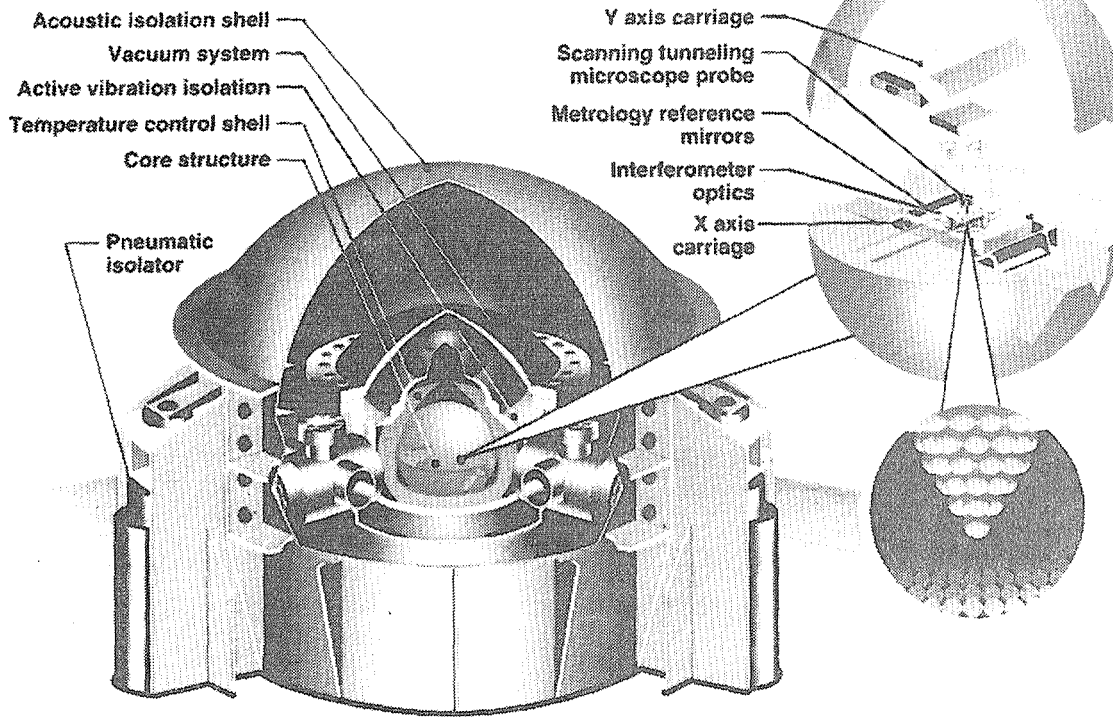


5.4 The Molecular Measuring Machine (M3)

Undoubtedly the most accurate 3D macro machine in the world at the time of writing is the Molecular Measuring Machine dubbed "M3" which has been developed at the USA's National Institute of Standards and Technology (NIST), [13]. This pioneering, world-leading project is aimed at imaging and measuring to nanometre accuracy, the positions of nanometre-size features located anywhere within an area of 50 mm x 50 mm and up to 100 µm high. This measuring machine, Fig. 19, is designed to meet the needs of industry working on next generation nano devices, particularly nano-electronics, ULSI (Ultra Large Scale Integration) circuits. It will provide calibration for the metrological standards by which to control the production of ULSI circuits with 100 nm line widths envisaged for widespread use within the next 20 years, e.g. line width standards and artefact standards for calibration of e-beam and x-ray production equipment etc.

Figure 19
Schematic of the NIST Molecular Measuring Machine
(Courtesy of National Institute of Standards and Technology, USA)

Molecular Measuring Machine



The maximum size of the specimen to be measured is 50 mm x 50 mm x 25 high; the vertical measuring range is 100 μm . It employs very high resolution scanning tunnelling and atomic force microscopes (STM and AFM). The target initial resolution of point-to-point measurement within the measuring volume was 0.1 nm (1 \AA). However, the X and Y interferometers that measure displacement with respect to the X and Y ultra precision metrology reference mirrors (a form of metrology frame, see section 4.2 above) have been improved to operate at a resolution of 0.05 nm i.e. 0.5 \AA or about one-fifth the distance of typical inter-atomic spacings.

The target of 0.1 nm accuracy over 50 mm explains the extraordinary care that has had to be taken in the design to isolate it from environmental effects such as seismic vibration (the structure has a first resonant frequency of 2 KHz) and active vibration isolation; acoustic vibration (acoustic isolation shell); temperature changes (temperature control shell); humidity, CO₂ content and temperature (vacuum system, 10⁻⁷ Pa). Control of temperature is maintained to better than 0.0005°C (0.5 mK).

The machine can be calibrated against an atomic length standard e.g. single crystal surfaces with known atomic order and spacing (0.54 nm in silicon). In designing this remarkable machine all of the Eleven Principles and Techniques listed in section 4.2 above were observed and employed.

6. Ultra-precision machining

6.1 Energy beam processes

As briefly indicated in section 1.3, 'atomic bit processing' for both material removal and accretion can be achieved by fine focusing beams of very high energy particles on to the workpiece (Figs. 5, 7).

The industrial applications of energy beam processing have rapidly increased over the last twenty years, driven in particular by the electronics and computer industries. Electron beam (EBM), focused ion beam (FIB) and reactive ion etching (RIE) are effectively swarf-free, virtually zero force processes. They can be used for material removal, accretion and surface transformation, if necessary on a molecular scale. The most widely used energy beam processes are listed in section 1.3.

Electron beam and ion beam techniques are essentially electro-thermal processes which can deliver high density energy to the workpiece in the order of 10^6 Joules/cm³ needed to remove material in the so called 'atomic-bit' processing mode. Holes of 4 nm have been produced at Cambridge University, where it has been demonstrated that with high speed raster scan electron beam drilling on a very thin substrate of silicon fluoride, it would be possible to write the complete 29 volumes of the Encyclopaedia Britannica onto the head of a pin (Fig. 6). A more typical application of these techniques is the use of focused ion beam machining, (FIB) for the cutting and repairing of individual tracks in integrated circuits (Fig. 8).

One of the brightest sources of finely focused high energy photons is the ultra violet light emitted by excimer lasers especially at the wavelengths of 193 nm to 353 nm. These intense 10 to 20 MW energy photons interact directly with the chemical bonds which hold most molecular based materials together causing rapid dissociation and material removal. They allow the cutting of delicate materials, almost without thermal damage. They can therefore be used by surgeons to correct short-sightedness by reshaping the surface of the cornea of the eye. Other applications include micromachining fibre optic clamps, microdrilling helical grooved holes in ink- drop forming nozzles for ink jet printers and minute holes in circuit boards that connect silicon chips in high speed computers. The delicacy of the process can be seen in (Fig. 20), which shows a human hair which has been micromachined, with no discernible thermal damage, by excimer laser mask projection.

Figure 20
Human hair micromachined, with no discernible damage, by excimer laser mask projection (Courtesy of Exitech Ltd., UK)



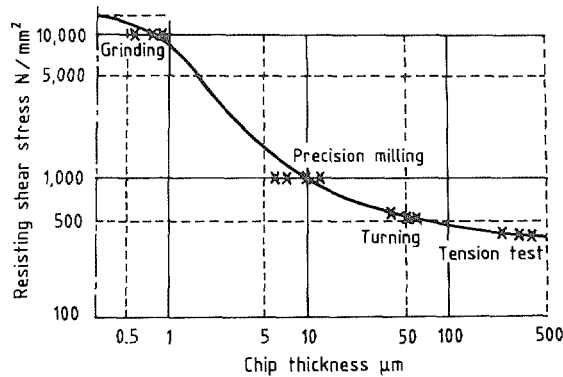
Other energy beam processes are listed in section 1.3 above; those of particular importance not described elsewhere in this paper are chemical vapour deposition (CVD, plasma and laser enhanced) and physical vapour deposition (PVD, sputtering and ion plating). CVD and PVD are both used for producing wear and corrosion resistant coatings. Other uses include the modification of component surfaces and the production of metallic and non-metallic (e.g. ceramics) powders, down to nanometre particles size. Virtually any metal, alloy or compound can be deposited and applications are found in the fields of electronics, semi-conductors, solar energy, optics and decorative coatings. These techniques, also known as vapour phase epitaxy, are used to produce semi-conductor super lattices. Thin film deposition systems with deposition rates controlled to better than 0.1 per cent are possible and layers can be deposited as small as one atom thick [2]. These thin film deposition techniques represent the ultimate in micro-structural control.

The energy beam processes are particularly appropriate for microengineering and nanotechnology applications. However the cutting and grinding processes, that have been greatly refined and developed over the last 20 years, will continue to have wide application in ultra precision machining.

6.2 Solid tools

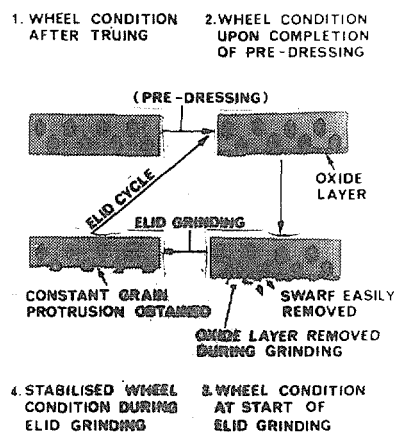
The last 30 years has seen a great development in advanced CNC machines for diamond turning, and the grinding of brittle materials e.g. glasses and ceramics with very low levels of sub surface damage. During this period the number of applications for single point diamond turning has increased significantly for components such as computer magnetic memory disc substrates, high performance aspherical mirrors and lenses, x-ray mirror substrates, etc. Solid tool machining of metals using cutting tools or abrasive grains, for chip thicknesses less than 1 μ m (Fig. 21) results in high cutting forces and temperature at the cutting edges [3].

Figure 21
Resulting shear stress V 's chip thickness, showing high stresses with fine machining. (Ref. N. Taniguchi)



In the cutting depth range of 1 nm to 100 nm or so, this is caused by the need to overcome atomic cluster bonding forces. In the range of 0.1 μm up to 10 μm the cutting forces reduce because the machining results in subgrain slipping failure of moveable dislocations for ductile materials, or by subgrain crack fracture of existing microcrack defects in brittle materials. For depths of cut in excess of 10 μm the defects are caused by the more conventional ductile failure in the multigrain range, or brittle fracture at the grain boundaries. It is clear therefore that solid tools are not ideal for atomic bit processing. Most of the newer high performance materials that have been developed over the last 25 years are essentially hard and brittle. However, a new fixed abrasive grinding process using very fine diamond (or CBN) grits has been developed called 'ductile regime' or 'shear mode' grinding. This is starting to replace conventional lapping and polishing to produce mirror surfaces on glasses particularly for higher cost, non-flat, non spherical optical components. However fixed abrasive grinding wheels with very small grits can be inefficient through 'loading' i.e. the grinding surface can become filled or 'clogged' with swarf or chips of the workpiece material. This would require frequent dressing of the wheel; a new abrasive surface would have to be produced by removing the clogged or loaded layer. A new 'Electrolytic In-Process Dressing' (ELID) technique [14] has been developed to overcome this loading problem, (Fig. 22); this can continuously electro-etch the metal bond of the grinding wheel in a highly controlled manner to unclog the wheel and enhance tool life. Ductile mode CNC grinding now appears to be a major contender to produce complex optics in minimum time and at low cost. With the use of advanced CNC machines both transmission and reflecting elements can be generated with very little (or even effectively zero) sub-surface damage or subsequent polishing. This is particularly advantageous for manufacturing complex optics, including off axis optics such as segments for very large telescope mirrors.

Figure 22
The 'Electrolytic In-Process Dressing' (ELID) technique, used for 'ductile mode' grinding



In order to achieve ductile mode 'damage free' machining, of brittle materials, extremely small chip thicknesses are necessary, typically in the order of 100 nm [15]. These fine chip thicknesses are necessary to keep the stress in the material small enough so the material yields in a 'ductile manner' rather than by brittle fracture. Fig. 4 illustrates the mechanism of ductile, or shear mode, grinding of a brittle material. This ability to machine brittle

materials in a ductile manner is also appropriate for machining complex macro-components in ceramics and bimetallic materials such as turbine blades, nozzle guide vanes, ink jet nozzles, etc. to high accuracy without inducing microcracking which would lower ultimate rupture strength and fatigue life. Clearly the type of machines capable of producing such components with the required mechanical properties, to accuracies in the sub-micron and approaching the nanometre regime, necessitate the development of appropriate ultra precision CNC machine tools. High loop stiffness between the tool and workpiece, for the close control of the tool-to-workpiece position under vibration inducing grinding is essential to achieve 'ductile mode' machining, (see section 4.4 above).

6.3 Ultra-precision machine tools

Examples of ultra-precision machine tools capable of nanometric tolerances on macro components which have been developed in the last 25 years include the Nanocentre (UK) (Fig. 23), the Large Optics Diamond Turning Machine (LODTM) (USA) shown in Fig. 24, and Disk and Wafer Edge Grinding Machines (UK) shown in Fig. 25. The Nanocentre, a 3-axis ultra precision CNC aspheric generator, is thought to be the world's most accurate machine tool of its type. It is designed for very high dynamic loop stiffness between tool and workpiece in both single point diamond cutting and 'ductile mode' grinding. This is achieved through an optimised machine configuration and the use of high stiffness servo drives and hydrostatic bearings throughout. Servo resolution of the X and Z linear axes is 1.24 nm; the rotary 'B' axis for "tool normal" single point turning has a resolution of 0.3 arc sec. An upgraded research machine has produced surface finishes superior to anything reported elsewhere in the single point diamond turning of optical germanium (0.8 nm Ra), Fig. 18 and in the ductile regime grinding of glasses and ceramics [16].

In order to maximise the geometrical accuracy of the Nanocentre, the research machine built under the UK's National Initiative on Nanotechnology (NION), incorporates a 'metrology frame' (Fig 26) to optimise the in-process measurement of the linear motions. This automatically compensates for Abbe offset errors for both pitch and yaw in the XZ plane at the height of the workspindle centre line. This system also measures straightness and orthogonality errors, which are also software compensated. The highly stiff metrology frame structure utilises two ultra precision Zerodur 'stick' mirrors for 3 laser interferometer air paths which are software compensated on line, for refractive index changes, by a differential refractometer. The use of these computer software error compensation techniques broadly described in section 4.3 above, improves the intrinsic horizontal straightness error motion of the XZ motions from 200 nanometres to 50 nanometres (Fig. 27).

The Large Optics Diamond Turning Machine (LODTM) was developed at the Lawrence Livermore National Laboratory, in the USA, to diamond machine large optical components with a surface finish of the order of 2 to 4 nm Ra. Temperature control in a specially environmentally controlled room includes a high velocity air shower controlled to 0.01°F. Refractive index errors, caused by environmental effects such as changes in temperature, pressure and humidity, are minimised by the use of evacuated steel bellows which enclose the interferometer beam paths.

The edge grinding machines, utilise the 'ductile' grinding technique for the finish machining of ceramic and glass disc substrates for the computer magnetic memory disk drive industry; more recently they have been developed for the precision finishing edge grinding of silicon wafers up to 300 mm in diameter.

Figure 23
Nanocentre: for the direct machining of complex aspheric optics, etc. (Courtesy of Cranfield Precision, UK)

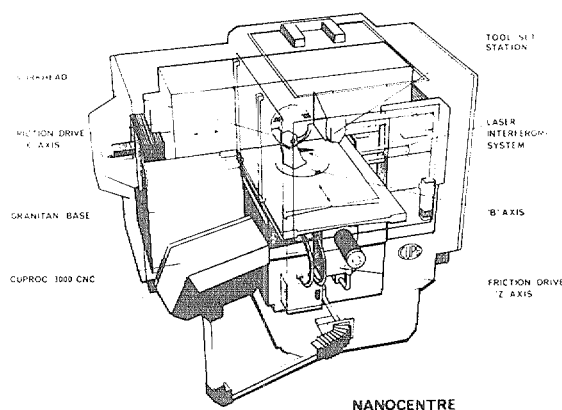


Figure 24
Large optics diamond turning machine. (Courtesy of Lawrence Livermore National Laboratory, USA)

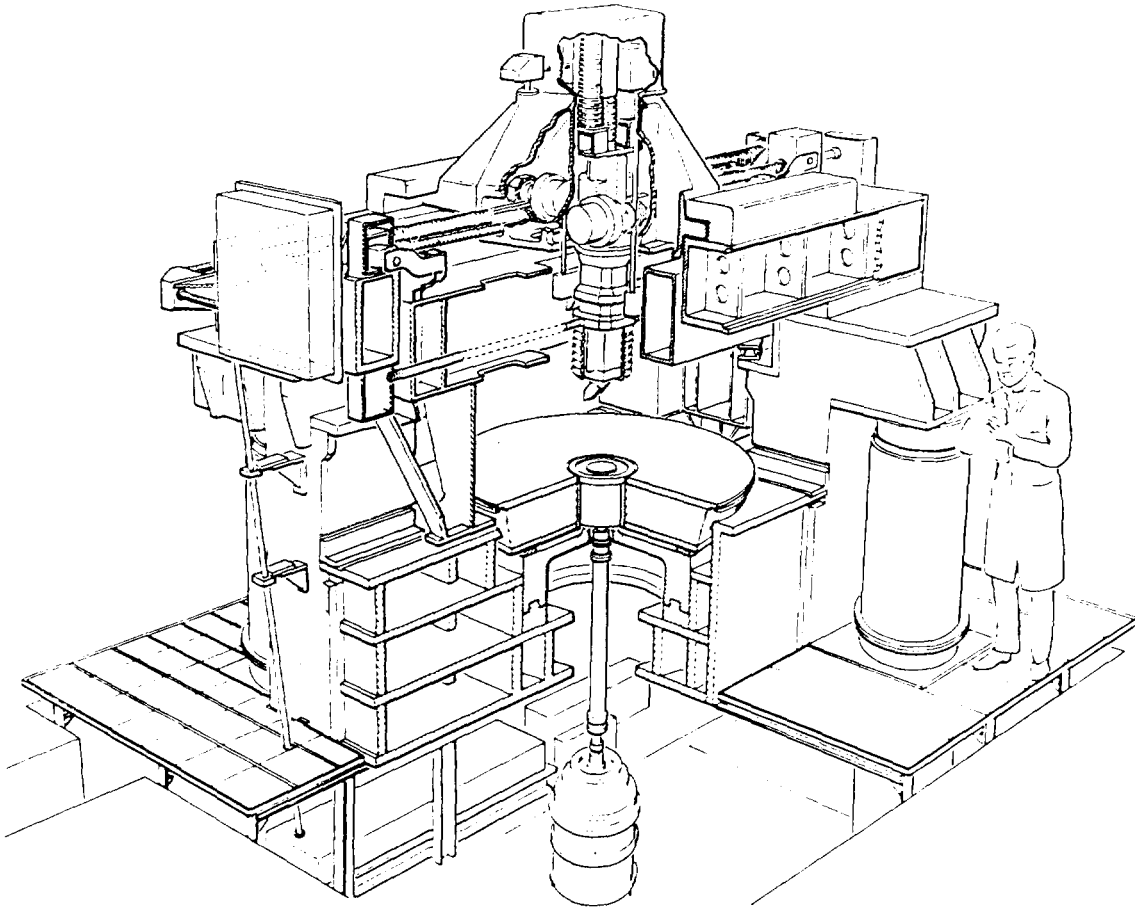


Figure 25
Computer memory disk edge finishing machine for alternative disk materials, e.g. glass ceramic. (Courtesy of Cranfield Precision, UK)

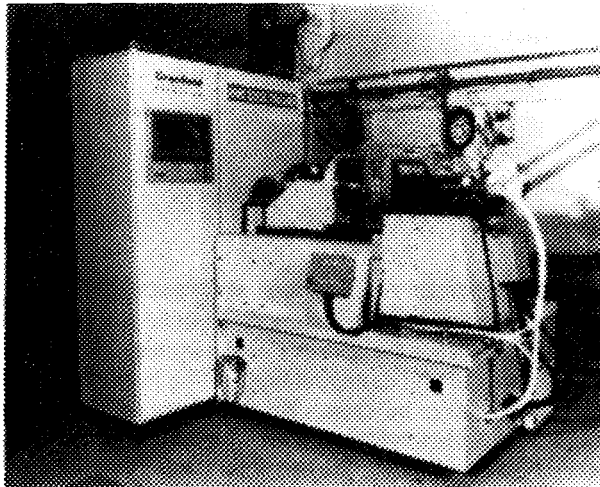


Figure 26

The use of a metrology frame to improve the geometric accuracy of the Nanocentre, built under the UK's National Initiative on Nanometrology (NION)

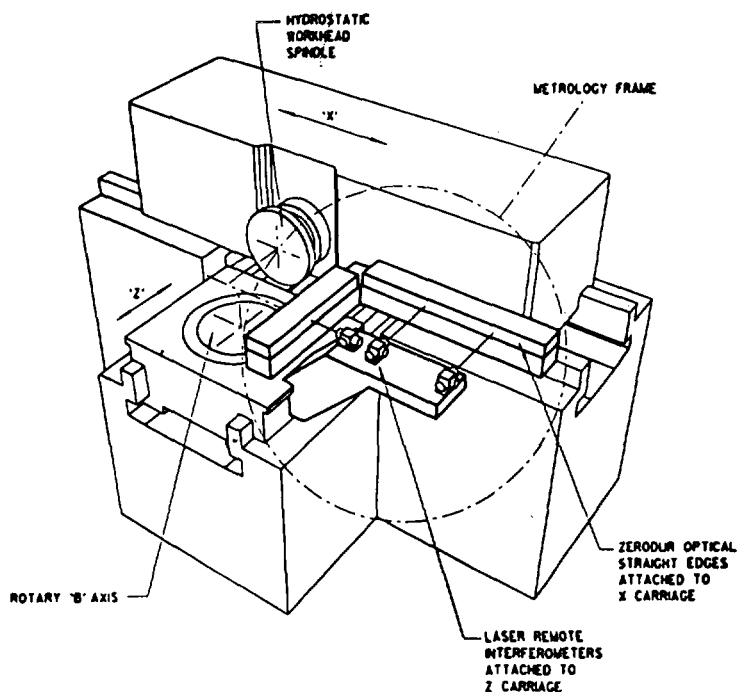
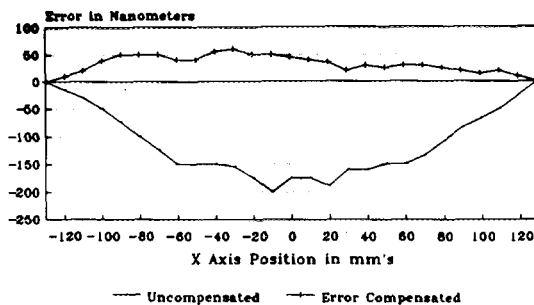


Figure 27

Nanocentre performance in horizontal straightness error motion obtained with the use of a metrology frame and computer software error compensation techniques



7. Scanning probe microscopy (SPM)

7.1 Background - from optical to electron microscopy

In 1873, Abbe [16] showed that the minimum feature that can be resolved in a conventional optical system is determined by a simple relationship involving the aperture of the system and the wavelength of the light used. For many years it was believed that this was the fundamental limit to the resolution of any microscope and that features smaller than this "Abbe Limit" could never be seen. By this formula, the resolution of a conventional optical microscope is limited to about 1 μm . However many developments in 'microscopes' that have much greater resolving power have been made resulting in instruments with atomic resolution; they can 'see' atoms. They are essentially electronic. The transmission electron microscope (TEM), which was invented in 1931 by Ernst Ruska and Max Knoll, uses electrons which have been accelerated to an energy of hundreds of KeV when they have an equivalent wavelength of about 0.001 nm (1 pm), and because of this short wavelength, with electromagnetic lenses analogous to those in an optical microscope, resolutions of 0.1 nm i.e. atomic resolution can be achieved. In this type of microscope, the Abbe Limit is not exceeded and the resolution is limited only by aberrations of the electro magnetic lenses.

The principle of making a 'microscope' by scanning a physical probe across the surface of an object and measuring a consequential effect due to its features has been known for many years. Probably the first of these microscopies was the Scanning Electron Microscope (SEM). In this an electron beam is focused into a small spot on the object and electromagnetically raster scanned across it. Images can be formed by collecting the secondary electrons generated by the impact of the impinging electron beam, by detecting the backscattered electrons or by detecting the x-rays generated. In this way, several different aspects of the object can be characterised, including morphology, average atomic number and composition and some of these aspects to a resolution of a few nm. Here again, however, the wavelength of the probing radiation is much less than the minimum observable feature size. The resolution is limited by the electron beam spot size which in turn, is limited by the electron source and electromagnetic lens aberrations.

