

Material

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MATERIALS ENGINEERING WITH NATURE'S BUILDING BLOCKS

This article provides a brief overview of the major events, science, tools and processes in nanotechnology. Part One highlights the history of nanotechnology, Part Two discusses the tools used to study such small-scale materials and the underlying mechanisms responsible for the novel properties observed, and Part Three covers the major processing methods of nanomaterials.

Part One: History of Nano Materials Engineering

Nanotechnology is definitely one of the most talked about "new" areas of study today. The idea that nanostructured materials would exhibit unique properties is accredited to Gleiter and Turnbull for the work they did independently in the early 1980s [1]. Since then the field of nanotechnology has expanded dramatically due to the realization of its potential technological benefits.

Nanotechnology basically is the process of manipulating matter at the atomic scale. There are many interpretations of what this includes, but for our purposes and our inherent focus on materials engineering, we will include the purposeful creation, manipulation and machining of engineered materials at the nanoscale. Typically, nanomaterials have some critical dimension more than 1 and less than 100 nanometers, where a nanometer is one billionth of a meter, or one thousandth of a micron. The typical "yardstick" at small scales - a human hair - is about 200,000 nanometers in diameter. (A more thorough description of nanoscale is included in Part Two.)

Humans have been utilizing nanomaterials for centuries, dating back to Roman potters and possibly before. Ancient craftspeople utilized nanoscale particles in glazes to create unique colors that changed with incident lighting. In the 19th century, the use of metals in new and astounding structural applications drove the importance of microstructure to the front of engineers' minds. With this focus, empirical methodologies for processing metals yielded massive improvements in strength, toughness, ductility and hardness. Much of this work was microtechnology, even before the field existed, and one could argue that it bordered on the nano regime with the empirical manipulation of microstructure, precipitates and crystallographic grains.

Eventually, innovations such as the automobile demanded more durable consumables like rubber, glass, and ultimately, plastics. Rubber may have been the first modern example of nanotechnology, with the addition of carbon black and sulfur to improve durability. Even though the ability of engineers to study the exact mechanisms at work did not exist, carbon black's nanoscale particles served to modify the behavior of rubbers, thus assisting the growth of one of the most important manufacturing booms of the late 19th and early 20th centuries. While nanotechnology claims its roots in chemistry, the fields of physics and engineering have joined the fray and are pushing the boundaries of the possible.

Many trace the roots of modern nanotechnology and nanomaterials to a 1959 talk given by Dr. Richard Feynman at a meeting of the American Physical Society.* It was in this now-famous lecture that Feynman proposed the all-too-

simple hypothesis that there was "plenty of room at the bottom." He speculated that future scientists and engineers would build complex structures from atoms and molecules.

It wasn't until 1974 that the field was given the name "nanotechnology" however, and that is widely attributed to University of Tokyo researcher Norio Taniguchi. He made the distinction between engineering at the micrometer scale (the basis for modern microelectronics that was just starting to hit its stride in the 70s) and the new field of sub-micrometer engineering that was beginning to emerge†[2]. Nascent nanotechnology began to "grow up" and enter the mainstream consciousness in the mid-1980s with work by Richard Smalley at Rice University on what would be called Buckminster Fullerenes (now famously known as "Buckyballs") and MIT researcher K. Eric Drexler's publication of "The Engines of Creation."

While Smalley tantalized chemists and physicists with the discovery of a "new" form of one of the building blocks of nature, Drexler outlined a future dominated by a new form of manufacturing done at the molecular, and even atomic level. The study of Buckyballs has led to the discovery of tube-like structures of carbon atoms which are basically sheets of graphite rolled up with their edges connected to form a cylinder. They can be thousands of times longer than they are in diameter. It is these carbon nanotubes (CNTs) which have piqued the interest of so many engineers as they may hold the promise of extremely high tensile strength inclusions for nanocomposites, structural beams for nanomachines, and possibly even conductors (wires) for nanoelectronics. CNTs and other molecular-level structures form the bases of what Drexler describes as a coming revolution in molecular manufacturing.

What is most amazing about the development of micro- and then nanotechnology is the pace at which it has advanced. Taniguchi predicted in 1974 that within 15 years there would be machining methods capable of sub-100 nm dimensional precision. This prediction was largely proven correct. By the early 1990s, nanotechnology's capabilities could not only image and probe atomic structures, but move individual atoms, one at a time, around on a substrate. The "quantum stadium" image generated by IBM researchers provides a poignant example of this in Figure 1. The image consists of 76 iron atoms on a copper substrate. Wave patterns in the interior are from the density distribution of trapped electrons.

