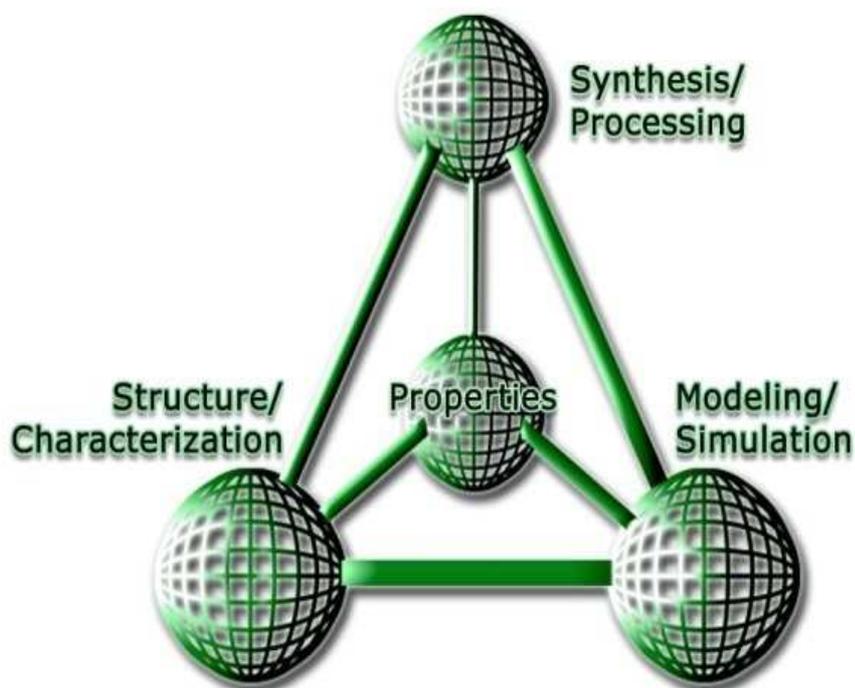


Progress in Fibrous Material Science



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Progress in Fibrous Material Science



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PREFACE

The current book is the first in a series of manuscripts under the title “PROGRESS IN FIBROUS MATERIAL SCIENCE”. These books are collection of chapters dealing with state of the art technologies and the science of polymeric materials in specific.

Research in this area primarily focuses on efforts to design materials at a molecular level to achieve desirable properties and applications at a macroscopic level. With this broad focus, research ranges from fundamental scientific investigations of the interactions, properties and assembly of such molecular constituents to applied, engineering efforts that translate such fundamental information to futuristic technological advances.

Much of the research is highly interdisciplinary, involving collaborations with chemists, biochemists, materials scientists, mechanical engineers and biologists. Moreover, most of the researchers and graduate students are affiliated with Department of Material Engineering, Faculty of Textile Engineering, Technical University of Liberec, facilitating the use the world-class equipment and resources established in the laboratories.

The first section deals with “ADVANCED FIBRES”. There have been considerable changes in the nature of the fibres being produced, the production methods and in consumers' values and expectations. Since 1990, the march of high-tech fibres has continued, with an ever increasing sub-division to meet specialized applications, as in high performance, high-function and high-sense fibres. New research and development has produced fibres with high tenacity and modulus to give the super-fibres now used as industrial materials. The more aesthetic and comfortable modern lifestyle has given rise to improved fibres. The special feature of this section is not only in giving an understanding and account of known possibilities, but also in describing the invention of new fibres and fibrous materials with unexpected and interesting properties and applications.

The second section is “ADVANCED NANOMATERIALS”. It is a collection of highly selected, peer-reviewed chapters; this showcases the research of an international standard. It covers nanomaterials with emphasis on synthesis, characterization, and applications. It also presents emerging developments in nanotechnology in areas as diverse as textile materials themselves. In addition to engineering aspects, the chapters discuss the physics, chemistry and biotechnology behind the fabrication and designing nanoscale technology in fibrous materials.

Third section is focused to “ADVANCED THERMAL INSULATION MATERIALS”. From the fundamental point of view, the measurement of the temperature variation of the thermal conductivity of a crystalline solid allows the investigation of structure defects, phonon-phonon interactions and eventually, in good electrical conductors, electron-phonon interactions. Textile materials have found a range of applications in the field of thermal insulation. Combination of different types of fabrics with various coatings and treatments are being studied to understand and improve the effectiveness of textile materials as thermal insulators. In this regard, measurement of thermal properties of textile materials plays an important role. It is imperative that we study the standard measurement techniques to best understand their strengths and weaknesses and also endeavour to fabricate and try new equipment to compliment or replace existing measurement techniques and equipment. The

section also includes simulation technique used for prediction of thermal insulation behavior.

The fourth section titled “MISCELLANEOUS FIBROUS MATERIALS” is a combination of diversified fibrous materials used in a wide range of applications. One chapter is dedicated to textiles used in book binding. Second chapter is about basalt woven hybrid fabrics and their applications in composite reinforcement. The potential of 3D spacer fabrics in multifunctional applications is discussed in the 3rd chapter. Image processing and Plasma technology used in fibrous materials is discussed in some details.

The editors and the authors have put their best efforts to enable the reader to have a broad understanding of these advanced fibrous materials aimed at futuristic areas of application.

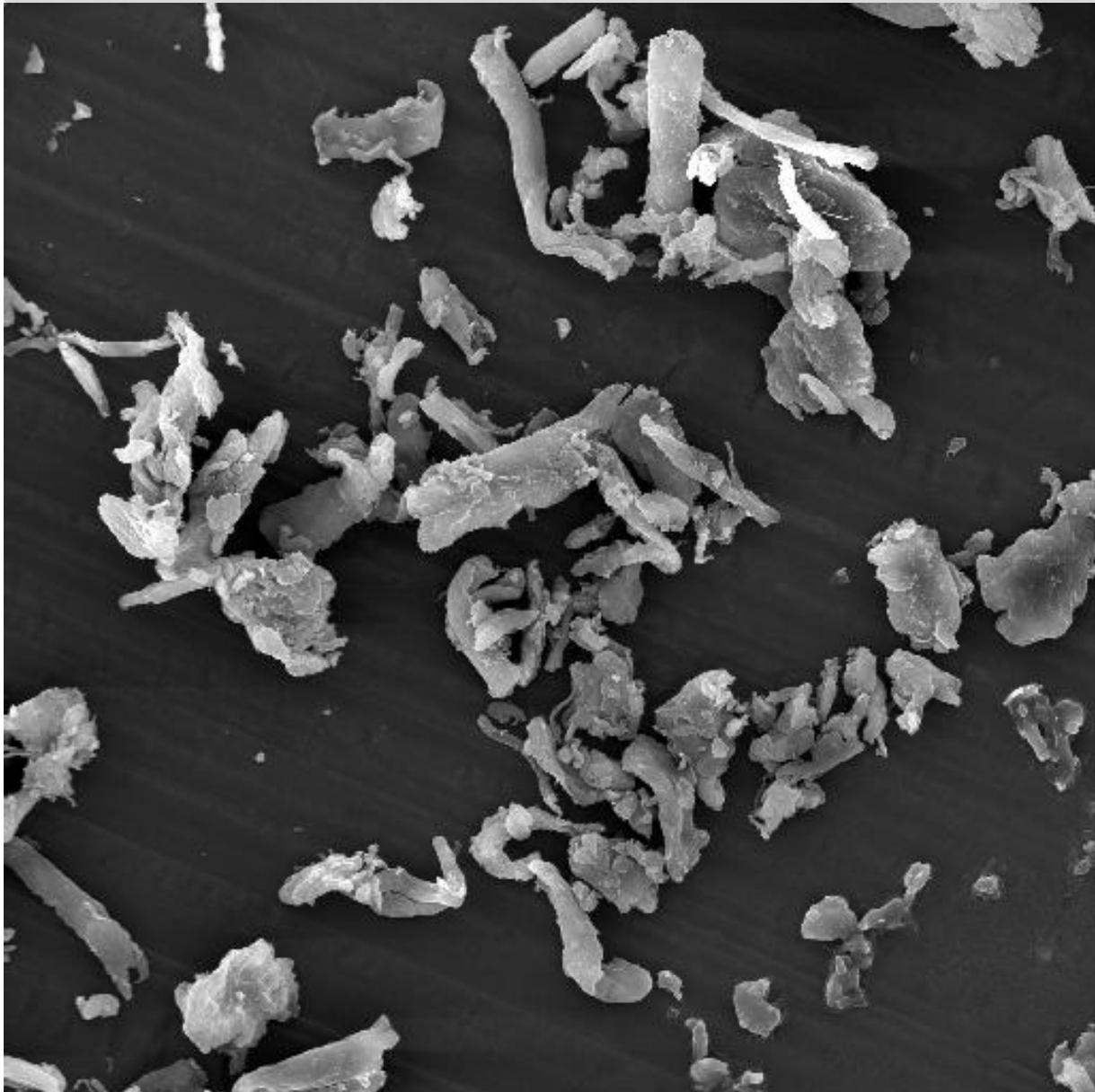
Dana Křemenáková
Jiří Militký
Rajesh Mishra

FT, TUL, December 2014.