The first suggestion of a form of super-resolution microscope (in which the resolution is less than the probing wavelength) was made by Syngé in 1928 [17]. He suggested that it might be possible to fabricate a tiny, sub-wavelength-sized aperture at the end of a glass rod and, by sending light down the rod and scanning the tip raster-fashion across a surface, build-up an image of a surface with a resolution equivalent to the size of the aperture. However, the principle was not demonstrated experimentally until 1972 when Eric Ash from University College in London [18] showed that it was possible to form images of physical features with an electromagnetic probe whose wavelength was many times their size. This was done using 10GHz (30 mm wavelength) electromagnetic radiation in an open resonator formed by a parabolic reflector and a metal plate with a small hole in it. While there is no radiation from the hole, there will be an electromagnetic wave of exponentially-decaying amplitude, called an "evanescent wave", extending beyond the hole. Any object placed within that field will affect the amount of radiation reflected from the plate and hence the impedance of the resonator. If the object is mechanically scanned in front of the hole, the changes in resonator impedance can be used to detect and measure features on the object which are of the same order of magnitude in size as the aperture. They demonstrated that they could resolve a grating with a $\lambda/60$ period (0.5 mm) using a 0.5 mm aperture.

7.2 Scanning tunnelling microscopy

The Scanning Tunnelling Microscope (STM) uses a similar principle [19]. Here, the evanescent wave is an electron wave function with an intrinsic wavelength of about 1 nm which extends beyond the surface of a sharp metal tip, as shown in Fig. 9. If a conducting surface is brought to within about 1nm of the tip and a potential difference is applied between them, then a 'tunnelling current' will flow; this was first discovered by Young and Teague at the USA's National Bureau of Standards in the 1960s. The magnitude of this current is an exponentially-decaying function of distance and is also dependent upon the difference between the work-functions of the two materials. Thus, information can be derived about both the topography of the surface and its chemical make-up. The tunnelling current is about 1 nanoamp and decreases by 10% of its value for each 0.1 nm increase in distance. The tunnelling current is measured and compared with a reference; the height of the tip (z-direction) is servoed by a piezoelectric device to maintain a constant current. The practical realisation of the microscope also uses a piezoelectric device (see Fig. 9) to scan the tip across the specimen surface in X & Y directions. It is possible in this way to obtain spatial resolutions of about 0.01 nm (10 pm) in 3 dimensions and thus to image individual atoms on a surface. One of the first discoveries using the microscope was the 7 x 7 rearrangement of atoms on the surface of silicon, long-predicted theoretically but never observed directly until the invention of this technique. A wide range of scanning probe 'microscopes' have been developed since Binnig and Rohrer of IBM, Zurich, shared the Nobel Prize for physics in 1986 for achieving atomic resolution with their STM, see Fig. 10.

Several extensions to STM techniques have been demonstrated, including Scanning Noise Microscopy [20], in which no bias field is applied to the tip, but the broadband rms noise (which is proportional to the tip-to-sample resistance) is used to control the tip-to-sample distance. This can be used to permit measurements of parameters such as thermoelectric voltage which would not normally be measurable by the STM.

A further modification is the Scanning Tunnelling Potentiometer [21]. Here, an ac voltage signal (usually a few KHz) is applied to the tip to generate an ac tunnel current, which is used to control the tip-to-sample spacing. An independent control loop is used to maintain a zero dc tunnel current so that the tip voltage then tracks to surface voltage on the sample. This can be used to measure the nanoscale potential variations in, for example, Schottky barriers or even on insulating substrates.

Other important extensions to the STM principle use the fact that the asymmetry of the junction makes it a rectifying contact. This has led to experiments in which radio frequency (RF) [22] and even optical signals [23] have been rectified and mixed. Photon emission by tunnelling electrons was first observed in 1988 [24] and it has proved possible to map the bandgap luminescence of semiconductors.

The potential of STM as a surface characterisation tool thus extends far beyond the observation of topography and enables one to gather information about the surface chemistry and electronic structure.

7.3 Scanning force microscopy

The limitation of the STM is that it can only work with conducting surfaces. The Scanning Force Microscope usually referred to as the Atomic Force Microscope (AFM), was developed to overcome this limitation [25]. The initial instrument used a diamond stylus on a gold foil cantilever scanned lightly across the surface of the specimen, with the repulsion being detected using a tunnelling tip. This was replaced with an opto electronic sensor [26] and atomic resolution imaging has been demonstrated [27]. An attractive-force sensing technique was developed [28] which uses an ac excitation of the probe tip. The change in cantilever resonance frequency is sensed as the tip

approaches the sample surface and is affected by the Van der Waals attraction. This type of microscopy has been used for a very wide range of surface characterisation, including:

- magnetic force microscopic imaging of magnetic domains [29] in hard and soft materials e.g. for information storage.
- electrostatic force microscopy for studying charge injected into electrets or voltages on semiconductor circuits.

It is fair to say that this type of ac AFM is probably the most-commonly used mode in commercial instruments.

7.4 Other scanning probe microscopies

A wide range of scanning probe techniques has been explored and it is impossible to review them all here. The reader is referred to reference [30] for a more-complete summary. Two of the more useful are Scanning Thermal Microscopy, which detects the flow of heat from the tip to the sample using a fine thermocouple as the probe [31] and Scanning Near Field Optical Microscopy (SNOM) [32] following the original ideas of Synge [17]. Here, an optical fibre is drawn down to a fine tip. The light reflected from the tip of the fibre as it is scanned is used to reconstruct the surface image. This is useful for obtaining an image at optical frequencies but at sub-wavelength resolution. Scanning Electrochemical Microscopy [33] is being used to map electrochemical potentials.

7.5 Non-imaging applications

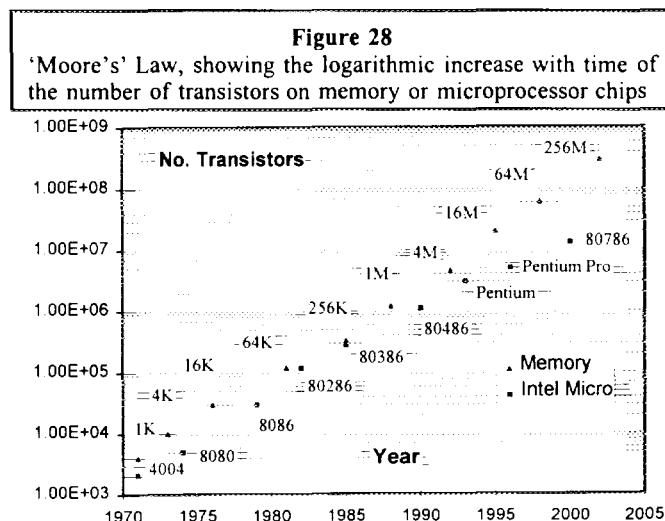
The huge sensitivity of the scanning probe techniques is now being applied to a wide range of other applications. These include:

- accelerometers using the sensitivity of a tunnelling probe to detect very small movements of a proof mass. In some versions of these, electrostatic attraction is used to servo the mass to a null displacement position [34].
- chemical and calorimetric sensors using the flexure of an AFM cantilever as the sensing element [35, 36]
- manipulation of atoms on a surface by both STM and AFM probes. In the former, atoms on a clean surface have been picked-up, moved and replaced in new positions by applying potential pulses to the probe tip. As indicated in section 2.4, this method was used in 1990 to produce the letters IBM, picked-out by 35 xenon atoms on the surface of a nickel substrate (Fig. 11). Whereas this work was performed at 4.2K, AFM probes are now being used to manipulate more complex molecules such as porphorins on surfaces at room temperature [37] whose conformations on the surface can be identified and modified [38].

8. Semiconductor devices

8.1 Background

Everyone is familiar with the impact which semiconductor technology is having on the sophisticated electronic systems which assist in almost every aspect of modern society, especially health care, medicine and safety. In Feynman's prophetic lecture "There's Plenty of Room at the Bottom" [4] he correctly foresaw the emergence of the integrated circuit with many components on a single piece or "chip" of semiconductor and the impact that miniaturisation would have on the capabilities of electronic computers. He also predicted the emergence of the fabrication methods which are now used to make integrated circuits, including electron beam writing. The silicon chip has been doubling its capability every 18 months since its invention in the late 1950s (Moore's Law [39] - see Fig. 28) and has penetrated our every-day lives to the extent that even quite humble domestic appliances such as toasters or irons can have a computing power within them that, 30 years ago, would have been found only in the mainframe computers of large corporations. Huge benefits have accrued from this "silicon revolution", not only economically but also in quality of life for many people. The semiconductor industry is now of enormous world-wide economic importance, with a size of the order of US\$100 bn, and which is expected to double by the year 2000.



8.2 Smaller dimensions and photo lithography

Until now, the enormous growth in silicon chip capability has been based on the continuous shrinkage of the dimensions of the individual transistors and the connections between them, which increases the number of components on the chip, increases their speed and reduces their power consumption, while at the same time reducing the time delay for electrical signals travelling between them. The physics upon which the transistors is based, predicts that they will work as-now at dimensions down to (0.03 μm) and thus, the drive to make higher performance chips reduces to the problem of how to make ever-smaller features. The smallest structural dimension in the present (1997) generation of 16 Mbit commercial memory chips, traditionally the type of chip that leads the development of the technology is 0.35 μm . These dimensions are printed using a process known as photolithography, in which ultraviolet (uv) light from a mercury vapour lamp is shone through a mask containing the features of the chip and projected onto the surface of the silicon wafers in a machine known as a photolithographic "stepper" - so-called because it prints an image of one chip and then 'steps' to the next location on the wafer to print the pattern for the next, and so-on. There are two mercury spectral lines in use, at 436 nm and 365 nm, known as the "g" and "i" lines respectively. With a variety of technical innovations, it has proved possible to achieve the 0.35 μm line widths using the i-line, but this is now considered to be the practical limit. Thus further technical solutions to the problem of printing fine lines need to be found if the pace of silicon technology improvement is to be maintained in accordance with Moore's Law. In particular, if the target for introducing 1Gigabit (10^9 bits) memory chips is to be met early in the next century, it will be necessary for feature sizes to be between 0.2 μm and 0.1 μm . To go beyond this and to achieve memory sizes of 16 Gbit in production, it will be necessary to go below 0.1 μm (100 nm), which is well into the region of nanotechnology [42]. Many companies are currently assessing the use of photolithography using the light from an excimer laser with a wavelength of 193nm as one possibility to achieve feature sizes of about 0.18 μm , but there are severe technical difficulties associated with this; one example is problem of making lenses work at this wavelength. It has been pointed-out that while prototypes have been demonstrated using early steppers working at this wavelength, the task of bringing these to production is lagging about 3 years behind the schedule outlined for 1Gbit chips. It may be that 193 nm photolithography will not prove to be a practical proposition. The two main alternatives are X-ray and electron-beam lithography.

8.3 X-ray lithography

In X-ray lithography, much shorter wavelength radiation of about 1 nm is used, hence the term nanolithography [40]. In principle, this should be the obvious choice for the very fine line widths as it does not suffer from the problems of diffraction associated with the ultra violet (uv) steppers. However, there are many formidable technical problems here. The first is associated with sources of radiation. One possibility is to use the X-radiation emitted by electrons as they circulate in a storage ring, known as a synchrotron. Until recently, such facilities have been so expensive that providing them has been the province of national governments. However, the UK manufacturer, Oxford Instruments have made and sold a small number of commercial storage rings. These are still very expensive (US\$ 20 m - 50 m each), although each machine will supply radiation to up to 16 stepper stations. Alternatives include the use of high energy laser pulses to vaporise metals, the plasma then emitting 'soft' X-rays. The second problem is associated with the lack of available lenses to focus the X-rays, so that the mask image can be demagnified onto the chip. This means that each mask must have the same dimensions and dimensional tolerance of the chip itself. There is research in progress into the use of multilayer coatings on mirrors to reflect the X-rays, but the mirrors themselves need to be shaped and smoothed to 0.1 nm precision over an area of many square millimetres, a severe challenge for the machining aspects of nanotechnology outlined in section 6 above. Finally, the masks themselves need to be made from a combination of materials which are either transparent or opaque to X-rays. Achieving the required opacity in the lines is very difficult. Gold and tungsten are the preferred materials, but they need to be laid down in layers up to 0.5 μm thick on a thin silicon membrane. If the required feature is only 100 nm wide then there is a real problem with the height to width aspect ratios of the features on the mask.

8.4 Electron beam lithography

An alternative process which certainly permits the fabrication of lines less than 100 nm wide is electron beam lithography. This uses a machine similar to a scanning electron microscope (Fig. 5) to "write" a pattern into a plastic resist with a finely-focused electron beam. The fundamental problem with this process is that it is inherently slow, because each line on every chip on a wafer must be written sequentially. Hence, the process has only been used commercially for very small runs of special or prototype devices or for semiconductor devices on which there are very few lines which need to be so narrow. An example of the latter is the high electron mobility transistors fabricated on gallium arsenide for microwave applications, where there are very few lines on a chip and most of the device can be made using conventional photolithographic processing.

A further application of the electron beam (EBM) process is the production of masks for optical and X-ray lithography. A related process, focused ion beam (FIB) lithography, in which the electrons are replaced by high energy ions (Fig. 7), may hold some advantages in resolution in that the secondary electrons produced in the resist by ion bombardment are of very low energy in comparison with those produced in electron bombardment. Research is in progress on the use of improved projection systems for both EBM and FIB techniques to overcome the problem of very slow serial scan and thus low productivity [41].

8.5 SPM possibilities

Finally, the invention of the scanning probe microscope as described in section 7, has led to a number of researchers exploring the possibility of using a scanned tip to "write" patterns on a silicon chip at the nanometre

level. It has been shown to be possible to use electron emission from an STM tip to write patterns in a polymer resist, which can subsequently be replicated in metal. Gold lines as narrow as 20nm have been produced in this way [43]. In order to make such patterning techniques viable, it will be necessary to produce arrays of large numbers of tips, each of which can be individually steered. Such arrays have been made for vacuum microelectronic display applications and it has been demonstrated that piezoelectric layers deposited on cantilevers can be used to steer STM tips over quite large areas [44]. Basic feasibility of the generation of lines has thus been demonstrated but a great deal of research needs to be done before a process could even be used to make chips in research, let alone production.

8.6 An uncertain way forward?

It can be seen from the above that there are formidable technical obstacles to the fabrication of chips on a commercial basis with line widths down to 100nm and less. Even for a 200 nm line width

| | |
|---|-------|
| Tolerance on linewidth is | 20 nm |
| Killer defect size is | 40 nm |
| Mask registration tolerance is | 50 nm |
| Mask accuracy (feature to feature) is | 10 nm |
| and on the normal 10 : 1 manufacturing to metrology rule, then, | |
| Metrological accuracy needed is | 1 nm |

This represents an enormous challenge in large scale manufacture. The required metrological accuracy shows the need for fundamental nano-metrology equipment such as the NIST 'Molecular Measuring Machine' described in section 5.4 above.

Furthermore is not clear which of the lithographic routes of those outlined above, if any, will be successful on a commercial scale. It has been pointed out that we may be approaching a fundamental technological limit to the continuing development of the semiconductor industry in the direction of ever-increasing performance based on higher packing densities and smaller linewidths and that the future in the next century may lie in greater diversification of the functions that can be achieved on a single chip. Certainly, the costs of the present generation of silicon fabrication plants is prohibitively expensive for all but the largest companies or even countries, with US\$1 bn to US\$ 2 bn becoming the norm. It is clear, nevertheless, that the market rewards of continuing to push the barriers forward are also very large, with a world market of US\$100 bn, doubling every few years. Therefore there is a very powerful impetus to maintain the present technical improvement rate. If this is to be done, the full armoury of nanotechnology techniques and processes outlined in this article will need to be applied to solving the technological problems.

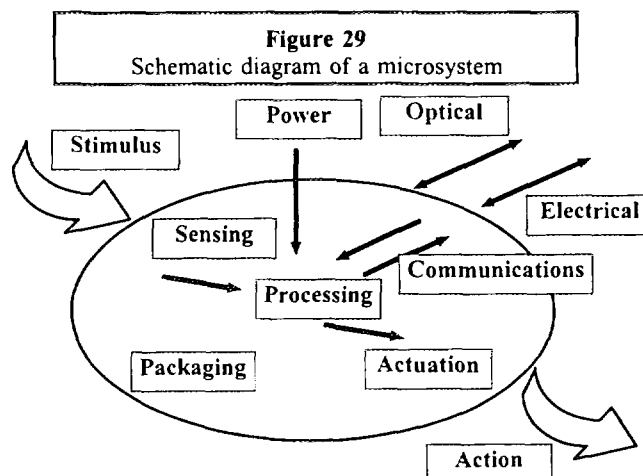
For further reading on this topic, please see references [40 to 43].

9. Microsystems Technologies (MST) and Micro-electro-mechanical Systems (MEMS)

9.1 Background

The power and the capabilities of silicon integrated circuits was expounded in section 8, but it is fair to say, however, that powerful as these chips are, they are only useful if given information which they can process and then act upon. This is frequently achieved via a range of external sensors. In many cases they will communicate the results of their computations either with other chips or ourselves through a variety of links such as wires, optical fibres, visual display units etc. They may also demand the generation of some mechanical function via an actuator such as a motor or a pump. These peripheral functions are currently provided as separate components which must be individually manufactured and then assembled into a complete system; this can be a costly process which also greatly increases the size of the finished system.

Microsystems engineering [45] aims to exploit the increasing signal processing power which can be integrated on a single silicon chip by putting it together with the sensing, actuation and communications functions into a single microassembled or integrated package, as shown in Fig.29.



The sensor and actuators can either be assembled with separate silicon chips into a package to make the microsystem using hybridisation methods (e.g. chip-and-wire or flip-chip bonding) or these functions can be made on the same chip of silicon as the processing circuits. There are clear advantages of cost, system size and achievable manufacturing volume in doing this. Either way, there is a requirement to make very small mechanical components which can move and/or to integrate materials for sensing or actuation functions which are not traditionally part of the integrated circuit (IC) engineer's repertoire. This is called microsystems technology (MST) or in the USA, micro-electromechanical systems (MEMS); it presents new challenges in terms of process integration and device design which go well beyond the normal requirements of IC manufacture. There can be, for example, a need to design and make freely-suspended or moving 3-dimensional structures in which the individual components may be only 1 μm - 2 μm thick. These may incorporate materials which would not be compatible with a normal silicon processing environment; the challenges posed by this are addressed below.

Examples of microsystems which are already bringing real benefits to society include:

- Accelerometers and gyroscopes for automotive applications, particularly for air-bag sensors, automatic suspension and navigation systems
- Uncooled passive infra-red (PIR) sensor arrays which image the infra-red radiation (IR) emitted by people are now used in applications such as firefighting and retrieving survivors from collapsed buildings.
- "Smart" pills and catheters incorporating pressure, temperature, chemical and acoustic sensors for diagnostic use.
- The concept of the "laboratory-on-a chip" incorporating means for synthesising, pumping, handling and testing chemicals is one that is gaining great interest in the pharmaceutical field.

9.2 Microsystems and nanotechnology

The critical dimensions in many microsystems are of the order of 1 μm and thus are not strictly in the nanotechnology range. However, many authors consider the topic to fall into the general area of nanotechnology and there is, indeed, a significant area of overlap between the fields. In particular, there are contributions which MST and MEMS can make to nanotechnology. For example, there is considerable promise in integrating the components of a highly miniaturised scanning probe microscope (sensors, actuators, amplifiers etc) onto a single chip and significant progress has been made in this direction [46]. The likely benefits of this work will be the reduction of the cost of these tools to the extent where they may be used in, for example, individual molecular sensors.

Several processes used in microsystem fabrication have nanometric accuracy capability.

9.3 Processing technologies for microsystems

The vast majority of processing technologies which have been applied to the fabrication of microsystems have evolved from those developed originally for IC manufacture and packaging. Hence, all the traditional processes of thin film metallisation and photolithography are used. The need to fashion 3-dimensional structures is being met in a number of ways, which have been reviewed in [45] but are summarised as follows:

- **bulk micromachining** based on chemical etching processes; it has been known for many years that single crystal silicon can be etched using a variety of chemicals, for example potassium hydroxide/propanol or ethylene diamine/pyrocetechol solutions [47]. These etchants tend to attack certain facets of the cubic silicon structure much more rapidly than others. For example, an etch rate ratio of 50:1 can be achieved between the (100) and (111) direction crystal facets. The etch rates can also be controlled by doping the silicon, so that a heavily p-doped region will act as an effective etch stop or by electrically-biasing a p/n junction. Thermally or chemically deposited glass layers, which can also be patterned by photolithography and etching, can be used as etch masks or as mechanical components in their own rights. Polysilicon layers, which can also be chemically doped to provide etch selectivity, can also be used as mechanical components. This crystallographic and chemical selectivity can be used to create quite complex structures in the silicon such as cavities, membranes and suspended masses, which can be used for devices such as pressure sensors and accelerometers.
- **surface micromachining** - a process related to bulk micromachining but instead of etching structures into the bulk of the silicon, it seeks to grow all the components on the surface of the wafer. Examples of the materials used include phospho-silicate glasses for the sacrificial layers and polysilicon [48] or silicon nitride for the mechanical components. The advantage of this type of process relative to the bulk micromachining is that the silicon itself remains undamaged so that it can all be used for electronic signal processing components.
- **LIGA** - (from the German: *L*ithographie *G*alvanformung und *A*bformung) is a deep etching process based on lithography, electroplating and moulding [49, 50] The LIGA process in its original form uses X-rays from a synchrotron to expose a thick layer of photoresist, to a depth of up to 1000 μm (1 mm) but with a lateral resolution of better than 1 μm . Thus, very high vertical aspect ratio components can be formed in the resist. These are generally replicated by an electro-plating process to make a master, which can then be used to mass-produce the original object by embossing or injection moulding, in a fashion related to the process that was used for making vinyl gramophone records. A variety of process have been devised which are related to this but which do not use the high-cost radiation from a synchrotron to create the prototype shape; they use either conventional photolithography to pattern a thick photoresist or use an ultra-violet laser such as an excimer laser to etch a pattern directly into a plastic to act as a micro mould.

9.4 Sensing and actuation materials for microsystems

The range of sensing materials which will turn a stimulus from a measurand into an electrical or an optical signal and which have been, or are being, applied to microsystems is shown in Table 1. The corresponding measurands are also shown in this table. The practicality of many of these systems has been established, although not all have yet been commercialised.

Sensing materials

- Oxides and other inorganic compounds
- Semiconductors
- Metals
- Polymers
- Organics
- Biochemicals
- Biologicals

Measurands

- Light or infra red (IR)
- Magnetic/Electrical Fields
- Sound
- Pressure
- Acceleration/Rotation
- Pollutants
- Antibodies/Toxins
- etc

Table 1 Sensing materials and measurands for microsystems applications

In the case of actuation the range of techniques, and the corresponding materials, for turning an electrical signal into a physical motion is much more restricted. The prospective candidate materials are listed in Table 2 and their characteristics in Table 3 [51].

Actuation Techniques

- Piezoelectric
- Electrostatic
- Electromagnetic
- Electrostrictive
- Magnetostrictive
- Ionic flow (cf muscle)
- Martensitic transitions (thermally or electrically driven)

Materials

- Ferroelectric oxides
- Polymers
- Magnetic materials
- Biological materials
- "Memory" metals/ceramics

Table 2 Actuation techniques and possible materials for use in microsystems

| Class | Stress NM/m ² | Stiffness GN/m ² | Strain % | Strain Rate s ⁻¹ | Power W/kg | Energy kJ/m ³ | Efficiency % |
|--------------------|-----------------------------|--------------------------------|-------------|--------------------------------|---------------|-----------------------------|-----------------|
| Piezo- electric | 35 | 40 | 0.09 | >10 | >1000 | > 10 | > 30 |
| SMA | > 200 | 78 | > 5 | 3 | > 1000 | > 10 | > 3 |
| Electro- static | 0.04 | < 0.01 | > 10 | > 1 | > 10 | 1 | >20 |

Table 3 A comparison of piezoelectric, SMA and electrostatic actuation for microsystems applications

The most promising of these for microsystem applications are the Shape Memory Alloy (SMA) materials [52] which are characterised by moderate displacements and high forces but with low thermal efficiencies, electrostatic actuators (which are characterised by high displacements and good efficiencies but with low forces) and piezoelectric/electrostrictive materials (which are characterised by low displacements and high forces with good efficiencies).

Of all the new materials being assessed for microsystems, the ferroelectric materials are the most interesting because they allow the designer to combine a variety of useful properties by virtue of their polar crystal structures. These useful properties of ferroelectrics are:

- **piezoelectric** - which means that they will generate electrical signals when stressed and can be used in sound, pressure and acceleration sensors. A piezoelectric material will also undergo mechanical strain when an electric field is applied to it and thus can be used for actuation in pumps, vibration control components, motors etc. In its most accurate servo control application it is used with picometre precision in scanning probe microscopes. This effect can also be used for generating electro-mechanical resonances

which can be used for frequency control and filtering as well as chemical and biochemical sensors. Ferroelectrics such as lead zirconate titanate (PZT) exhibit some of the strongest piezoelectric effects known.