Since 1990, significant advancements have been made, both in actual materials fabrication and in computational simulation. The manufacturing of CNTs and many other nanomaterials has been improved and scaled up,

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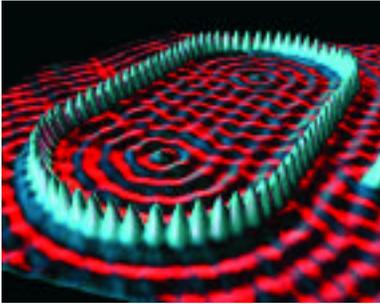


Figure 1. Quantum Stadium
(Courtesy: IBM, Almaden
Research Center. All rights reserved)

making affordable materials available for test and evaluation which did not exist just 5 years ago. Computer power has improved dramatically over the past 10 to 15 years, enabling atom-level simulation of many new nanomaterials before significant resources are invested in producing them. Researchers are now able to theorize, simulate,

manufacture, test, and evaluate new materials in much shorter times, further expanding the field and reducing wasted effort. While many of the ideas surrounding engineering at the atomic and molecular level are still in a highly speculative stage, experts agree that there seem to be no physical laws barring further advances.

Many of the advancements in nanotechnology were made possible by concurrent advancements in analysis tools capable of resolving atomic, molecular and crystallographic structures. Just in the last 20 years, various forms of scanning probe microscopy (SPM) and transmission electron microscopy (TEM) have become available to researchers on a reliable basis. These tools, described more fully in Part Two, have provided views into the inner workings of atomic bonding, molecular assembly, and the structure of materials at the smallest scales in history. It is these tools that have taken atomic manipulation out of the chemistry beaker and into the realm of engineering.

Part Two: The How and Why of Nanotechnology A Sense of Scale

To grasp what nanotechnology encompasses, it is critical to have a mental concept of what “nano” really means. Roger Whatmore of Cranfield University in the UK provides an excellent example in which he proposes that one imagine a human hair is roughly the size of a large tree trunk, one meter across. Now a typical bacterium (1 micrometer) would be about as big as a caterpillar on that tree and a typical virus (which is about 100 nanometers long) would be the size of an ant. And that is where nanotechnology is just getting started. Figure 2 presents an overview of items from the macro to the nanoscale. One can see that multiple technical disciplines of chemistry, physics, engineering, biology and others converge as we approach the lower reaches of size.

Analysis and Tools

In order to study materials on the nanoscale, instruments capable of very fine resolution must be utilized. Many of the microscopy tools used for characterizing nanostructures can easily fit inside a shoebox. Advances in microscope probe tip technologies have come about from the study of nanostructured materials, creating even better tools for characterization. Figure 3 compares some microscopy techniques and illustrates the incredible recent growth of tools with micro- and then nanoscale resolution.

Scanning Probe Microscopy One of the first tools used for nanomaterials investigation was the scanning tunneling microscope, or STM, developed by Gerd Binnig and Heinrich Röhler at IBM in 1981. (They would win a Nobel Prize in 1986 for this work.) The STM formed the basis for what would later become a family of scanning probe tools, or scanning probe microscopy (SPM), the key features of which are depicted in Figure 4.

Scanning probe microscopes use an atomically “sharp” tip (typically pyramidal in shape and coming to a point that is literally one or two atoms across) which is moved over a sample’s surface a few Angstroms from making contact. The tip is then scanned across the surface in a regular pattern (much as one would mow a lawn) and the electrical current, which traverses the gap between the tip and the sample, is monitored. By keeping the height of the tip constant and measuring the changing electrical current (more when close to the sample, less when farther away), or by keeping the current constant and measuring the deflection of the tip, a topographical map can be generated of the sample’s surface. The device relies upon the small amount of electric current which crosses the gap between the tip and the sample (“tunneling”).