*The book is dedicated to our beloved colleague and friend
prof. Ing. Sayed Ibrahim, CSc. for his ever lasting memory.
May his soul rest in peace.*

PART - II

ADVANCED NANOMATERIALS



SEM MAG: 500 x
HV: 30.0 kV
VAC: HiVac

DET: SE Detector
DATE: 11/06/11
Device: TS5130

100 μ m

Vega ©Tescan
TU Liberec



CHAPTER 4

NANOMATERIALS AND TEXTILES

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1. INTRODUCTION

Apart from the stereotypical ways of introducing nanotechnology, we would like to start the discussion with some questions and the accuracy of their probable answers. Is it accurate to call the current state of knowledge about materials at their nano-scaled as “nanotechnology”? What is the impact of the nanotechnology on our lives? What is the future of that field? Responding to these questions begins with the definition of the term “nanotechnology” which has two parts; the prefix “nano” and the word “technology”. The prefix “nano” refers to the 10^{-9} th fraction of the unit and, as other prefixes, it can be added to other words to give a special meaning. Therefore, a nanosecond is a one of 10^9 parts of the second, a nanogram is 10^{-9} of the gram, and a nanometer is a one part from 10^9 of the meter..., etc. Out of the three fundamental quantities (mass, length, and time) the field of nanotechnology is more oriented towards studying materials at the nano-dimensional level, or the material that has at least one dimension less than 100 nm [1].

Based on Webster’s dictionary, the word “technology” is defined as “*the practical application of science to commerce or industry*”. Or it is “*the discipline dealing with the art or science of applying scientific knowledge to practical problems*”. These definitions distinguish between the two terms “technology” and “science” and consider one as a premise to the other. According to Encyclopedia Britannica, “science” refers to “*any system of knowledge that is concerned with the physical world and its phenomena and that entails unbiased observations and systematic experimentation. In general, a science involves a pursuit of knowledge covering general truths or the operations of fundamental laws*”. It can be briefly said; science deals with "understanding" while technology deals with "doing". In this sense, the question “do we live in the era of “Nanotechnology” or “Nanoscience”?” becomes more legitimate since there are many challenges in understanding the behaviour of the material, how to control it, and to overcome the problems that only appear at nano scale.

This discussion about the definitions in the introduction aims at making two points clear; firstly, we should not be surprised by the moderate applications available nowadays of nanotechnology in textiles (as the era of nanotechnology is still in its infant stage). Secondly, the stacks of expectations are really high for the future of nanotechnology in textiles (as the era of nanoscience is booming and growing very fast). It is clear now that most of the hopeful wishing and the “science fiction” that doesn’t achieve yet in the applications of nanoscience will achieve in the near future with the intensive research and interest in this promising field. Once the progress in nanoscience reaches its milestones in applied products, the nanoscience will create another revolution that might change the way in which we are thinking today. This

revolution after nanotechnology might be used to name the future years in a way similar to the naming of old ages as “stone age”, “Bronze age”, and “iron age” where these materials were used as main sources for tools production.

2. THE MAGIC OF NANOSTRUCTURES

Nano materials gain their exceptional properties because of their size as the properties of the same material vary significantly with its size. Gold, for instance, is known at its bulk scale as a shiny, yellow noble metal that does not tarnish, has a face centred cubic structure, is non-magnetic and melts at 1336 K. Reducing the size of the same gold to the nano-scale (below 100 nm), however, results in totally different behaviors and dramatic change in its properties. The melting temperature of gold found to decrease as the size of the particle goes down to the nano-scale. Gold Particles of size *ca.* 10 nm appear in a red color as they become able to absorb the green light at that size. Gold becomes catalytically active at nano size and 2–3 nm nano-particles are excellent catalysts and also exhibit considerable magnetism. Gold particles at this nano-size are still metallic, but smaller ones turn to be insulators. Their equilibrium structure changes to icosahedral symmetry, or they are even hollow or planar, depending on the size [2]. All these changes in the material properties can be observed after by only changing the size of the gold to the nano-scale which poses the question; why size matters? Reasons behind such effects have two origins: the surface effects and the quantum confinement effects [3]. These two general reasons need some explanation and will be addressed in the following paragraphs.

2.1. Surface effects:

In general, all surface properties depend on the dispersion of a particle and scale linearly with the inverse of the particle diameter (D^{-1}) or the third root of the total number of atoms ($N^{-1/3}$).

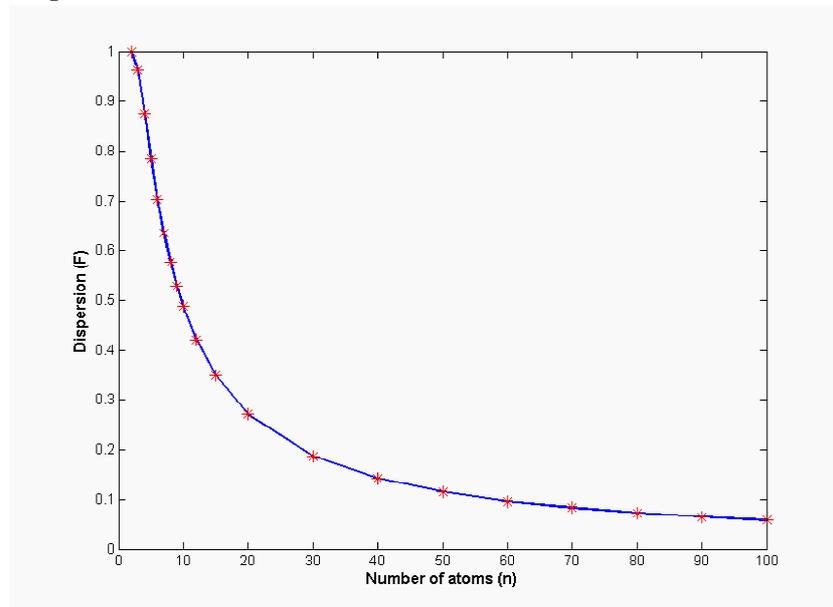


Fig. 1 The dispersion (F) of a cubic particle as a function of the number of atoms on each edge of the cube (n)

This relation can be seen graphically in Figure 1 which shows that the dispersion decreases with the increase the particle size (as indicated by the increase of the edge size and the number of atoms per edge) until it asymptotically approach a constant value at bigger sizes of the particles.

According to this scaling law, changes of some properties with the size of the particles can be observed such as the change in the cohesive energy, melting temperature, and the thermodynamic phase transitions. All of these material properties were found to scale from the bulk-size to the nano-size at the same rate of proportionality; that is $1/D$ or $1/N^{1/3}$. Examples of these changes include the cohesive energy of magnesium clusters which is shown in Figure 2 as calculated for clusters of different number of atoms. The extrapolation of the of the plotted values at higher number of atoms results in a binding energy per atom of 1.39 eV, which is close to the bulk experimental value of 1.51 eV [2]. It should be noticed that the cohesive energy values shown in Figure 2 are average values and the individual values differ per atom and depending on the atom position in the particle's bulk and atoms with higher cohesive energy are forming more bonds and more stable than those of lower energy values. The atoms at the corners (of a cubic particle) have the least cohesive energy values and are the least saturated (i.e. more unstable) followed by the edge atoms, the in-plane surface atoms, and finally the interior atoms. Therefore, the physical and chemical activity of the atoms varies in a particle according to their position and it follows the same order; where the corner atoms are more likely to form bonds with adsorbate molecules followed by the edge then the in-plane surface atoms. Due to their high activity and instability, the corner atoms might be missing on single crystals even in states of thermodynamic equilibrium [2].

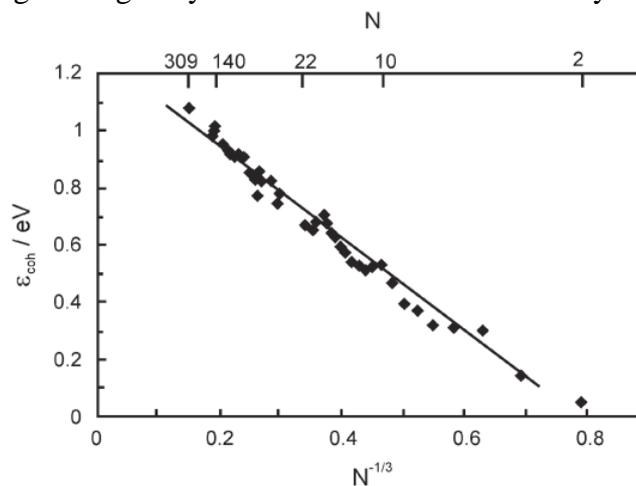


Fig. 2 Calculated cohesive energies of various size magnesium clusters as a function of the total number of atoms N (at the top horizontal axis) and $N^{-1/3}$ (at the x-axis)[2]

Another example of the surface effect is the decrease in melting temperature with the reduction of the particle size which is in compliance with the Gibbs-Thomson relation that phrases this inverse relation between the melting temperature and the radius of a particle.

Among those size effects is the change in the thermodynamic behavior and the “break-down” of the thermodynamic laws in a way that we cannot observe phase transition as sharp as can be observed in bulk and the nano-sized clusters might be thought of as different “structural isomers” that coexist over a range of temperatures. In these situations, part of the cluster

might exist in solid state as frozen part (with atoms that does not dynamically exchange) while another part of the same cluster might exhibit in a liquid-like state and the size and state of each part might fluctuate in a small range of temperatures. Particles at the nano-scale might show *negative heat capacity* which means a drop in the measured temperature of a particle while it is heated. This can be explained by the dependence of the heat capacity on the total energy while the measured temperature depends on the kinetic energy and the negative heat capacity can be observed in situations where partial reduction of the kinetic energy of the particle (converted into potential energy) takes place.

Numerical example of size effect:

Consider a 2 nm spherical silver nanoparticle.

How many silver atoms are on the surface?

What percent of the total number of atoms are on the surface?

Plot the ratio of the surface area to the volume of silver atom clusters as well as the percentage of atoms on the surface (dispersion) as a function of the particle spherical diameters in the range $0.1 \text{ nm} < D_p < 100 \text{ nm}$.

Show any equations and assumptions you might use in your calculations.

