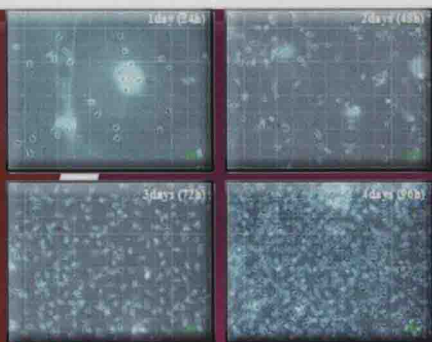
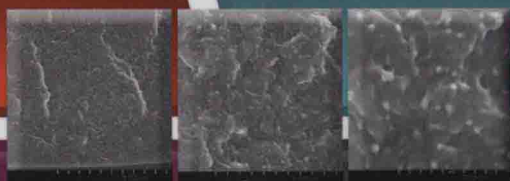
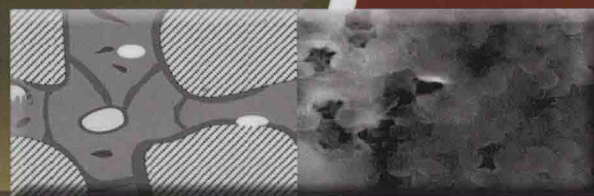


New [redacted] Developments in Polymer Composites Research



Polymer Science and Technology

Stephan Laske • Andreas Witschnigg
Editors

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POLYMER SCIENCE AND TECHNOLOGY

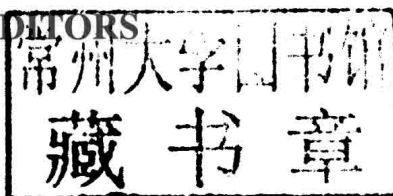
NEW DEVELOPMENTS IN POLYMER COMPOSITES RESEARCH

STEPHAN LASKE

AND

ANDREAS WITSCHNIGG

EDITORS



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POLYMER SCIENCE AND TECHNOLOGY

**NEW DEVELOPMENTS IN POLYMER
COMPOSITES RESEARCH**

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PREFACE

The field of polymer composites is one of the most studied and published areas in polymer engineering and science. A lot of excellent research has been done and is going on, nevertheless, there are new outstanding results and findings all over the world nearly every day. The reason for this is that polymer composites offer almost endless possibilities in changing material properties. By combining at least two materials with different properties and behavior it is possible to generate materials with complete different characteristics and even to establish new material classes (e.g., wood plastic composites or nanocomposites). The ample potential and wide range of polymer composite is also observable within this book as the topics range from new processing strategies over new material combinations to matrix-filler-composite structure-property-relationships. Thereby the different chapters cover the fields of biodegradable, conducting, nano- or wood plastics composites as well as generating new (nano-) composites with sophisticated properties in terms of thermal or electrical conducting, tribology and radiation shielding.

The excellent 13 chapters within this book can be divided into material, processing and properties. Within every topic the newest developments are shown giving an impression of the comprehensive and diverse work of international scientists in the field of polymer composites. The results mark only the tip of the iceberg of polymer composites and leave a mark of what we can expect in future. After the Stone, Bronze and Iron Age we just started the age of polymer composites as material of the 21st century.