The other most widely used member of the SPM family is the atomic force microscope or AFM. Similar to the STM, it uses an atomically sharp tip, but instead of carefully moving it just Angstroms from the sample, the AFM actually drags its tip along the sample’s surface. (This exaggerates the interaction at this scale - the atomic forces actually prevent atoms from “dragging,” they simply are pushed into very close proximity.) The tip is mounted on a cantilever

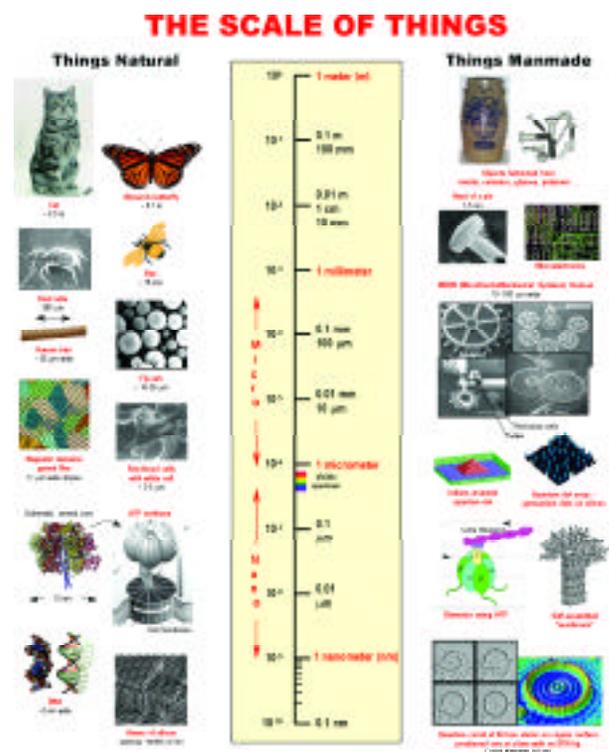


Figure 2. Scale of Common Natural and Manmade Items
(Reprinted with permission of the Office of Basic Energy Sciences,
Office of Science, US Department of Energy)

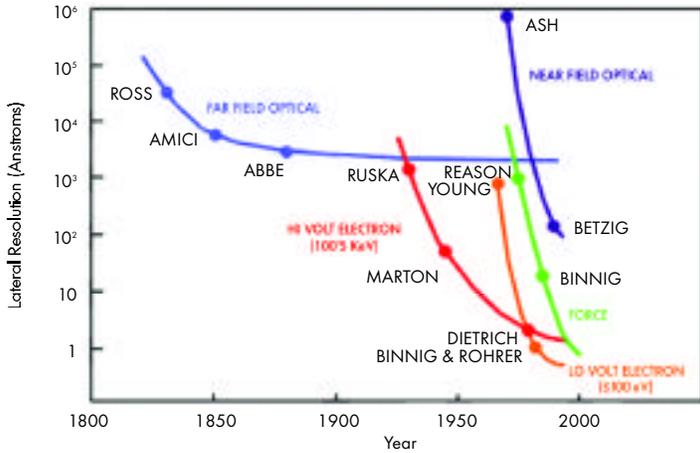


Figure 3: History of Lateral Resolution in Microscopy
(Courtesy of J. Murday, Naval Research Laboratory)

which flexes as the tip moves up and down the surface topography. Its deflections can be tracked by bouncing a laser beam off the cantilever into a split photodiode or by other techniques.

Magnetic Force Microscopy (MFM) is another technique of SPM, but uses a magnetic probe to sense the magnetic field above the surface of a material. The probe is standard silicon- or silicon nitride-coated with a thin magnetic film, such as cobalt, capable of mapping the magnetic domain of a specimen with a resolution up to 20 nm. Two passes are required to distinguish the topography of the sample. The first pass is in contact or semicontact with the sample and the second pass is at a specific constant height above the first pass.

Electron microscopes have been instrumental in the study and characterization of materials since the invention of the first electron microscope in the 1930s. High resolution transmission electron microscopy can now provide structural characterization at better than 0.2 nm spatial resolution. In its simplest form, a TEM consists of an electron beam that is projected through a thin sample, generating a diffraction pattern onto a receiving device, such as a phosphorescent screen or CCD camera. At low resolution, amplitude contrast images are used to map material structures in the 0.5 nm to 1 mm range. Specimens must be < 10 nm thick to provide the highest resolution

Nanoscience

The promise of nanotechnology is based upon the ability to create nanostructured materials that will produce novel properties on the macroscale. That ability now exists, however in many cases the mechanisms behind these observed properties are not yet clearly understood. Despite this incomplete understanding, it is still possible to attribute, by inference, the novel qualities of these nanomaterials to the following changes in internal structure.