- **pyroelectric** - by which effect, electrical charges are produced when the temperature of the material is changed. Extremely small changes in temperature (less than one tenth of one millionth of a degree Celsius, $<1 \times 10^{-7}^{\circ}\text{C}$) can be detected and, with the appropriate thermal structure and electrical amplification, they can be used as very sensitive detectors of infra-red radiation. Arrays of such detectors can be used to create thermal images from heat (infra red) radiation. Unlike more conventional detectors of such radiation, they need no cooling and are thus low power and easy to use. Again, compositions related to PZT are particularly interesting in this area.
- **electro-optic** - an effect which can be used to modulate light signals passing through the ferroelectric material and hence to communicate information. Lanthanum-doped PZT compositions show some of the strongest electro-optic effects known.

Ferroelectrics also show high dielectric constants and dielectric hysteresis which can be used in electrical capacitors and non-volatile memories in microsystems.

Over the last 10 years, a range of processes have been used to grow films of materials such as PZT onto silicon and other substrates. These have included deposition from solution, from the vapour phase and by evaporation or sputtering in a vacuum. The critical issue for applications to microsystems will be how best to obtain good quality thin films (1 μm to 5 μm thick) on silicon devices at a processing temperature of less than 500°C [53].

9.5 Applications of microsystems

Microsystems, incorporating sensing, actuation, signal processing and communications functions incorporated in a micro-assembled package or integrated into a single chip, are rapidly emerging as devices which are likely to be of major economic importance in the future [54]. This has been recognised in several European countries and the topic has received a substantial boost in the Framework 4 research programme in the European Community [54]. Currently-available commercial microsystems incorporate only sensing, signal processing and communications functions.

- **automotive applications** - examples include sensors for various automotive functions [55], such as manifold air pressure (MAP) sensors which are used, together with exhaust oxygen content sensors in closed-loop control of engine fuel systems. The MAP sensors use a micromachined diaphragm of silicon which is exposed to the inlet manifold pressure. The displacement of the diaphragm is either measured using piezoresistive sensors implanted in it or from the change in capacitance relative to a capacitor plate placed adjacent to it. In the last two decades these sensors have helped to raise the average fuel consumption of US cars from 4 to 12 km per litre. The accelerometers which are used for controlling air-bag inflation command a large and growing market. These use a proof mass of silicon suspended on a silicon membrane and the displacement of the diaphragm is again sensed either capacitatively or piezoresistively. Other automotive applications include sensors for 'smart' suspension systems, anti-skid braking etc.
- **medical applications** - in the field of medicine, similar technologies have also brought great benefits. The MAP technology inspired manufacturers to develop a disposable blood pressure transducer (DPT), leading to a drop in unit price relative to the previous technology, from US\$600 to US\$ 10. Other applications include intra-uterine pressure (IUP) sensors, angioplasty pressure sensors, sphygmomanometers and many others.
- **consumer applications** - in the consumer market, applications for smart pressure sensors range from scuba diving computers through altitude sensors to vacuum cleaners. One very exciting and potentially large market is for digital projection TV displays. In this case, actuation is fully incorporated. Texas Instruments [56] have developed a Digital Micromirror Device, which produces a modulated image by tilting thousands of tiny mirrors integrated onto chips. The device produces much better resolution and contrast than conventional projection displays for digital imaging especially on large displays.

In all these examples the properties of silicon are used to provide both the sensing and the actuation mechanism in the case of the digital micromirror device. The use of silicon restricts the range of properties which could otherwise be obtained. If, however, novel materials can be integrated onto silicon, then the field of pyroelectric infra-red detection and thermal imaging, for example, could be greatly expanded [57]. Arrays of more than 10,000 IR detectors made by hybridising ceramic detectors onto silicon readout chips, can give thermal resolutions of better than 0.1°C and excellent thermal images [57]. One of the prime objectives for technical assistance to firefighters is seeing through smoke thus overcoming the fire-fighter's greatest hazard: long-wavelength IR thermal imaging achieves this as well as showing where the seat of the fire is. Other IR microsystem applications include thermographic surveys of buildings, machines etc. by industrial users [58], doctors using medical thermography [59, 60] and IR vision enhancement for cars [61, 62]. It has been pointed out that IR vision enhancement can make a major contribution to driving safety [62], and not only on the road. Equipping boats navigating crowded waterways at night with thermal imagers could lead to a marked improvement in safety there as well [63].

- **actuators on a chip** - it is possible to provide some actuation by using electrostatic attraction between two movable components on a chip. These can be made by etching the silicon in a carefully controlled way into complex 3-dimensional structures. Micromotors approximately 100 μm in diameter have been made experimentally in this way [64]. However, the forces which can be generated by electrostatics are very small and very few useful devices have been made using this principle. If actuation with useful

force and displacement could be introduced by, for example incorporating a piezoelectric layer on the silicon, then a much wider range of applications and markets would be generated, including micropumps for medical applications and environmental air and water analysis etc. [65], novel fuel injection systems, ink jet printers, motors [66] and microrobotics [67].

9.6 Markets for microsystems

The market for smart silicon sensing devices has grown enormously since 1983, at a compounded growth rate of 22% pa to about US \$500m in 1994, much faster than the 3-5% rate of the US\$5 bn sensor market as a whole; by 1996 it was estimated to be well into the billion dollar range [68]. The market is now estimated to grow to US\$14 bn pa by the year 2000, with the automotive applications - mainly pressure and inertial sensors - accounting for 41% of the total.

The market for uncooled thermal imaging systems is expected to grow markedly [69], to a multi billion US\$ pa business by 2005, although this entirely depends upon the reduction in unit price of detector arrays; this can only be achieved by a move to a fully integrated structure using ferroelectric thin films as the sensor elements. An example of one application is given in Fig. 30 which shows a thermal imaging system made by Cairns & Brother in the USA for fire-fighting applications using a solid-state pyroelectric array manufactured by GEC Marconi (UK). Being fully solid state, these are small enough to be helmet mounted and enable fire-fighters to see through smoke without having their hands encumbered by bulky thermal imaging equipment.

Figure 30

Cairns IRIS pizeoelectric thermal imager for fire fighters manufactured by GEC Marconi and marketed by Cairns Inc. (Courtesy of Cairns (UK) Ltd.)



10. Nanostructured materials

10.1 Introduction

The topic of "Nanostructured (or Nanophase) Materials" can be defined as the fabrication of materials in which there is some aspect of dimension (for example grain size or layer thickness) which is in the nanometre range. It has enabled scientists to access new ranges of electronic, mechanical or optical properties. Over 2000 years ago, the Romans discovered that the inclusion of very small amounts (approximately one part in 50,000) of gold in glass in the form of either metal or metal salts produced a deep red colour if the glass was appropriately heat treated. On dissolution, and cooling to room temperature, the gold produces a straw colour in the glass, but after annealing the colour "strikes" and a deep ruby-red is formed [71, 72]. Similar effects can be produced by using copper or

selenium. The phenomenon is due to the formation of nano-sized particles of metal crystals in the glass, which form during the annealing process. These are far too small to scatter visible light so the glass retains its transparency. Instead they strongly absorb light of below a certain wavelength, giving the colour observed. Clearly, the physics of this phenomenon would not have been understood by the early glass makers, but it is just one example of how the deliberate nanophase structuring of materials can open-up new options for materials and product design. The following sections summarise the different ways in which such materials can benefit society.

10.2 Optical materials

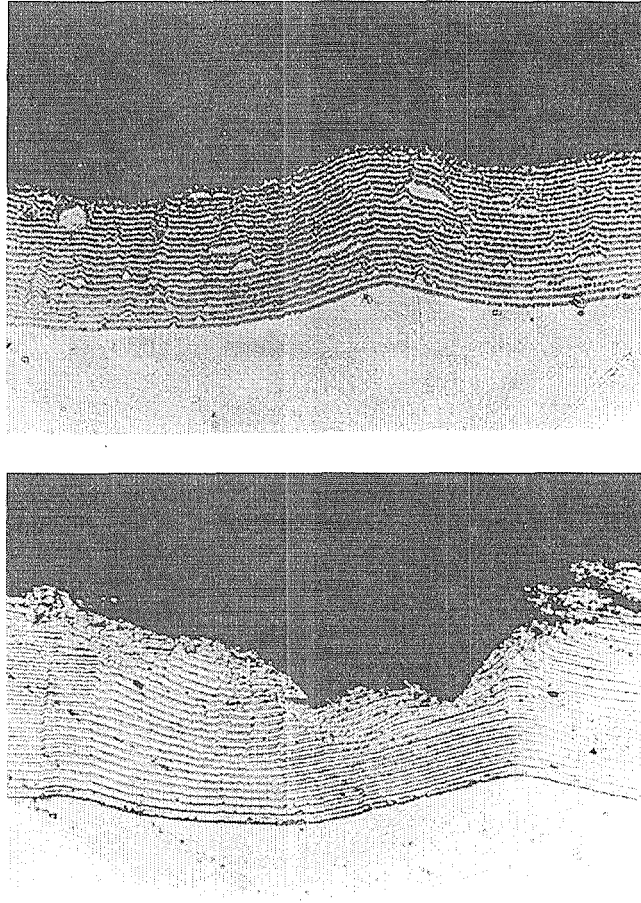
In the example of the formation of coloured glasses, it is quantum-confinement of electrons in the small particles of the conductor that shifts the cut-off edge for the absorption spectrum in the metal into the blue region of the spectrum, which gives the red colour to the glass. Typically, the smaller the crystallite, the wider the gap between the valence and the conduction bands and the shorter the wavelength of the absorption edge. Hence, it is possible to produce powders of a single material, and the semiconductor cadmium selenide is a particular example which has been extensively studied, in which the particle size determines the absorption cut-off wavelength and hence the material's colour. In this material, crystallites of approximately 1.5nm in size will appear yellow, while 4nm particles will appear red. Still larger particles appear black. This principle is now being applied to tailoring the sizes of particles of zinc oxide and titania for sun-screen cosmetics where it is desirable to shield the skin from particularly damaging wavelengths of the ultra-violet component of sunlight. A further promising application of this quantum confinement principle is in the development of semiconductor lasers for the fibre-optic communications market, where it is highly desirable to be able to tune the wavelength of emission, so that many channels of optical information can be sent simultaneously down a single optical fibre. In this case the technology consists of making very small "quantum-wires" or "quantum-dots", usually in a stack of nanometre-thick layers of different compositions of gallium arsenide in solid solution with aluminium or indium arsenide or selenide. The different compositions enable the technologists to alter the band-gaps of the semiconductor layers, leading to one dimension of quantum electron confinement in a single layer. The physical restriction of the other dimensions of the layers by photolithographic pattern definition and plasma etching is used to create wires or dots. Other layers of materials with different refractive indices can be used to confine the light into the regions of the semiconductor material containing the quantum dot. The technological problems of fabricating these structures and making electrical contact to them are considerable. The fabrication of two and three dimensional arrays of such quantum dots, effectively making a structure with its own set of allowed and excluded optical and electronic states and therefore band structure, also offers new possibilities for active and passive devices. Lasers using quantum well structures based on layers (i.e. with one dimension of electron confinement) are now commercially available and are used in high data-rate fibre-optic communications systems, but the technology for making optoelectronic devices based on quantum wires or quantum dots is still in its infancy and it will be some time before they appear on the market.

10.3 Structural Materials

While the mechanical properties of materials (e.g. yield stress) are ultimately determined by the strength of the bonds holding their constituent atoms together, the actual parameters that are measured for a bulk material are strongly related to their physical form, and particularly the presence of defects. Thus, a thin glass fibre with a defect-free surface can sustain spectacularly high levels of strain without failure while a piece of plate window glass will sustain hardly any strain before breaking, due to the presence of microcracks in the surface. Similarly the hardness and ductility of a metal is largely determined by the ease with which dislocations can propagate through the crystallites which make up its structure. Richard Siegel and co-workers at Renselaer Polytechnic Institute (USA) have demonstrated that the hardness of pure copper is increased by a factor of up to five times when the grain size is reduced to 6nm. This is because the small crystallites cannot sustain dislocations and thus are relatively difficult to deform. An important potential application of this principle is in the design of coatings for advanced damage tolerant surfaces. At Cranfield University (UK), layered microstructures with individual layer thicknesses of between 10 and 1000 nm have been used to produce erosion protection systems for applications as diverse as gas turbine compressor blades and magneto-optical storage disks. In the former case, a 50 μm coating is produced which consists of a stack of metal/ceramic layers with individual layer thicknesses of between 100 and 1000 nm. In the latter case, a ceramic/ceramic multilayer coating is produced which consists of 2000 layers, each 15nm thick. In both cases, the protection is obtained by tailoring the sub-layer thicknesses to the footprint which is generated by the Hertzian contact between an impacting particle and the surface. Thus, larger impacting particles require more-widely spaced layers than smaller ones. Fig. 31 shows a cross section through a 50 μm multilayer coating after having been impacted by a 600 μm particle travelling at 250ms⁻¹. The coating has not fractured and all of the energy has been dissipated in the elastic rebound of the particle, fracture of the particle or plastic deformation of the substrate. In service exemplified by these conditions, this type of layer can provide a 300-fold improvement in component life when compared with uncoated metal.

Figure 31

A multi layered Ti/TiN coating produced commercially by Multi-arc Ltd. shown here a) before and b) after impact by a $600\ \mu\text{m}$ sand particle travelling at $250\ \text{ms}^{-1}$. The impact of the particle causes the top layers to form into a tough composite, absorbing energy and protecting the underlying substrate. (Courtesy of Prof. J.R. Nicholls, Cranfield University (UK))



10.4 Self-Assembling Materials

Nature has provided many ways of organising materials into ordered structure, with a reduction in the net free energy being the driving force. An example is the growth of a crystal in which the atoms exhibit long range spatial order from the liquid phase in which they only exhibit short range order. There are many other examples of such thermodynamically-driven ordering, which are starting to be exploited technologically, in which structures are ordered on the nano-scale.

10.4.1 Langmuir-Blodgett (LB) Films

Certain long-chain organic molecules which have a group on one end which is water-loving (hydrophilic) and a water-hating (hydrophobic) tail will organise themselves if floated on the surface of water so that the tail is oriented perpendicular to the surface. (See fig. 32) Such layers can be compressed to form a molecular monolayer which shows long-range crystalline order in the layer plane. Such a layer will be transferred to a flat surface dipped into the water and successive layers can be deposited to build-up structures monolayer-by-monolayer. The technology for this is well advanced, to the point where different materials can be deposited in successive monolayers and their orientation can be controlled. A wide variety of molecules have been configured into such layers, exhibiting a plethora of effects such as optical non-linearity, piezoelectricity and semiconducting behaviour. Potential applications include optoelectronic devices, resists for nanolithography and active layers for gas sensors. (See reference [72] for further information.)

10.4.2 Liposomes

Long-chain molecules, such as phospholipids, which possess hydrophilic heads and hydrophobic tails can also show interesting effects if dispersed in water. They tend to form double or bi-layer membranes with their heads on the outside and the tails on the inside (see Fig. 32-). These membranes will wrap around to enclose tiny spherical volumes, which can be in the nano-size range. These tiny capsules can be made to enclose a wide variety of materials ranging from drug molecules to the cadmium sulphide clusters referred to in section 10.2. The technology

promises to be an important underpinning-tool for the manufacture of nano-sized powders. Further discussion of this topic follows in section 11.1

10.4.4 Buckminsterfullerenes

The discovery by Harold Kroto of Sussex University (UK) and his co-workers of the so-called "third-form" of carbon in which the atoms are arranged in spheroidal molecules, and of which C_{60} is the prime example, has led to the development of a whole new range of physics and chemistry based upon these "fullerenes" [78]. Recently, there has been much interest in manipulating these molecules on surfaces using STM methods. One of the interesting developments for the field of nanotechnology is the discovery that these compounds can also form long cylindrical tubes with nano-sized diameters. These can form the smallest electrically conducting "wires" ever made and could be a component in future novel nanoelectronic circuits.

10.5 Further Information

For further information on the topic of nanophase materials, the reader is referred to the references numbered from [72] to [80].

11. Molecular, biological and medical aspects of nanotechnology

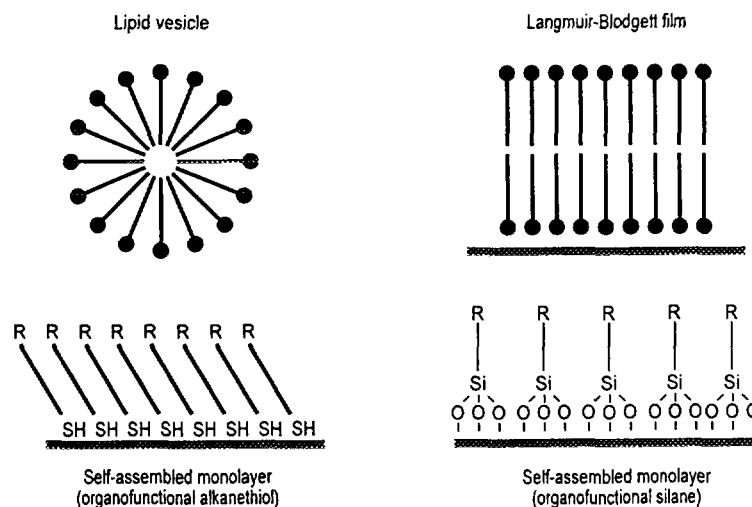
11.1 Self-assembly - thin films, liposome-based systems

Previous sections have focused on an extension of traditional approaches to nanotechnology which exemplifies the "top-down" approach to nanotechnology. The alternative "bottom-up" approach relies upon exploitation of the fields of chemistry and biology and comprises the formation of structures, both microscopic and macroscopic, from molecular building blocks. The advantages of this approach centres around the ability to produce complex nano-structured materials and entities via the relatively simple macroscopic handling of liquids and solids. The challenge becomes the design and synthesis of the molecular building blocks and the understanding of their interactions with liquids, solids and other molecular building blocks.

Biology is a highly complex and feature-rich example of the "bottom-up" approach and is discussed further in sections 11.3 and 11.4. Biology fundamentally relies upon the self-assembly of molecular components at nanometre scales and modern chemistry has now appreciated this approach and is applying such concepts in a number of areas. Molecular self-assembly can be summarised as the spontaneous association of molecules into stable, structurally well-defined aggregates or surface-coatings joined commonly by non-covalent bonds. A wide variety of non-covalent interactions are possible including ionic, hydrophobic, hydrogen bonds and van der Waals interactions. The formation of vesicles from a solution of amphiphilic molecules (Fig. 32) is a classic example that has been studied for over a century; Langmuir-Blodgett films is further example. The self-assembly of bifunctional molecules onto solid surfaces (Fig. 32) are examples that have developed recently. All the preceding approaches rely upon a knowledge of how, at molecular dimensions, molecules, solvents and interfaces interact and also on the subsequent design and synthesis of appropriate molecules to exploit this and to result in the desired final nanostructured materials that fulfil the needs of specific applications.

Figure 32

Schematic representation of self-assembled systems showing the molecular arrangement within lipid vesicles and Langmuir-Blodgett films (• represents a polar region of a molecule and — represents a non-polar, region of a molecule) and within two forms of self-assembled monolayers where R represents a variety of possible chemical groups. The alkanethiols typically self-assemble on gold and similar surfaces while the organofunction silanes self-assemble on oxide surfaces such as silica



The potential applications of self-assembled approaches to commerce is broad and includes applications within the electronics, medical, analytical and materials industries.

Langmuir-Blodgett (LB) films are typically formed by spreading a molecularly thin layer of molecules at a liquid-gas interface and where molecules of appropriate design, orientate due to preferential interactions with either, or both, the liquid or/and gas. The passing of a surface through the interface, if appropriate conditions exist, results in the transfer of a layer of single molecule thickness, to the surface. Repeated passage can result in the addition of further layers. Although this technique has been known for many decades, only recently have applications with commercial potential been developed. The design and synthesis of molecules that enable defined structuring during the LB deposition technique and that introduce application specific functions to the films are the challenges in this area. Application areas include, sensors, electronics, optics, optoelectronics, formation of resist materials for lithography and use in biological and medical areas [72].

While the LB technique is very broad in the range of films and surfaces that can be utilised, the spontaneous formation of thin film coatings on surfaces directly from solution is appealing. A range of systems have been investigated over the past decade and have become generally known of as self-assembled monolayers (SAMs). These consist typical of bifunctional molecules with one functionality designed to interact specifically with, and bind to, a particular surface, e.g. thiols with gold and silanes with oxide surfaces, resulting in the formation of monolayer coatings (Fig. 32). The remaining functionality is chosen to impart desired properties upon the resultant monolayer coating, and hence surface [79]. Applications are being developed in areas similar to LB-films, *i.e.* sensors, electronics, optics, optoelectronics, lithography resist materials and biomedical.

Liposome-based systems are important in the context of self-assembly and 'delivery' of material to a target site. Liposomes are vesicles typically comprised of a small number of spherical lipid bilayer shells enclosing a volume of unstructured material. Liposome size can be varied ranging upwards from diameters of less than 100 nanometres and with size dependent on the materials used and method of production [80]. The material enclosed within liposomes can be varied widely and for many applications the lipid shells of the liposomes serve to protect the contents. Liposomes are thus commonly used to protect and deliver material to a site of interest with applications or potential applications existing within the pharmaceutical industry for drug and gene delivery (see section 11.5), the food industry, e.g. liposomes containing flavour oils, agrochemical industry and for cosmetic applications; the latter example being a significant commercial application.

11.2 Supramolecular chemistry

Supramolecular chemistry, another recently established example of self-assembly depends upon the specific recognition and interaction of complementary molecules to direct the formation of structured materials and assemblies. Supramolecular chemistry offers a potentially broader range of applications as the self-assembly of three-dimensional structures is possible and is not reliant upon surfaces and interfaces to direct assembly. Although the breadth of supramolecular chemistry is great, a limited number of demonstrations that fall within the accepted concept of nanotechnology, have been reported. As an example, a series of interlocking and intertwined molecular structures have been formed exploiting the molecular recognition between p-electron rich and p-electron deficient molecular units. The novel molecular architectures formed have been shown to have switchable properties on the nanometre scale suggesting the future possibility of molecular-based electronic circuitry [81].

An area of supramolecular chemistry with more immediate application is that of dendritic polymers. Dendrimers are specific types of molecules grown by repetitive reactions such that each reaction step adds a small and defined number of molecular fragments to a growing molecular framework. The most common example is in the production of nanometre-sized spherical particles with each reaction step adding an additional "shell" to the particle. Particles with highly defined sizes and substructures can be achieved, *i.e.* size is determined by the number of reactions steps and hence number of "shells", and structure by varying the individual reaction steps. Applications based on these structure-controlled architectures can be found in many areas including life and materials science [82] and with their use in microscopy for size calibration at nanoscales an immediate application.

- Molecular-based manufacture: the Drexler approach

A possible high profile application of supramolecular chemistry and self-assembled systems is the Drexler concept of nanotechnology that relies upon the use of molecular assemblers to select and structure molecular building blocks together [83] (also see section 2.4). Although basic concepts within this area are being investigated, a commercial impact of this approach is unlikely to occur in the foreseeable future due to both technological [84] and economic [85] considerations. Drexlerian concepts are further discussed in the next section (section 11.4) within a biological context.

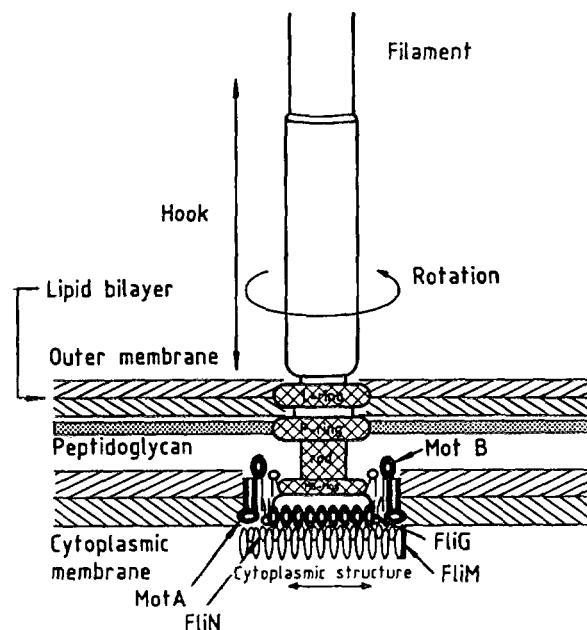
11.3 Molecular nanotechnology

- Biology as a molecular nanotechnology

Biology is often used both as an example and proof that molecular-based or "bottom-up" nanotechnologies are possible, including those proposed by Drexler and his supporters [84]. Biological examples can easily be found that demonstrate molecular-based information storage, information processing, molecular replication and nanoscale "mechanical" motors. The "pre-programmed" sequences of amino acids and nucleotides in protein and nucleic acid molecules represent routine information storage at a density far greater than currently achievable by modern technologies; *i.e.* approximately 6×10^{18} bytes.cm³ [86]. The transcription of a single DNA sequence into multiple RNA molecules and their subsequent translation into protein molecules represents molecular processing of information and molecular replication. Chemo-mechanical molecules such as kinesin, a motor protein, that directionally transports cell contents along the internal skeleton structure of cells, represent single molecule chemo-

mechanical motors able to individually generate forces of 5 pN [87]. Further examples can be found in animal muscle - the actin-myosin system - and in the bacterial flagellar [88] (Fig. 33).

