

# Abstract book for the proceedings of the 1st International conference on structural nano composites (NANOSTRUC 2012), 02-04 July 2012, Cranfield, UK.

SACHSE, S., NJUGUNA, J. (eds.)

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# NANOSTRUC 2012

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## ABSTRACT BOOK



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THIS ABSTRACT BOOK CONTAINS ABSTRACTS SUBMITTED TO THE FIRST INTERNATIONAL CONFERENCE ON STRUCTURAL NANO COMPOSITES. THIS ABSTRACT BOOK DIFFERS FROM THE ABSTRACT BOOK GIVEN AS HARD COPY DURING THE CONFERENCE AND CONTAINS ADDITIONAL ABSTRACTS.

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# PREFACE



Dear Colleagues,

It is a great pleasure to welcome you to Nanostruc2012 at Cranfield University. The purpose of the 2012 International Conference on Structural Nano Composites (NanoStruc 2012) is to promote activities in various areas of materials and structures by providing a forum for exchange of ideas, presentation of technical achievements and discussion of future directions.

NanoStruc brings together an international community of experts to discuss the state-of-the-art, new research results, perspectives of future developments, and innovative applications relevant to structural materials, engineering structures, nanocomposites, modelling and simulations, and their related application areas. The conference is split in 7 panel sessions, Metallic Nanocomposites and Coatings, Silica based Nanocomposites, safety of Nanomaterials, Carbon based Nanocomposites, Multiscale Modelling, Bio materials and Application of Nanomaterials.

All accepted Papers will be published in the IOP Conference Series: Materials Science and Engineering (MSE), and included in the NanoStruc online digital library. The abstracts will be indexed in Scopus, Compedex, Inspec, INIS (International Nuclear Information System), Chemical Abstracts, NASA Astrophysics Data System and Polymer Library.

Before ending this message, I would like to acknowledge the hard work, professional skills and efficiency of the team which ensured the general organisation.

As a conclusion, I would like to Welcome you to the Nanostruc2012 and wish you a stimulating Conference and a wonderful time.

On behalf of the scientific committee,



James Njuguna

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## SESSION CHAIRS

**Panel session 1.1 - Metallic Nanocomposites and Coatings (Chaired by Krzysztof Pielichowski, Cracow University of Technology, Poland)**  
**Panel session 1.2 - Silica based Nanocomposites (Chaired by Rob Dorey, Cranfield University, UK)**  
**Panel session 1.3 - Safety of Nanomaterials (Chaired by James Njuguna, Cranfield University, UK)**  
**Panel session 2.1 - Carbon based Nanocomposites (Chaired by Aravid Dasari, Nanyang Technological University, Singapore)**  
**Panel session 2.2 - Multiscale Modelling (Chaired by Jianqiao Ye, University of Leeds, UK)**  
**Panel session 2.3 - Bio materials (Chaired by Kambiz Kayvantash, CADLM, France)**  
**Panel session 3.1 - Application of Nanocomposites (Chaired by Davide Roncato, Centro**

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**Jenny Kay** - Cranfield University, UK

# PROGRAMME AT A GLANCE

## NANOSTRUC DAY 1 - 2<sup>ND</sup> JULY 2012

Time	Programme point
9.30-10.00	Registration
10.00-10.30	Welcome and Opening session
10.30-12.30	Panel session 1.1 -Metallic Nanocomposites and Coatings ( Chaired by Krzysztof Pielichowski, Cracow University of Technology, Poland)
12.30-13.30	Lunch break
13.30-15.10	Panel session 1.2 - Silica based Nanocomposites (Chaired by Rob Dorey, Cranfield University, UK)
15.10-15.40	Coffee Break
15.40-16.50	Panel session 1.3 - Safety of Nanomaterials (Chaired by James Njuguna, Cranfield University, UK)
18.00-20.00	Poster Session with wine reception