1. The increased total surface area of grains, as grain size is decreased, will alter the physical properties of nanomaterials.
2. The increased volume of grain boundaries relative to unit structure, as grain size is decreased, will alter the physical properties of nanomaterials.
3. Discrete electronic levels (quantum behavior) will alter the electrical and optical properties of nanomaterials as grain sizes approach the molecular scale. The discrete energies associated with electron orbits become more evident as grain size nears the molecular scale (approximately < 5 nm), creating non-linear property effects.

Mechanical Properties The most widely accepted hypothesis to explain the mechanical properties in polycrystalline nanomaterials is that the strength and hardness follow the Hall-Petch relationship, increasing with smaller grain size, down to a critical grain size, $d_c \approx 10$ nm, where a decrease in strength and hardness results thereafter, as depicted in Figure 5 [3].

The increase in strength is based upon the piling up of dislocations at grain boundaries; i.e. by increasing the total surface area of grains, the dislocation density increases and in turn increases strength. However, pileups cannot occur when the grain size is less than the dislocation spacing in the pileup [4]; and so the Hall-Petch relationship will no longer be valid on this scale. The strength of nanoscale-thickness, multi-layered materials also follows the Hall-Petch relationship, replacing grain size with layer thickness.

By decreasing grain size, the grain boundary volume relative to the unit volume will increase. At roughly 5 nm, 50% of the volume will be grain boundaries [5], which may then dominate the properties observed in the material. The decrease in strength at this small end of the nanoscale may be attributed to grain boundary sliding due to the high defect density allowing fast diffusion of atoms and vacancies in the stress field [6]. Superplasticity [+] has been observed in nanostructured metals and ceramics at about 200°C lower than microstructured materials. This creates improved formability of nanostructured materials, which is especially important for ceramics.

Thermal Properties The enhanced diffusivity observed in the grain boundary structure of nanostructured materials is the mechanism thought responsible for the changes in thermal properties. In metals, thermal conductivity and melting point have been observed to decrease (such as a 27°C lower melting point for gold) while thermal expansion coefficients have been observed to increase. The decrease in thermal conductivity of nanostructured ceramic materials such as yttria stabilized zirconia may broaden their use as thermal barrier coatings.

Chemical Reactivity An increased chemical activity can be obtained by the large number of atoms on the surface of nanocrystallites providing active sites for reactions. For instance, nonstoichiometric CeO_{2-x} has a high chemical reactivity resulting from an unusually high oxygen vacancy concentration [7]. Nanostructured CeO_{2-x} has been demonstrated to offer catalytic activation for SO_2 reduction and CO oxidation at significantly lower temperatures as well as enhanced poisoning resistance (resistance to the loss of catalytic reactivity).

A second class of reactive materials are nanostructured porous materials. Conventional porous materials such as aluminosilicates and phosphates are

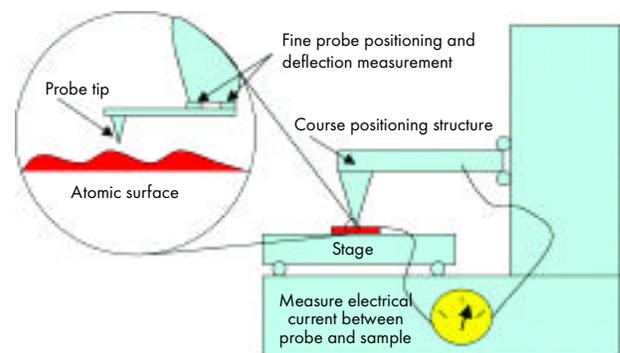


Figure 4: Graphical Depiction of the Key Features of an SPM

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used for catalytic reactions and gas absorption applications, but they are limited due to their small pore sizes of less than 15 Å [8]. Nanostructured aluminosilicates with optimized pore sizes in the range of 20–100 Å have been developed which extend their use in these markets. Similar research is being applied to the use of transition metal oxides in petrochemical production, pollution control, and pharmaceutical and fine chemical synthesis. Extending the pore sizes beyond the 20–100 Å range could provide benefits for enzyme catalysis, bioseparation and biosensing.