Figure 33
Schematic representation of a bacterial flagellar motor that demonstrates a molecular-based rotary motor powered by electrochemical gradients present across the bacteria cell membrane. The motor resides in the bacterial cell wall with the flagellar (hook and filament) projecting out from the membrane and where rotation of the flagellar provides bacteria with propulsion. Various molecular components of the motor are labelled. Figure adapted from [89]



Whilst these obvious biological examples have been “designed” by millions of years of evolution to fulfil particular roles, it is hoped that a rational-design approach based-upon our developing understanding of biology and chemistry can improve upon nature and overcome the limitations of natural biological systems such as (i) poor performance at elevated temperatures, (ii) poor structural performance (for some applications) and (iii) a limited range of naturally occurring starting materials.

However, the reality is that while the evocative long-term goals of molecular nanotechnology are well publicised, doubts exist as to the real possibilities of Drexlerian approaches, *i.e.* nanomachines as scaled versions of macroscopic systems. They include arguments concerning thermodynamics and information processing [95]. The goal of molecular-based manufacture of common-place macroscopic items, such as domestic vacuum-cleaners, is, as yet, a distant dream! The immediate applications of molecular-based and traditional nanotechnologies relating to biology are found most commonly within the biomedical, pharmaceutical and analytical sectors. In addition, realistic long-term possibilities have been highlighted by a number of novel demonstrations and which include the demonstration of techniques for the observation and characterisation of individual molecules.

- Single molecule observation

Of the fundamental components required for Drexler-type nanotechnology, the ability to communicate information from the macroworld to the nanoworld and *vice versa* is crucial. This is required both for eventual use in nanotechnology applications and immediately, for characterisation and observation during the development of nanotechnologies. Thus methods for single molecule detection, observation and manipulation is an important area of current investigation. Typical examples include scanning probe microscopy (see section 7) where techniques such as atomic force microscopy (AFM) can image individual biomolecules [89] (Fig. 34), the use of modified AFM probes to measure the force interaction between individual biomolecule pairs [90], observation of the movement of single motor proteins [91], optical detection of single molecules [92] and the controlled manipulation of single macromolecules such as a particulate-labelled DNA molecule via optical tweezers [93] (Fig. 35).

Figure 34
 Schematic representation (approximately to scale) of the measurement of single molecule interactions. An AFM probe is modified with a biological molecule (streptavidin) and brought into contact with a surface modified with a complementary biological molecule (biotin) and a molecular complex is formed. The ability of the AFM to measure forces, in the range of nanonewtons and less, enables the force required to break the molecular interaction to be measured as the AFM probe is retracted from the surface. The small diameter of the AFM probe limits the number of molecules able to interact to only a few, or even a single molecule

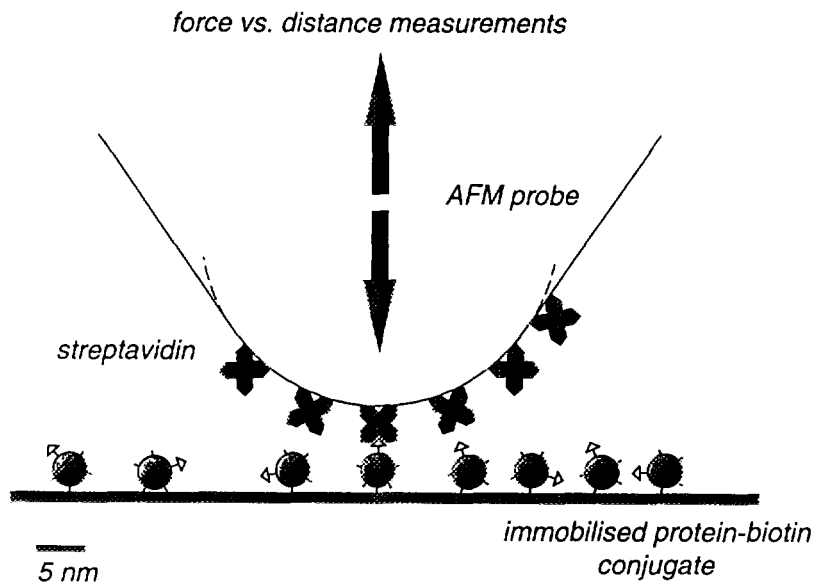
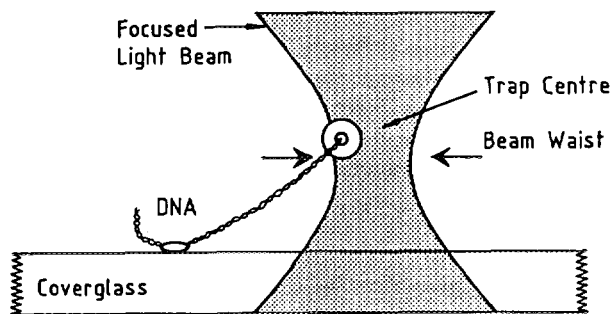


Figure 35
 Schematic describing the physical manipulation of a single molecule. In this example a single DNA molecule is attached to a surface and to a 0.5 μm polystyrene bead. The individual DNA molecule can be stretched by using an optical tweezer to apply force to the bead and hence the DNA. An optical tweezer comprises a focused, high intensity light beam in which gradients of radiation pressure enable the bead to be trapped. Figure adapted from [93]



11.4 Medical and pharmaceutical applications

The possibilities of nanotechnology within medicine is almost endless with futuristic suggestions including injectable nanomachines that would eliminate the requirement for invasive surgery. The immediate reality is less radical.

- Biocompatible textured surfaces

The effect of morphology and topography of surfaces upon cell-surface interactions is now appreciated and the deliberate nano- and micro-texturing of surfaces to produce biocompatible textured surfaces to optimise interactions is being pursued. Applications listed by Meyer and Biehl in 1995 [94] of biocompatible micro-textured surfaces include cell culture substrates, neural implants, prostheses, microsubstrates for bioartificial organs and the regeneration of biomaterials such as bone, tendon and nerves. Specific examples include (i) an observed increase in biosynthetic activity and mobility of bone cells on polymer films cast in micromachined moulds, *i.e.* a textured surface, when compared to a smooth control surface [95], (ii) the observation of *in vitro* regeneration of severed rat tendons only on textured surfaces and which suggests the possibility of *in vivo* use of textured "bandages" to accelerate healing [96] and (iii) the directed growth of nerve cells on micromachined structures [97] with suggestions of eventual artificial neural implants [98].

- Drug delivery: nanoparticles

The use of nanoparticles to direct drug delivery to treatment sites within patients is currently the most active application of nanotechnology to medicine. Nanoparticles have been shown to accumulate at various sites depending on the size and chemical nature of the nanoparticles and mode of administration, *e.g.* intravenous injection can result in accumulation in the liver, spleen and bone marrow. The incorporation of drugs into the particles therefore directs the drugs to the nanoparticle accumulation sites where the incorporated drugs are released, *e.g.* if comprised of biodegradable polymers, the polymer typically degrades at a predetermined rate releasing the drug. Many examples can be found [99] with fabrication being commonly achieved by (i) polymerisation from microemulsions of precursor molecules, (ii) formation from preformed polymers via microemulsions or precipitation or (iii) from naturally occurring polymers such as polysaccharides and proteins.

11.5 Analytical applications

The desire for miniaturised sensors is driven by the ever increasing requirement for information to quantify almost all aspects of human endeavour and is especially true in medical situations. The demand for real-time information for efficient control of processes dictates *in situ* measurements and therefore sensor-type approaches with small size a common requirement for practical use. Nanotechnology impacts upon this situation via (i) the microengineering of sensors (see section 9.4) and (ii) in the design and control required at molecular scales to transduce measurable parameters into easily detectable, typically electronic, signals. The latter item is fundamental to biological applications such as biosensors.

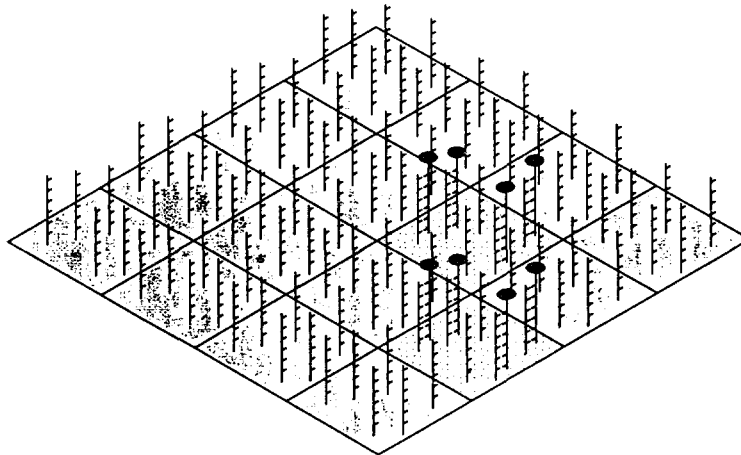
- Biosensors

Biosensors exploit the exquisite selectivity exhibited by biological systems that enables the recognition of one molecular species in the presence of complex mixtures of other, often closely related molecular species found commonly in sample matrices. The integration of biological systems, typically enzymes and antibodies, with suitable physical transducers enables the transduction of biorecognition events into measurable signals. The details of this last point requires design and control at the nanometre scale. For example, systems based upon enzymes that perform oxidation and reduction reactions - redox enzymes - such as glucose oxidase for the determination of blood glucose levels, require the efficient communication of electrons generated at the enzyme's active site during the recognition and catalytic events to an electrode for measurement. Various approaches are being developed to enable direct electronic communication between the active site and electrode [100] with "molecular wiring" a leading example [101]. The means of integration of the biological component of biosensors is central to their eventual performance and advances in surface science such as self-assembled systems (see section 11.1) are being actively investigated to ease production of devices, increase device stability, reduce interferences and maximise signal output.

- Biochips and high-density sensor arrays

Many examples exist where large numbers of individual biological analyses, *i.e.* biological assays, commonly 10^3 to 10^6 , need to be performed and include the screening of libraries of potential pharmaceutical compounds and various protocols for the screening and sequencing of genetic material. Such large numbers dictate the parallel processing of assays to enable completion in reasonable timescales and the common availability of only small sample quantity dictates small size. Thus microfabricated high-density arrays of biosensor-like sensor elements have been investigated where the size of individual elements approaches the nanotechnology domain. Such approaches are often termed "biochips" generally meaning an integration of biology with microchip type technologies (Fig. 36). For example, devices are being developed for genetic screening that contain two dimensional arrays with greater than 1×10^3 elements each comprising a differing DNA sequence and where each element is optically examined for specific interaction with complementary genetic material [102].

Figure 36
 An example of a biochip used for DNA analysis. A schematic of a 4 x 4 array of 16 different DNA molecules (represented by the vertical "ladder" structures) immobilised to a surface. 2 of the array points show interaction with other complementary labelled DNA molecules ("ladders" with •) which can be detected by the presence of the label, e.g. a fluorescent label. Scaling to high density arrays of greater than 10 000 array points has been demonstrated and requires the utilisation of microfabrication techniques



- Other microfabricated bioanalytical systems

The use of microfabricated structures within biological applications are mainly directed towards analytical measurements and often for those that require multiple steps and complex liquid handling. Thus microfabricated systems have been investigated to (i) determine the quality of sperm and to perform *in vitro* fertilisation [104], (ii) perform DNA analysis by a combination of polymerase chain reaction (PCR) and electrophoretic separation of the resultant amplified DNA [104] with applications including rapid detection of genetic mutations in medicine and DNA typing in forensics and (iii) the characterisation and separation of cells using dielectrophoresis and microfabricated electrode and microfluidic structures [105] with potential applications in the detection of food and water-borne pathogens and the separation of human cell types in medicine.

11.6 Other biological applications

- DNA-based computing

A novel technological application of nanoscale biology is the recent demonstration of DNA-based computing [106] where the tools of molecular biology were used to solve a mathematical problem. The problem was encoded in the sequences of DNA molecules, with the operations of the computation performed by the self-assembly of complementary DNA sequences and the answer output by characterising the resulting self-assembled DNA, *i.e.* hybridised DNA, with standard molecular biology tools. Although the calculation required days of laboratory work, the parallel nature of the "computation" and scale-up possibilities suggest further developments will be achieved [107].

- DNA construction sets

The ability of DNA molecules to hybridise to complementary molecules and modern molecular biology's ability to synthesise known DNA sequences has led to the demonstration of DNA as a molecular construction set [108]. Sequences have been designed that spontaneously self-assemble into geometrical structures such as cubes and octahedra. This approach demonstrates a molecular framework or scaffold that could be used to assemble other entities in a defined spatial arrangement at nanometer scales; is this a precursor of artificial nanomachines?

12. National and international activities and initiatives

12.1 National programmes in Europe

Studies undertaken in Germany and the UK have highlighted the emergence of nanotechnology as a major technological development with a likelihood of having a major economic impact in the medium to long term [109, 110]. Although nanotechnology was not explicitly identified in the UK study, a number of key nanotechnology areas were indicated. These included sensor technology, drug delivery systems and the requirement for new cost-effective nanofabrication processes.

The UK launched a National Initiative on Nanotechnology (NION) in 1986 to: a) represent and coordinate government, industrial and academic interests in nanotechnology, and b) to advise Government in all aspects of nanotechnology. A subsequent LINK Nanotechnology Programme (LNP) ran from 1988 -1994 and was closely associated with NION. The programme supported 27 projects, with a government funded budget of around US\$ 18 million, with matching funds from industry giving a total of approximately US\$ 40 million. The topics covered were:

- ultra precision machining and surface finishing
- nano positioning and control
- nanometrology
- nanostructures
- surface treatment and analysis
- ultra fine ceramic powder materials
- medicine
- biosciences
- micromechanics

The programme was successful in bringing together universities and industry to work collaboratively on the above mentioned topics. The NION programme concluded with a 'technology transfer' phase implemented primarily by Cranfield University (UK).

Since 1994 when research councils, within the UK, were re-organised the Engineering and Physical Sciences Research Council (EPSRC) has funded some projects within its own Nanotechnology Programme, and these will be completed in 1999. The Government contribution to this programme was US\$ 7.5 million, and major successes, to date, include [111]:

- ultra precision 'ductile' mode grinding of brittle materials, with extremely low levels of sub surface damage
- nanoparticles and in particular a 'world leading' zirconia powder for the fabrication of toughened zirconia ceramics etc
- direct experimental observation of diamond growth mechanisms
- the realisation that, in the field of biosciences, nanotechnology can be regarded as a 'toolbox' for cellular engineering.

The UK Government's Technology Foresight Panels have recently identified technologies which fall in the context of nanotechnology. For example two of the panels ('Information Technology, Electronics and Computing', and 'Defence and Aerospace') identified semiconductor manufacture, sensors, computer peripherals, display devices, mobile communications and micromechanical devices as being areas in which nanotechnology is considered as a fundamental 'enabling' technology. The Materials Panel micromaterials systems, fullerenes, bioelectronics and bioengineering in a similar way. Nanotechnology was also considered to be an important wealth creating technology by the Construction Panel. However the specific areas identified (e.g. conventional materials with unconventional properties) were considered to need long term programmes and to be at least 10 years from commercial exploitation.

Several German research institutions are running programmes in key areas of nanotechnology, even though the country does not have any specific nanotechnology programme, as in the UK, to raise industrial awareness of its technological potential. The German Government is funding nanotechnology related programmes in 14 Fraunhofer Institutes together with other universities and industrial research organisations. In addition a Government Microsystems Technology programme, with an annual budget of around US\$ 60 million is being funded until 1999.

A recent Delphi study carried out in Germany predicts that the time of realisation of technologies associated with nanometre size structures will be as indicated in Table 4. [112]

| Technology | Time of realisation |
|---|---------------------|
| Processing technology for supersmooth metal mirrors | 2002 |
| STM or AFM for analysing molecular structures | 2003 |
| Widespread use of atomic layer etching in semiconductors | 2004 |
| Embedding impurities and repairing silicon surfaces with STM | 2004 |
| Polymer processing for controlling nanostructures in 1 to 10nm size | 2007 |
| Mass production of new materials constructed by using ions and particle beams with controlled characteristics | 2010 |

Table 4 - Estimated time to realisation of nanometre size structures - German Delphi study [114]

France has recently formed the 'Club Nanotechnologie' which has brought together many industrialists and academics to identify areas which have national strategic importance. In addition one of the country's seven interdisciplinary government funded programmes, Ultimotech, is aimed at the development of advanced technologies and instrumentation for making nanoscale structures.

Other European countries undertaking Government funded programmes in nanotechnology areas include:

- (i) **Italy**, which has a government funded programme in nanotechnology aspects of biotechnology at the Elba Foundation.
- (ii) The **Netherlands** Foresight Committee reported recently that nanotechnology research was generally founded on single disciplines and suggested that in future the possibilities of stimulating interdisciplinary research in nanotechnology should be explored [113]
- (iii) **Russia** is reported to have several individual groups working on molecular/bio-electronics [114] and other aspects of nanotechnology.
- (iv) **Switzerland** has a national research programme which includes chemistry and physics of surfaces, as well as more general nanotechnology activities, e.g. scanning probe microscopy notably at the IBM Research Laboratories, Zurich.

12.2 USA

As with other major technologies, progress in nanotechnology is being pushed in the main by industry, and in particular the larger firms. However Government funding bodies supporting micro engineering, especially Micro-Electro-Mechanical Systems (MEMs) are:

- a) the Advanced Research Projects Agency (ARPA) (US\$ 40 m pa),
- b) the National Science Foundation (NSF) (US\$ 5 m pa), and
- c) the National Institute of Standards and Technology (NIST) (US\$ 5 m pa).

In addition the US Department of Defence Initiative on MEMs funded special 'sensitive' projects with US\$ 46 m in 1996 and this has risen to US\$ 63 m in 1997.

Thus the main activities in USA micro and nanotechnology programmes are:

- (i) MEMs which encompasses all microsystems technologies and devices
- (ii) Nanofabrication for manufacturing devices with nanometre scale structures, and,
- (iii) Aspects of the traditional science base (e.g. physical, chemical and biology) appropriate to 'nanoscience', including biotechnology.

A National Nanofabrication User Network (NNUN) has been established by in which a key aspect is aimed at bringing together key industrialists and academics to create an 'environment of self help'. A core group of successful projects in the areas of biology, chemistry, medicine and physics are presented in appropriate forums. The NNUN has a hub of 2 user facilities available, one on the east coast (Cornell University) and one on the west coast (Stanford University). In addition three additional sites offer expertise in the specific areas of a) wide band semiconductors (Howard University), b) novel materials (Pennsylvania State University, and c) semiconductor etching (University of California Santa Barbara).

Centres of excellence in nanotechnology and micro engineering exist at MIT, and the universities of Stanford (California), North Carolina (Charlotte and Raleigh) and California (Berkeley).

Dr Eric Drexler (see section 2.4) is President of the Foresight Institute (Palo Alto, California), whose mission and fundamental goal is the "betterment of the human condition, especially as it is related to molecular nanotechnology". The Institute runs regular molecular nanotechnology conferences and publishes 'Foresight Update' to promote an understanding of nanotechnology, and report on worldwide nanotechnology activities.

12.3 Japan

The importance placed, by the Japanese Government, on nanotechnology can be judged by the large amount of government funding it has and is continuing to provide. Funding is made available by 3 separate ministries: Ministry of Trade and Industry (MITI), Science and Technology Agency (STA), and the Ministry of Education.

In 1992, MITI started a major 10 year project 'Research and Development of Ultimate Manipulation of Atoms and Molecules' with total government funding of the order of US\$ 250 m. The research consortium currently includes 26 Japanese and 4 foreign companies and the broad range of applications expected to emerge from the programme range from computation e.g. higher density computer memory, to new materials, gene manipulation and new catalysts for environmental clean up.

A further large 10 year project funded by MITI to over US\$ 200 m is in the field of 'Micromachines'. Twenty eight companies and institutions, including 3 foreign groups are involved in the programme which is undertaking research in sensors, actuators, energy supply, system control and to bring into being a prototype micro factory, one in which very small machines are grouped within table top dimensions for the manufacture of very small (millimetric and sub millimetre) industrial products.

STA Projects have focused on various aspects of nanotechnology over recent years, including Nano-Mechanisms (1985-90), Solid Surfaces (1985-90), Molecular Dynamic Assembly (1986-91), and more recently the Atom Craft project (1989-94) which is investigating the behaviour of atoms and molecules on surfaces and techniques for precision deposition, based upon the application of STM technology. STA is also responsible for the Institute of Physical Chemical Research (RIKEN), which ran a 'Frontier Research Programme' in the areas of molecular electronics, bioelectronics, and quantum electronics. One objective of this programme is the development of an 'artificial brain'. The Ministry of Education supplies research funding for universities, and this includes several nanotechnology programmes.

The Applied Technology Development Department within Japan's New Energy and Industrial Technology Development Organisation (NEDO) has several current nanotechnology and microengineering related projects which are undertaking research and development aimed at:

- a) medical applications (micromachines capable of undertaking advanced coeliac diagnosis and treatment with minimal damage to the human body),
- b) electronics, information and communications (including quantum functional devices to realise ultra high speed and ultra functional information processing technologies), and
- c) biotechnology (molecular assemblies for functional protein systems and evolutionary molecular engineering).

12.4 Europe - collaborative

There are no formal nanotechnology programmes funded by the European Commission (EC) although there are several programme areas in which nanotechnology is highly relevant. For example, protein engineering, bio-sensors, etc. are included in the Biotechnology Action Programme. The current BRITE/EURAM programme in Materials Science have identified 17 projects which involve nanotechnology [114] as well as others in the area of 'microtechnology'. Nanotechnology is also a part of the Biomedical Technology Programme, e.g. artificial limbs, nerves, hearing, sight, etc. [115].

Within the 4th Framework Programme, running from 1994 to 1998, which supports Microsystems Technology, a microsystem is defined as 'an intelligent miniaturised system comprising sensing, processing and/or actuating functions, normally combining two or more electrical, mechanical or other properties on a single chip or multichip hybrid'. The EC has made available around US\$ 33 m for an ESPRIT Advanced Research Initiative in Microelectronics.

Other EC funding has been directed towards the establishment of micro and nanotechnology networks to support research and development to improve European competitiveness. Networks of Excellence which bring together relevant industrial and academic research groups, are aimed at bridging the cultural differences that exist between European Countries. Three relevant Networks of Excellence funded by the EC are:

- (i) PHANTOMS which is coordinating work on Mesoscopic Physics and Technology. The research covers the areas of nano-electronic components, nanotechnology fabrication, opto-electronics and novel architectures for switching. There are 18 partner countries in this programme.
- (ii) NEOME (Network Excellence on Organic Materials for Electronics) has run since 1992 and is coordinating work in the area of organic materials for electronics as the basis for nanostructures, optical devices, electro-optical switches, sensors and for potential applications in information storage and information processes. Ten countries are currently directly involved in this network.
- (iii) NEXUS (European Network of Excellence in Multi-functional Microsystems) was established in 1992. Its brief includes:
 - a) coordinating work in microsystems technology (MST),
 - b) the strategic assessment of international MST developments, and
 - c) the formation of links to small and medium sized enterprises.

Other Networks include:

- (i) EURORACTICE which has been established to stimulate the exploitation of state-of-the-art microelectronics technologies by European industry to improve industrial competitiveness.
- (ii) NANO to coordinate research and development in nanostructured materials.
- (iii) INPEC, an international network of protein centres, which is concerned with information exchange between countries, related to changing and studying the properties of molecules.

A proposal is being submitted to the EC during 1997 for the establishment of the European Society for Precision Engineering and Nanotechnology (EuSPEN). The Society aims to strengthen industrial and research potential in precision engineering and nanotechnology by establishing a society, organising events, initiating transnational research projects, building a comprehensive database and helping develop education and training programmes in precision engineering and nanotechnology. The EuSPEN Foundation Committee comprises one industrialist and one academic from 6 countries, namely Denmark, France, Germany, Italy, The Netherlands and the UK.

12.5 Rest of the World

Two other countries investing in nanotechnology related areas are **Taiwan** and **South Korea**. Taiwan has had a strong IC industry since the mid 1980s and research is being funded, through the National Nano Device Laboratories, in the area of ultra-large scale integrated circuits. The research started with 250nm feature sizes but is now concerned with lower feature sizes of 180nm and intends to progress to smaller scales. Taiwan is also strong in the area of information technology and is now the main producer of 'merchant motherboards' for personal computers.

South Korea has strong government funded programmes in microelectronic and precision engineering which are starting to bear fruit. For example, companies such as Samsung, are currently producing advanced flat panel displays dependent on nanotechnology production processes.