## NANOSTRUC DAY 2 - 3<sup>RD</sup> JULY 2012

Time	Programme point
9.00-9.30	Good Morning Coffee
9.30-12.00	Panel session 2.1 - Carbon based Nanocomposites (Chaired by Aravind Dasari, Nanyang Technological University, Singapore)
12.00-13.00	Lunch break
13.00-15.30	Panel session 2.2 - Multiscale Modelling (Chaired by Jianqiao Ye, University of Leeds, UK)
15.20-15.50	Coffee Break
15.50-17.40	Panel session 2.3 - Bio materials (Chaired by Kambiz Kayvantash, CADLM, France)
16.40-17.20	Panel discussion (Chaired by Dirk Lehmkus, University of Bremen)
19.00-23.00	Gala Dinner at the Luton Hoo

## NANOSTRUC DAY 3 - 4<sup>TH</sup> JULY 2012

Time	Programme point
9.00-9.30	Good Morning Coffee
9.30-13.15	Panel session 3.1 - Application of Nanocomposites (Chaired by Davide Roncato, Centro Ricerche Fiat, Italy and Dirk Lehmkus, University of Bremen)
11.10-11.30	Coffee Break
12.50-13.15	Best Poster Award
13.15-13.30	Closing Words
13.30-14.30	Lunch

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### Silver Nanowires/Polycarbonate Composites for Conductive Films

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Transparent conductive thin films are widely used in electronic devices, such as touch screen panel, solar cell, thin film transistors and field emission displays. One-dimensional nanostructured materials, such as nanotubes, nanorods and nanofibers have received much research interests as conducting nanofillers for polymeric matrices, especially carbon nanotubes. Even though silver nanowires are particularly interesting as filler because bulk silver has the highest electric conductivity among all metals, there are very few publications dealing with AgNW/polymeric composites. In this work synthesized silver nanowires were introduced in a polycarbonate matrix in order to obtain transparent electrical conductive films.

Silver nanowires have been synthesized via a solvothermal method where the nanowires growth in a polyol process assisted by PVP as capping agent. The obtained silver nanowires were characterized using several techniques. From electron microscopies, they were found to be around 110 nm in diameter and 10  $\mu\text{m}$  in length. In addition, XRD, SAED, TGA, UV-Vis spectroscopy techniques have been used to investigate the structure, morphology and composition of these nanowires.

Nanocomposites of polycarbonate as matrix and silver nanowires as filler were prepared using casting combined with a pull rod in order to obtain thin films. Nanocomposites with loading of AgNW between 0,04wt.% and 4,35wt.% were prepared. The distribution of the nanowires in the matrix was even and uniform for all the loadings as shown by electron microscopies and optical microscopy, no agglomerates were observed. Also, TGA indicates an increase in the degradation temperature of the polymer. In order to measure the electron conductivity of the prepared films, an impedance analyser combined with a conductivity cell was used. The presence of the wires strongly increases the conductivity of the polymeric matrix. Percolation has been obtained at low filler concentration and maximum conductivity obtained was about  $10^{-2}$  S / cm.

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### 3-Dimensional Free Standing Micro-Structures by Proton Beam Writing of Su 8-Silver Nanoparticle Polymeric Composite

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Proton beam lithography a maskless direct-write lithographic technique (well suited for producing 3- dimensional microstructures in a range of resist and semiconductor materials) is demonstrated as an effective tool in the creation of electrically conductive freestanding micro-structures in an Su 8 + Nano Silver polymer composite. The structures produced show non-ohmic conductivity and fit the percolation theory conduction model of tunneling of separated nanoparticles. Measurements show threshold switching and a change in conductivity of at least 4 orders of magnitude.

The predictable range of protons in materials at a given energy is exploited in the creation of high aspect ratio, free standing micro-structures, made from a commercially available SU8 Silver nano-composite (GMC3060 form Gersteltec Inc. a negative tone photo-epoxy with added metallic nano-particles(Silver)) to create films with enhanced electrical properties when exposed and cured. Nano-composite films are directly written on with a finely focused MeV accelerated Proton particle beam.