Optical Properties The size of dispersion materials in a composite can alter the wavelength of light that is absorbed by the particulates. The quantum effect is responsible here where the discrete electron energies in the particulates determine the wavelength of light absorbed. Altering the size of a particle can change the associated energy and wavelength of light absorbed. Cadmium selenide is one material extensively studied where crystallites of ~ 1.5 nm will appear yellow, 4 nm will appear red, and larger particles will appear black [9]. This discovery is now being applied to sunscreen lotions using zinc oxide and titania to filter ultraviolet radiation.

Electrical Properties Quantum effects in nanomaterials have been shown to produce a non-linear dependence of electrical conductivity on electric field and can produce electron tunneling characteristics. Quantum mechanical interference between separate paths an electron may take through a material can strongly enhance or suppress electrical conductivity. Most of the research to date in nanostructured electronic devices comes from fabrication via lithography, but inroads are being made into molecular-level structures fabricated from chemical reactions or self assembly. For instance, quantum dots are pyramid- or faceted dome-shaped clusters of atoms, typically between a few nanometers and hundreds of nanometers in diameter. They self-assemble from the deposition of a large lattice constant material onto a substrate with a small lattice constant, whereby the compressive strain in the deposited material relieves itself by causing the material to spontaneously coalesce or clump into islands on the substrate. They may be utilized as three-dimensional potential wells if they are overgrown by a material with a larger bandgap and have applica-

tion potential in nanoelectronics. (See the article in this issue by Dr. Amirtharaj et al for further information and application of quantum dots.)

CNTs (the tubes formed from rolled up sheets made of carbon atoms joined in hexagonal arrays) have shown some interesting electrical properties as well. The way in which the two ends of the sheet wrap and meet can modify the longitudinal conductivity of the tube. In fact, they can be made to conduct freely like metals or behave like semiconductors. And further advancements have shown that various-sized tubes can be nested inside one another for structural or electrical performance modifications.

Magnetic Properties Magnetic nanoparticles exhibit unusual behaviors resulting from the size effects and charge transfer characteristics. Changes seen in these materials include Giant Magneto-Resistance (GMR) in some layered composite materials and granular solids, spin valves, and spin injection in ferromagnet/insulator/ferromagnet sandwich materials.

Part Three: Processing of Nanomaterials

Bulk Methods

A number of processes are used for producing nanomaterials in bulk powders, coatings, thin films, laminates, and composites. However, there are two fundamental approaches to fabricating nanomaterials. The “bottom-up” approach represents the concept of constructing a nanomaterial from basic building blocks, such as atoms or molecules. This approach illustrates the possibility of creating exact materials – materials that are designed to have exactly the properties desired. The second approach, the “top-down” method, involves restructuring a bulk material in order to create a nanostructure.

INERT GAS CONDENSATION Considered a “bottom-up” method, inert gas condensation (IGC) was the first method used to intentionally produce a nanostructured material, and has become widespread. It can be used to produce nanostructured metals, alloys, intermetallics, ceramic oxides, and composites. The process begins by energizing a source material, which generates a vapor of atoms. This evaporation process can be performed through electron beam heating, laser ablation, sputtering, or plasma methods, but is most often done through Joule-heating. Due to condensation, the vaporized atoms agglomerate and form very small clusters when they are introduced to the inert gas. These

clusters are carried by a convection current, induced by thermophoresis, to a tube cooled by liquid nitrogen where they accumulate.

Adjusting certain parameters, such as the gas used (i.e. He, Ar, Kr, Xe), the gas pressure, the precursor evaporation rate, and the residence time, allows some control over the particle size and distribution. The smallest particle sizes produced using this method are about 5–25 nm using a precursor with a low evaporation rate