13. The effect of nanotechnology on world development

13.1 World markets

There are huge potential markets for nanotechnology related products. For example in areas such as semi-conductors, computer peripherals, bio-medical engineering and pharmaceuticals the potential markets are US\$ multi-billions in each case [115]. Because of the wide diversity of products which can be considered as being in the nanotechnology domain it is difficult to accurately predict future potential markets. However in 1994 the German Ministry for Research and Technology produced a detailed estimate of the market potential for what they considered to be specific nanotechnology products - see Table 5 [116]. This shows the largest area to be for devices using lateral structures, e.g. microelectronic components, precision nozzles and filters, micro sensors and some optical components. Table 5 also indicates the ascendancy of ultra precision engineering.

Table 5 Global market potential US\$ bn

| <u>TECHNOLOGY</u> | 1991 | 1995 | 2000 |
|-----------------------------|------|------|------|
| Thin films | 9.2 | 13 | 18 |
| Lateral structures | 70 | 139 | 223 |
| Ultra-precision engineering | 5.2 | 9.7 | 18.2 |
| Nanocrystals | 0.05 | 0.08 | 0.11 |
| Nanometrology | 0.62 | 0.83 | 1.1 |

Source: VDI Germany [116]

It is easier to assess individual product potential. For example, one German company has patented a micro-fabricated CFC-free nebuliser, and estimates that it has a global market potential of US\$ 6.4 bn p.a. Another product - the air bag sensor, which currently costs around US\$ 160, is set to be replaced by mass produced sensors costing around US\$ 5. The global market potential for MST (microsystems technology) products has been quoted, from several sources, as being set to reach US\$ 25 bn by the year 2002 [115].

13.2 Research and development

The high cost of developing marketable products from basic research findings dictates that it will involve mainly larger firms. However small and medium sized firms are often best suited to serve the niche market areas, although this might be limited by the large capital investment required. To counteract this the successful USA 'Small Business Innovative Research' programme is making available high risk venture capital for high technology (including nanotechnology) start ups. However, there is little evidence of similar programmes being initiated in Europe or elsewhere. The international trend in funding research and development appears to be directed more toward short term competitive application oriented research, which in turn could reduce the funds available for more basic longer term research. Examples of research and development which are needed to extend microengineering and nanotechnology include:

- (i) **Materials and Processes:** the integration of these areas is essential, as methods of forming parts will require additional techniques which allow the efficient removal or accretion of material down to the atomic scale.
- (ii) **Mechanisms and Structures:** the microscopic tools and motions require the research, design and manufacture of fine mechanisms which can accurately position and manipulate objects of appropriate scale. Critical analysis of microstructures e.g. stability, friction, wear, chemical, electrical and magnetic properties is needed.
- (iii) **Nanometrology:** the successful application of nanotechnology depends upon the ability to accurately measure minute displacements. Examples of specific research includes longer range X-ray interferometry, providing picometre resolution and 'absolute' interferometry based on multi-wave length solid state lasers and sensors.

13.3 Opportunities for exploitation

Drexler predicts [117] that developments in nanotechnology will result in the production of nano robots that will be able to reproduce themselves and clean up the environment, repair damaged living tissue, etc. However this is thought, by many experts, to be very futuristic and that more realistic opportunities for exploitation lie in

advancing the capabilities of products such as microprocessors, disc drives, display devices and printers, etc. Further areas for exploitation include minimally invasive surgery tools, sensors for environmental and automotive application, and medical diagnostic and analysis tools. Many observers see nanotechnology as being a key technology to ensure competitiveness, into the 21st century, for a wide range of products. Likely future application of 'nanotechnology' products are shown in Table 6.

Table 6 Likely future applications of nanotechnology

| Product | Comment |
|--|--|
| Micro/macro optics (including fibre optic devices) | Improved telecommunications |
| Prosthetic devices | Lighter, cheaper, longer life, - increased functions |
| Catheter based medical diagnostics | Improved imaging systems and analysis |
| Micro surgical devices | Therapy systems and micro surgery non-trauma surgery; faster wound healing; efficient drug targeting in the body |
| Medical diagnostics, using colloidal particles | Blood sampling in ante-natal care and cancer screening |
| Photonic products | Miniaturisation |
| Micro filters | Many environmental applications |
| Micro optical systems | High speed beam positions. Scanning shutters |
| Compliant and active probe micro-assays | Applicable to micro electronic manufacture, biological and neural investigation |
| Micro switches | Faster operation, lower power consumption |
| Deformable structures | Valves, micropositioning analogues of muscle |
| Semi-solid fuel injectors | Cost and control low weight |
| Mass storage | Even denser packing, higher speed devices |
| Inks incorporating colloidal nanoparticles | Ink jet printers |
| Automotive components (pistons, camshafts, crankshafts, etc) | Improved wear, life and reduced environmental pollution |
| Advanced optics (including aspherics) and opto electronics | Improved performance, smaller and cheaper |
| Nanofabricated surface coatings | Coatings for keeping windows clean; protective 'skins' for many applications |
| Thin film sensors and actuators | Environmental monitoring and nano-manipulation |
| High resolution display systems | Using quantum well technology, or based on molecular structures |

13.4 Developing countries

The high cost of establishing nanotechnology research and development programmes will undoubtedly severely limit activities in developing countries, for the foreseeable future. However the technology and products arising from programmes in developed nations will have many important applications in the developing countries, as well as elsewhere. Thus developing countries may wish to have well qualified scientists and engineers further educated and trained in nanotechnology so that they can advise their governments on priorities in applications - and later, developments in their own countries (see 13.5 below).

Appropriate applications include controlling and cleaning the environment and improving healthcare. Cheap sensitive and accurate sensors are emerging for monitoring the atmosphere and water quality, and opportunities exist to apply biological 'machines' to clean up the environment by the extraction of pollutants. In the area of healthcare nanotechnology is enabling minimal invasive surgery and effective drug delivery systems. Research programmes are targeting intravenously administered drugs to different organs, such as the lung, liver, spleen, sites of inflammation and to tumours [118]. These drugs, coated with antibodies, resist attack by the body's own defence cells, and can lock on to the appropriate target such as a tumour, thus avoiding the release of toxic drugs on healthy tissue. New developments, taking account of economic and social factors are placing an emphasis on prevention rather than corrective medicine and surgery.

13.5 Needs for education and training

Nanotechnology demands multi-disciplinary inputs from scientists and engineers and recognition is needed with regard to the important role of education, in maximising the future wealth creation possibilities. The necessary advanced skills in this heavily science based area can only realistically be developed through intensive, multi-disciplinary postgraduate courses. The education and training of young physicists, mathematicians, chemists, materials scientists and engineers is essential in order to ensure a flow of high calibre postgraduates into industry. Never before have these been at such a premium and those nations and the postgraduate courses which harness these skills will surely reap large benefits.

Nanotechnology Centres of Excellence within UK universities are given in Table 7 below [115].

| Organisation | Principal Areas | Organisation | Principal Areas |
|------------------------|---|------------------------|--|
| Birmingham University | nanoparticles; surfaces nanostructured materials | Imperial College | MST/microsystems |
| Cambridge University | microelectronics; energy beam processes | Nottingham University | semiconductors, bio- physics, pharmaceuticals |
| Cranfield University | precision engineering; engineering nanotechnology; nanostructured materials biosensors and nanobiotechnology | Southampton University | silicon fabrication |
| Glasgow University | nanoelectronics nanobiotechnology | Warwick University | precision engineering; nanometrology |
| Heriot Watt University | microsurgery | | |

14. Conclusions

The authors of this paper have set out to explain what nanotechnology is, to describe the current state of the art, emphasise its essential multi-disciplinary nature and convey the enormous impact it will progressively make on product and process research and thence on to manufacturing technologies, industry and the economies of countries throughout the world. Undoubtedly, nanotechnology is a major, new technological force destined to have major socio-economic effects. It promises many benefits in standards of living and quality of life.

The authors hope that they have succeeded in making the topic interesting and informative to a wide readership from those with curiosity and a limited scientific base to scientists and engineers some of whom may be stimulated to engage in further study.

Owing to the very wide range of disciplines and thus the breadth of understanding needed, the necessary skills in this heavily science-based, technological thrust can be developed, only through intensive, multi disciplinary post-graduate courses, closely related to social and industrial needs.

The evolution towards the 'Nanometric Age' is gathering pace. It has the potential to provide revolutionary benefits.

Editor's note: The authors have indicated they would be willing to answer any specific questions readers may have on this article. Please contact them directly:

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GLOSSARY

1. **Accretion** - growth or increase by the additive assembly of atoms or molecules.
2. **ASPE** - American Society for Precision Engineering
3. **Atomic Force Microscope (AFM)** - a SPM which uses the Van der Waals attraction between a tip and the substrate as the probing signal.
4. **Biological assay** - a process to measure or assay a particular biological function.
5. **Capacitance gauge** - non contact measuring device that determines the distance (gap) between a probe and the target surface, by measuring changes in capacitance.
6. **CBN** - cubic boron nitride, the second hardest man-made material after diamond. Unlike diamond, it will resist chemical attack by iron during machining.
7. **Chemo-mechanical molecules** - molecules that convert chemical energy in mechanical energy.
8. **Chip** - a semiconductor device with 2 or more active devices (e.g. transistors) defined on it and connected together.
9. **CIRP** - College International de Recherche de Production
10. **CNC** - computer numerically controlled.
11. **'Ductile regime' grinding** - see 'shear mode' grinding.
12. **Eddy current probe** - non contact measuring device that determines the distance (gap) between the probe and the target surface, by measuring changes magnetic fields generated by eddy currents in the surface.
13. **Electro-optic effect** - the effect by which a transparent material's refractive indices are changed by the application of an electric field.
14. **Electron beam lithography** - the use of a focused electron beam to define a pattern in a polymer resist on the surface on a wafer.
15. **Electrostatic Force Microscopy (SFM)** - a SPM which uses electrostatic forces between the tip and the substrate as the probing signal.
16. **Electrostatic Actuator** - an actuator which exploits electrostatic attraction or repulsion between two charged components.
17. **Electrostrictive Material** - a material which undergoes mechanical strain when subjected to an electric field where the strain is proportional to the square of the applied field.
18. **Enzymes** - proteins that catalyse various chemical and biochemical reactions.
19. **Epitaxy** - The phenomenon whereby one crystal will grow upon another such that their lattices are in registration.
20. **Evanescent Wave** - a wave whose amplitude decays exponentially with distance into a medium.
21. **Excimer** - A molecule (such as ArF⁺) which exists only in an excited state in, for example, an electrical discharge and which decays by breaking up to emit a uv photon. Used in excimer lasers for generating uv light.
22. **Ferroelectric Material** - a polar dielectric in which the direction of the polarisation can be switched between 2 or more directions which are related to each other through the symmetry of a higher temperature non-polar (paraelectric) phase.
23. **Frequency Tracking Fabry Perot Etalon (FPE)** - device to measure and compensate for variations in the refractive index of air i.e. temperature, pressure and humidity.
24. **Gbit (Gigabit)** - a billion (10⁹) bits of information.
25. **Interferometry** - technique of dividing a beam of light into a number of beams and re-uniting them to produce interference fringes. Uses include the accurate determination of wavelengths of light, displacement and velocity.
26. **Joule** - The SI unit of energy, being equal to the work done by moving a force of 1 newton through a distance of 1 metre.
27. **JSPE** - Japan Society for Precision Engineering
28. **KeV - Kiloelectron volt** - the energy attained by an electron when it accelerated by a potential differences of 1000 volts.
29. **Laser interferometer** - very accurate displacement measuring system; many current systems use the wavelength of helium-neon laser as a measuring standard. (See also OHI)
30. **Lead zirconate titanate (PZT)** - a solid solution between the two compounds lead zirconate (PbZrO₃) and lead titanate (PbTiO₃) which possesses a crystal structure isomorphous with the mineral perovskite.
31. **LIGA (Lithographic, Galvanforming und Abforming)** - the use of X-ray lithography to expose a pattern from a mask in a thick polymer photoresist followed by a replication in nickel using electroplating.
32. **Linear variable displacement transformer (LVDT)** - displacement measuring contact probe which employs electromagnetic induction to sense linear motion; can have sub-nanometre resolution.
33. **Magnetic Force Microscopy (MFM)** - a SPM which uses a magnetic tip and the magnetic forces between the tip and the substrate as the probing signal.
34. **Mbit (Megabit)** - a million (10⁶) bits of information.
35. **Mercruy "i" line** - uv light with a wavelength of 365 nm.
36. **Mercruy "g" line** - uv light with a wavelength of 436 nm.
37. **Metrology frame** - a stationary reference frame, uncoupled from the machine structure, by which the tool or probe position of a machine are measured with respect to the workpiece.
38. **Micro-electro-mechanical systems (MEMS)** - see "microsystem".

39. **Micromachining** - techniques for making three dimensional structures on the micrometer scale on a microsystem.
40. **Microsystem** - a device consisting of sensors and actuators integrated together with a semiconductor signal processing capability in a single micro-assembled package.
41. **Microsystems engineering** - see MST and MEMS.
42. **Microsystems Technologies (MST)** - the technologies for making microsystems.
43. **mK** - milliKelvin
44. **Molecular wiring** - pathways to enable the transmission of electrons.
45. **Motor protein** - enzymes that convert chemical energy into directed movement of molecules.
46. **Nanophase Material** - see Nanostructured Material.
47. **Nanostructured Material** - a material which there is some aspect of the structure deliberately defined to be in the nanometre range and which has a useful effect upon the material's properties.
48. **Optical grating scale** - linear displacement measurement device which operates on the principle of counting and sub-dividing scale lines with the use of a light source and photodiodes.
49. **Optical Heterodyne Interferometry (OHI)** - two frequency laser, used in most laser interferometers
50. **Pa** - Pascal: The SI standard unit of pressure, being equal to 1 newton per square metre.
51. **Photolithography** - the use of light to transfer the pattern from a mask to a polymer resist on the surface of a wafer.
52. **Photon** - The quantised unit of energy of an electromagnetic wave.
53. **Piezoelectric material** - a material whose crystal structure lacks a centre-of-symmetry and which will generate electrical charges when stressed. Conversely, it will undergo mechanical strain when subjected to an electric field in which the strain is proportional to the applied field.
54. **Polysaccharides** - naturally occurring polymers of various sugar molecules.
55. **Quantum wire** - a nanostructured conductor in which the electrons are confined to move in one dimension.
56. **Quantum dot** - a nanostructured conductor in which the electrons are confined in all three dimensions.
57. **Quantum confinement** - the use of nanostructuring in a material to confine the conduction electrons and thus to generate new energy bands.
58. **Radio-frequency** - an electromagnetic signal with a frequency between 100 KHz and 1 GHz.
59. **Resolution** - The smallest distance between two objects that can be measured using a measurement system (e.g. a microscope or interferometer).
60. **RNA** - a naturally occurring biological polymer, similar to DNA, that encodes information in the sequence of its constituent monomers.
61. **Scanning Probe Microscope (SPM)** - a microscope which uses a probe scanned in two dimensions across the surface of an object to be studied and which forms an image through an interaction between the probe and the surface.
62. **Scanning Thermal Microscopy (SThM)** - a SPM which uses a tip (such as a piece of Wollaston - PtRh alloy - wire) capable of generating a thermal signal and detecting a temperature change.
63. **Scanning Near Field Optical Microscopy (SNOM)** - a SPM which uses an evanescent optical wave emerging from a drawn optical fibre as the probe.
64. **Scanning electron microscopy (SEM)** - a microscope which uses a focused spot of electrons scanned across the object under study. The image is formed by collecting electrons or X-rays scattered from, or produced by, the object under study.
65. **Scanning Noise Microscopy** - A STM in which the tunnelling current is explored without an electrical bias field by means of the broadband r m s noise.
66. **Scanning Tunnelling Potentiometer** - A STM in which an a c signal is used to generate a constant top-to-sample spacing.
67. **Scanning Tunnelling Microscope (STM)** - A SPM in which the probe is a sharp conducting tip and the interaction is a tunnelling current generated as a consequence of an electric bias.
68. **Semiconductor** - a material in which the valence band is filled and conduction band empty. Conduction is dominated by electrons in the conduction band or holes in the valence band due to the presence of impurities acting as either electron donors or acceptors.
69. **Servo** - A device or system for controlling one physical variable (e.g. position) by means of measuring that variable, comparing it with a separate standard, taking the difference between the two measurements and using the difference signal in a feedback system (usually, but not always electronic) to make the difference as small as possible (ideally zero).
70. **Shape memory alloy (SMA)** - an alloy which returns to a pre-determined shape when heated through a martensitic phase transition temperature.
71. **'Shear mode' grinding** - a grinding technique for machining hard and brittle materials with a 'continuous' type chip and with zero or minimum sub-surface damage.
72. **Synchrotron radiation** - the radiation emitted by high energy electrons confined to circulate in a ring as they are accelerated around a curve.
73. **Transcription** - the biological processes by which the information present within DNA (genes) is copied or transcribed into RNA.
74. **Translation** - the biological processes by which the information present within RNA is converted or translated into a specific protein molecule.

75. **Transmission Electron Microscope (TEM)** - a microscope which uses electrons transmitted through the specimen rather than light. The electrons are focused (in a similar manner to light in an optical microscope) using electromagnetic lenses.
76. **ULSI** - Ultra large scale integration
77. **Ultraviolet (uv)** - light with a wavelength between approximately 450 and 100 nm.
78. **Vapour deposition** - a surface coating process in which the workpiece surface is subjected to chemical reactions by gases that contain chemical compounds of the material to be deposited.
79. **VLSI** - Very large scale integration
80. **Wavelength** - The length over which any periodic object or disturbance (e.g. a wave) repeats itself.
81. **X-ray lithography** - the use of X-rays to transfer a pattern from a mask into a polymer resist on the surface of a wafer.

2. NANOTECHNOLOGY OVERVIEW

Nanotechnology's many disciplines

In the development of nanotechnology, the interface between biology and technology will be hugely important. The acquisition of the tools of nanotechnology will be part of the next phase of evolution of biotechnology; without them many aspects of biological function will remain unexplored.

It is becoming increasingly obvious that systems that combine biological and chemical molecules on the one hand, and physical devices and electrodes on the other, have a huge potential in many applications. However, the fragility of the biological systems that are readily manipulatable (cells, tissues) has meant that biotechnology's efforts to interface with physical systems have, thus far, been crude. Researchers can entrap cells in polymeric matrices or juxtapose slices of tissue and sensor surfaces. Using micromanipulative techniques, biotechnologists can even move individual cells and organelles around. At the molecular level, however, they have had to be content with manipulating populations of molecules.

Nanotechnology is changing that. Physicists and chemists have developed tools that operate at dimensions similar to those of biological macromolecular structures. Much of the progress in the biological application of nanotechnology will depend on using them.

Physical methods for observing and manipulating single molecules lie at the heart of all nanotechnology. Scanning tunnelling microscopy (STM) and atomic force microscopy (AFM) were both first applied to observe nanostructures of polymers in chemistry and to resolve structures around the angstrom level. In the last few years, there have also been many papers dealing with the observation of biological molecules and macromolecules (DNA, proteins) and, in the case of AFM, even of live cells.

Nanotechnology demands the organization of atoms and molecules in two- or three-dimensional space. Once an organizing framework has been established, molecules or atoms may subsequently be allowed to self-assemble around or within it. As the microelectronics industry has demonstrated, physical techniques can be used to provide the framework for this organization through the fabrication and machining of silicon materials. Arranging atoms through the use of nanomanipulative technologies is, in essence, just an extension of the manipulation. This approach to the production of a framework is closely linked to the ability to observe both the manipulations and the resulting structures, and there is clearly a reciprocity between the two.

Nanotechnology in basic biology

Progress in nanotechnology has meant that there is now almost no clear border between (bio)chemistry and physics. Biotechnologists have depended, to a great extent, both in research and in the development of products, on their ability to constrain macromolecules. One only has to think of affinity chromatography, ELISA, and solid-phase synthesis or sequencing to understand how useful it is to be able to restrict the position of just one macromolecule in space. Now, through nanotechnology, biotechnologists can start to control two (or possibly more) interacting entities.

We can bring together two molecules, not by letting them wander randomly around in the same general three-dimensional space (as we do when mixing reagents), but by physically increasing their proximity.

This will bring a revolution in biochemistry. No one currently works with one molecule or one interaction (except those undertaking virtual biochemistry *in silico*). But through AFM, for instance, it would be possible to arrange and study directly the contact between a ligand and a protein structure. Traditional approaches in studying protein-ligand binding may become obsolete.

Another way of providing a direct link between interacting macromolecules is through the exploitation of the mechanical properties of some biological molecules. Consider, for instance, the work done by researchers at Stanford University School of Medicine (Stanford, CA) and King's College London (London, UK). They have been able to measure the nanometer displacements and piconewton forces involved when a single actin filament, stretched between two latex beads held in optical traps, interacts with a single (or very few) immobilized myosin molecule. Beyond what it tells us about the actin-myosin interaction, this study was important because it demonstrated coupling between a mechanical process and a biochemically well-defined process.

Nanotechnology can also provide considerable insights into catalysis. In 1993, a group at the University of Liverpool (Liverpool, UK) used STM to study the oxidation of carbon monoxide at the surface of an oxygen-covered rhodium surface (the process is related to the catalytic removal of carbon monoxide from exhaust fumes). They showed that the catalysis at high temperatures was sensitive to the nature of the structure and that the reaction tends to proceed in a particular direction with respect to the geometry of the catalyst. Thus, the nanotechnological perspective enables one to progress beyond mass transfer and diffusion in catalysis.

Nanotechnology can provide biotechnology with a direct way of following a biocatalytic reaction by studying a very limited number of molecules. It should be possible not only to describe the activity globally and numerically, but also to obtain information about the mechanistic aspects of the catalysis.

At some point in the near future, as a result of nanotechnological approaches, biochemistry will have to reconcile its descriptions of the behaviour of single molecules with the descriptions we already have (affinity, for example) for interactions of populations. There will be a period of transition before these ways of thinking about the biological world come together, just as there was in reconciling quantum and classical physics.

Only the imagination limits the potential applications of bionanotechnology. And, at present, except for biosensors, it is only in the imagination that most of them perform. Rather than speculate upon nonexistent specifics, it is preferable to stress the significance of something that has already been demonstrated. The understanding of the actin-myosin system could lead to the use of these molecules as the components of nanoengines. These may have

potential medical applications as components of artificial muscles. However, of more fundamental importance is the fact that the actin-myosin engine provides direct coupling between the biochemical energy (ATP) and mechanical energy of movement. There are few devices in any field in which such direct coupling occurs; heat or electricity usually make energetic links between (bio)chemical energy and motion. For more information contact: The Director, Department of Enzymatic and Cellular Engineering, Université de Compiègne, BP649, 60206 Compiègne Cedex, France (e-mail: chantal.david@mx.univ.compiègne.fr). (Source: *Biotechnology*, Vol. 13, May 1995)

Protein crystals and bioelectronic arrays

Nanotechnology requires organizing principles, framework to provide K. Eric Drexler's (one of the founders of nanotechnology) "positional control" over atomic and molecular arrangements. Most current nanotechnology uses a "top-down approach", refining modern lithographic and machining techniques to make finer divisions. The alternative "bottom-up approach" is exemplified by self-assembled molecules like those designed by Nadrian Seeman at New York University (New York), who has trained DNA to form a range of geometric shapes. DNA was chosen for a number of reasons: the predictability of the intermolecular reactions, the ability to synthesize with ease desired sequences, and perhaps most importantly, the ability to use "off-the shelf" reagents to make unusual bases.

One of the designs synthesized is a polyhedron shaped like a structure seen in zeolite silicates. The truncated octahedron was synthesized using a solid support-based methodology. The squares were added in intermolecular addition reactions, and the hexagons were formed by seven successive intramolecular closure reactions. The synthesis involves a variety of molecular biology techniques, including hybridization, ligation and the use of restriction enzymes.

The polyhedron has a large enough internal volume to act as a host for macromolecular guests. Molecules attached to the outside of the "cage" could be used to direct the complex to a target location. Another longer-term option would be to construct artificial multienzymes or multi-protein complexes by attaching fusion proteins to different edges of the polyhedron.

If the polyhedron can be catenated, forming a sort of molecular chain mail, one relatively near-term (next three to five years) application would be in facilitating the analysis of proteins that are hard to crystallize, including membrane proteins. The idea would be to produce fusions of the required protein with site-specific DNA-binding proteins. The DNA-binding elements would orientate the target protein within the lattice.