The energy loss of the incident proton beams in the target polymer nano- composite film is concentrated at the end of its range, where damage occurs; changing the chemistry of the nano-composite film via an acid initiated polymerization - creating conduction paths. Changing the energy of the incident beams provide exposed regions with different penetration and damage depth – exploited in the demonstrated cantilever microstructure.

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## A New-Developed Nanostructured Mg/Hap Nanocomposite by High Frequency Induction Heat Sintering Process

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The objective of the present study was to investigate the effect nano-hydroxyapatite contents on the mechanical and microstructural properties of magnesium nanocomposites in order to develop a new biodegradable hard tissue substituent. Mg/HAp nanocomposite with various HAp contents (0-10 wt %) were prepared using pure magnesium and HAp nanopowder as raw materials. The starting material was super fast densified by high frequency induction heat sintering (HFIHS). The results indicated that, a uniform distribution of HAp particles was observed along the boundary between matrix particles. Nano-crystalline grains with a crystal size range of 37-50 nm were obtained. The relative densities and microhardness of the composites initially increased with increase the amount of HAp addition. Despite the short dwelling time when the current was applied, the relative density and microhardness of the sintered samples reached as high as 99.7 % and 70 HV respectively, in the composite containing 1 to 3 wt% HAp. Addition of 1 to 3 wt% of HAp improved compression strength of Mg by 16%. Addition of HAp decreases the crystal size of the nanocomposites. The mechanical properties, i.e. hardness and compressive strength are evidently increased with increasing HAp content up to 2 wt%. However, when the HAp content was larger than 2 wt%, the compressive strength decreased due to the agglomeration of HAp particles. The hardness, compressive and ultimate stress this composite

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## Corrosion Behaviour of Pristine and Added MgB<sub>2</sub> in Phosphate Buffered Saline Solution

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We have obtained by Spark Plasma Sintering (SPS), dense samples of MgB<sub>2</sub> added with Ho<sub>2</sub>O<sub>3</sub>. Starting composition was (MgB<sub>2</sub>)<sub>0.975</sub>(HoO<sub>1.5</sub>)<sub>0.025</sub> and we used addition powders with an average particle size below and above 100 nm. For Mg, pristine and added MgB<sub>2</sub> samples we measured potentiodynamic polarization curves in Phosphate Buffered Saline (PBS) solution media at room temperature. MgB<sub>2</sub> based composites show corrosion/ degradation effects. This behavior is in principle similar to Mg based alloys in the same media. Our work suggests that the different morphologies and phase compositions of the SPS-ed samples influence the interaction with corrosion medium; hence additions can play an important role in controlling the corrosion rate. Pristine MgB<sub>2</sub> show a significant improvement of the corrosion resistance, if compared with Mg. The best corrosion resistance is obtained for pristine MgB<sub>2</sub>, followed by MgB<sub>2</sub> with nano-Ho<sub>2</sub>O<sub>3</sub> and μ -Ho<sub>2</sub>O<sub>3</sub> additions.

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### Cast Iron Cutting with Nano TiN and Multilayer TiN-CrN Coated Inserts

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During the past decade great success has been achieved in the development of duplex and multilayer multi-functional surface systems. Among these surface systems outstanding properties have nanoscale multilayer coatings. Within the framework of the M3-2S project funded in the 7th European Framework Programme, several nanoscale multilayer coatings have been developed and investigated for experimental and industrial validation. This paper shows the performance of TiN and TiN/CrN nanoscale multilayer coatings on WC cutting inserts when machining GJL250 cast iron. The thin films have been deposited by cathodic arc evaporation in an industrial PVD system. The multilayer deposition characteristic and its properties are shown. The inserts have been investigated in systematic cutting experiments of cast iron bars on a turning machine specifically equipped for force measurements, accompanied by wear determination. Furthermore, equivalent experiments have been carried out on an industrial turning unit. Industrial validation criteria have been applied to assess the comparative performance of the coatings. The choice of the material and the machined parts is driven by an interest in automotive applications. The industrial tests show the need to further optimise the multi-scale modelling approach in order to reduce the lead time of the coating development as well as to improve simulation reliability.