Assumptions:

Consider the diameter of the silver atom as its van der waals diameter that is 0.344 nm, Assume the atom shape as a regular sphere with volume = $1/6 \pi D_a^3$,

Neglect the crystal structure of the silver atoms and assume maximum stack of the atoms in the silver produced particle (i.e. neglect the volume of the voids between the atom spheres,

Assume the particle is completely spherical with a volume = $1/6 \pi D_p^3$ and a surface area = πD_p^2 ,

Assume the volume of the surface layer will be the volume of the particle – the volume of sphere with diameter lower than the particle diameter with two atom diameters ($D_s = D_p - 2 * D_a$).

Based on the given information, the following parameters can be calculated:

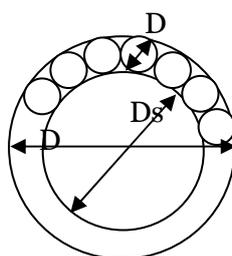


Fig. 3 Numerical example of size effect

Table 1 Numerical example of size effect

	Diameter (nm)	Volume (nm ³)
Atom	0.344	0.021
Particle	2	4.189
Particle after the surface layer	1.312	1.183

Therefore:

The total number of silver atoms (N) is the ratio of the total volume of the particle (V_P) and the volume of the atom (V_A), that is:

The number of surface atoms (N_s) can be calculated as the ratio of the volume of the surface layer (V_{sl}) and the volume of the atom (V_A) that is:

The percentage of the surface atoms to the total number of atoms will be:

To plot the relation between the particle's surface area to its volume ratio and the percentage of atoms on the surface (dispersion) as functions of the particle diameter, the above calculations should be repeated and extended to cover the given diameter range. Results of these calculations are shown in Figure 3. The domination of surface atoms at small particle size can be observed from the given results while at bigger diameters the dispersion levels off at a specific value that slightly changes with the particle diameter.

Note: In the above calculations, since the silver atom diameter is 0.344 nm, the smallest cluster of atoms started at this diameter of one atom rather than starting at 0.1 nm as stated in the problem.

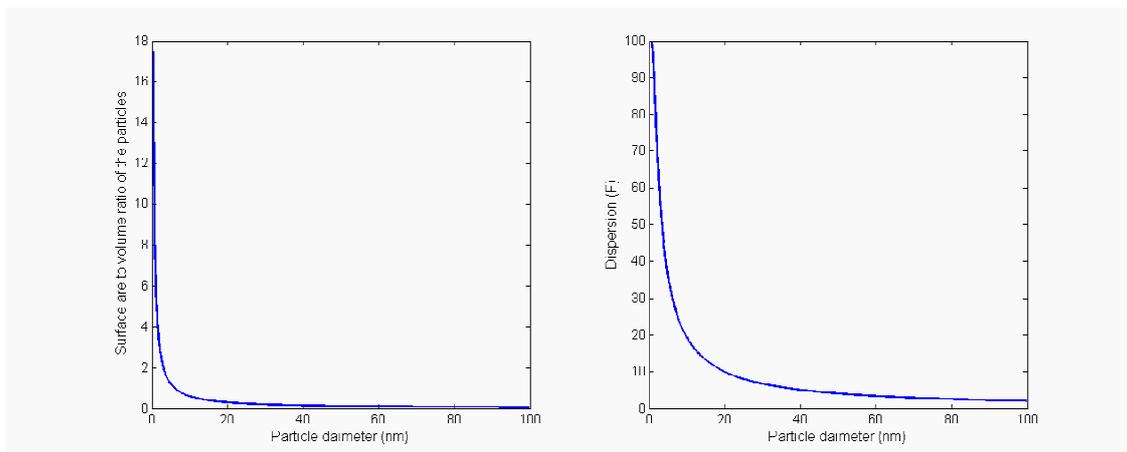


Fig. 4 Calculated surface area to volume ratio (left), and dispersion (right) as functions of silver particles diameter

2.2. Quantum confinement effects:

Before starting with the scientific details of the quantum confinement effects, let us start by looking at these words and their meaning. We started our learning for math with integer numbers where no fractions are allowed, then, and after growing up, we start to learn about rational and real numbers. With a similar analogy, the energy levels at the atomic level are “quantized” where only some specific levels are allowed and electrons of such energies cannot be found in between these levels. It is common to analyse objects of this situation with a “particle in a box” model where the object is confined in a “box” that depends on the size of the particle and generated by the applied potential field. By increasing the number of atoms and taking the average values for the energies, an overlapping start to occur and “quasi-continuous” energy levels can be measured at the bulk level (where high numbers of atoms

exist). It is the averaging effect (that diminishes with approaching the nano-scale) that affects the behaviour of materials at these levels. The word “confinement” just means what it says; it is the situation where objects (e.g. electrons) are confined between certain levels and governed by the quantum nature of these levels.

The energy of the highest occupied and lowest unoccupied states is very important in governing the smoothness of transition between states where the difference between these two energy levels is called “Kubo gap δ ”.

This gap is affected by the total number of atoms participating in each energy level and the Kubo gap decreases as the number of the atoms increase until reaching zero with infinite number of atoms at the bulk scale.

The size of Kubo gap as a function of the number of atoms in the material cluster is demonstrated in Figure 4 where the change in material’s electronic and magnetic properties can be observed with the size of the particle due to the effect of the thermal energy ($k_B T$) on the molecule.

Electrons can transfer, or lack the ability to transfer, across the Kubo gap depending on the thermal energy introduced to the system and if it sufficient to overcome the energy gap at different scales which allows the material to be metallic, semi-conductor or insulator at these scales.

If a cluster of atoms was considered as a molecule, which is a preferred approximation by chemists, this gap can be evaluated as the difference between the highest occupied molecular orbital (HOMO) and the lowest unfilled molecular orbital (LOMO) and incompletely filled degenerate HOMO leads to magnetism in the cluster.

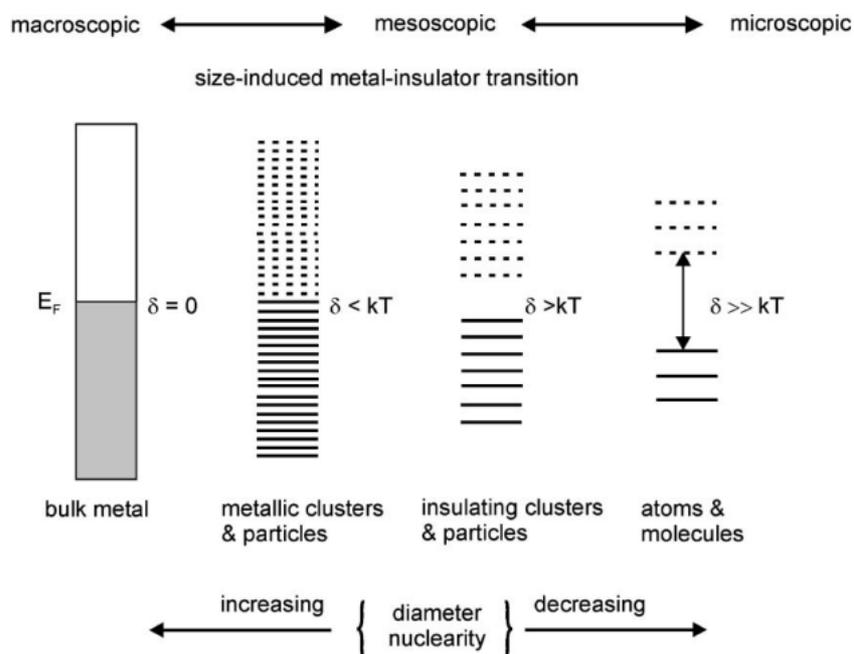


Fig. 5 Evolution of the band gap and the density of states as the number of atoms in a system increases (from right to left) [2]

The size dependence of the HOMO–LUMO band gap is best seen in the luminescence properties of semiconductor nanoparticles. It is illustrated in Figure 5-a for the fluorescence of colloidal CdSe–CdS core–shell nanoparticles. By adjusting the particle size, the fluorescence

can be tuned between blue for particles of 1.7 nm diameter and red at 6 nm. The difference in wavelengths between absorption and fluorescence occurs because the system relaxes to adapt to the new charge distribution after absorption, initiated by creation of excitons (the electron–hole pairs generated after electron excitation). The size dependence is shown schematically in Figure 5-b where the valence and conduction bands are drawn inside a circle that represents particle size. It is analogous to the well-known shift of colour of conjugated polyenes with increasing chain length. It should be noted that already at a size of a few nanometers a system contains several hundred atoms so that discontinuities are no longer observed and the wavelength shift with size appears smooth. It is nevertheless a quantum effect since it is governed by the nature of standing waves of a confined system rather than by the fraction of atoms at the surface [2].

Colloidal gold nanoparticles have been already well known in the middle ages and were used as dyes in stained glass windows of cathedrals and palaces. 10 nm gold particles absorb green light and thus appear red, smaller ones would even fluoresce. In contrast to the above semiconductor particles this absorption of metallic gold particles is due to surface plasmons (a collective excitation of electrons near the surface that represents standing waves on a surface) and their interaction with light is well described by Mie theory. Surface plasmons obey somewhat different rules, and their wavelength in some cases increases and in others decreases with size [2].