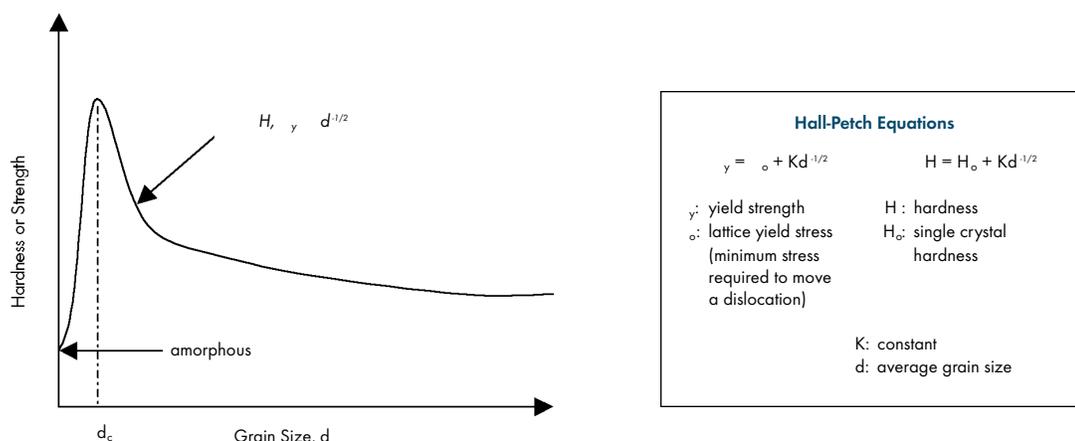


Figure 5: Strength/Hardness of Nanostructured Materials [3]

and a light inert gas at low pressure [10]. Since the process takes place in ultra-high vacuum systems (UHV), there is very little contamination of the material, and therefore, it remains highly pure.

MECHANICAL ALLOYING Another process commonly used to produce nanostructured materials is mechanical alloying, which is an example of a "top-down" method. There are two basic versions of this process. The first, more specifically called mechanical attrition, is a process where high-energy ball milling is used to grind and refine the structure to produce highly mixed, ultra-fine powders. Grain sizes of the material are partially dependent on time, where extended milling times result in more uniform grain sizes. The second version, reaction milling, involves in-situ solid-state chemical reactions between precursor materials while they are mixed and milled.

Producing large quantities of nanostructured material is one advantage of the mechanical alloying process. Moreover, various types of nanomaterials, such as metals, ceramics, intermetallics and composites, can be produced using these straightforward processes. However, contamination from the milling hardware and environment, non-uniform particle sizes due to short milling times, and non-homogeneous chemical composition due to incomplete milling reactions are problems for mechanical alloying processes. One way to lessen the effects of environmental contamination is to perform the process in the presence of liquid nitrogen, which is also referred to as cryogenic milling.

SEVERE PLASTIC DEFORMATION Severe plastic deformation (SPD) is used to fabricate nanocrystalline metals and intermetallics. There are several different types of SPD processes, but the three most common processes are equal-channel-angular extrusion (ECAE), torsional straining and accumulative roll-bonding (ARB) [10]. Nanosized grains are created when the material is subjected to a very large deformation causing a modification of its structure by fragmenting the existing phases. Recrystallization of these broken phases results in structures with significantly reduced sizes. The materials formed by SPD generally have average grain sizes of about 100 nm, although grain sizes down to 20 nm have been obtained for metals [11]. The grain size controlling parameters in this process include temperature, strain, strain rate, and applied pressure. Severe plastic deformation is capable of forming nanostructured materials with little contamination and little or no porosity. A further advantage is that SPD is a scalable process, meaning that it could be used for industrial applications. However, since there is significant straining of the material, high residual stresses may be present in the end product.

SOL-GEL The sol-gel method is a solution phase processing technique that is used more often to fabricate nanostructured materials than any other liquid phase process. This is a process capable of producing nanostructured ceramics and nanocomposites. There are essentially two phases present during this technique, where one is a homogeneous solution phase and the other is an elastic, gel-like, solid phase. The homogeneous solution is dried, thereby transforming it into a gel, while maintaining a constant volume. Subsequent drying causes a phase transformation of the gel along with a corresponding reduction in volume, ultimately resulting in the desired phase. The key to obtaining a nanostructured material using this process is to control the processing parameters. One advantage to solution phase processing is the excellent control it provides over the chemical composition, which leads to a more homogeneous composition [10]. A disadvantage to this process is that the starting materials can be expensive.

Coating and Laminate Methods

Nanostructure coatings have been primarily deposited via thermal spraying. Processes used to fabricate laminates include: Radio Frequency sputtering, DC magnetron sputtering, chemical vapor deposition, electroplating, and physical vapor deposition methods including electron beam, cathodic arc, and jet vapor deposition [12].

THERMAL SPRAYING Thermal spraying is a process capable of producing nanostructured materials and nanostructured coatings. Control over the composition and structure of the final material is very important when producing nanostructured materials, and for this reason powders are typically used as the starting materials. The powders are carried by a gas (i.e. air, N₂) and heated such that they are at least partially melted. Deposition onto a substrate results in the deformation and solidification of the particles.