The immediate use of Seeman's DNA polyhedron structure is hampered by its lack of rigidity. As a result, the angles in the structure are sometimes indeterminate and the DNA tends to form inappropriate cyclic trimers or pentamers that disrupt growth of the lattice. Having tried to overcome this by protecting the DNA sticky ends, and having found that this approach was unfeasible in a large arrays, Seeman is now looking for more rigid molecules to use as components before he attempts to scale up the synthesis. (Source: *Biotechnology*, Vol. 13, May 1995)

Nanotechnology helping to shape the future

One technology sure to help shape the future is nanotechnology, engineering at dimensions one-ten-

thousandth the diameter of a human hair. At its ultimate expression—molecular engineering—we enter a realm one-billionth that size, one of atoms and molecules arranged in precise fashion to fabricate structures on the ultra-microscopic scale. It is Lego at the atomic level, building from the bottom up, the way nature does, for perfect structure and function. These tiny structures can then be assembled into larger structures.

The possibilities are staggering. For example, if you could grow computer processing power atom by atom, you could exceed all the computational power ever created in the world to date in a desktop-sized package, according to Eric Drexler of the Institute for Molecular Manufacturing, Palo Alto, CA. Predicted applications of molecular engineering are endless because it can be applied to automobiles and jet planes as easily as microscopic medical devices.

Tiny molecularly engineered devices of a particular shape could be injected into the body or circulatory system to find their way to specifically shaped docking sites on particular individual cells. Once docked, they could inject microdoses of toxin into cancer cells, deliver therapeutic doses to malfunctioning cells, even perform microsurgery on severed nerves—true healing at the molecular level. Carbon atoms arranged in precise diamond-like fashion create a material that is 100 times the strength-to-weight ratio of steel. Thus, automobiles or anything else made of today's steel could be 10 times stronger and 10 times lighter at the same time.

Often materials created in nanophase—that is, from clusters of thousands of atoms rather than the millions of atoms in traditional material grains—exhibit unusual properties. This leads to predictions of transparent metals, flexible ceramics, polymers with electrical conductivity exceeding copper, materials with a controllable index of refraction, and plastics with the barrier properties of glass, for example.

While some tiny machines and components are fabricated from silicon at the nanoscale today, the methods for actually manipulating atoms—scanning tunnelling microscope and atomic-force microscope—are clumsy. Fabrication techniques will likely evolve, but the greater challenge is to impart intelligence to these atoms and molecules so they will *self-assemble* into the desired components and machines.

Perhaps the key objective is to understand the concept of self-organization—nature's way of building life from the bottom up, one molecule, one cell at a time, yet without a central control mechanism. In nature, from the start every molecule responds to whatever it feels in its neighbourhood, somehow ultimately forming incredibly complex organizations such as trees, complete ecosystems, and human beings.

Artificial-life researchers hope to unravel the secret of self-organization so these principles can be applied not only to self-assembly in molecular engineering, but also to self-organization of large-scale man-made distributed systems such as transportation, communication, and power distribution, and socio-technical systems such as traffic.

To date, artificial-life researchers have been able to create man-made artificial cell membranes that, given building-block material, will grow. The membranes will divide in a manner similar to a cell, creating self-replicating spheres. Natural and man-made elements introduced into the spheres also have been observed to self-assemble into elements seen in living cells. (Source: *Industry Week*, 21 August 1995)

Microscope

When people want to understand how something very small works, they often put it under a microscope. Physicists at Hitachi's Advanced Research Laboratory at Hatoyama, outside Tokyo, Japan have taken this approach a step further. To investigate how a microscope works, they have shrunk it to a microscopic size and placed it under another microscope.

The device being studied is a scanning tunnelling microscope (STM). Unlike conventional microscopes, which form an image from light or electrons passed through or bounced off the object under investigation, an STM builds up its image by feeling its way across a surface. It works by placing a sharp metal tip less than a millionth of a millimetre from the surface in question and passing an electric current through it to the surface. As the tip scans back and forth, tiny variations in the current can be amplified to reveal details of the surface as small as single atoms.

Despite much research, however, some aspects of the way STMs work remain mysterious. Sometimes, for example, the image suddenly becomes blurred. Frustrated microscopists usually assume that something—perhaps a single molecule—has attached itself to the tip, to the detriment of the microscope's resolution.

This is not merely an academic issue. STMs are being touted as tools for building future generations of ultra-miniaturized electronic circuits one atom at a time. They

would do this by picking up and depositing atoms with the tip—a deliberate version of the accidents that appear to be fuzzing up the image.

Normal STMs are too big to put under an electron microscope, so a small one was built. With help from Hitachi's chip makers, they managed to carve a diminutive STM, including all the mechanics for scanning the tip, out of a chip only 2.5 mm wide. The thickness of the tip and of the sample it scans is less than a thousandth of a millimetre.

This will fit neatly into the specimen holder of a commercial transmission electron microscope (TEM). When the researchers placed their tiny STM in a TEM, they were able to look sideways-on at the gap between the tip of the STM and the sample, resolving single atomic layers on either side of the gap. As they did so, they saw the tip jump away from the surface by a small amount every five seconds or so, a telltale sign that individual molecules were attaching themselves to the tip.

The molecules, though, came not from the surface being felt, but from the residual gas in the vacuum chamber in which the mini-STM was sitting. The researchers hope to exercise better control over which atoms or molecules attach themselves to the tip, and when they do so. This should allow them to catch the perpetrators of fuzzy STM images in the act—and possibly help pave the way for atomic-scale electronics. (Source: *The Economist*, 23 March 1996)

3. TRENDS IN RESEARCH AND DEVELOPMENT

Scanning probe microscopy

In virtually every field of scientific endeavour, there is a growing need to investigate materials on the nanometre scale. From semiconductors to biomaterials, features of critical importance to product performance or the understanding of physical and chemical processes are shrinking in size. The ability to measure these features, define their properties and characteristics, and manipulate a material's structure at the molecular and even atomic level, is becoming a necessity, not only for scientific studies, but also for manufacturing and analytical applications. The scanning probe microscope (SPM) is a relatively new tool that is enabling scientists and engineers to advance the state of the art in nanotechnology.

In the little more than a decade since the invention of the scanning tunnelling microscope (STM)—the technique has expanded to include a family of related probe microscopes that can be used to measure a host of physical characteristics of surfaces. Members of the SPM family all share a set of unique performance features that differentiate them from other analytical methods:

- Magnifications as high as $10^9\times$ (higher than that of any other microscopy technique) allow visualization of individual atoms.
- True three-dimensional (3-D) magnification provides quantitative height or depth information about features of interest.
- Operation in any environment permits imaging in the conditions best suited for the specimen, including air, vacuum or liquid.
- Proximity measurements allow physical or chemical information to be obtained in addition to topographic data.

All SPMs operate similarly. A very fine probe tip is placed close to the surface and then raster-scanned across it to generate an image. Piezoelectric ceramic elements, which have sub-angstrom resolution, are used to control the x , y , and z motion of the probe. As the probe is scanned in the x - y plane, it "senses" the surface and is controlled in the z axis to maintain a constant relationship with the surface. These movements are electronically recorded and stored in the instrument's computer to generate a 3-D representation of the surface.

SPM techniques differ from one another only in the physical relationship that is measured between the tip and the surface. The STM measures the local electronic state density, whereas atomic force microscopy (AFM) measures the short-range interatomic forces between the surface and a probe mounted on a flexible cantilever.

Other SPM techniques include near-field scanning optical microscopy (NSOM), a new method that overcomes the diffraction-limit barrier to resolution in conventional optical microscopy ($\sim 0.5\ \mu\text{m}$) via use of an optical aperture measuring $<50\ \text{nm}$, which is much smaller than the wavelength of light. NSOM brings to scanning probe microscopy the ability to combine optical-contrast techniques such as absorption and polarization with nanoscale resolution. NSOM also provides a method for optical spectroscopy of nanoscale structures as small as single molecules.

The ability of SPM methods to directly visualize surfaces at the atomic and molecular level with stunning resolution has provided a wealth of new information about materials ranging from bare silicon wafers to biological molecules such as proteins and chromosomes. The most important potential long-term benefits of these techniques may lie in their ability to perform nanoscale proximity measurements. Since SPM operation is based on various interactions between a probe and specimen, the nature of this interaction can be exploited to measure physical or chemical properties of the surface.

SPM technology continues to evolve. A current trend is to combine SPMs with other analytical instruments. For example, an SPM mounted on an inverted fluorescence optical microscope provides high-resolution images in biological applications, while an SPM inside a scanning electron microscope offers unique ways to study advanced materials.

For more information: TopoMetrix Corp., 5403 Betsy Ross Drive, Santa Clara, CA 95054-1162. Fax: 408-982-9751. (Extracted from *Advanced Materials & Processes*, July 1994)

Waiting for breakthroughs

Nanotechnology is the manufacture of materials and structures with dimensions that measure up to 100 nanometres (billionths of a metre). Its definition applies to a range of disciplines, from conventional synthetic chemistry to techniques that manipulate individual atoms with tiny probe elements. In some visions, current nanoscale fabrication methods could eventually evolve into techniques for making molecular robots or shrunken versions of nineteenth-century mills. In the course of a few hours, manufacturing systems based on nanotechnology could produce anything from a rocket ship to minute disease-fighting submarines that roam the bloodstream. Finished goods in this new era could be had for little more than the cost of their design and of a raw material—such as air, beet sugar or an inexpensive hydrocarbon feedstock. The "Drexlerian" future posits fundamental social changes: nanotechnology could alleviate world hunger, clean the environment, cure cancer and guarantee biblical life-spans.

Scientific visionaries have shifted their attention from outer to inner space, as the allure has faded from dreams of colonizing another planet and travelling to other galaxies. Computer mavens and molecular biologists have replaced rocket scientists as the heroes that will help transcend the limits imposed by economics and mortality.

In recent years, however, intricately constructed pictures of the next century and beyond have begun to be overtaken by real investigations into nanotechnology. What inspires actual researchers at the nanoscale is infinitely more mundane than molecular robots—but also more pragmatic. Nanotechnology, in this guise, may not contain the ready promise of virtually limitless global abundance and human mastery of the material world. But it may move beyond mere speculation to produce more powerful computers, to design new drugs or simply to take more precise measurements.

Researchers can now manipulate atoms or molecules with microscopic probe elements, marshal the 20 basic amino acids to form new proteins not found in nature, or help organic molecules spontaneously assemble themselves into ordered patterns on a metal surface. This work certainly presents the prospect of providing new tools for the engineering community. Ironically, it also demonstrates the difficulties of using individual atoms or molecules as building blocks, given the presence of a host of physical forces that may displace them.

Combining nanocomputers with molecular machines would allow almost anything that can be designed to be made from a variety of inexpensive raw materials, perhaps even dirt, sunlight and air. Assemblers could string together atoms and molecules so that most goods could be made from diamond or another hard material, giving the most ordinary objects a remarkable combination of strength and lightness.

The cost per kilogram of goods produced by nano-manufacturing would equal the price of potatoes. The resulting nanoworld, in which everyone is wealthy because of the drastic reduction in the cost of goods, would flummox economists, those scientists of scarcity. A jumbo airliner could be purchased for the current price of an automobile. A homeowner would pour acetone into a household manufacturing system, similar in appearance to a microwave oven. An hour later, out would come a computer, a television set or a compact-disc player. A home food-growing machine could rapidly culture cells from a cow to create a steak, a godsend to the animal rights movement.

Minuscule submarine-like robots made by assemblers would extend life or reverse ageing by killing microbes, by undoing tissue damage from heart disease or by reversing DNA mutations that cause cancer; the nanomachines would help revive bodies preserved in cryogenic storage by repairing frostbite damage to the brain and other organs.

On the dark side, assemblers would streamline the production of superweapons, allowing rapid fabrication of a tank or a surface-to-air missile. And then there is the "grey goo" problem—the possibility that nanodevices might be designed to replicate uncontrollably, like malignant tumour cells, and reduce everything to dust within days.

Most researchers whose work moves beyond computer simulations and into the laboratory do not view the challenges of nanotechnology as leading towards the goal of other nanoists. A number of them, some of whom even capitalize on the "nano" label in promoting their work, pursue a series of more modest objectives.

The complexity of making objects with individual molecular building blocks may eliminate any of the dramatic cost savings envisioned by the nanoists, except in

a few clearly delineated technological areas. Fabricating computer chips has already become a form of engineering the small, with the tiniest circuit elements measuring less than a micron. The cost of a new semiconductor plant now reaches into the billions of dollars, in part because of the technical challenges posed by the need to craft even smaller features onto the surface of a chip. Chipmakers can still justify the added expense because packing circuits more densely leads to higher computational performance and ultimately lower costs. For most other goods, nanotechnologies may receive tough competition from Mother Nature.

A researcher in the department of chemistry at the University of Newcastle describes the contortions often required to achieve atomic control of matter. In 1989 two IBM researchers penned their employer's acronym by manipulating 35 xenon atoms with a scanning tunnelling microscope—a device that dragged the atoms across a nickel surface. The atoms moved because of chemical bonding interactions that occurred when the microscope's tungsten tip came to within a tenth of a nanometre or so of each atom. A difficulty involved: the IBM logo was created in an extremely high vacuum at the supercooled temperature of liquid helium using inert xenon atoms. Outside this rarefied environment, the world becomes much less stable.

Some researchers believe that the nanoists fail to take into account critical questions about the thermodynamics and information flow in a system of assemblers. How do the assemblers get their information about which atom is where, in order to recognize and seize it? How do they know where they themselves are, so as to navigate from the supply dump [where raw atomic material is stored] to the correct position in which to place it? How will they get their power for comminution [breaking up material] into single atoms, navigation and, above all, for massive internal computing?

The present inability to build an assembler—coupled with elaborate speculation about what the future may hold—gives nanotechnology a decidedly ideological or even religious slant.

Chemistry has distant roots in alchemy, the belief that transmutation of materials will bring health and wealth. Nanoism resembles a form of postmodern alchemy—and one that awards cash for molecular machine parts. A prize is to be awarded for the fundamental breakthroughs that will usher in the era of molecular nanotechnology: a robot arm and a computing component for an assembler.

For the time being, the nanoists can only wait for these breakthroughs to arrive, while continuing to formulate their computerized models of molecular machine parts. It may be a long time coming. (Extracted from *Scientific American*, April 1996)

4. NATIONAL PROGRAMMES

Nanotechnology materials in India

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Abstract

In the production of the new generation of electronic devices, a major role will be played by low-dimensional structures. By using sophisticated techniques such as MBE and MOCVD, one can now easily grow two, one, and zero-dimensional multi-layer structures. The author describes the current R&D scene in various research laboratories and academic/technical institutions engaged in the characterization and development of electronic materials for nanotechnology applications; leading to the capacity- and capability-building in this new and emerging field of micro-electronics/micro-miniaturization in India.

Miniaturization (or size effect) has attracted enormous support from industry, as well as from the physicists and engineers. When the size of the semiconductor crystals (in nano-scale) is compared to an effective Bohr radius of an exciton, the optical/electrical and mechanical properties differ considerably from that of the bulk value—which could be attributed to the size confinement of kinetic motion of electron/hole/exciton-pair resulting in the enhancement of the semi-conductor band gap.

The majority of the nano-scale semiconductors studied with their interesting properties belong to the II-VI group. Recent developments in techniques such as MBE, MOCVD and MOVPE have enabled scientists to reduce structures in the realm of one and zero dimensions, which forces electrons to different energy states. Under the state of quantum confinement in low dimensions, important changes occur in the density of electron states, band structure phonon spectrum, charge carrier, scattering mechanisms, exciton binding energy and impurity ionization energy etc. It has a significant effect on the electrical, optical and physical characteristics of low-dimensional structures. Predictable energy changes in the electron states can, therefore, be produced by controlling the physical dimension of a structure, enabling scientists to pick or tune the desired electronic properties, leading to the fabrication of nano-structures such as lasers, transistors, photo-conductors and optical integrated circuits.

Several schools of physics in universities and technical institutions which have been conducting research basically in experimental solid state physics in the past have provided a strong foundation for investigating the characteristics of semiconducting materials in India. Even the national R&D laboratories became an extension of academic institutions. The contributions of Dr. V.G. Bhide (NPL, New Delhi), Dr. W.S. Khokle (CEERI, Pilani, 1964), Dr. S. Ranganathan (IISc, Bangalore, 1968) and Dr. K.L. Chopra, Dr. A.K. Barua (IACS, Calcutta, 1974), Dr. Krishan Lal, Dr. E.S.R. Gopal/Dr. T.S. Anantharaman and others also made a great impact on materials research in India. The arrival of Prof. K.L. Chopra from the USA to the Indian Institute of Technology heralded many milestones in thin-film deposition techniques and processes, physics of the nano-state and a whole range of applications

in many diverse optical, electronic and mechanical fields. It identified quantitatively the role of various scattering processes prevailing in polycrystalline and epitaxial metal films. The observed existence of a size effect in the Hall coefficient was a surprise.

The following paragraphs will focus on the current development of physics of the nanotechnology materials in India.

Electronic material—transport mechanism

Newer lithographic techniques using electron-beam, X-ray and ion beams as the exposure radiations have been developed by the Central Electrical and Electronics Research Institute, Pilani (Rajasthan). Plasma etching is also employed there to facilitate semiconductor device fabrication, and lithographically imaged low-dimensional structures in the transfer layers, which are generally imprinted on the underlying thin film.

The physics of resonant tunnelling are based on (a) the time-independent, and (b) the time-dependent quantum measurement approaches which have been under investigation at IIT, Delhi. The quantum transport in a semiconductor nano-structure, especially in a double barrier resonant tunnelling diode (DBRTD) has currently drawn the attention of both theoreticians and experimentalists alike. A negative differential conducting region is found to appear in its I-V characteristics, which is responsible for its high frequency amplification, oscillation, fast switching and logic applications.

Due to the variation of the crystallite size (2 nm to 50 nm) and the heterogenous nature of the porous silicon material, as well as the discovery of the emission of visible light from it, a tremendous amount of semiconductor research over the past few years has been carried out at the University of Delhi and the Solid State Physics Laboratory (SSPL), Delhi. It has been found to be very useful in making three-dimensional micromechanical devices. These structures are finding applications in automobiles, aerospace, defence and many other civilian sectors. It has capabilities to control large aircraft, and also to provide a large number of applications in the medical sector. Microbotics is another area where these devices are in great demand. A combination of microsensor, actuator, and electronic control on the same chip (called "smart structure") work analogous to the biological pattern of functioning. Fabrication of MEMS devices, micromotors and 3-D devices by using a variety of processing techniques including bulk micromachining, surface micromachining of silicon using isotropic or anisotropic techniques have been developed for making 3-D structures at SSPL, New Delhi.

The study of one electron energy levels in some sub-micron structures is being carried out at IIT, Madras. In addition, they are investigating the "Stark" effect in low-dimensional semiconductors where the tuning of the frequency with the field funds uses electro-modulators and electro-optic devices in several applications.

The Stark effect is the electrical analogue of the "Zeeman" effect. Heterostructure lasers emit light when these are forward-biased and lase when the current is

beyond the threshold value. In this regime, the electric field is high enough to produce a shifting in the frequency of the peak intensity of the line. The energy shift is quadratic with the field strength. Of late, the growth of ultrathin semiconductor wires and whiskers has attracted much attention because of their potential use in improving the development of 1-D quantum wire high-speed FETs and LEDs with an extremely low power consumption. A variety of techniques for the growth of semiconductor whiskers, 1-D quantum wires, quantum dots, superlattice structures, arrays of minute cones as field-emitters and nano-/micro-structures, include electron/ion beam (both focused and non-focused) microlithography, photolithography, vapour-liquid-solid (VLS) growth techniques, metal organic vapour phase epitaxy (MOVPE) methods etc., to quote a few. With the emergence of heavy ion-polymer/dielectric (called Solid State Nuclear Track Detectors—SSNTDs), interaction-based techniques involving phase change development along the track trails followed by the controlled creation of etched channels of desirable dimensions through “chemical amplifiers”, nuclear track or ion track filters (ITFs) have been produced at GNDU, Amritsar. These ion track filters can potentially be used for the template synthesis of metallic, metal-semiconductor nano-/microstructures or ensembles and tubules using electrochemical techniques.

Opto-electronic processes

Opto-electronic processes in semiconductors have been investigated due to their technological importance in optic-fibre communications. The understanding of optical processes, such as emission and absorption, are very important for development of technology. In this respect, it will be interesting to note the investigations carried out by Jadavpur University, Calcutta. Photoelectric emission from a two-dimensional electron gas (2-DEG), formed in the ultrathin film in the presence of a quantizing magnetic field, due to a complete three-dimensional quantization, ultimately forms a zero-dimensional electron gas. The subject of non-linear optical properties of excitons due to Exciton collisions in GaAs quantum wells is under investigation at the Tata Institute of Fundamental Research, Bombay. Non-linearities in excitonic optical transitions in GaAs Quantum Wells (QWs) have been seen in the past, mainly in the form of saturation of excitonic absorption under resonant excitation in the presence of either cold excitons or free electron-hole (e-h) pairs. These have been attributed to effects such as the Fermi exclusion principle and collision broadening. Exceptions can also be formed under nonresonant conditions from an initial excitation of free e-h pairs.

Mesoscopic

The length scale associated with the sample dimensions in systems are much smaller than the inelastic mean free path, or the phase-breaking length of the electrons. Thus, the electron motion becomes phase coherent over the entire sample. Studies in this field have led to the possibility of a new generation of quantum devices. The feasibility of using quantum devices is being examined at the Institute of Physics, Bhubaneswar.

Various processing steps are involved in the fabrication of chrome masks for MMIC using an electron beam microfabricator system. The system has the capability of writing the minimum feature size of the order of 0.25 micron with positional accuracy: .026 micron at 30 KV. Patterns of the size of 0.5 micron are successfully

delineated. Polymethylmethacrylate and Isopoly E-B Negative resist -NS are used as electron positive and negative resists respectively for chrome masks. Dry plasma technology has been used for chrome etching. A set of masks have been fabricated for various devices involving submicron geometries with a feature size ranging from 10 μm to 0.5 μm , at the SSPL, New Delhi.

The unseating of the once unbeatable conventional supercomputers, such as Crays and Convexes, by cheap parallel processing alternatives is the latest upheaval in the ongoing information revolution. Scientists at BARC (Bhabha Atomic Research Centre), Bombay have managed to make a 64-node machine (called ANUPAM) fully functional. It can calculate electronic structures and total energies of molecules using quantum mechanical equations; solving these equations earlier was so formidable that it was generally considered beyond the power of most computers.

IIT, Bombay has concentrated on thin oxide materials, such as radiation hardened ones, with characterization tools, simulation software and plans to develop IR and biosensors.

Several other laboratories and R&D centres are engaged in activities which would facilitate micro-electronic device processing. The Central Scientific Instruments Organization (CSIO), Chandigarh is running a comprehensive programme on the development of capital instruments, which typically constitute a fabrication line. The Centre for Advanced Technology (CAT), Indore is developing Synchrotron Radiation Source, whose miniature version could be tapped for submicron lithography. Work on plasma polymerization, dry resists and e-beam writing is being carried out at the University of Pune. NPL, Delhi has also been active, through some of its programmes on the characterization of micro-electronic materials, collaborative programmes and assembly of process equipment. Several other institutions, such as IISc, Bangalore, TIFR, Bombay and NCL, Pune, have also contributed to specific sectors of the entire fabrication process either directly or through their collaboration with other institutions in India.

Conclusion

India may be said to have an electronic policy since 1970, when the Department of Electronics was set up. It was followed by the setting up of the Electronics Commission in 1971. The large-scale production of electronic goods was concentrated in public sector companies, primarily the Electronic Corporation of India Ltd. (ECIL), Bharat Electronics Ltd. (BEL), Central Electronics Ltd. (CEL) and Indian Telephone Industries (ITI). The liberalization of delicensing called for the restructuring of the import policy, and was introduced in 1985 and 1991 respectively. The pattern of technology change and the development of indigenous technology to produce electronic products for industry would require R&D efforts and innovations in the new and emerging fields.

New developments are constantly taking place, all over the world, in the field of fabrication of electronic devices. Spectacular developments in imaging technology, material quality, measurement techniques, device architecture and its stagewise simulation have led to the fabrication of a variety of sophisticated, energy-efficient, low-dimensional semiconductor devices, for example, 64 bit DRAM, integrated sensors, neural networks, single electron devices and so forth. India's contribution in this area is well appreciated by the world.

This trend in micro-miniaturization extrapolates that, in approximately two or three decades, the size of an electronic circuit will ultimately reach the size of a large molecule where various device activities could be modulated by manipulating the molecule. The molecular structure of such a circuit will dominate all elementary physical processes and thus influence the behaviour of each functional unit. In future, the device resulting from extreme miniaturization may not be completely based on silicon technology due to effects such as clustering, dopant diffusion and channelling which set a lower limit to the structural dimensions and power dissipation. Compound semiconductors, such as GaAs, have been found more suitable for high-speed, high-frequency and slightly higher-temperature operations. The ejection of electrons or the emission of lasers from crystalline whiskers fabricated by conventional processes have been marred by inherent defects. Recent advances in crystal growth have facilitated

the fabrication of 200 Angstrom thick, 2 micron long, defect-free GaAs whiskers having a p-n junction to boost the emission efficiency 100-fold.