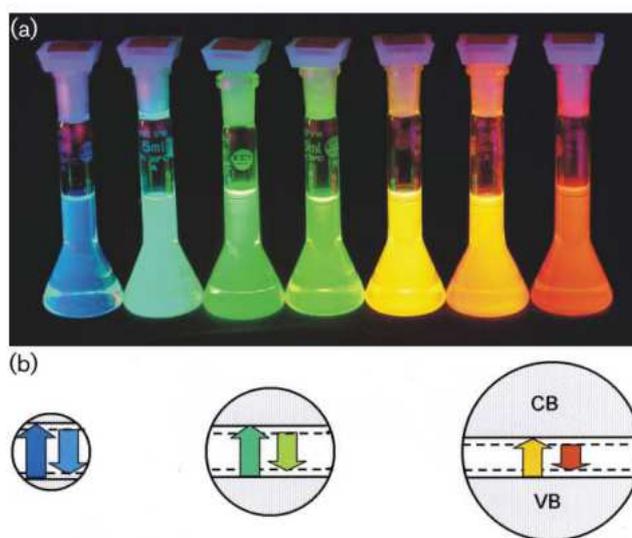


Fig. 6 a) Fluorescence of CdSe–CdS core–shell nanoparticles with a diameter of 1.7 nm (blue) up to 6 nm (red). b) Schematic representation of the size effect on the gap between the valence band (VB) and the conduction band (CB) and the absorption (up arrow) and fluorescence (down arrow) [2]

3. REASONS FOR THE NANO-BOOMING IN THE RECENT YEARS

We talked in the last sections about the old struggle of human beings to manipulate the nanostructures and get the most benefit out of it and how old people used nano-gold particles in the decoration of glass for windows of old cathedras. We also talked about the reasons behind the dramatic changes in material properties when the material, size approaches the nano-scale. The next logical question that comes to mind, “if the application of nanostructures is so old, and we know the great effect of this size scale on the material properties, so *why*

does it become a very hot topic in research only nowadays and not from the time of the conception of the importance of nanostructures?”. Answering this question requires us to look at the facilities available for researchers at the old time and whatever available for them nowadays. It is very obvious that there are many advances in such facilities that have a great effect on the research and utilization of nanostructures. Among these advances, we select explicitly three major reasons, shown in Figure 6, that influenced the nanoscience and nanotechnology in the recent years, those are:

Advances in manufacturing nanostructures

Advances in computational capability

Advances in the sensitivity of the characterization instruments

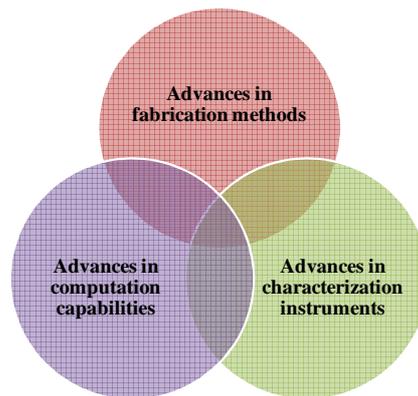


Fig. 7 Reasons for the booming of nanotechnology in the recent years

3.1. Advances in manufacturing nanostructures:

The fabrication of nanostructures is rapidly developing and, only over the most recent years, these methods and their perfection are going up. The precession of methods such as the *lithography* (the use of a radiation-sensitive layer to form well-defined patterns on a surface), and the *molecular beam epitaxy* (the growth of a crystalline material on the surface of another material) to control the materials at the nano-scale is growing. In this part, we will discuss the “quantum dots” as one example of the nanostructures that become recently of a great interest in many applications. Quantum dots can be synthesized through many different methods and each method of synthesis has its own advantages and disadvantages which should be considered in deciding on their production. Generally, these methods can be classified as shown in Figure 7.

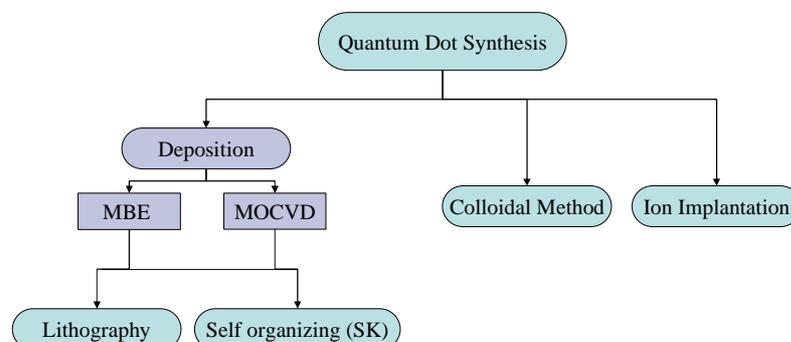


Fig. 8 Methods of quantum dots fabrication

3.1.1. Deposition techniques

The two principal techniques in widespread use today for the deposition of quantum dots are the molecular beam epitaxy (MBE) and metallorganic chemical vapor deposition (MOCVD). Both MBE and MOCVD have produced a wide range of very high-purity quantum dot materials for semiconductor applications with excellent optical and electrical properties.

The word "epitaxy" is derived from the ancient Greek words "epi," meaning on, and "taxis," meaning arrangement. Thus, an epitaxial layer is one that takes the same structure of its substrate, i.e. the same crystal symmetry and lattice constant. If the layer is of the same material as the substrate it is said to be homoepitaxial (e.g. GaAs/GaAs) and in case of a different material it is called heteroepitaxial (e.g. AlGaInP/GaAs, InSb/GaAs).

The molecular beam epitaxy (MBE) was developed as a means of growing high purity epitaxial layers of compound semiconductors. In MBE, the solid source materials are placed in evaporation cells to provide an angular distribution of atoms or molecules in a beam [4]. The substrate is heated to the necessary temperature and, when needed, continuously rotated to improve the growth homogeneity. This process takes place in ultrahigh vacuum (UHV) so that the beams are not scattered which guarantees that the mean free path (λ) of the particles to be larger than the geometrical size of the chamber. Figure 8 shows both a schematic for the MBE process as well as how mean free path varies with regard to pressure.

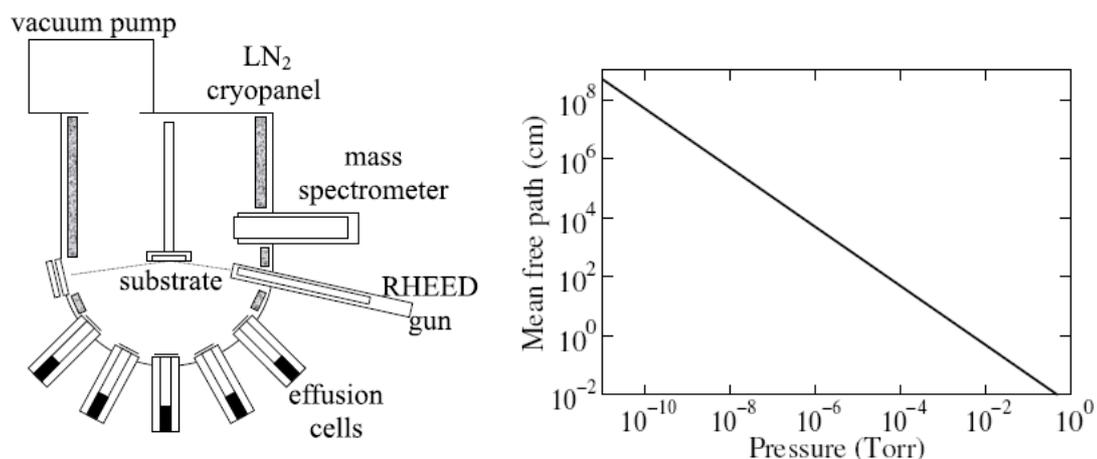


Fig. 9 Molecular Beam Epitaxy system (left) and the change in mean free path of nitrogen molecule with respect to pressure (right) [4]

The most important aspect of MBE is the capability for controlling growth of heterostructures with layer thicknesses down to a single molecular layer (ML) with a slow deposition rate (1 to 300 nm per minute) which allows the films to grow epitaxially. MBE can produce high-quality layers with very abrupt interfaces and good control of thickness, doping, and composition. MBE technique has a high compatibility with many in-situ characterizing techniques at ultra-high vacuum (UHV) such as mass spectroscopy and reflection high energy electron diffraction (RHEED). On the other hand, the vacuum chamber of the MBE needs to be periodically opened in order to add source materials to the evaporation sources that turn the process to be time consuming. There are also limitations on the materials that can be used in

MBE, most notably phosphorous, and the low growth rates (~1ML/s) of the process means that it can take long periods of time to grow thick structures.

3.1.2. Metallorganic chemical vapor deposition (MOCVD):

MOCVD is also referred to, and used interchangeably with, metallorganic/organometallic vapor phase epitaxy (MOVPE/OMVPE). MOCVD is a broader term that is applicable to the deposition of crystal, polycrystalline and amorphous materials. Here, it is used to create controllable epitaxial layered structures by atomic deposition over a substrate material.

In MOCVD, compounds of the desired material are transported to a heated substrate where a chemical reaction takes place at the surface. The substrate wafer is placed on a graphite susceptor inside a reaction vessel and heated by a radio frequency (RF) induction heater. The temperature depends on the type of compounds grown, but it is usually between 500°C and 700°C. Growth can occur in an atmosphere of hydrogen at a pressure between 2×10^{-4} and 760 torr (atmospheric pressure). The growth precursors decompose on contact with the hot substrate to form epitaxial layers. Figure 10 shows a schematic representation of the mechanism behind MOCVD.