CONTENTS

Preface		vii
Chapter 1	Evolution of Novel Size-Dependent Properties in Polymer-Matrix Composites due to Polymer Filler Interactions' <i>Sheila Devasahayam and Sri Bandyopadhyay</i>	1
Chapter 2	New Development in Polymer Composites Research and Applications of Polymer-Cement Matrices for Solidification/Stabilization of Radioactive Wastes <i>S. B. Eskander and M. E. Tawfik</i>	33
Chapter 3	New Fabrication Techniques of Conductive Polymer/Insulative Polymer Composite Films for Printed Electronics <i>Mitsuyoshi Onoda</i>	67
Chapter 4	Nanocomposites Based on Block Sulfonated Polyarylenethioethersulfone Copolymers and In Situ-Generated Nanoparticles of Zirconium Phosphate <i>Zongwu Bai, Marlene D. Houtz and Barney E. Taylor</i>	151
Chapter 5	Conducting Polymer Composite Films Based on Polyaniline: Dielectric and Electrical Properties <i>Kateryna Fatyeyeva, Christele Vanga Bouanga, Alexander Pud, Jean-François Bardeau and Mohamed Tabellout</i>	169
Chapter 6	Thermosetting Biopolymers Reinforced with Natural Fibres: A Review <i>Carlo Santulli and Fabrizio Sarasini</i>	197
Chapter 7	Fundamental Investigations of Reinforcement of Expanded Perlite in Polypropylene <i>Hannelore Isabella Mattausch, Stephan Laske, Kristin Cirar, Helmut Flachberger and Clemens Holzer</i>	213
Chapter 8	New Thermally Conductive Liquid Crystalline Polyester and Its Composites <i>Shusuke Yoshihara</i>	227

Chapter 9	Determination of Binodal Curves in Ternary Polymer Systems: Co-/Polymers Compatibility <i>Francisco Torrens and Gloria Castellano</i>	243
Chapter 10	Influence of Chemical Stress on Sulfur-Polymer Composite Structure <i>Milica M. Vlahović, Predrag B. Jovanić, Sanja P. Martinović, Tamara Đ. Boljanac and Tatjana D. Volkov Husović</i>	257
Chapter 11	Effect of Contact Temperature on the Tribological Behavior of PTFE Composites <i>Marcello Conte, Bihotz Pinedo and Amaya Igartua</i>	279
Chapter 12	Injection Molding Compounding of Polymer Nanocomposites <i>Markus G. Battisti and Walter Friesenbichler</i>	301
Chapter 13	Structural Organisation and Biomimesis of Nature's Polymer Composites <i>Parvez Alam</i>	325
Editors' Contact Information		381
Index		383

Chapter 1

EVOLUTION OF NOVEL SIZE-DEPENDENT PROPERTIES IN POLYMER-MATRIX COMPOSITES DUE TO POLYMER FILLER INTERACTIONS'

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ABSTRACT

Engineering polymers, despite experiencing rapid increases in many applications including in space, nuclear reactors and in the medical field, suffer from limitations due to lack of sufficient strength and stiffness, electrical and thermal conductivities as well as thermal stability, etc. The advances in polymer nanocomposite technology, with the filler dimensions in nanometer scales, have made inroads into engineering polymers' applications in the clean energy, semiconductor, medical and construction industries owing to their significantly larger surface to volume ratio and the highly reduced particle-particle distance compared to their micro-sized particle counterpart.

The quantum effects which begin to dominate the behaviour at the nano-scale, particularly at the lower end affect the fillers' optical, mechanical, electrical and magnetic behaviour. The scale at which discontinuity compared to bulk emerges at room temperature is different for different properties, e.g., for magnetic properties is typically ~50nm. The Bohr radius defining the optical and electronic properties is ~10nm. The critical nucleus size is 5 nm above which new crystalline phase will form, below which the clusters tend to dissolve rather than grow.

When the polymeric materials are reinforced with smaller amounts of nanofillers, due to the evolution of extraordinary range of size dependent properties on these nanofillers, the filler-polymer matrix exhibit superior barrier properties, toughness, tribological and mechanical performance, optical properties, lower melting point, increased surface area, higher specific heat, increased electrical conductivity and magnetic properties showing decreased coercive field and increased remnant magnetization, which are not possible in conventional polymer composites. For example, bulk gold appears yellow in colour while