Diamond film is another new material that has prospective application as a semiconductor material at high temperatures in highly radioactive environments. But the synthesis of high-purity diamond films, with good crystallinity onto a different substrate, especially at temperatures lower than—say 800° C—has been a problem. Plasma deposition of atomic hydrogen-assisted growth of high quality diamond films at 130° C has made the use of this material as semiconductor films a reality. Porous silicon is another new material which has scope for various applications, including SOI, sensors and micromechanical devices etc.

It is therefore worthwhile diverting R&D efforts towards the investigation of new and emerging semiconducting materials.

Table 1. Hybrid micro-electronics in India

(a) R&D institutions

1. Central Electronics Engineering Research Institute (CEERI), Pilani.
2. Defence Electronics & Research Laboratory (DIRL), Hyderabad.
3. Vikram Sarabhai Space Centre (VSSC), Trivandrum.
4. ISRO Satellite Centre (ISAC), Bangalore.
5. Space Applications Centre (SAC), Ahmedabad.
6. Electronics & Radar Development Establishment (LRDE), Bangalore.
7. Indian Institute of Technology (IIT), Bombay.
8. Banaras Hindu University (BHU), Varanasi.
9. Indian Institute of Technology (IIT), Kharagpur.
10. University of Poona, Pune.
11. Indian Institute of Technology (IIT), Delhi.

(b) Organizations

1. Nuclear Fuels Complex, Hyderabad.
2. M/s Eltech Corporation, Peenya, Bangalore.
3. M/s Jyoti Refinery, Bombay.
4. M/s Kerala State Electronics Development Corporation (KELTRON), Trivandrum.
5. Centre for Development of Materials, Pune.
6. Central Glass and Ceramic Research Institute (CGCRI), Calcutta.
7. Indian Institute of Science, Bangalore.
8. Indian Institute of Technology (IIT), Kharagpur.

(c) Major production units

1. Bharat Electronics Limited (BEL), Bangalore and Ghaziabad.
2. Indian Telephone Industries (ITI), Bangalore, Naini, Allahabad, Mankapur, Gonda.
3. Hindustan Aeronautics Limited (HAL), Hyderabad.
4. Electronics Corporation of India Limited (ECIL), Hyderabad.
5. Gujarat Communications and Electronics Limited (GCEL), Vadodara.
6. Western Indian Enterprises Limited, Pune.
7. Mini-circuits Limited, Bangalore.
8. Vikas Hybrids and Electronics Limited, New Delhi.
9. Film Electronics, Meerut.
10. Anvi Electronics, Valsad.
11. Usha Rectifiers Corporation India (P) Limited, Shimla.

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UK needs to invest more in micro technologies

The UK is not doing enough to exploit nano-technology—the design and manufacture of microscopic engineering structures that is increasingly regarded as a vital future element in such key industrial areas as information technology (IT), electronics, computing and precision manufacturing. The technology is being “left behind” as a priority area for publicly-funded research, despite the efforts made over the last couple of years in the Government's Technology Foresight programme to identify technologies that will prove crucial to economic competitiveness and quality of life over the next 5-20 years. Existing initiatives in the field have now allocated all their available funding, but Foresight did not identify nano-technology as a priority for future activity. The result could be a further slippage of the UK's already tenuous position as a major force in nanotechnology research, with consequent loss of economic opportunities.

Those are the major conclusions of a report published by the Parliamentary Office of Science and Technology (POST). The report says that the term nanotechnology generally refers to structures with dimensions in the range 0.1-100 nm (1 nm = 1 millionth of a millimetre), but in this case the report also takes in “micro engineering” which it defines as operating at a scale up to 1 mm.

Altogether three major initiatives have sought to stimulate activity in this “small engineering” area over the last few years. These are: the National Initiative on Nanotechnology (NION) set up in 1986 by the National Physical Laboratory (NPL) and the DTI to promote awareness of the technology; a LINK Nanotechnology Programme (LNP) launched in 1988 by the DTI with £6 million of initial funding, subsequently supplemented by £1.5 million from the Science and Engineering Research Council (SERC); and a SERC Nanotechnology Managed Programme managed by the Engineering and Physical Sciences Research Council

(EPSRC), which had funding of £4.7 million. But these initiatives are now in their final stages and closed to further applications, though work in the Managed Programme may stretch out to the end of the decade.

Nevertheless the UK does now have a groundbase of around 1,000 companies, 30 universities and 7 research establishments with expertise in the field. Application areas include medicine, metrology, manufacturing, aerospace, automotive, IT/communications and home automation. The main technologies involved are surface mounting, fibre-optics, thin films, micro machining and micro assembly. Moreover this work has provided the UK with some 13 acknowledged "centres of excellence".

But at the same time the UK is already apparently lagging behind its major rivals in most relevant areas. An assessment prepared by the German Ministry of Research and Technology indicates that the UK trails Japan, the USA and Germany in the areas of ultra-thin films and nanocrystals and is behind the USA and Germany in ultra-precision engineering. Only in the area of nanometrology

does it achieve a higher ranking—joint second with Germany and France with the USA again out in front.

In this context the report says that the Foresight programme "was remarkable for its lack of mention of the subject". Nanotechnology was only cited in two of the 15 Foresight panels—those for materials, mainly in respect of new nanocomposites, and for construction. The report suggests that because Foresight was deliberately structured around application areas it was weak at identifying key "generic" technologies.

The likely future applications of nanotechnology, however, and the size of the respective markets are considerable. Moreover many potential applications, the report points out, have yet to be realized commercially, meaning that every country has the chance to establish a market presence or even dominance. These markets include some sensor and actuator applications, ceramic structures using nanopowders and high resolution display systems. Source: *Engineering*, November 1996

5. RECENT DEVELOPMENTS

Russia science in nanocrystal advances in US

A Denver-based company and a group of Russian scientists are producing metal and ceramic nanocrystal materials which it claims can improve strength and ductility and provide more flexibility than conventional materials.

Ultram International Inc. said it is the "first US-based firm producing and marketing high-quality nanocrystal and nanocrystalline structured materials".

The tiny materials consist of discreet, extremely small particles—between 10 and 100 nanometres in size—of ordinary metals and ceramics, according to a company statement.

The technology for producing the nano-materials came from the Soviet Union more than 15 years ago.

The company expects to see tougher steel casting materials that offer greater wear resistance than conventional metals, ceramics with improved properties and enhanced filtration membranes that will allow for gas separation.

Danaloy DSC, a dispersion-strengthened copper alloy produced by Ultram, is a nanocrystalline-structured material that assumes the unique properties missing in conventional materials.

Products utilizing the unique properties of these revolutionary materials can be more flexible, ductile, or harder or have electrical, magnetic, mechanical and optical properties that conventional materials lack.

Ultram uses Russian-developed continuous production processes to produce large quantities of the nanocrystal materials.

The company produces the tiny metals in facilities in Russia and Denver which together have "multi-ton" capabilities. (Source: *American Metal Market*, 6 July 1994)

Silicon oxide nanostructure emitting blue light

H. Morisaki, S. Nozaki, H. Ono and their colleagues at the University of Electro-Communications have developed a blue-light emitting material containing chains of silicon oxide regions tens of nanometres in diameter. Ultraviolet illumination causes the material to emit light-bright enough to be seen by the eye in a normally-lit room. Since a few years ago, when the luminescence of silicon-based materials was discovered, many types of light-emitting materials have been proposed. However, none has the same structure as the new material.

The new material is made by a gas-evaporation process. Silicon powder is put on a boat-like container made of boron nitride. The powder is heated in a gas mixture of 99 per cent argon and 1 per cent oxygen, and vaporizes. The silicon vapour ascends to a quartz substrate over the boat, and solidifies to form particles 0.01-0.02 μm across. The particles form chains on the substrate.

When the gas is pure argon, the particles are too large to emit blue light, and only glow orange. To reduce the diameter of the particles, the research team added a little oxygen to the ambient gas to oxidize the silicon during evaporation. The particles obtained have been analysed, and consist of silicon oxide with no trace of pure silicon.

Light-emitting semiconductors are common in compact disc players and display equipment. Most are expensive

compound semiconductors such as gallium arsenide. However, since researchers found that silicon emits visible light when shaped into a particle or wire with an extremely small diameter, extensive work on silicon-based light emitters began.

A light-emitting device made of compound semiconductor is hardly compatible with a silicon LSI because of the different material properties and production process, and they are difficult to integrate in a monolithic chip. When a silicon light emitter is available, the problem will vanish, and optical coupling can be included in a chip so that data can be transferred by light in an LSI.

For more information contact: The University of Electro-Communications, Department of Communications and Systems, 1-5-1, Chofugaoka, Chofu City, Tokyo 182. Tel.: +81-424-83-2161; Fax: +81-424-80-6072; e-mail: NOZAKI@TTL.CAS.UEC.AC.JP (Source: *JETRO*, January 1995)

NIRIM fabricates silicon carbide nanoceramics

Through low-temperature liquid phase sintering of a fine powder with uniform particle size (average diameter of 0.09 microns), Japan's National Institute for Research in Inorganic Materials (NIRIM) of the Science and Technology Agency (STA) has succeeded in making a silicon carbide nanoceramic material that has an extremely fine, uniform structure. It has been maintained that precision manufacturing of ceramic parts with complex shapes is difficult with silicon carbide because there is 15 to 20 per cent firing shrinkage during sintering. However, the nanoceramics produced by NIRIM demonstrated superplasticity with a deformation rate of 10^{-4} per second (equal to a deformation rate of 36 per cent per hour) at a temperature below 1,700° C, and this opens the way for the manufacturing of complex shaped silicon carbide ceramic parts through the same plasticity processing used with metal. This is the first time in the world that this sort of nanoceramic material has been manufactured.

Silicon carbide ceramics have superior resistance to heat, oxidation and abrasion and are used for many gas turbine and other precision machine parts.

Generally, however, processing of this ceramic material is difficult because it is sintered at high temperatures of over 1,900° C. One problem is the expense, because diamond tools, etc., must be used.

NIRIM took commercially available type- β silicon carbide powder, adjusted the particle size through wet grinding and classification processing, and made a fine powder which had an average particle diameter of 0.09 microns and which did not contain any coarse particles over 0.5 microns. To this, sintering assistants alumina (7 wt. per cent), yttria (2 wt. per cent) and calcia (1 wt. per cent) were added, and hot press sintering was performed for 15 minutes under 20 MPA pressure and at a temperature of 1,750° C.

By adjusting the particle size distribution of the raw material powder and performing liquid phase sintering at a low temperature, NIRIM achieved a silicon carbide nano ceramic material with an unprecedented uniform, fine struc-

ture. This will be effective as a matrix for conducting structural control and is also noteworthy because now that superplasticity has been demonstrated, plasticity processing, which was considered difficult for silicon carbide, has become possible, and will expand its application into the manufacturing of parts with shapes that are complex and require precision. (Source: *Tokyo KAGAKU KOGYO NIPPO*, 4 April 1995)

Properties at nanoscale

"Small is different", concludes Dr. Uzi Landman, director of the Georgia Institute of Technology's Center for Computational Materials Science, Atlanta. "In the desire to reduce the size of electronic devices, there are certain effects pertaining to size and dimensions that must be considered. If you go below a certain limit of size ... the behaviour of the system may not be what you would expect on the macroscopic level". (Source: *Industry Week*, 1 May 1995)

Carbon nanocapsules grown on carbon fibres

M. Kusomoki and colleagues at Japan Fine Ceramics Centre (JFCC) have discovered that carbon nanocapsules can be produced just by heating carbon fibres. No arc discharge is necessary. The research team performed microscopic investigations of amorphous carbon nanoparticles at high temperatures, and ascertained that the particles become graphite at the surface.

Since 1990, when C60, a soccer ball-like carbon molecule, was found in soot, many researchers have examined cages of carbon atoms, or fullerenes. Many possible applications are being pursued in different fields. The most attractive of the fullerene molecules are nanometre-size tubes and capsules made of carbon atoms because of their properties.

Fullerenes were thought to be produced only in plasma with a strong electrical field and a very high temperature. Most conventional processes use arc discharge. The new research shows that nanometre carbon tubes and capsules can be produced in a purely thermal process.

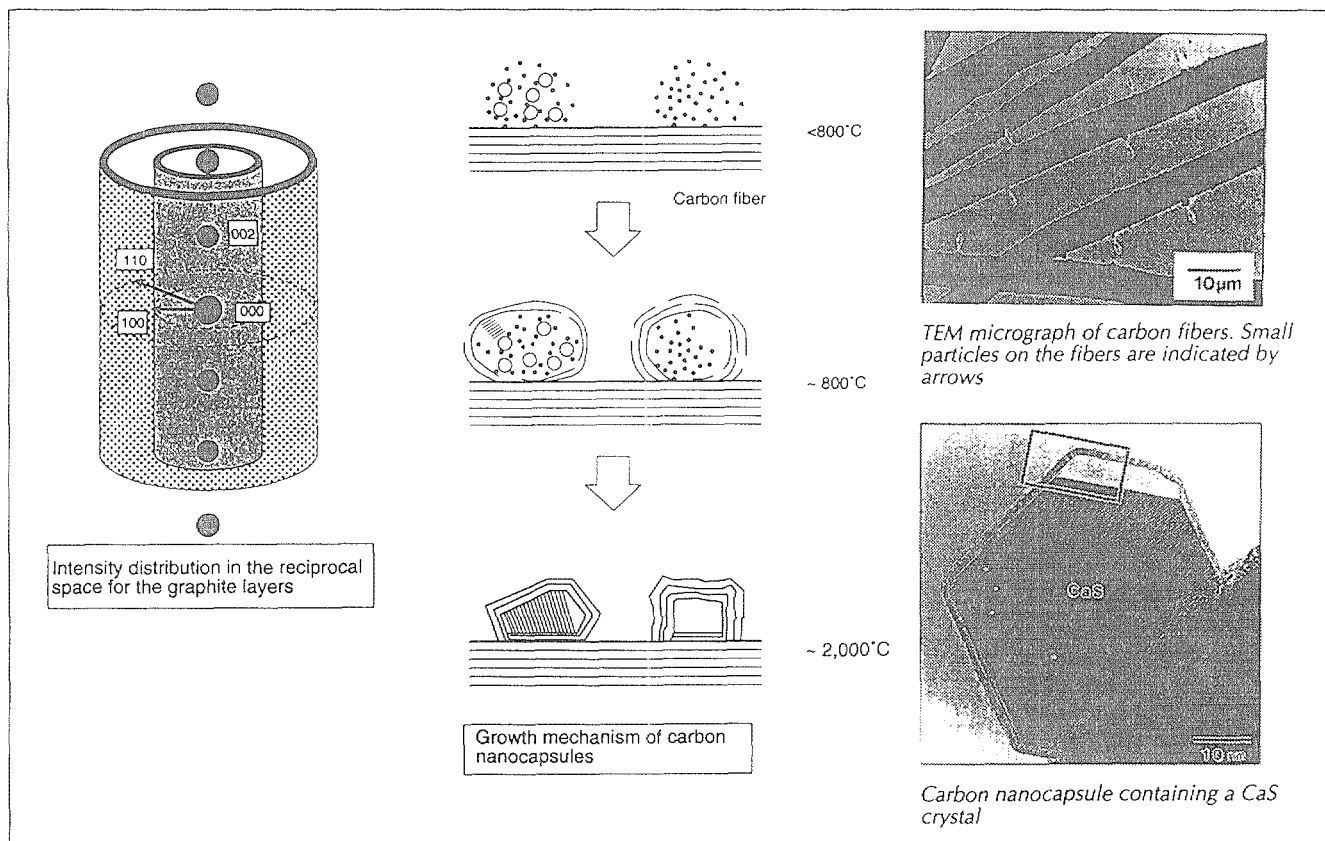
The research team realized that carbon nanocapsules were formed on sintered carbon fibres. Specifically, after heating carbon fibres at 2,000° C under tension, carbon nanocapsules were found on the fibre surface. Although some capsules are hollow, most have a shell made of six graphite sheets, and contain a single crystal of calcium sulphide (CaS) which seems to come from impurities in the source carbon.

The investigation has revealed that amorphous carbon particles grow on the surface of a carbon fibre being heated, and then become graphite at the surface and hollow inside at 800° C. Each particle turns into a carbon nanocapsule, either cubic or polyhedral, when the temperature rises to 2,000° C.

The capsules are so easy to shake off carbon fibres using ultrasonic wave that the capsules can be obtained in the form of powder. Because so many carbon nanocapsules will be available, their physical properties will be easy to determine. Masses of carbon nanocapsules may serve as an elastic lubricant.

The new method promises to be a source of high-purity carbon. When a material is added as an impurity to source carbon, the method will produce capsules enclosing the material. Possible applications of such capsules are the drug delivery system (DDS), and delivery of contrast medium for medical imaging.

For more information contact: Japan Fine Ceramics Centre, 2-4-1, Mutsuno, Atsuta-ku, Nagoya City, Aichi Pref. 456. Tel.: +81-52-871-3500; Fax: +81-52-871-3599. (Source: *JETRO*, August 1995)



(Source: *JETRO*, August 1995)

Silicon nitride based ceramic nanocomposites

Nanocomposites ($\text{Si}_3\text{N}_4/\text{SiC}$) were studied by combined high-resolution transmission electron microscopy and electron energy-loss spectroscopic imaging (ESI) techniques. In ESI micrographs three types of crystalline grains were distinguished: Si_3N_4 matrix grains ($\sim 0.5 \mu\text{m}$), nano-sized SiC particles ($< 100 \text{ nm}$) embedded in the Si_3N_4 , and large SiC particles (100-200 nm) at grain boundary regions (intergranular particles). Amorphous films were found both at Si_3N_4 grain boundaries and at phase boundaries between Si_3N_4 and SiC. The Si_3N_4 grain boundary film thickness varied from 1 to 2.5 nm. Two kinds of embedded SiC particles were observed: type A has a special orientation with respect to the matrix, and type B possesses a random orientation with respect to the matrix. The surfaces of type B particles are completely covered by an amorphous phase. The existence of the amorphous film between the matrix and the particles of type A depends on the lattice mismatch across the interface. The mechanisms of nucleation and growth of $\beta\text{-Si}_3\text{N}_4$ grains are discussed on the basis of these experimental results.

It is well known that Si_3N_4 ceramic materials exhibit outstanding mechanical and thermomechanical properties at high temperature and can be used as components in heat engines and gas turbines. In recent investigations of Si_3N_4 ceramics, particular attention was paid to the improvements of these properties. Researchers have demonstrated that Si_3N_4 matrix composites with a dispersion of nano-sized SiC particles show enhanced properties compared to monolithic Si_3N_4 materials, i.e., superplastic behaviour and high strength and fracture toughness. The improvement in mechanical properties should be strongly related to the change of microstructure and chemistry of the composites, such as grain morphology, the distribution of SiC particles, and structure and chemistry of grain boundaries, phase boundaries and intergranular phases.

Very recently the microstructure and chemistry of grain boundaries and grain boundary phases in many different Si_3N_4 ceramic materials have been extensively investigated by high-resolution transmission electron microscopy (HRTEM) and/or analytical electron microscopy (AEM). It has been shown that an amorphous intergranular phase always exists at grain boundaries and at triple junctions and that the amorphous film at the grain boundary exhibits an equilibrium thickness in a given material, which is dependent only upon the chemistry of the system (impurities, sintering additives, oxidation layer on the starting powders, etc.). However, fewer studies have been made for nanocomposites. In research undertaken by the Max-Planck-Institut für Metallforschung and the Japanese Institute of Scientific and Industrial Research, experimental studies on composites comprising nano-sized particles have been conducted. Grain morphology, phase distribution, and the morphology and distribution of SiC particles were studied using AEM. The structure of grain boundaries and phase boundaries was investigated using the HRTEM lattice imaging technique.

For more information contact: the Max-Planck-Institut für Metallforschung, Institut für Werkstoffwissenschaft, D-10174, Stuttgart, Germany and the Institute of Scientific and Industrial Research, Osaka University, Osaka, Japan. (Source: *Journal of the American Ceramic Society*, Vol. 79, No. 3, 1996)

Ultrafine superstrength materials

Pioneering work on nanocrystals has shown that materials with novel and improved properties can be

obtained when their microstructures approach nanoscale dimensions. For example, the fracture strength of consolidated Fe-nanocrystals alloyed with 1.8 wt. per cent of carbon, is an order of magnitude larger than the same Fe-alloy produced by conventional casting methods. Besides dramatic increases in strength and toughness, significant changes in physical properties have also been observed in nanocrystalline solids when compared with the corresponding conventionally produced coarser-grain counterparts. These properties include: thermal expansion coefficient, magnetic susceptibility, saturation magnetization and superconducting properties. Although the detailed mechanism responsible for these large changes in the physical and mechanical properties of nanocrystalline materials are not as yet clearly understood, most researchers in the field agree that they are connected with the much larger boundary area in nanocrystalline materials. However, the microstructure of metallic nanocrystalline materials is unstable against grain growth at high temperatures, with concomitant loss of the material properties. This instability makes these materials unsuitable for high-temperature applications.

Before the production and the consolidation of nanocrystalline materials, it was known that the strength of polycrystalline metals follows the Hall-Petch relation given by the empirical formula

$$\sigma = \sigma_0 + \frac{k}{\sqrt{d}}, \quad (1)$$

where k is a material constant, d is the average grain dimension, and σ_0 the flow stress. For an average grain dimension in the range of 10 nm another strengthening mechanism becomes dominant. This mechanism is associated with the stability of a dislocation loop and is expressed by the relation,

$$\sigma \propto \frac{\mu b}{d}, \quad (2)$$

where μ is shear modulus, b is the magnitude of the Burgers vector and d , as in equation (1), is the average grain size. Equations (1) and (2) above express the fact that the strength of polycrystalline metals increases with decreasing grain size. Although these strengthening mechanisms are effective at low and moderate temperatures, they become inoperative at high temperatures due to the onset of thermally activated processes, such as creep and grain coarsening. These thermally activated processes have the effect of degrading the strength quite rapidly. Both the faster diffusion rates and the larger boundary area in nanocrystalline materials have the effect of increasing the rates of thermally activated processes even further. Consequently, the rate of strength degradation increases and the temperature at which the strength of the material approaches zero decreases as the average grain size decreases. That is to say, the very nanoscale microstructure that produces dramatic increases in strength and toughness at low and moderate (below half the melting temperature of the material) temperatures, accelerates the rate of mechanical properties degradation at elevated temperatures. The important question is then the following. How is it possible to take full advantage of the significantly

improved mechanical properties at low and moderate temperatures provided by the ultrafine-grained (ideally nanosize) materials and still be able to retain a substantial fraction of this higher strength at elevated temperatures? As has been explained, a satisfactory answer to this question was provided by Louat through the formulation of his theory of superstrength.

According to this theory, materials with superior strength can be obtained by embedding high volume fraction (greater than 50 per cent) of ultrafine particles in a ductile matrix. Further, the theory predicts that the strength of such materials increases with decreasing particle size. Specifically, at temperatures below the melting temperature of the matrix and over a wider range of particle size, the strength, σ , should follow a Hall-Petch relation similar to the one given by equation (1). In addition, for the case where the particles are so closely packed as to form a rigid skeleton, the expected minimum strength when the matrix melts is given by

$$\sigma = \frac{2\gamma\cos\theta}{r}, \quad (3)$$

where γ is the surface energy, θ the contact angle between the particle and the matrix and r is the effective radius of the interstices between the particles; r is proportional to d , the average particle size. Taken together equations (1)-(3) implicitly contain the following basic requirements: (1) that there is good bonding between the matrix and the particles; (2) both phases deform plastically, but one phase is more rigid than the other. In addition to these two requirements, it is also important for stability against Ostwald ripening that the particles and the matrix should be immiscible.

Louat's approach takes full advantage of the dramatic increases in strength, at low and moderate temperatures, that can be achieved in nanocrystalline materials while at the same time avoiding their major drawback. Because, in contrast to monolithic crystalline materials, the materials strengthened in accordance with Louat's theory retain a significant portion of their strength even when the matrix melts. Moreover, the level of high-temperature strength retention increases with decreasing particle size.

An initial experimental confirmation of this theory was first obtained on Fe-Hg system. For this system, a yield strength of 32.4 MPa was obtained at 90 per cent of the melting temperature of mercury. Following this work, the research was extended to include materials which are technically more relevant. The materials included in that study were various carbide particles embedded in a copper matrix. For these material combinations, micron and submicron size commercially available carbide powders were used. Consistent with the theory, the room temperature strength of the consolidated specimens followed the Hall-Petch relation given in equation (1). Moreover, a yield strength of 2,413 MPa was obtained for 0.5 μm size TiC particles in a copper matrix. This strength value is more than 30 times that of annealed copper.

The results presented in this paper refer to iron or copper particles embedded in a lead matrix and to the case of tungsten particles embedded in a copper matrix. These particle-matrix combinations were chosen because: (a) both iron and copper are immiscible in lead; and, similarly tungsten is immiscible in copper; (b) the types of particles are, in varying degree, deformable at low temperature; (c) a lead-based material can be considered to be in the high temperature regime even at room temperatures; and, finally,

(d) the copper-tungsten material would have technological importance in terms of high temperature strength and high conductivity. In summary, therefore, all the material combinations are appropriate model materials to further test the superstrength theory and have practical applications.