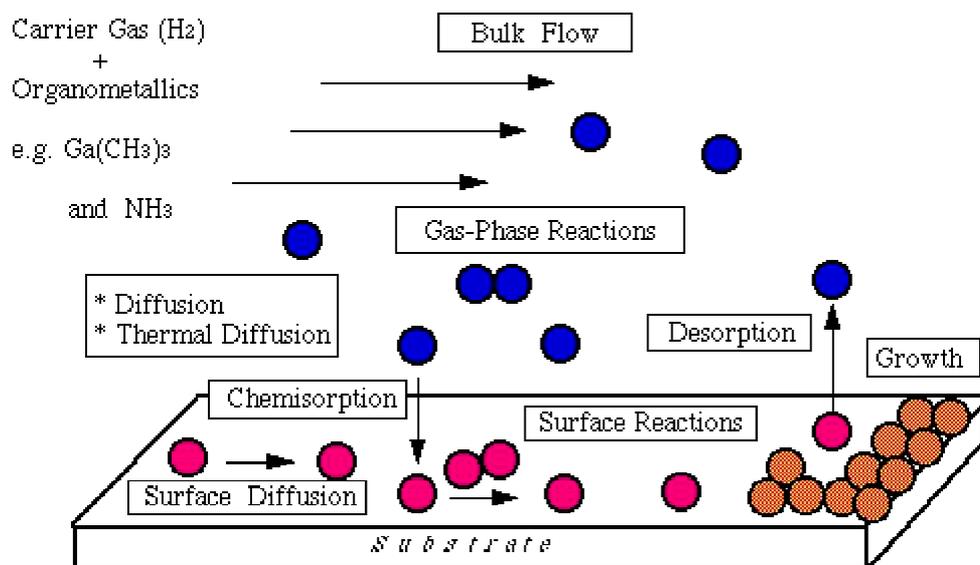


Fig. 10 Schematic diagram of MOCVD mechanism

MOCVD is a very versatile technique and has been used to deposit materials that are difficult to grow using MBE, such as phosphides and nitrides. One disadvantage of MOCVD is that it requires the storage and use of large quantities of hydrides for the group V source, sophisticated gas handling systems for gas delivery, and scrubbing systems for the cleaning of material after every time the system is used.

3.1.3. Lithography

Lithography is typically the transfer of a pattern to a photosensitive material by selective exposure to a radiation source such as light. A photosensitive material is a material that experiences a change in its physical properties when exposed to a radiation source. If we

selectively expose a photosensitive material to radiation (e.g. By masking some of the substrate from the radiation), the pattern of the radiation on the material is transferred to the material exposed. This causes the properties of the exposed and unexposed regions to be different. When the photoresist material is exposed to a radiation source of a specific wavelength, the chemical resistance of the photoresist changes with respect to the developer solution. If the photoresist is placed in a developer solution after selective exposure to a light source, it will etch away one of the two regions (either the exposed region or the unexposed region). If the exposed material is etched away by the developer and the unexposed region is resilient, the material is considered to be a positive resist. If the exposed material is resilient to the developer and the unexposed region is etched away, it is considered to be a negative resist. The photosensitive layer is often used as a temporary mask when etching an underlying layer, so that the pattern may be transferred to the underlying layer. Photoresist may also be used as a template for patterning material deposited after lithography. The resist is subsequently etched away, and the material deposited on the resist is "lifted off". Figure 11 shows a schematic representation of these different processes.

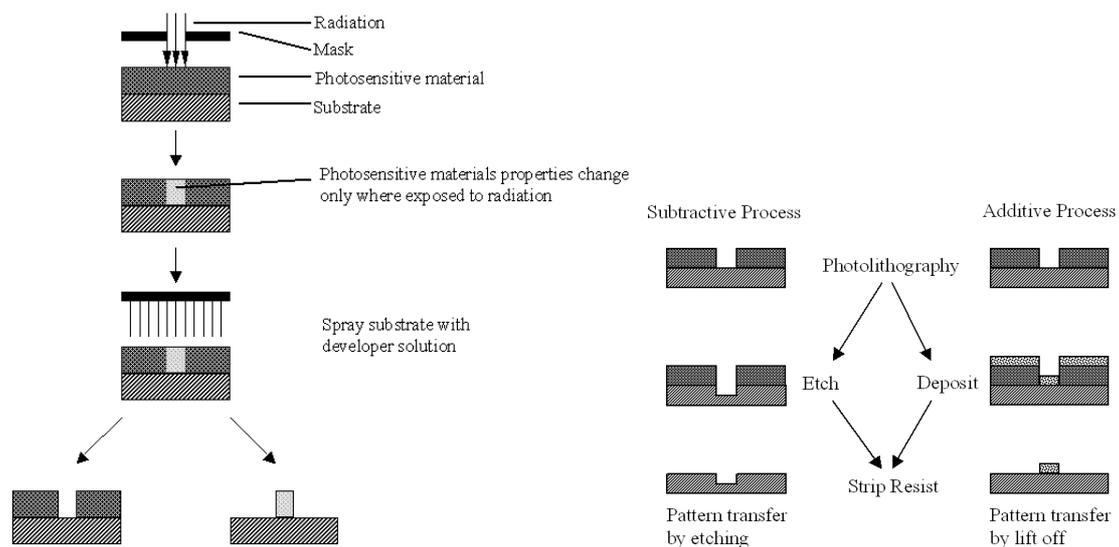


Fig. 11 The sequence of lithographic process with positive resist (far left), negative resist (middle left), an etching process (middle right), and a deposition process (far right)

Lithography is used widely in fabricating quantum dots [5]–[7]. However, one of its disadvantages is that there is a probability that the surface of the sample will experience some degradation. This can lead to the grown quantum dots experiencing some degradation during the several processing steps required.

3.1.4. Self organizing Stranski Krastanov (SK) growth:

The Stranski Krastanov (SK) growth method [8]–[10] is used to fabricate defect-free quantum dots in lattice mismatched systems of about less than 10 nm in diameter. This technique can be implemented using MBE or MOCVD. In the SK growth mode, mismatched epitaxy is initially accommodated by biaxial compression in a layer-by-layer (two-dimensional) growth region, traditionally called the wetting layer. After deposition of a few monolayers, the strain

energy between the two lattices of matched materials increases exponentially, then three-dimensional islands of the deposited material grow.

The advantages of the SK technique are that no further etch or implantation induced processes are necessary since quantum dots are grown during the deposition. Thus, a homogeneous surface and quantum dot/substrate interface morphology is maintained and defects due to exterior impurities are avoided. However, some of the problems associated with the SK method are the size non-uniformity and the lack of control of the position of the quantum dots. The self-assembled growth of quantum dots avoids these complex processing steps accompanied with other methods, e.g. lithography, and is also very time-efficient with a complete growth sequence as short as one hour. Therefore, self-assembled quantum dots are a viable candidate for a commercially relevant growth process. However, several parameters need to be better controlled such as size, density and quantum dots distribution over the surface of the substrate.

3.1.5. Colloidal methods:

The principle of colloidal synthesis [11], [12] of semiconductor nanocrystals is based on observing that a temporally short cluster nucleation event followed by controlled slow growth on the existing nuclei results in the formation of monodisperse colloids. In practice, reagents are rapidly injected to a vessel charged with hot coordinating solvent, thus raising the concentration above the nucleation threshold. A schematic representation of this process is shown in Figure 12.

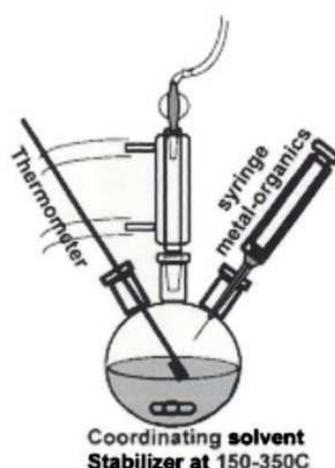


Fig. 12 Schematic representation of the apparatus used for the preparation of nanocrystals

It is possible to form a supersaturated solution of species when the temperature is sufficient to decompose the reagents. Supersaturation is followed by a short nucleation period that partially relieves the supersaturation. This results in a drop in concentration of species below the critical concentration for nucleation, and the clusters "bang" out of solution. As long as the rate of addition of precursor does not exceed the rate at which it is consumed by the growing nanocrystals, no additional nuclei will form. Since the growth of each of the nanocrystals is similar, the size distribution is mainly governed by the time over which the nuclei are formed and continue to grow. This simply means that; by increasing the reaction time, larger and

more uniform nanocrystals can be formed which, therefore, gives a considerable control over the size in this process.

3.1.6. Ion implantation method:

Producing the quantum dot materials via ion implantation is quite straightforward: an intense ion beam [13]–[15] of an appropriate semiconductor species is bombarded into a glass target, typically, silicon dioxide (SiO_2). For example, the growth of Si quantum dots in SiO_2 matrices has been achieved by implanting Si ions (dose $\approx 10^{17}$ ions cm^{-2}) at energies of 200 keV into a 0.65–0.75 μm thick SiO_2 layer grown on a Si (100) wafer. After implantation, the Si/ SiO_2 substrate is annealed at temperatures as high as 1100°C. Depending on the Si^+ beam dose and energy, well formed quantum dot materials with various sized Si quantum dots (20–60 Å in diameter) can be fabricated. Figure 13 shows a schematic representation of the ion implantation method.

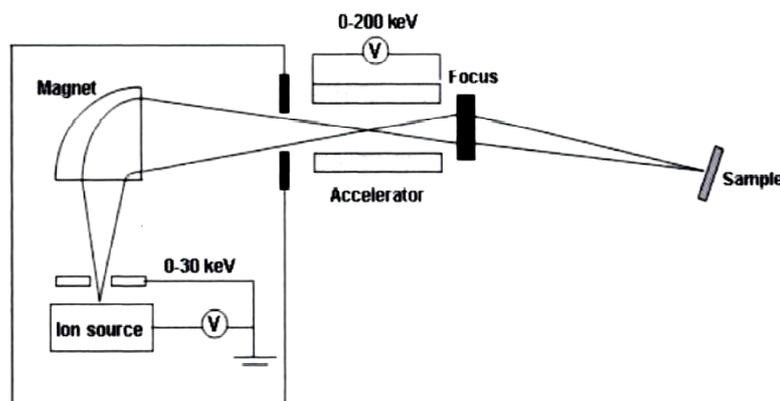


Fig. 13 Illustration of high-energy ion implantation process to fabricate quantum dots

The formation of these quantum dots occurs as follows, when an energetic ion is implanted into the substrate, it will lose energy very quickly by interacting with the substrate. The ions will collide with substrate atoms in a random fashion until they slow down to an energy that is comparable with the thermal energy ($kT = 0.025$ eV). Because of the extreme conditions of the initial implantation event, it is possible to have the substrate supersaturated with the ion dopant. Supersaturation refers to the situation in which a larger quantity of solute (the ions) is dissolved in a solution (the matrix) than would normally be possible. The supersaturated substrate is then annealed slowly to promote the nucleation and growth of the quantum dots.