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## Silver Nanoclusters Silica Composite Coating on Polymers for Space Application

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The microbiological contamination on board of spacecraft and orbital stations is a relevant problem in prolonged space exploration<sup>1</sup>. The interior of a space structure can be easily colonized by microbes and fungi because of the suitable environment created by the presence of humans and conditions<sup>2,3</sup>. At the same time, the polymeric materials are widely used in the space field even if they are susceptible to colonization of microorganisms and subjected to deterioration and degradation<sup>2</sup>.

For this purpose, an antibacterial silver nanocluster silica composite coating<sup>4,5</sup> was deposited on commercial polymers suitable for aerospace application, using the radio frequency co-sputtering technique. Silver as antimicrobial agent is currently exploited under several forms in a wide range of applications<sup>6</sup>. A multilayer film composed of polyethylene, nylon and ethylene vinyl alcohol called Combitherm<sup>®</sup> and a fabric of aramidic fibers are used as substrates.

The coating structure was confirmed by energy dispersion spectrometry (EDS), x-ray photoelectron spectroscopy (XPS) and localized surface plasmon resonance (LSPR). The atomic force microscope (AFM) evidenced the coating morphology. The antimicrobial behavior was verified through evaluation of the inhibition halo against several bacterial and fungal species discovered into the space station<sup>2,3</sup>. The coating enhanced the nano-hardness and the resistance to tensile and perforation tests; the coating wear resistance was measured by abrasion test against Kevlar. A folding procedure on the coated

<sup>1</sup> D.L. Pierson, *Gravit. Space Biol. Bull.* 14 (2001) 1-6

<sup>2</sup> J. Gu, *Int. Biodeterior. Biodegrad.* 59 (2007) 170-179.

<sup>3</sup> N. Novikova et al., *Res. Microbiol.* 157 (2006) 5-12

<sup>4</sup> M. Ferraris et al., *Mater. Chem. Phys.* 120 (2010) 123-126

<sup>5</sup> M. Ferraris, et al., *Adv. Eng. Mater.* 12 (2010) B276-B282.

<sup>6</sup> N. Masuda et al., *J. Biomed. Mater. Res. Part B: Appl. Biomater.* 83B (1) (2007) 114-120



Combitherm<sup>®</sup> and storage in air for three months was also carried out without deterioration of the measured properties. The coating deposition did not influence the air permeability of the substrates.

This work was funded by the project “NABLA-Nanostructured Antibacterial Layers” from Regione Piemonte, Italy.

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## Al-Al<sub>3</sub>Ti Composite Produced In Situ by Two-Step Hot-Press Sintering

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Aluminum reinforced with a large amount (up to about 55 vol.%) of micrometer-sized Al<sub>3</sub>Ti particles can be fabricated from Al-20Ti elemental nanometer-sized powder mixture via in-situ two step hot press sintering (TSS). For production of intermetallic reinforced in-situ composite, TSS can provide: elevated temperature to facilitate the formation of intermetallic phase in situ and hot consolidation to form a fully dense solid. The first step sintering was employed at a higher temperature to obtain an initial high density, and the second step was held at a lower temperature by isothermal sintering for more time than the first one to increase bulk density without significant grain growth. The optimum TSS regime consisted of heating at 625 °C for 5 min (1st step) and 470 °C for 40 min with applied pressure of 470 MPa, resulting in the formation of near full dense microstructure (0.97 TD) with suppressed grain growth.