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nanosized gold appears red in colour. Large ZnO particles scatter visible light, and appear white, while nanosized ZnO particles, due to their smaller dimensions compared to the wavelength of visible light don't scatter it and appear clear. This is attributed to the shift in optical absorption toward shorter wavelengths as the size tends to be smaller. There is also deviation in melting point from the bulk value greater than a couple of hundred degrees when the particle size goes down to below 10 nm. Fracture strength of nanostructured Cu-Fe alloys, 2.8 GPa (at ~38 nm) is about 5 times the fracture stress of iron with larger grain sizes, ranging from 50 to 150 μm . Saturation magnetization, M_s , of zinc ferrite increases significantly below 20 nm from 1 to 4.5. The coercive field of Nd₂Fe₁₄B with nanograin structure decreases significantly and the remnant magnetization increases below ~40 nm. Silica micro-particles at 16 % volume show increased energy release rate by 40 %, while silica nano particles at 5 % and 10 % volume show increased energy release rate by 60 % and 86 % respectively, i.e., from 0.162 kJ/m² to 0.26 and 0.3 kJ/m². Electrical conductivity of neat HDPE is $1 \times 10^{-9} \text{ Sm}^{-1}$. However, with the addition of 10 wt % single-walled carbon nanotube (SWCNT), the electrical conductivity of HDPE increases up to 100 Sm^{-1} . Similarly thermal conductivity of neat HDPE is only $0.52 \text{ W m}^{-1} \text{ K}^{-1}$. However, it increases by 573% with an addition of 0.2 vol% SWCNT reinforcement. The elongation at break becomes 2.3 times higher when the standard composites are reinforced with nanoparticles. Impact strength increases by 1.7 times as well as the bending strength and the modulus also increase due to the incorporation of the nanofillers.

The emergence of the novel properties in polymer composites very much depend on the interfacial interaction between the polymer and these fillers. Thermodynamic and kinetic barriers inhibit the dispersal of inorganic nanoparticles with generally high surface energies within hydrophobic polymer matrices. In this chapter we discuss the effects of different types of interactions, including the intrinsic vs extrinsic properties of the nanofillers, as a result of their functionality, shape, orientation and size of the particles governing their properties.

Keywords: polymer nanocomposites, nanofillers, quantum effects, quantum confinement, size dependent-novel properties, interfacial interactions: attractive interactions, non attractive interactions, nanoconfined polymers, hierarchical morphology

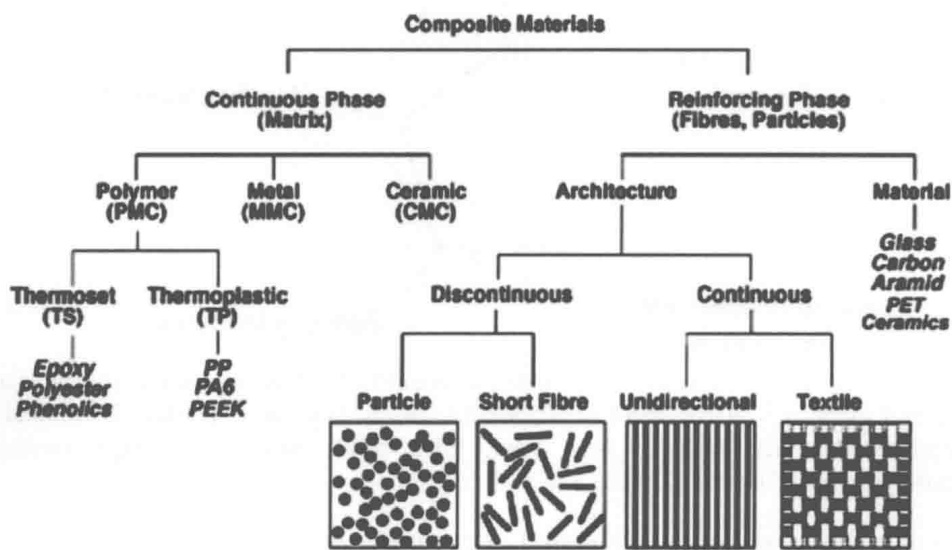
INTRODUCTION

Polymer composite materials are replacing metals in various engineering applications due to their high specific strength, specific modulus, low densities [Gibson, 1994, ASM International, 1992, Zhang SW, 1998, Thomas Selvin et al. 2012, Sengupta et al. 2011], excellent friction & wear characteristics and corrosion resistance [Ning, 2011, Durand, 1985]. They have been designed and manufactured for various applications like naval ships, war planes and sea vehicles, fabrication of devices for non-linear optics, for optical data storage and for optical switching semiconductor [Reisfeld, 1996, Peyghambarian, et al., 1993].