3.2. Advances in computational capabilities

Since the early 1970's, there is a continual advancement in technology that follows Moor's law where the number of transistors incorporated on memory chip doubles every year and a half. That rapid increase in computational capabilities from a year to the next provided high resources for the most expensive computations of quantum mechanics. It is known that the basics of quantum physics were laid at the beginning of the twentieth century, however, Schrödinger equation, for instance, cannot be "exactly" solved for a big molecule and it is just restricted to solve this equation for small atoms (e.g. Hydrogen atom). Given this background about the requirements for modeling the material at the electronic level and the rapid growing in computational algorithms (hardware as well as software) that should be able to compensate

for these requirements, it is obvious that nanostructures recently become more accessible in terms of their modeling and simulation.

3.3. Advances in the characterization instruments

We, as human, relay mainly on our sensorial system to communicate with the “objects” around us and we need to “see”, “touch”, and “feel” them. Fortunately, the advances in the characterization and measuring instruments in the very recent years allow us to “see” and “touch” objects as small as atoms. A brief of the most effective characterization methods is shown in Figure 14.

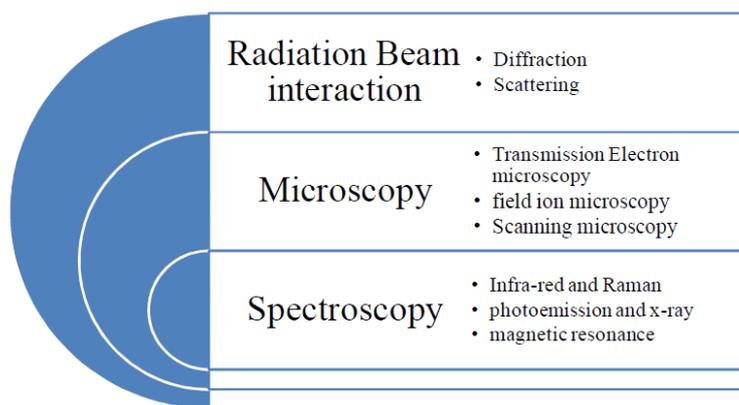


Fig. 14 Characterization techniques that affected the current state of nanoscience

With the aid of the radiation sources (e.g. x-ray beams), we become able to better understand and dissolve the structure of the material. The **interaction** of the radiation beams in the form of *diffraction* from the material’s atoms in a *diffractometer recording* gives enough information about the crystallographic planes and their distances. On the other hand, the *scattering* of radiation beams with higher wavelengths allows determining the size of a cluster of atoms and *dynamic light scattering* (DLS), for example, is one of the methods that enabled us to determine the size of small atom clusters. To get information about the surface of material, special diffractometers are used to let the electron beam to penetrate a very short distance from the surface and *low energy electron diffraction* (LEED) is in use for that purposes.

Advances in the resolution of **microscopy** had the greatest impact on researchers to “see” very small objects. Among the very useful microscopic techniques are the three major families: the transmission electron microscopy (TEM), the field ion microscopy, and the scanning microscopy. The latest family of microscope (scanning microscopy) has different famous forms that include: the scanning electron microscope (SEM), the scanning tunneling microscope (STM), and the atomic force microscope (AFM).

Transmission electron microscopy (TEM) is a technique analogous, to some extent, to the optical microscopy, but instead it utilizes an electron beam and electrostatic and/or electromagnetic lenses to penetrate the object rather than the light beam and the glass lenses. The resolving power of TEM approaches 0.1 nm and by adjusting the power of the intermediate lenses, it is possible to obtain both the image of a sample, as well as its

diffraction pattern. In addition, as with optical microscopy, bright-field and dark-field imaging can be used to change the contrast of the scanned sample. Samples need to be in the form of very thin films - no more than 200 Å in thickness - so that the electron beam can readily pass through the sample and ultra-microtomes are used to cut material's samples to the appropriate thicknesses.

Scanning electron microscopy (SEM) is a technique that is used to produce an image of the material's surface where a fine electron beam is scanned across the sample surface in synchronization with a beam from a cathode-ray tube. The scattered electrons produced can then result in a signal which modulates this beam, which produces an image with a depth-of-field that is usually 300-600 times better than that of an optical microscope, and also allows a three-dimensional image of the object to be obtained. Most scanning electron microscopes have magnification ranges from 20X to 100,000X. As polymers tend not to be good conductors, they need to be coated with a thin layer of a conducting material such as gold. However, in recent years a new *environmental scanning electron microscopy (ESEM)* technique has emerged which enables samples to be investigated without the need to apply any conducting coating. SEM has been used for a broad range of material studies and applications, including surface roughness, adhesive failures, fractured surfaces, networks, and phase boundaries in blends.

Scanning tunneling microscopy (STM) was invented in 1981 by Gerd Binnig and Heinrich Rohrer, while working at IBM Zurich Research Laboratories in Switzerland, and was the reason for them to win the Nobel Prize in physics in 1986. The STM works by bringing the tip of a very sharp metal wire very close to the material surface, and by applying an electrical voltage to the tip or sample, we can image the surface at an extremely small scale – down to resolving individual atoms. An example of the STM images for the surface of different materials is shown in Figure 15 which allows the “visualization” of the atoms.

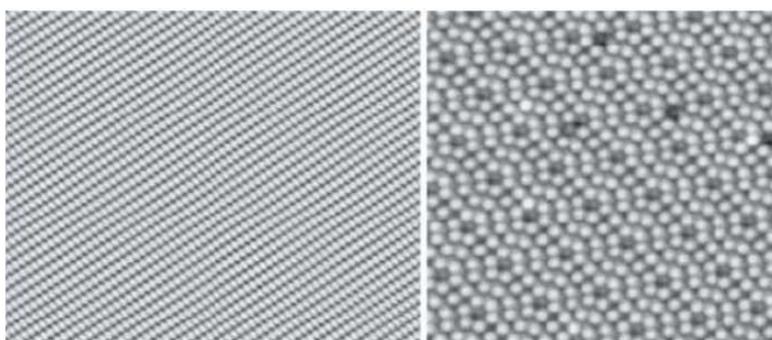


Fig. 15 STM atomic scale images of: the (110) surface of Ag (left) and the (111) surface of Si (right). The width of view in each image is about 15 nm [16]

The STM is based on several principles that include: the quantum mechanical effect of tunneling which allows us to “see” the surface. Another principle is the piezoelectric effect which allows us to precisely scan the tip with angstrom-level control. Lastly, the feedback loop which monitors the tunneling current and coordinates the current and the positioning of the tip [17].

Atomic force microscopy (AFM) was developed by the same inventors of STM to overcome its basic drawback which is that it can only image conducting or semiconducting surfaces. The AFM, on the other hand, has the advantage of imaging almost any type of surface, including polymers, ceramics, composites, glass, and biological samples [17]. Analogous to how an STM works, a sharp tip is raster-scanned over a surface using a feedback loop to adjust parameters needed to image a surface. Instead of using the quantum mechanical effect of tunneling, atomic forces are used to map the tip-sample interaction. AFMs uses a laser beam deflection system where a laser is reflected from the back of the “reflective AFM lever” and onto a position-sensitive detector. AFM tips and cantilevers are micro-fabricated from Si or Si₃N₄ and the typical tip radius is from a few to 10s of nm. The force between the AFM tip and sample is not measured directly, but calculated by measuring the deflection of the lever and knowing the stiffness of the cantilever according to Hooke’s law:

$$F = -kz$$

Where F is the force, k is the stiffness of the lever, and z is the distance the lever is bent. Often referred to as scanning probe microscopy (SPM), there are AFM techniques for almost any measurable force interaction - van der Waals, electrical, magnetic, thermal. For some of the more specialized techniques, modified tips and software adjustments are needed [17]. **Spectroscopic** techniques are also among the developments that facilitate the discovery of material at small scales. Infra-red and Raman spectroscopy, photoemission and x-ray spectroscopy, as well as the magnetic resonance spectroscopy are the names of the major spectroscopic techniques that provide information about the chemical structure of the material as well as some of its physical properties. Discussion of these techniques is beyond the scope of this introduction and the interested readers can find many sources [1] that explain them.

CLOSING REMARKS ON THE FUTURE

Obviously, the question about the Nanotechnology’s future is controversial, since the human curiosity will not stop at the nanometer limit. Although traces of using the nanomaterials go many centuries back, some researchers dated the onset of nanoscience to the 1959 when the Nobel Laureate Richard Feynman laid the vision for this research in his speech about the “plenty of room at the bottom - invitation to enter a new field of physics”. It is obvious that the scale does not stop at the nano level as Ahmed Zewail received the Nobel Prize in 1999 for his studies of the transition states of chemical reactions using femto-second spectroscopy. The femto-second is a 10^{-15} of the second, hence the question about the future might be; do we expect more exciting materials when they are studied and controlled at the femto-meter scale? The future is wide open for answering such a question.

TEXTILE AND NANO TECHNOLOGIES, WHERE DO THEY MEET?

The size of different physical objects around us can be measured with different instruments, but we cannot use the same instrument for measuring everything because of the sensitivity limitations of the instruments to different scales. While we use a measuring tape to get the dimensions of human being for deciding their clothing fit, we do not use it to measure the hair thickness, for example. This highlights the idea of the size variation on the dimension

spectrum as demonstrated in Figure 15 where the human beings can be found at one extreme of the spectrum, while the atomic handwriting are on the other extreme.