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## **Polymer (PTFE) and Shape Memory Alloy (NiTi) Intercalated Nano-Biocomposites**

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Engineering on a nano-scale has been undertaken to mimic a biomaterial by forming an intercalated nano-composite structure by PVD sputtering of a polymer with a nickel-titanium (NiTi) shape memory alloy (SMA). A PTFE polymer has been selected due to its elastic properties, low interactions with water, optimum surface energies, stability and chemical resistance. NiTi SMAs allow the coatings to be energy absorbent and thus suitable in load bearing situations. The coatings are aimed to constantly withstand variable adverse biological environments whilst maintaining their characteristics. The nano-intercalated structures have been characterised for their wettability, friction coefficients, chemical composition, and morphology. Intercalation of a polymer with energy-absorbing alloys uncovers a set of material systems that will offer characteristics such as self-healing of hierarchal tissue in the body. The reformation of PTFE following sputter deposition was confirmed by FTIR spectra. According to SEM analysis PTFE shows a promising surface interaction with NiTi, forming stable coatings. Surface interactions are evident by the hydrophobic behaviour of films as the composite's water contact angle is around  $86^{\circ}$ , which lies in-between that of PTFE and NiTi. The nano composite films are lubricious and have a measured CoF below 0.2 which does not vary with layer thickness.

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### Thermoplastic Polymer Nanocomposites with Montmorillonite - Lab Vs Industrial Scale Fabrication

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Fabrication of polypropylene or polyamide-6/montmorillonite nanocomposites in a laboratory- and industrial-scale by melt processing has been described. Depending on the fabrication scale, different technological problems were observed that need to be taken into account during production of PP and PA-6-based nanocomposites with MMT which constitute a promising class of modern materials for engineering and packaging applications. Fabrication of PP and PA-6 nanocomposites with montmorillonite both on laboratory and industrial scale needs to be optimized in terms of processing conditions that include pre-mixing parameters, feeding rate, temperature of the barrel zone and extrusion die, screw speed and head pressure. Proper cleaning procedures need to be applied and the selection of the 'main' fraction enables to get nanostructured materials with enhanced properties.

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## Fire Retardancy Behavior of Polymer Nanocomposites

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The usage of conventional flame retardants (particularly based on halogen compounds) in polymers and the ultimate disposal of these composites are associated with multi-faceted negative effects on the eco-system. This has instigated a search for alternatives. With high aspect ratio nanoparticles in polymers, inspiring preliminary flame retardancy results like reduced heat release and mass loss rates and delayed burning were obtained. Despite this, they are unable to meet the requirements of fire safety standards. Briefly, in my talk, I will highlight the reasons behind this and the issues that have to be resolved. Further, I will present our recent efforts to understand some of these issues. Emphasis will be on understanding some of the important parameters in the combustion behavior of polymer nanocomposites such as thickness of samples, ease of ignition, catalytic activity of clay particles, loading of nanoparticles *versus* reductions in heat release rates and delay in burning.

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## Elastic Behavior of Silica/Poly(dimethylsiloxane) Nanocomposites. Nano Size Effects

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Elastomeric materials require the incorporation of reinforcing fillers in order to improve their mechanical properties. The enhancement of properties is very much dependent on the size and any surface modification of the reinforcing agent. It is widely accepted that the reinforcement effects are primarily due to molecular interactions of the polymeric matrix and the filler inclusions and it involves both chemical and physical interactions.

Herein, we have incorporated silica nano fillers (Stober silica) into poly (dimethylsiloxane) (PDMS) elastomeric networks. The mechanical and swelling properties of the networks were investigated as a function of filler sizes (50, 130, and 170 nm), volume fraction of the filler inclusions, and surface treatment of the particles by were analyzed to give the Mooney-Rivlin constants  $2C_1$  and  $2C_2$ . These properties were found to be dependent on the size of the nano inclusions. Thus, a nano size phenomenon had been discerned and was one of the highlights of this investigation. This phenomenon was largely attributed to the high specific surface area of the nano fillers used that leads to significant increase in the interfacial interactions. Also, and as would be expected, the properties of the polymeric networks filled with unmodified particles were different than those obtained for the polymeric networks filled with surface-modified particles. This has been primarily attributed to changes in the surface properties, and as would be expected, the elastic properties of the networks were thus shown to be strongly dependent on type and concentration of the filler.