ELECTRODEPOSITION Electrodeposition has traditionally been used as a coating process, although recently it has also been used to form bulk nanocrystalline materials. This process is capable of producing a nanocrystalline material with grain sizes down to 5 nm. The factors affecting the resulting grain sizes include pH, temperature, current density, and type of current among others [10]. The process can be carried out at room temperature and the time of fabrication is of course dependent on the size of the material to be produced. For example, as one would expect, larger materials take longer, but compared to other deposition processes, electrodeposition is capable of rapid deposition. Moreover, electrodeposition is a less expensive process that can be used for large-scale production [10].

JET VAPOR DEPOSITION Jet vapor deposition is a relatively cost-effective technique capable of rapidly depositing multilayered films with alternating material layers to produce a nanostructured composite [13]. Nanostructured multilayered films consist of layers with nanoscale thickness. Sonic or nearly sonic gas jets, typically helium, carry an atomized material and deposit it as a film on a substrate. Uniform deposition occurs with a rotating or oscillating substrate which is mounted on a carousel. Alloys and multilayered composites are formed by employing two or more jets simultaneously or in sequence, respectively. An optimum deposition temperature needs to be maintained to ensure minimal interdiffusion between the layers and a continuous nature of the layers without impurities forming in between.

SPUTTERING Sputtering is a well-known and widely used technique for producing thin films. It is a vacuum method capable of producing nanostructured materials. In this technique, plasma, which is generated by energizing a low-pressure gas such as argon, strikes a target transferring its momentum which causes the target material to be ejected and subsequently deposited onto a substrate. The main disadvantage to the sputtering method is that it is difficult to control. DC-magnetron sputtering and RF-diode sputtering have been used to prepare nanolaminate materials. These methods are capable of depositing layers of a material with a thickness below 100 nm.

CHEMICAL VAPOR DEPOSITION The process of chemical vapor deposition (CVD) involves a gas-phase chemical reaction which forms a solid material on a substrate. Nanostructured ceramics and composites are the most common types of nanomaterials produced by CVD (Carbon nanotubes are also fabricated using CVD). Vapors of the precursor materials are transported by a carrier gas to a heated substrate where they are deposited and subsequently react with the substrate to form a solid material. Although it is a relatively slow process, chemical vapor deposition offers good control over the chemical

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composition and is capable of deposition over a large area. There are several variations of the CVD process, such as plasma assisted CVD and laser assisted CVD.

CHEMICAL VAPOR INFILTRATION Chemical vapor infiltration (CVI) is a technique that is used to form nanostructured fiber-reinforced composite materials. A very similar process to CVD, CVI uses a transporting gas to carry vaporous precursors to a porous fiber preform, while diffusion takes the precursors inside where they react and form the nanostructured matrix. An important issue with CVI is ensuring that the outer parts of the fiber preform are not sealed off prior to the interior being fully deposited. To prevent premature surface densification, the chemical vapor infiltration process is slowed by adjusting the process parameters, such as temperature, pressure, and reactant concentration. Another factor to CVI being a slow process is that it is limited by the diffusion mechanism of the gaseous precursors. The advantage to CVI is that it is capable of producing a fiber-reinforced composite without damaging the fibers, which is often a problem in other composite processes.

CHEMICAL VAPOR CONDENSATION Another chemical vapor process used to produce nanomaterials is chemical vapor condensation (CVC). A precursor vapor carried by a gas stream is sent through a heated (hot-wall) reactor tube, where the pyrolysis of the precursor occurs to form nanoparticles. The nanoparticles are then collected on a liquid-nitrogen cooled tube. The CVC process is a low-pressure technique that requires a low concentration of the precursor in the carrier gas stream. Process parameters such as temperature, pressure, and residence time can be adjusted to determine the size of the nanoparticles. A variation of this process is the combustion flame version which replaces the hot-wall reactor with a combustion flame reactor. The combustion flame reactor supplies a significantly higher temperature which increases the reaction rate.

For further coverage of the subject matter, the interested reader can find more information on nanostructured materials in "Nanomaterials: Synthesis, Properties and Applications" [14].

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† Norio Taniguchi shows the trend in ultraprecision machining from 1900 and projects that it will reach "Atomic lattice distance" (0.3 nanometers) around 2017.

+ Superplasticity is the ability of a material to exhibit large ductility, usually > 200%.