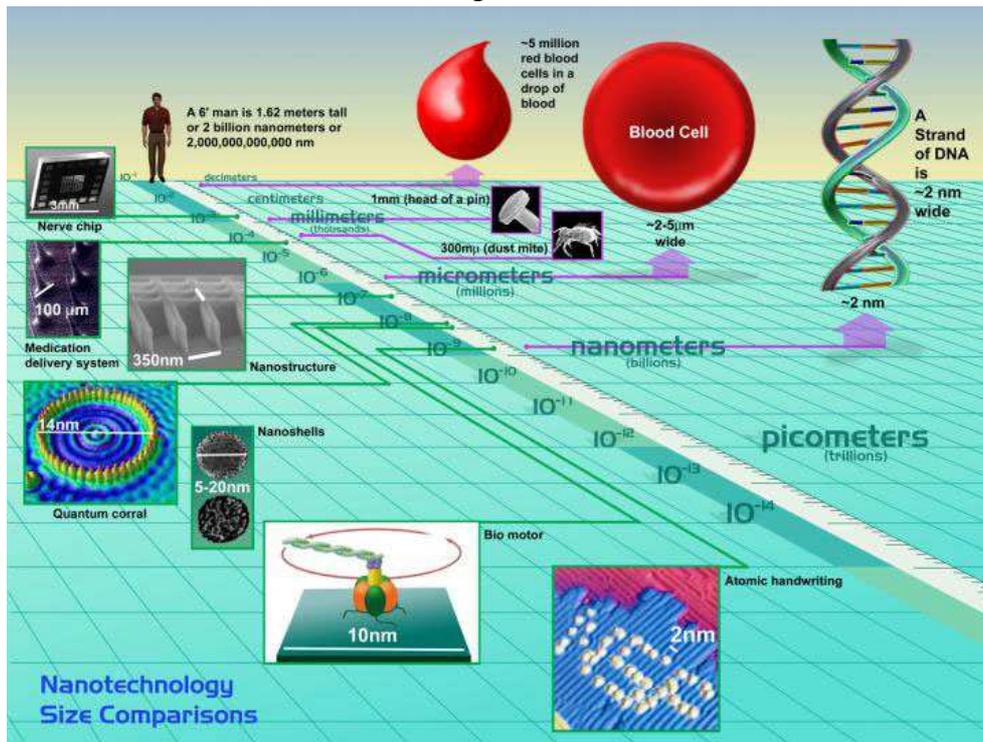


Fig. 16 The allocation of different objects on the dimension/space scale and the comparison of their sizes [18]

There are many objects with dimensional sizes in between these two extremes in the figure and there are many other objects that are many folds larger than the size of the biggest object in the spectrum (e.g. buildings, stars...etc) as well as there are many other objects that are many folds lower than the size of the smallest object (e.g. atoms, quarks...etc).

The traditional textile products, such as garments and clothing, having dimensions in the macro-scale and there is no way to introduce nanotechnology to the textile industry by producing nano-shirts, for example, because of an obvious reason which is; those nano-shirts will not fit in! On the other hand, textile technology shares an important feature with nano technology because textile industry is one of the oldest industries that utilize the *bottom-up* approach. For instance, the old traditional textiles start with discontinuous natural fibres such as cotton that is collected from the cotton field with a high degree of variability to go through some important opening and cleaning operations and produce a continuous stream of fibres with a high linear density known as “sliver”. The sliver goes through “drawing” stages that work on reducing the linear density and increasing the homogeneity of the sliver by mixing (technically known as “doubling”) with higher number of slivers (six or eight slivers). Later on, some consequent processes handle the sliver to reduce its linear density to have a few number of fibres in the cross-section then giving those few fibres some consolidation through twisting. This continuous product is known as a “yarn” and it has the fibres in helical shapes and oriented in one direction with a relatively acceptable degree of regularity that allows the yarns to withstand mechanical stresses in that continuous direction. Transforming this one-dimensional (1D) fibrous structure (i.e. the yarn) into a two dimensional (2D) structure (i.e.

fabric) is done through different technologies such as the knitting and weaving. The produced 2D fabrics are then processed to form three-dimensional (3D) structures such as the garments that humans (who are 3D) wear. This journey from discontinuous 1D fibres to the 3D final product is one of the oldest bottom-up approaches of building structures and, therefore, provide a rich experience for those who are seeking to create a material starting from its simple building blocks.

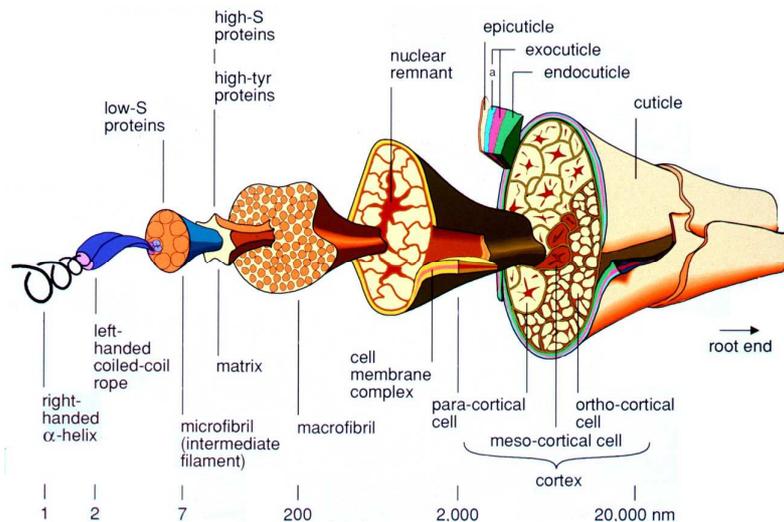


Fig. 17 Schematic diagram of a Marino wool fibre structure [19]

It should be notified here that we intentionally overlooked in the above description the “zero-dimensional” starting points which are the polymer molecules that build the 1D fibres. A schematic representation of the internal (fine) structure of a wool fibre is shown Figure 16 and demonstrates the hierarchy of the complicated building blocks for an individual natural fibre. In fact, engineering the molecular structure to produce fibres of specific properties can be considered new (if it compares to the life of the textile industry) as it started in the twentieth century after the introduction of man-made fibres. It is, however, a very attractive field in the nano science as the control of the polymeric molecules is done as a work at the nano level and that is where the second point of intersection between the textile technology and the nano technology comes to play.

6. NANO-TECHNOLOGY IN TEXTILE FIELD

Although garments and clothing are the first things that come to mind when we mention the word “textile”, textile industries are not restricted to those traditional forms. Textile industries can be defined in a broader way as “any industry that has inputs in fibrous form” (excluding the food products that based on “digestible” natural fibres). This broader definition of textiles, as fibrous structures, covers many of the traditional textiles as well as the “technical textiles” which might also known as “industrial textiles”. Technical textiles extend to cover fibrous structures in many applications and might have some nano-technology in practice. However, our discussion in this section excludes the technical textiles and focuses only on the application of nanotechnology with “traditional textiles”.

As we mentioned earlier, it is not rational to bring the size of traditional textile products to the nano size, it is more common, however, to follow one of the following three approaches, shown in Figure 17, for bringing the nanotechnology into the traditional textiles:

Scale the fibres down (nanofibres)

Build nano-composite fibres

Use nanostructures in finishing

These approaches are intuitively suggesting that the producer of the textile material should use the finest fibres possible (nano-fibres), or reinforce the fibres at their current micro-size with nano-particles, or produce the textile material in its traditional way but to enhance its performance using nano-structures during the finishing process.

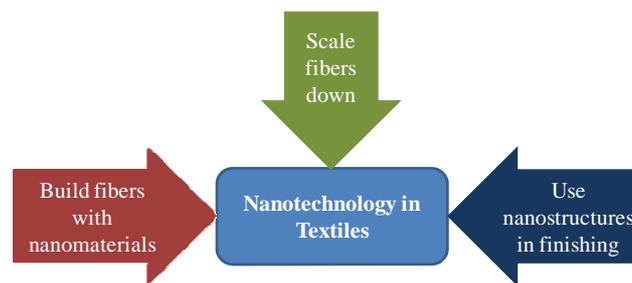


Fig. 18 Common practical approaches for bringing in nanotechnology to traditional textiles

6.1. Scale the fibres down

The diameter of the most common natural and synthetic fibres are in the range of micrometers, which is, at least, a hundred times the order of the required diameters to get into the nano-scale. Therefore, producing sufficient amount of fibres at nano-meter range of diameter and at the same time having sufficient properties to be applicable in the traditional textile products is one of the challenges that face the implementation of nano-technology in textiles. Among the solutions suggested for this challenge was the introduction of the electro-spinning systems where polymeric solution with a reasonable viscosity being injected under a strong electric field that charges the solution droplet and draws it away toward a surface with an opposite charge causing the formation of a fibre with a diameter in the nano-meter range, as can be seen in Figure 18. The produced fibres have high aspect ratio (ratio of the length to the diameter), high specific surface area (ratio of the surface area to the unit mass), and high potential for mimicking biomaterials.

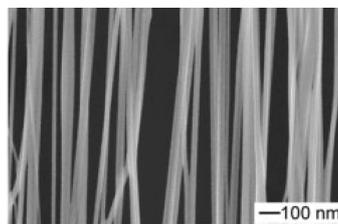


Fig. 19 SEM image of uniaxially aligned nanofibre made of polycrystalline NiFe_2O_4 [20]

The electro-spinning process depends mainly on the electrostatic atomization of polymer solutions based on the well known physical phenomenon of instability (therefore, distortion) of liquid droplets under the effect of electric charges. The distortion of the spherical polymer droplet is a result of the competition between the Coulomb repulsion of like charges (which works on the droplet distortion) and the surface tension (which works on keeping the droplet from division). The pressure balance on the surface of the droplet between these forces can be expressed mathematically in the form:

Where the first term represents the pressure from the surface tension (σ) and the second term is the electrostatic pressure resulting from the total droplet charge (e) on the droplet of radius R at vacuum permittivity ϵ_0 .