In comparison to widely used conventional microscale particles, submicron particles have some unique features. When particle size is changed to nanoscale, properties such as melting point, fluorescence, electrical conductivity, magnetic permeability, and chemical reactivity change as a function of the size of the particle (<http://www.nano.gov/nanotech-101/special>). In this chapter we discuss the unique properties of the nanoscale materials, polymeric materials and the factors dominating the evolution of unique properties in polymer nanocomposites (PNCs).

POLYMER NANOCOMPOSITES

Nano composite (NC) is a conventional material reinforced by nanoscale particles or nanostructures dispersed through the bulk material. NC has at least one component in nanoscale. PNCs are nanoparticles (spheres, rods, and plates) dispersed in a polymer matrix. The principle composition of typical composite materials is illustrated in Figure 1.



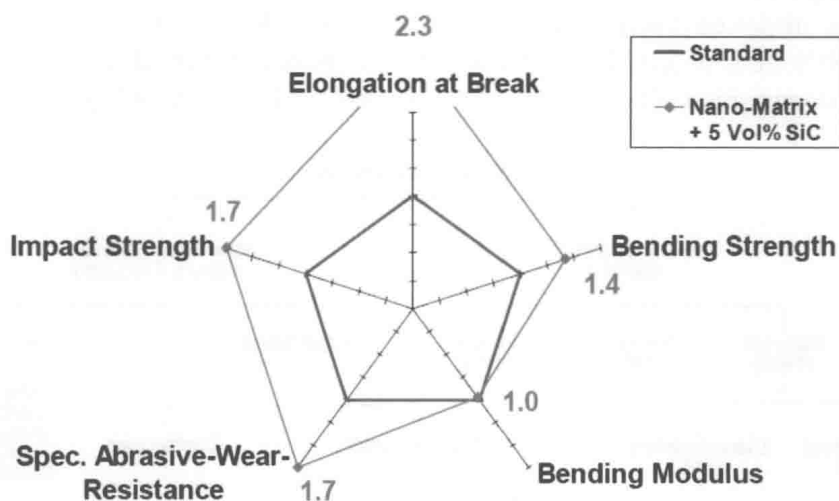
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Figure 1. Systematic illustration of the structural components of composite materials.

In the light of global polymer production, worth 100-200 million tonnes annually, nanoparticle additions to plastics affords one of the commercially largest and diverse applications of nanotechnology. PNCs exhibit different mechanical, electrical, optical, catalytical and structural properties different from that of individual components. Nanoparticle dispersion has been shown to enhance physical (e.g., barrier, erosion resistance, and reduced flammability), thermomechanical (e.g., heat distortion temperature, thermal expansion coefficient, and stiffness) and processing (e.g., surface finish and melt strength) characteristics [Vaia and Maguire, 2007].

Performance enhancement of polymer nanocomposites capitalizes on advantages afforded by up to a 3 orders of magnitude spatial refinement of morphology relative to traditional micrometer-scale filled polymers and composites. This contrasts nanotechnology in electronics, optics, and data storage, where the nanoscale provides access to new physical processes based on quantum phenomenon. The mechanical, electrical, thermal, optical, electrochemical, catalytic properties of the nanocomposite differ markedly from that of the component materials (Figure 2). The length scale of the morphology becomes critically important as the dimensions of particle and polymer, as well as the interfacial curvature and interparticle distance, become comparable. Size limits for novel properties are typically < 5 nm for catalytic activity, < 20 nm for making a hard magnetic material soft [Banerjee et al.

2013], <50 nm for refractive index changes, and <100 nm for achieving superparamagnetism, mechanical strengthening or restricting matrix dislocation movement.



Reproduced with permission: Friedrich, K., Chang, L., Hauptert, F.: 'Current and Future Applications of Polymer Composites in the Field of Tribology', in: L. Nikolais, M. Meo, E. Miletta (eds.): Composite Materials, Springer, New York, USA, 2011, <http://www.springer.com/materials/special+types/book/978-0-85729-165-3>.