For more information contact: Materials Science and Technology Division, Naval Research Laboratory, Washington, DC 20375-5000. (Extracted from *Nano-Structured Materials*, Vol. 1, pp. 89-94, 1992)

Nanocomposite magnetic materials

When materials possessing long-range magnetic order (e.g., ferromagnetism, antiferromagnetism) are reduced in size, the magnetic order can be replaced by some other magnetic state. Conceptually, this can be understood as a consequence of the increased uncertainty in the momentum, p , and in the energy of an electron (possessing a magnetic spin and orbital angular momentum) in the ordered region as the dimension d , of the magnetic region decreases in accordance with the Heisenberg relation $\Delta p \approx \hbar/d$ (\hbar is Planck's constant). When this energy uncertainty is equivalent to the magnetic ordering energy ($\approx k_B T_C$, where k_B is the Boltzmann constant and T_C is the ordering temperature), the long-range order is no longer energetically favourable. For Fe ($T_C=1043$ K), $d \approx 1$ nm. One way to reduce the dimensions of the ordered magnetic regions is to isolate them inside non-magnetic matrices by precipitation from solid solution. Examples of many such studies can be found in the literature of metallic alloys. Another way is to form a composite of nanometre-sized magnetic and non-magnetic species, e.g. by cosputtering immiscible materials. For the latter studies, the magnetic species have usually been metallic and the non-magnetic species have been nonmetals. For these "nanocomposites", the gross magnetic behaviour became either paramagnetic or superparamagnetic, as the size of the magnetic species decreased to the nanometre size range, when the composition was adjusted to keep the magnetic species well separated. Because of the ease in control of the magnetic behaviour by control of the processing parameters, such materials present great possibilities for the atomic engineering of materials with specific magnetic properties. Such materials are potentially important to the magnetic recording industry for achieving high-density information storage, and in the refrigeration industry for their potential for enhancing the efficiency of magnetic refrigeration cycles.

Nanocomposites form a class of materials in which the magnetic properties can be tailored by means of composition and processing variables to range from that of the bulk magnetic constituent to that which is identified with the nanometre dimension. They can be prepared in both bulk and thin-film forms. Because these materials possess electrical and optical properties similar to their constituents, they can also be prepared with regard to choosing a unique combination of electrical, optical and magnetic properties. Three types of nanocomposites exist: (1) thin films containing small magnetic particles, (2) bulk materials containing small magnetic particles, and (3) thin-film multilayers with nanometre layer thicknesses. They all are comprised of a magnetic and a non-magnetic species and the interface is important in each system. Their magnetic behaviour is modified significantly by the processing conditions, and their unique magnetic properties (e.g., superparamagnetism, spin-glass behaviour, magnetic viscosity) are due to the nanometre dimension. Use of the atomic engineering laboratory provided by the class of

nanocomposites makes possible the development of materials with specific magnetic properties. (Extracted from *NanoStructured Materials*, Vol. 1, pp. 83-88, 1992)

Synthesis of polymerized preceramic nanoparticle powders by laser irradiation of metalorganic precursors

Laser chemical processing provides an attractive route for the synthesis of ultrafine, high-purity ceramic powders. Silicon nitride powders, for instance, have been synthesized by gaseous-phase reactions, induced by CO₂ laser radiation, using ammonia and silane precursors. Another recent approach, however, involves the ultrasonic injection of a liquid metalorganic monomer into the beam of a cw CO₂ laser. Intense interaction of the beam with the strong radiation-absorbing aerosol results in plume, of ~2000° K estimated temperature. Using a silazane monomer precursor, an intriguing discovery with this process is the synthesis of nanophase particles of preceramic silicon nitride. By appropriate molecular design and process parameter selection, this method can, in principle, be used for the synthesis of intermetallic, silicide, boride, nitride, carbide, and oxide nanophase materials.

It should be emphasized that the rapid polymerization of the silazane monomer (CH₃SiH₂NH)_x to form nanoparticles does not occur via conventional chemical processing. A key feature in the laser-facilitated process evidently is the high degree of reactivity of Si-H and N-H functionalities.

It has been shown that a metalorganic liquid precursor can undergo conversion to a nanophase preceramic powder. The rapid cross-linking involved in this process is unique, since this is not achievable by conventional chemical processing. The process is highly efficient with an overall efficiency of ≥ 60 per cent. It can also be carried out at atmospheric pressure and avoids using toxic chemicals. Two possible consolidation routes appear to be viable for converting synthesized powder into bulk material. The fully converted ceramic material could be consolidated by hot isostatic pressing; alternatively, the preceramic materials could be die-extruded into a desired form, and then converted into ceramic material. (Extracted from *Nano-Structured Materials*, Vol.1, pp. 21-25, 1992)

Materials with ultrafine microstructures: retrospectives and perspectives

Materials with ultrafine microstructures are solids that contain such a high density of defects (point defects, dislocations, grain boundaries, interphase boundaries, etc.) that the spacings between neighbouring defects approach interatomic distances. Ultrafine microstructures have already been recognized more than a century ago (e.g., by Sorby, Tschernoff, Osmond, Howe, Sauveur, Wilm, Merica et al.) to exhibit remarkable and technologically attractive properties. The physical reasons for these properties were elucidated when modern defect theory and high-resolution methods (TEM, FIM) became available. Since about 1970, the interest of materials scientists refocused on solids with ultrafine microstructures when it was recognized that specifically tailored ultrafine microstructures permit the generation of materials with new atomic and/or electronic structures. So far, the following two developments of this type have emerged. In the area of semiconductors, quantum well structures and superlattices consisting of thin layers with different dopings or compositions were generated by means of two-dimensional ultrafine microstructures. Research activities aiming towards the synthesis of new

atomic (and electronic) structures in metals, ceramics and semiconductors by means of three-dimensional ultrafine microstructure were initiated by the proposal to generate solids, a large volume fraction of which consists of the cores of grain and/or interphase boundaries. These solids have been called nanocrystalline or nanophase solids. Such solids differ structurally from crystals and glasses with the same chemical composition, because the atomic arrangements formed in the cores of grain or interphase boundaries deviate from crystalline or glassy structures. Recent studies of nanocrystalline solids by X-ray/neutron diffraction, EXAFS, different spectroscopies, as well as property measurements support these ideas. Presently, the following areas of research on nanocrystalline materials appear to be of particular interest: nanocrystalline alloys, the ductility of nanocrystalline ceramics, ferromagnetism in nanocrystalline metals, nanoglasses and submicron materials. In this paper, a material will be called a material with an ultrafine microstructure if it is a solid and if it contains such a high density of defects (point defects, dislocations, grain boundaries, interphase boundaries, etc.) that the spacings between neighbouring defects approach interatomic distances. Macromolecular materials and solids, the atomic structure and/or properties of which are dominated by free surface effects (e.g. thin films, thin wires, small isolated crystals), will not be considered. According to the title, the paper will be divided in the following two sections: (i) retrospective and historical development, and (ii) recent developments and future perspectives.

Retrospective and historical development

Historically, two periods of development in research and application of ultrafine microstructures may be distinguished. During the first period, ranging from about 1870 to about 1970, the microstructure of materials was recognized to be the crucial parameter controlling many mechanical, magnetic and electronic properties. This line of thought seems to start with the pioneering work on the mechanical properties of iron alloys by Sorby, Tschernoff, Osmond, Howe, Sauveur and others before the turn of the century. Their studies led to the conclusion that the fine-scale microstructure retained after the allotropic transformation of iron alloys gives martensite its hardness. The discovery of precipitation hardening by Wilm in 1906 was the first observation suggesting that the correlation between microstructure and properties (originally proposed for ferrous alloys only) applies to non-ferrous materials as well. Wilm quenched an Al-Cu-Mg-Mn alloy and noticed (after a long weekend) a substantial hardening relative to the as-quenched state. However, it was not until 1919 when Merica, Waltenberg and Scott proposed that the hardening resulted from precipitation of a new phase on a sub-microscopic level. Numerous observations in the subsequent years substantiated and generalized this view and led to the classification of the properties of solids with different types of chemical bonding and to microstructure-sensitive and non-sensitive ones.

In the following years, this classification played an important role in promoting the idea of lattice defects and their significance for crystalline solids. In fact, the physical understanding of the mechanism by which ultrafine microstructures affect the properties of solids received a remarkable boost after the Second World War, from the advent of the theory of lattice defects—in particular, dislocation theory—and from the availability of new high-resolution research techniques such as electron and field ion microscopy. Both developments helped to elucidate the

physical basis for understanding the correlation between the structure-sensitive properties and the microstructure of solids. As a matter of fact, the development of most high-strength and high-temperature materials available today is based on the results of those studies. When it was recognized that dislocations play a similar role for mechanical behaviour of materials, as do domain walls or flux lines for ferromagnetic or superconducting properties, respectively, it became apparent that ferromagnetic and superconducting properties can be manipulated, too, by suitably varying the microstructure. In fact, the pinning of ferromagnetic domain walls or of flux lines in type II superconductors by finely dispersed precipitates leads to magnetic materials with high coercive forces and superconductors with high critical current densities. Instead of reducing the motion of dislocations by precipitates from supersaturated solid solutions, small second phase particles introduced by means of powder metallurgy or by extrusion of two-phase coarse-grained structures may be used as well. The enhanced defect density in irradiated, in highly cold-worked, as well as in fine-grained materials results in similar effects, since the defects inhibit the dislocation motion. A closely related approach in the field of ceramics generated by the sol-gel method was the proposal to use heterogeneity on a nanometre scale. This approach was based on the hypothesis that diphasic or multiphasic ("nanocomposite") xerogels which are heterogeneous on a nanometre scale store more energy than a single-phase gel and thus exhibit new properties.

About 1970, the second period of developments in the area of ultrafine microstructures started, when it was recognized that specifically tailored ultrafine microstructures permit the generation of solids with new atomic and/or electronic structures. These developments seem to bring Feynman's dream of nanotechnology closer to reality. Up to now, the following two types of solids with new electronic and/or atomic structures have been generated by means of tailored ultrafine microstructures. In the area of semiconducting materials, extensive research activities were initiated on a new two-dimensional class of materials with a nanometre-scale microstructure by the proposal of an "engineered" semiconductor superlattice, consisting of alternate thin layers with different dopings, different compositions leading to new electronic structures.

These superlattices represent an extension of double- or multibarrier structures where quantum effects prevail because the layers have a thickness of a few lattice constants. Several recent reviews of the present state of understanding of the physics and application of quantum well structures and superlattices have been compiled. A recent modification and extension of this approach is the alternating growth of coherent multilayer structures of components with large lattice parameter mismatch. This opens the possibility of combining superlattice effects with those associated with strain to design materials of suitable band gaps and electronic properties. By growing thin-strained layers of crystalline quality, one obtains binary materials, the so-called pseudoalloys. Pseudoalloys permit the combination of strain effects with quantum size effects. Two-dimensional semiconductor materials systems exhibit the technologically important features of high quantum efficiency and rapid carrier capture efficiency. An example of such a structure is a thin pseudomorphic layer of InAs embedded between $\text{In}_{0.53}\text{Al}_{0.47}\text{As}$ layers which are lattice-matched to an InP substrate. These heterostructures contain a thin-strained InAs quantum well. The strain modifies the properties of the resulting material. For example, the wave-

length of the light emitted by the InAs layer is shifted from its usual bulk value of $2.95 \mu\text{m}$ to the range of $1.2\text{--}1.6 \mu\text{m}$ depending on the layer thickness. Optical investigations of highly strained (3.4 per cent lattice mismatch) isolated InAs quantum wells show not only a controllable emission in the $1.2\text{--}1.6 \mu\text{m}$ range but also an increase in the photoluminescence, by a factor of 2.5, relative to the bulk InAs value.

Research activities aiming towards the generation of new atomic and electronic structures by means of ultrafine microstructures were stimulated by a proposal to generate solids (metals, semiconductors, ceramics), a large volume fraction of which consists of the cores of grain (or interphase) boundaries. Solids of this type were argued to differ structurally and propertywise from the crystalline or glassy state with the same chemical composition because grain boundaries and interphase boundaries represent a special state of solid matter due to the fact that the atoms in the core of an interface are known to be subjected to the (periodic) potential field of the crystals on both sides of the interface. As a consequence, the arrangements formed by the atoms in the cores of interfaces differ from the glassy and the crystalline state. In the glassy state, the atoms are not subjected to the (periodic) boundary conditions like the atoms in the core of interfaces. In the crystalline state, the atoms relax to a structure of lowest free energy which is prevented in the cores of interfaces by the fact that the interface represents the transition between two crystals of different orientations. Hence, the atomic structure and the properties of solids in which the volume fraction of the cores of interfaces becomes comparable to or larger than the volume fractions of the residual crystals differ from the structure and properties of the same materials in the crystalline or in the glassy state. Direct experimental and theoretical evidence supporting this idea comes from studies of the structure and properties of defect cores, in particular, of grain and/or interphase boundaries.

Up to now, the research activities on materials with ultrafine microstructures have resulted in several hundred publications on quantum well structures and superlattices. More than one hundred papers have been published dealing with the various aspects of the structure and properties of materials containing a high density of defects, preferably grain boundaries.

Perspectives

One of the basic problems that has to be solved in order to permit the technological application of materials with nanometre-sized grains is the availability of economical methods to produce large quantities of such substances. The present situation seems to be somewhat analogous to metallic glasses. Shortly after the discovery of metallic glasses, several studies revealed technologically attractive properties of these materials. Nevertheless, it was the development of the melt spinning method which opened the way to the technological applications, because it allowed the production of economically large quantities. Similarly, the present methods to generate nanocrystalline materials seem too expensive for large-scale production. In fact, there are many chemical methods known for synthesizing economically large quantities of small crystals. Perhaps one of them can be utilized in the future. A second area which seems to deserve attention is the thermal stability of ultrafine microstructures. The high energy stored in these structures provides driving forces for recovery and recrystallization processes. However, ultrafine microstructures can be thermally stabilized in the form of

multiphase materials consisting of crystals with different chemical compositions. If these crystals are mutually insoluble and if the various crystals are arranged in a non-percolative manner, thermally stable, ultrafine microstructures may be generated. So far, ultrafine microstructures formed by incorporating a high density of grain boundaries have been studied predominantly.

Obviously, ultrafine microstructures by incorporating a high density of dislocations or stacking faults, etc. are likely to exhibit interesting properties as well.

For more information contact: Universität des Saarlandes und Institut für Neue Materialien, Gebäude 43, W-6600 Saarbrücken, Germany. (Extracted from *Nano-Structured Materials*, Vol. 1, pp. 1-19, 1992)

6. APPLICATIONS

Small worlds

Nanotechnology is the next big thing. Or so its proponents would have us believe. All too regularly predictions are published, ironically with very big headlines, which say more about the topical concerns of modern Westerners than about an achievable future.

What many of the statements are really saying, among all the boasting about self-replicating devices a few atoms in diameter, is that we now know an awful lot about very small things. We even know how to move single atoms individually. In fact, IBM showed all of us who own scanning tunnelling microscopes how to do it in April 1990 at its Zurich research laboratory when it rearranged 35 xenon atoms into the smallest advertisement yet seen.

We also know what might be achievable with these atoms if current theories of quantum physics are correct and if we can work out ways of manipulating the atoms. So we have a starting point; we have dozens of maps showing all the nanotechnology destinations and the highways which will get us there. But we have not got a car. Not only have we not got a car but we have barely got the bus fare to take us to the next stop. Nanotechnology is further from becoming an industrial technology than anyone would like to admit.

Biological structures are cited as examples of the kind of things which could be made by nanotechnologists, who emphasize that these are simply very small, natural machines. Synthetic molecular machines, for example, have a diameter of 100 nanometres and are programmable so that they can assemble other molecules into the required design. This has been given an attractively alliterative name, molecular manufacturing. According to at least one commentary, this technique will eventually replace most of our current industrial base, inevitably leading to large changes in the way the economy works.

The rise of nanotechnology can be ascribed to one theoretician, Eric Drexler. As an undergraduate at MIT in the late 1970s he was trying to draw connections between such diverse subjects as the systems engineering aspects of complicated projects in the aerospace industry, molecular biology and molecular machinery. His thoughts were published in 1986 as *The Engines of Creation* and the word nanotechnology was first used in the book. Rarely does a new discipline create such interest.

Apart from the wave of technophilia which has swept the industrial world, fuelled by the massive and obvious influence of information technology, the swift rise of nanotechnology has also been helped by references to Richard Feynman. It was he who, in a speech in 1959, said he saw no reason why atoms should not be moved around individually to build structures from the bottom up, as opposed to taking larger structures and cutting them down until left with the desired bit.

Feynman made a prediction that took more than 30 years to come true. Enthusiastic nanotechnologists are saying that molecular machines will be storing data, building chemicals, eating pollution and creating energy. But this is unlikely to happen in the next decade, nor even in the next 30 years. So where is nanotechnology now?

Some of it is out in space. The Space Vacuum Epitaxy Center (SVEC) in Houston creates advanced thin film materials and devices for commercial application through vacuum growth technologies using terrestrial and space vacuum environments. SVEC explores materials processing techniques using the ultra-vacuum of space because an ultra-vacuum allows for the critical reduction of contaminants in thin film materials. SVEC aims to produce new industry-driven electronic, magnetic and superconducting thin film materials and devices.

Some of it is in medicine. Recently researchers at Northwestern University, IL, showed how to cover tiny pellets of metal with graphite in a totally airtight fashion. Electron microscope pictures of the pellets, which measure less than a millionth of an inch across, show layers of single atoms of graphite running around them. Instead of being hollow tubes, the spherical layers totally encapsulate the round metal pellets. The researchers believe the capsules could be used in a wide range of applications, including medicine, since they could travel through the human bloodstream without ever exposing the body to the metal contained inside.

The research team discovered that the capsules, called encapsulated nanoparticles, form spontaneously when graphite is present during the process of vaporizing the metal particles using a tungsten-based electric arc. The capsules could be useful in magnetic resonance imaging (MRI), giving greater clarity to the MRI images inside the body, the researchers say, and in anti-cancer therapy and cell separations, which currently use larger magnetic particles. The capsules have a number of other potential uses, such as for magnetic strips on credit cards, magnetic ink on cheques and in cassette tapes.

Some nanotechnology research aims to harness the effects of quantum structures for use as electronic storage devices, for new types of computer. Researchers at the University of California, Santa Barbara have created a quantum wire superlattice that contains a network of parallel wires so tiny that more than six million would fit within a human hair. These wires proved small enough to restrict electron motion to a single dimension, generating new and potentially useful quantum properties. Moreover, in the process of creating the superlattice, the scientists made an unexpected discovery: when a mixture of equal amounts of the two compounds used is deposited on atomic stair-step surfaces, such as the one on which the superlattice is built, the substances organize themselves into alternating wafer-like bands of materials.

While quantum manipulation is, strictly speaking, an order of magnitude smaller than nanotechnology, microtechnology is one order larger but it is here that the greatest advances are being made. Researchers at the Sandia National Laboratories have built the first micro-motor to drive external gearing and they have made it entirely by microelectronic fabrication techniques—etching silicon.

The millimetre-square engine and its even tinier gearing has two holes to function as an optical shutter—a

beam of light can pass through its openings in some positions but not others. The smaller gear turns two similarly sized gears at a rate of 200,000 revolutions per minute. So far, several hundred million rotations have been demonstrated by the smaller gears. The motor, which develops 0.5 microwatts of power delivered through a gear 50 microns in diameter, could be used to operate tiny micromedical pumps that function as drug delivery systems inside the human body and to act as low-cost, high-performance gyroscopes that could have a dramatic impact on the design of future automobiles and military systems. (Source: *Engineering*, January 1996)

Carbon connections promise nanoelectronics

As the semiconductor industry inches towards integrated circuit technology based on silicon chips with features just 200 nm wide, researchers are trying to build carbon-based electronic devices that are hundreds of times smaller. These devices would be based on the remarkable electronic properties of carbon nanotubes and the junctions between dissimilar nanotubes.

Single-wall nanotubes were first proposed in 1991, when several groups suggested that a graphene sheet—a single atomic layer of a graphite crystal—could in principle be rolled into a cylinder about 1 μm long and 1 nm in diameter. Since the cylindrical nanotubes are conceptually terminated by half a fullerene molecule at each end, it was postulated that the smallest diameter for a nanotube is about 0.7 nm, equal to the diameter of the C_{60} molecule.

In fact the first nanotubes to be produced in the laboratory, in 1991, were multiwall structures made up of nested coaxial tubes. But such multiwall tubes are difficult to model theoretically, unlike single wall nanotubes, which can be defined by two integers (n , m). The carbon atoms within the cylinder are all found at the vertices of sixfold rings of a regular hexagon.

Two events greatly stimulated interest in single-wall carbon nanotubes. The first came in 1992, when researchers predicted that carbon nanotubes could be either metallic conductors or semiconductors, depending on their diameter and the relative orientation of the hexagons and the axis of the tube axis. This orientation is uniquely specified by (n , m). Then, in 1993, two groups successfully synthesized single-wall carbon nanotubes using a carbon arc discharge with a small concentration (about 4 per cent) of iron or cobalt catalyst. To date, however, there has been no direct experimental verification of the remarkable electronic properties of single-wall carbon nanotubes, although such properties have been observed in multiwall nanotubes.

Qualitative proposals for using carbon nanotubes in device applications quickly followed. Some groups envisioned inserting a metallic nanotube inside a semiconducting tube to form a shielded nanowire or perhaps even a nanometer coaxial cable. Others proposed using a semiconducting nanotube joined to a metallic nanotube as the basis for a heterostructure. This would act as a switch when appropriately perturbed in the junction region (e.g. by an optical pulse or the probe of a scanning tunnelling microscope). Nanodevices were also envisaged for rotating the polarization of an electric or magnetic field, or as a nanometer-sized semiconducting memory element.

Of these proposals, the heterostructure device attracted particular attention. This was due in part to the importance of heterostructures in the semiconductor industry and also to the experimental observation using high-resolution trans-

mission electron microscopy that nanotubes with different diameters could be joined.

The first theoretical modelling of the electronic properties of the nanotube junction was performed by researchers at the University of Notre-Dame de la Pais in Namur, Belgium. Independent calculations were carried out jointly by MIT and a group at the Electro-Communications University in Tokyo.

All three groups used different approaches to the calculation and studied complementary examples of nanotube junctions. However, all found that the semiconductor-metal junction of such a nanotube behaves sufficiently similar to the Schottky barrier, which is widely used in modern semiconductor devices, for applications to be envisaged.

More specifically, the electronic properties of metal-metal, metal-semiconductor and semiconductor-semiconductor junctions between two dissimilar nanotubes were considered, including junctions with and without bends. The density of electronic states was found to be essentially unchanged for the nanotubes on either side of the junction, except for the electronic states associated with a small region containing about three rows of hexagons near the junction. No localized states appear in the junction so there is no resonant tunnelling to localized junction states.

Tunnelling effects are predicted in all cases, which is similar to observations in conventional semiconductor quantum well heterostructures. Since the nanotubes under consideration have very small diameters, no significant electron scattering due to impurities is expected in the periodic region.

A number of recent experimental developments suggest that devices based on carbon nanotubes may be realized. At the American Physical Society meeting in St. Louis in March 1996, researchers at Rice University reported a new method based on laser ablation for synthesizing 10 μm long single-wall carbon nanotubes with diameters of 1.2-1.5 nm. A collaboration between Belgian groups in Louvain-la-Neuve and Leuven, and General Motors in the US, has succeeded in making four electrical contacts to an individual multiwall nested nanotube 50 nm in diameter. This result suggests that it may be possible to attach electrodes to very small single-wall carbon nanotube devices that are less than 1.5 nm in diameter. The ability to attach leads also suggests the possibility of applying a perturbation normal to the junction to produce switching action, as suggested by various authors.

Computational capabilities have now advanced to the point where an electronic structure calculation can be carried out for the 10^4 carbon atoms contained in two joined dissimilar nanotubes about 1 nm in diameter and 1 μm long, providing a self-consistent solution for the current density, charge density and voltage drop across the junction. Such calculations will provide a detailed understanding of the non-equilibrium transport in such nanotube junction systems and will determine whether the transport in these systems is ballistic or diffusive.

These recent experimental and theoretical findings point to the possibility of a host of exciting new device applications of nanometer dimensions and a large number of extremely challenging experimental and theoretical problems that must be solved before these applications of carbon nanotubes can become a reality. (Source: *Physics World*, May 1996)



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For more information, contact:

Mr. V.E. Ubalde, Senior Manager, Operations Engineering, Picop Resources, In. Center Point Bldg, J. Vargas cor. Garnet St., Ortigas Center, Pasig City, Philippines. Fax: +63 2 633 9959

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