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## Advanced Materials Based on Polymer Blends/Polymer Blend Nanocomposites

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Processability, morphology, mechanical properties and rheological behavior of poly(vinylchloride) (PVC)/poly(ethylmethacrylate) (PEMA) blends and PVC/PEMA/montmorillonite (MMT) composites, prepared by melt processing in a brabender mixer, were studied. Samples were characterized using SEM, mechanical testing, DMTA and a parallel plate rheometer. Plastograms show that there is noticeable drop of fusion times and increase in melt viscosity torque of both, polymer blend and polymer blend nanocomposite, in comparison with those of neat PVC. SEM images show that homogenous dispersions are obtained. Tensile tests indicate that PVC/PEMA and PVC/PEMA/MMT samples have greater tensile strength and elastic modulus and lower elongation compared to PVC. When solid viscoelastic properties are considered (DMTA), slightly higher storage moduli are obtained whereas more prominent increase of storage modulus is observed when nanoclay particles are added in a PVC/PEMA matrix. From the calculated area of tandelta peak of all tested samples, nanocomposites exhibit the lowest damping behavior. Oscillatory measurements in a molten state were used for determining the frequency dependencies of storage  $G'$  and loss  $G''$  moduli. It was found that  $G''$  curves of neat PVC lie above those of  $G'$  suggesting that PVC behaves like viscoelastic liquid. Similar results, but with significantly higher values of  $G'$  and  $G''$  over the whole frequency range for PVC/PEMA blends were obtained. Steady shear measurements show that the presence of PEMA and nanoclay particles increases the shear stress and shear viscosity of neat PVC. In order to define the rheological equations of state the three material functions were determined. According to these functions ( $\eta(\dot{\gamma})$ ,  $\psi_1(\dot{\gamma})$ ,  $\psi_2(\dot{\gamma})$ ) all samples exhibit shear thinning behavior and the curves obey the power law equation.

As rheological behaviour was found to be strongly dependent on blend's micro and macro structure and it is one of the main factors defining the end properties, attempt was made to use this material functions to define the structure-properties relationship of polymer blends/polymer blend nanocomposites.

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## Energy Absorption Characteristics of Nano-Composite Conical Structures

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The effect of the filler material on the energy absorption capabilities of polyamide 6 composite structures is studied in details in the present paper. The axial dynamic and quasi-static collapse of conical structures was conducted using a high energy drop tower, as well as Instron 5500R electro-mechanical testing machine. The impact event was recorded using a high-speed camera and the fracture surface was investigated using scanning electron microscopy (SEM). The obtained results indicate an important influence of filler material on the energy absorption capabilities of the polymer composites. A significant increase in specific energy absorption (SEA) is observed in polyamide 6 (PA6) reinforced with nano-silica particles (SiO<sub>2</sub>) and glass-spheres (GS), whereas addition of montmorillonite (MMT) did not change the SEA parameter.

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## Dielectric Behavior of Silica/Poly(dimethylsiloxane) Nanocomposites - Nano Size Effects

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The enhancement of properties of elastomeric composite materials is very much dependent on the size and the surface modification of the reinforcing filler inclusions. It is well accepted that the reinforcement effects are primarily due to molecular interactions of the polymeric matrix and the filler inclusions and it involves both chemical and physical interactions.

In the present study, we have incorporated silica nano fillers (Stober silica) into poly(dimethylsiloxane) (PDMS) elastomeric networks. The dielectric properties of the networks were investigated as a function of filler nano filler size, volume fraction, and surface treatment by hexamethyldisilazane. The broad-spectrum dielectric properties (in particular, the dielectric constant, the dielectric loss, and  $\tan \delta$ ) were characterized. These properties were found to be dependent on the size of the nano inclusions; thus, the results clearly showed a nano size phenomenon that was a highlight of the present research. Certainly, that can be largely attributed to the high specific surface area of the nano fillers, which significantly leads to a pronounced increase in interfacial interactions. Also, and as would be expected, the dielectric properties of the polymeric networks filled with unmodified particles were different from those for the polymeric networks filled with surface-modified particles. Again, this is mainly attributed to changes in the surface properties. The expected dependence of properties of the nanocomposite networks on the nature of the filler and its concentration has thus been demonstrated.