Figure 2. Illustration of property improvements by nanofillers.

FACTORS CONTROLLING THE NC PROPERTIES

The nanoparticles lose their quantum size effect as their concentrations increase in the matrix, with the dispersion state of submicron particles determining the final properties of the PNCs. Fine tuning of size dependent properties in PNCs depends on a) dielectric function of material [Thomas et al. 2012], b) the surroundings, c) the shape and the size of the nanoparticle, d) the distance between the nanoparticles, e) the filler concentrations, and finally, f) the interface.

The NC properties are defined by three major characteristics [Anandhan and Bandyopadhyay 2011, Starr et al., 2001; Starr, et al., 2002]:

- 1) the nanoscale inorganic component,
- 2) the nanoscopically confined matrix polymer, and
- 3) arrangement of the inorganic component within the matrix.

1. The Nanoscale Inorganic Component

Nano-scale is defined as the size range between approximately 1 nanometer and 100 nanometer. A restriction of the definition of nanoscience and nanotechnology is that new functionalities are made available by manipulation of matter at this scale or through specificities of the nano dimensions, where the physical, chemical and biological properties of materials

differ from those of the bulk matter. Nano-scale definition of 1-100 nm has incited a series of debates. It is argued that something can be described as nano-scale if it indeed shows a discontinuity, compared with the bulk, with respect to the number of phenomena emerging distinctively different from bulk behaviour [Ramsden and Freeman, 2009]. The search for discontinuities and crossovers around the size of a few nanometers revealed that the nano-scale itself is property dependent, the scale depending on external variables such as temperature and pressure. What appears indubitably as a nano-scale phenomenon at one temperature might cease to be thus distinguished when hotter or colder. Scale at which discontinuity compared to bulk emerges for different properties at room temperature is compiled in Table 1 [Ramsden and Freeman, 2009]. It is also debated that biological ‘unique properties’ are not constrained to sizes less than 100 nm, rather 1–1000 nm scale is recommended for the purposes of dosimetry and toxicological assessment [Teeguarden et al., 2007].

A fascinating and powerful result of the quantum effects of the nanoscale is the concept of “tunability” of properties [http://www.nano.gov/nanotech-101/special]. That is, by changing the size of the particle, a material property of interest can be literally fine-tuned. In this regard it is essential to consider the intrinsic and the extrinsic properties of the nanomaterials themselves.

Table 1. Summary of nano-scale defining lengths

Domain	formula	typical value / nm ^a
Surfaces	geometry	5
Nucleation	critical nucleus size (6)	5
Optics and electronics	Bohr radius (7)	10
Magnetism	single domain size	50
Mechanics	Griffith length (13)	50
Systems		
Electronic	electron mean free path	100
Photonic	evanescent field penetration depth	100
Fluidic	Debye length (30)	10
Mechanical	phonon mean free path	1000

^a At room temperature.

Ramsden and Freeman, 2009.

Intrinsic versus Extrinsic Nano-Scale Properties

Intrinsic properties of manufactured nanomaterials (MNs) concern the material itself. Chemical composition leads to intrinsic properties [Thomas Selvin et al. 2012 in JAPs and JMS]. Size and shape can also underpin some intrinsic properties. The extrinsic properties are concerned with the functionality associated with the MNs. (http://2020science.org/2009/09/11/living-in-a-post-chemistry-world-the-regulatory-challenges-of-emerging-nanotechnologies/ Living in a post-chemistry world – the regulatory challenges of emerging nanotechnologies, Science, 2009, 2020). For example, the intrinsic magnetic properties of the nanomaterial do not vary considerably with size. The new magnetic behaviour different from their bulk is attributed to their extrinsic

properties resulting from their interactions and is strongly dependent on their microstructure [Beke, et al., 2004; Banerjee et al. 2013].