It is clear from this relation that the total pressure on the droplet does not change monotonically with its size R where the electrostatic pressure term, for instance, dominates the behaviour of the total pressure at small radius of the droplet as the charge density increases on the surface at these small radii. The total pressure reaches a maximum value at certain radius and at smaller radius it vanishes (i.e. $\Delta P=0$) as the competing forces reach their equilibrium at that radius. This point is associated with the electrostatic Rayleigh criterion, which can be interpreted as “the maximum charge density that a droplet of a given diameter can withstand” [21].

The electro-spinning process uses polymer solutions; therefore, nano-fibres out of polylactides, polyethyleneoxide (PEO), polyvinylalcohol (PVA), polyacrylonitrile (PAN), and polyimides were produced. High polarity and electrical conductivity of the applied solvent are recommended because they improve the electrostatic process; however, the solvent toxicity, vapor pressure, flashing point, and explosivity are main safety concerns that should be considered during the selection of the solvent for this process. Hence, it is not surprising to find both PEO and PVA to be the most common and successful polymers during the experimentations of electro-spinning as these polymers dissolve in water and their aqueous solutions have no safety or environmental issues. In textiles, there is a direct thinking about the production of continuous and aligned fibrous structures with a controlled fibre orientation (i.e. yarns) instead of just the production of the short nano-fibres. This will allow the building of nano-fibrous structures with complex and controlled geometries as the traditional textile technologies can facilitate such production and it will be very useful in many applications. Therefore, researchers are exerting tremendous efforts to produce nano-fibre yarns where the two most successful methods depend on the application of a *rapidly rotating disk collector*, or a *gap alignment effect* [21]. In the rotating disk collector, a disk rotates with a very high speed that creates a shearing force on the fibres and causes their alignment at the direction of rotation. The aligned fibres can then be collected manually and twisted to produce the nano-fibre yarn as shown in Figure 19. On the other hand, charged electrospun fibres tend to align when they are deposited onto a collector that consists of two electrically conductive substrates and separated by an insulating gap. The application of this idea of depositing poly(ϵ -caprolacton) between two parallel grounded rings that were placed 80 mm horizontally apart resulted in a 1.26 μm diameter fibres, with a good alignment that allows the production of a

multifilament yarn with a diameter of less than 5 μm and a length of 5 cm, as shown in Figure 20 [21].

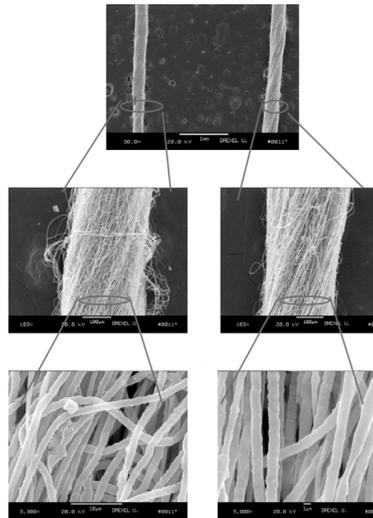


Fig. 20 Twisted poly(ethylene-oxide) nano-fibre yarns obtained from tows spun on a rotating disk collector [21]

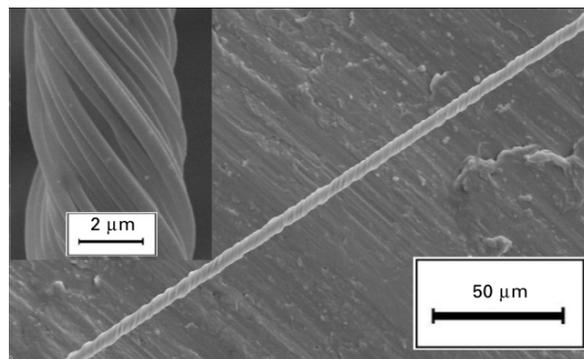


Fig. 21 Twisted poly(ϵ -caprolactone) yarn [21]

6.2. Build nano-composite fibres

The second approach of bringing in the nanotechnology to the traditional textiles is to keep the size of the fibre the same in the micro-scale range, but to use nano-fillers in the fibre's matrix and obtain nano-composite fibre. This approach spread quickly because it is similar to the well known compounding in injection molding and resembles, to a great extent, the powder injection molding (PIM) and the metal injection molding (MIM) which combines the structural benefits of metallic materials with the shape complexity of plastic injection molding technology. In the same fashion, different fillers are used during the production of synthetic fibres to produce composite fibres and twin-screw extruders are commonly used for that purpose with thermoplastic polymers. This approach, however, has some difficulties because of the agglomeration of the nano-filler due to the high attraction forces that dominate the interaction of the fillers at the nano-scale. Also, the effect of these fillers on the rheological properties of the melt should be considered and controlled effectively.

Different polymers are used as a matrix for producing the required composite fibres and include some amphiphilic polymers dissolved in water such as: poly(hydroxyaminoether)

(PHAE), poly(vinyl alcohol) (PVA), and PVA/poly(vinylpyrrolidone) (PVP) that have proved particular effectiveness, as well as other organic systems, such as polystyrene (PS), polyamide (PA), ultra-high molecular weight polyethylene (UHMWPE), poly(methylmethacrylate) (PMMA), and polypropylene (PP). Among the fillers that are commonly used: the carbon nano-fibres, the carbon nano-tubes in its different forms as single walled carbon nano-tubes (SWCNT) or multi-walled carbon nano-tubes (MWCNT). The application of these fillers affect the physical and thermal properties of the produced composite fibres as demonstrated in Figure 21 with the effect of filler content on the mechanical properties of a system of poly(ether ether ketone) (PEEK) matrix filled with carbon nano-fibres (CNF). Clay fillers are also commonly to be used as well as silica, oxides (e.g. TiO₂, Al₂O₃, ZnO...etc), and carbonate (CaCO₃) particles which change the fibre properties and have wide application especially to increase the fire resistance of the fibres.

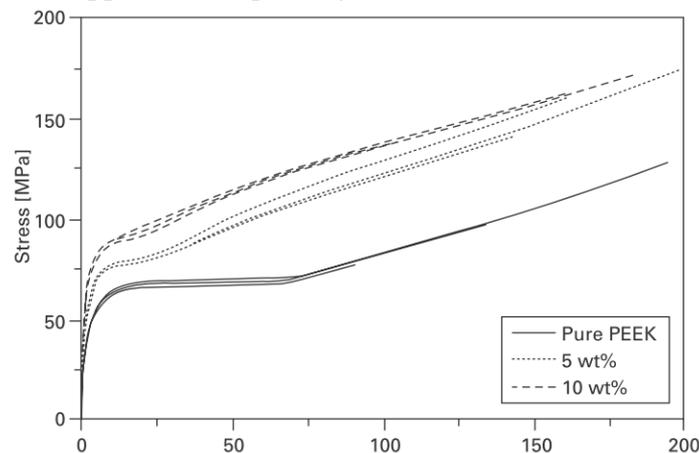


Fig. 22 Stress–strain diagrams of as-spun PEEK-CNF nanocomposite fibres as a function of the filler content [21]

6.3. Use nanostructures in textile finishing

This approach might be the most effective way in commercially bringing the nanotechnology to textiles where many surface treatments were found to be effective in transforming the functionality of traditional textiles. The application of nanostructure coating layers is widely used in textiles to affect the surface function and nano-particles such as antimicrobial silver, photo-active TiO₂, conductive or magnetic metals or metal oxides, and UV-absorbing particles (ZnO) are utilized.

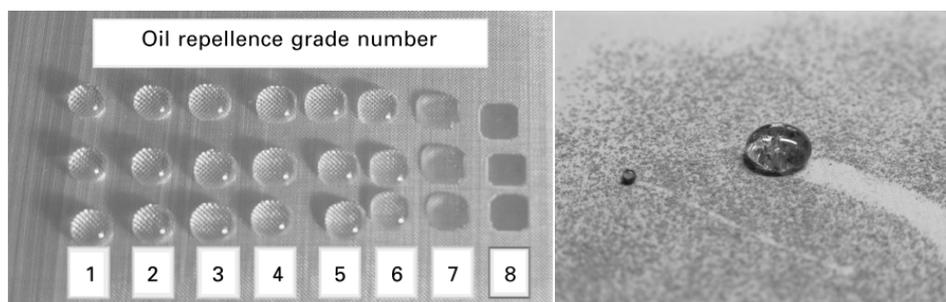


Fig. 23 Oil repellence grading for plasma-finished fabric (left), and a honey droplet on a fabric with self-cleaning surface characteristic (right) [21]

One of the most common applications for these nanostructures in finishing is the “liquid repellence” surface which can be oil or water repelling and implement resins based on fluorocarbons (FC) that are applied either at the yarn level or the fabric level. The goal of such application of material is to obtain super-hydrophobic or super-hydrophilic surfaces aiming for the Lotus-Effect® of self-cleaning materials. Demonstrations of oil-repellant surface as well as self-cleaning fabric are shown in Figure 23. Different technologies are in operation for applying this treatment and the coating layers where aerosol spraying and plasma treatment (especially atmospheric plasma) are commonly used in textiles for such purposes. Although this treatment is on the surface, the structure and construction of the fabric as well as its roughness affect the thickness of the required coating layer where, for instance, by decreasing the diameter of the fibre to 1 μm and keeping textile weight and liquor pick-up in the padding machine constant, the coating thickness on each fibre will decrease to 10 nm and lower.

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