Factors contributing to different properties of nano-scale materials compared to the macro-scale materials are discussed below [Filipponi and Sutherland, 2010]:

Gravitational Force

Gravitational force becomes negligible, as it is a function of mass and distance and is weak between (low-mass) nanosized particles, because the mass of nanoscale objects is so small.

Electromagnetic Forces

Electromagnetic forces dominate at nanoscale. Gravitational force is a function of mass and distance and is weak between (low-mass) nanosized particles. Electromagnetic force is a function of charge and distance, and is not affected by mass, so it can be very strong even when we have nanosized particles under dominance of electromagnetic forces.

Surface to Volume Ratio

Surface to volume ratio increases in nanomaterials, having a significant effect on the nanoparticles properties. Firstly, nanoparticles have a larger surface area when compared to the same volume of the material. For example, 1 kg of particles of 1 mm³ has the same surface area as 1 mg of particles of 1 nm³. [Trantakis, 2012] As particle size decreases, a greater portion of the atoms are found at the surface compared to those inside, having profound effect on reactions that occur at the surface such as catalysis reactions, detection reactions and the reactions that require physical adsorption of certain species for the reaction to occur. For example, a particle of size 3 nm has 50 % of its atoms on its surface, at 10 nm 20 % of its atoms and at 30 nm has 5 % of its atoms on its surface, thereby 3 nm size particles leading to chemically super- highly reactive nanoparticles.

The larger fraction of the atoms on the surface influences physical properties such as melting point, which happens to be lower for the nanosized materials. This is due to the ease with which the surface atoms, which require less energy to overcome the intermolecular forces holding the atoms together, can be removed easily from bulk atoms [Burda, et al., 2005]. The atoms and molecules at the interface enhance reactivity and high surface energy, with a greater tendency to agglomerate. At nanoscale weak forces (like electrostatic and Van der Waals) result in strong interactions when the surface area is large. Materials which are inert in their bulk form, e.g., gold are reactive in their nanoscale form. Therefore a given mass of nanomaterial will be much more reactive than the same mass of material made up of large particles.

Also, surface effects become significant because of the large surface to volume ratio of very small particles [Thielsch, 1998, Brus, 1984, Steigerwald, et al., 1990, Wang, et al., 1991]. The resulting physical properties are neither those of bulk metal nor those of molecular compounds, but they strongly depend on the particle size, interparticle distance, nature of the protecting organic shell, and shape of the nanoparticles [Brust and Kiely, 2002].

Random Molecular Motion

At the macroscale level random molecular motion (molecules move due to their kinetic energy above absolute zero) is very small compared to the size of the particles. At the nanoscale these motions can be of same scale as the size of the particles thus behaving differently, a typical example being the Brownian motion.

Wave-Particle Duality

As the size of the matter decreases, electron wave-like nature dominates and exhibits tunnelling effect (tunnelling is the penetration of an electron into an energy region that is classically forbidden) where the thickness of the barrier or the energy potential is comparable to the wavelength of the particle, observed only at nanoscale.

Electrical Properties

Semiconductor nanocrystallites exhibit interesting electronic, optical and photochemical properties differing from their bulk counterpart. The size induced metal-insulator transition is observed if the metal particle is small enough (about 20 nm), when that size-dependent quantization effects can occur [Daniel and Astruc, 2004].

The nanoparticles exhibit energy separation between the valence band and the conduction band, called the band gap, unlike in bulk metals. The band gap energy increases as the size of the crystallites decreases. The increase in bandgap is caused by the quantum confinement of freely mobile electrons trapped in quantum boxes leading to discrete energy states of electrons in a nanoscale metal structure - implying more energy states are lost due to shrinking size, increasing the energy band gaps in the diameter range 1-10 nm (intermediate between the size of small molecules and that of bulk metal) [Alivisatos, 1996].

Under spatial confinement, electron energies are quantized, leading to many novel effects, collectively known as quantum size effect (QSE) [Chen, et al. 2003]. This quantum size effect arises at dimensions comparable to the Bohr-diameter of the exciton when the particles behave electronically as zero-dimensional quantum dots (or quantum boxes). The quantized energy spectrum has important consequences on electronic applications. The quantum confinement also affects the mass transport and stability of the shape of the nano-interconnect lines.

Optical Properties

The nanoscale materials show different optical properties, unlike their bulk counterpart. The increase in bandgap seen in nanoscale material due to quantum confinement leads to absorption in shorter wavelength resulting in blue shifts. When the particle radius is less than the exciton radius, photon induced transitions in exciton energy levels produce series of discrete optical absorption levels. At such crystallite dimensions the photo generated 'electron-hole'- pairs are spatially confined if the diameter of the crystallites is smaller than the Bohr-diameter of the exciton. Freely mobile electrons trapped in such quantum boxes show a characteristic collective oscillation frequency of the plasma resonance, giving rise to the so-called plasmon resonance band (PRB) observed near 530 nm in the 5-20 nm diameter range. As a result colloids of metal nanoparticles such as gold and silver display colours different from their bulk counter parts like red, purple, or orange which depends upon the dielectric function of material and the surroundings and to the shape and the size of the nanoparticle. The PRB is also sensitive to the distance between the nanoparticles. [Pinchuk and Schatz, 2008]

Magnetic Properties

Magnetic materials such as Fe, Co, Ni, Fe_3O_4 show size-dependent magnetic properties. Magnetic behaviour of a material depends on the structure of the material and on its temperature. The typical size of classical magnetic domain is $\sim 1 \mu\text{m}$. When the size of

magnetic domain reaches nanoscale, these materials show new properties (Banerjee et al. 2103) due to quantum confinement like magneto resistance effect, a property of a material to change the value of its electrical resistance when an external magnetic field is applied to it. The strength of a particle's internal magnetic field and the 'coercive force' (or magnetic memory) needed to reverse an internal magnetic field within the particle is size dependent. The size of magnetic nanoparticles influences the saturation magnetization increasing significantly below a grain size of 20 nm [Chinnasamy, 2000]. For example, saturation magnetization, M_s , of zinc ferrite increases significantly below 20 nm from 1 to 4.5.

The effects of the size of the nanoparticle grain structure on $\text{Nd}_2\text{Fe}_{14}\text{B}$ indicate that in this material the coercive field decreases significantly below 40 nm and the remnant magnetization increases. This is because when the magnetic interactions extend to a distance of more than one neighbouring particle, the reversal of magnetisation becomes easier and leads to a drop in the coercivity [Manaf, et al., 1991].

Mechanical Properties

Deviations from the Hall-Petch behaviour have been observed for materials made of particles less than 20 nm in size, where the nanosize materials having smaller grain size exhibit increased yield strength. This is because the conventional dislocation-based deformation is not possible in bulk nano-structured materials with sizes < 30 nm as mobile dislocations are unlikely. The yield strength, σ_y , of a conventional grain-sized material is related to the grain size by the Hall-Petch equation:

$$\sigma_y = \sigma_0 + Kd^{(-1/2)} \quad (1)$$

where,

σ_0 is the frictional stress opposing dislocation movement, or yield strength at infinite grain size,

K is a constant, and

d is the grain size in micrometers.

One method of increasing the stress at which the brittle-to-ductile transition occurs is to impede the movement of the dislocations by introducing tiny particles of another material into the lattice. This process is used to harden steel, where particles of iron carbide are precipitated into the steel. The iron carbide particles block the movement of the dislocations. Stress needed to fracture bulk glass is around 100 MPa (14500 psi), while the theoretical stress needed for breaking atomic bonds is approximately 10,000 MPa (1,450,000 psi), indicating that the fracture stress increases as the fibre diameter decreases, hence the improved uniaxial tensile strength. For nanostructured Cu-Fe alloy fracture occurs at 2.8 GPa which is about five times the fracture stress of iron having larger grain sizes, ranging from 50 to 150 μm [He and Ma, 2000].

Polymers

Polymers are very light in weight with significant degrees of strength [Anandhan & Bandyopadhyay 2011 Sengupta et al. 2011, Thomas Selvin et al. 2012]. They show low density, low coefficient of friction, good corrosion resistance and poor tensile and