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## Physical Characteristics of Nanoparticles Emitted During Drilling of Silica Based Polyamide 6 Nanocomposites

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During the past decade, polymer nanocomposites have emerged as a novel and rapidly developing class of materials and attracted considerable investment in research and development worldwide. However, there is currently a lack of information available in the literature on the emission rates of particles from these materials. Therefore, the aim of this study was to investigate the effect on different fillers (nanoclay, nanosilica, foam glass crystal and conventional glass fibres) on the dust emission during a mechanical drilling process of reinforced polyamide 6 composites panels.

Airborne particle emission rates ranged from  $1.16\text{E}+07$  ( $\text{min}^{-1}$ ) to  $1.03\text{E}+09$  ( $\text{min}^{-1}$ ) and the size distribution peak diameters varied from 29.6 to 75.1nm. Airborne particles in the nanometer range (11.1-46.8 nm), in the ultrafine range (51.3-101.1 nm) and in the accumulation mode range (111.9-521nm) accounted for 34.1-76.6%, 8.3-47% and 4.1-24.2% of the total emission rates, respectively, depending on the type of filler. Additionally, particle deposition rate was found to be strongly influenced by the filler chosen. Deposited particles might cause emission to soil and water, and hence need to be considered and assessed in addition, to those released to air. Future work will involve a series of comprehensive chemical characterisation of the dust particles released during drilling of nanocomposites to make a better assessment of public health and environmental outcomes due to this exposure.

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### Ecological Assessment of Nano Enabled Super Capacitors for Automotive Applications

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New materials on nano scale have the potential to overcome existing technical barriers and are one of the most promising key technologies to enable the decoupling of economic growth and resource consumption. Developing these innovative materials for industrial applications means facing a complex quality profile, which includes among others technical, economic, and ecological aspects. So far the two latter aspects are not sufficiently included in technology development, especially from a life cycle point of view. Supercapacitors are considered a promising option for electric energy storage in hybrid and full electric cars. In comparison with presently used lithium based electro chemical storage systems supercapacitors possess a high specific power, but a relatively low specific energy. Therefore, the goal of ongoing research is to develop a new generation of supercapacitors with high specific power and high specific energy. To reach this goal particularly nano materials are developed and tested on cell level. In the presented study the ecological implications (regarding known environmental effects) of carbon based nano materials are analysed using Life Cycle Assessment (LCA). Major attention is paid to efficiency gains of nano particle production due to scaling up of such processes from laboratory to industrial production scales. Furthermore, a developed approach will be displayed, how to assess the environmental impact of nano materials on an automotive system level over the whole life cycle.

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## Life Cycle Assessment of the Application of Nanoclays in Wire Coating

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A life cycle assessment (LCA) is carried out to compare nanoclay-reinforced polymer wire coatings with conventional ones. While the conventional wire coatings contain standard halogen free retardants, in reinforced coatings, montmorillonite (nanoclay) is incorporated into electric cable linings as a rheological agent for an increased resistance to fire. In addition, a reduced load of standard halogen free retardants is obtained. The synergistic effect of the montmorillonite on traditional flame retardant additives (by the formation of a three-dimensional char network<sup>1</sup>) can lead to a revolution in wire production. The application of nanoclays contributes also to anti-dripping effect<sup>1</sup> and flexibility increase. Some producers have already started commercializing wire with nanotechnology-based coating; in the short term the use of nanoclay in wire coating production will probably reach a significant market share replacing traditional formulations.

The main aim of this study is to compare the environmental impacts along the life cycle of a traditional wire coating (mineral flame retardants like ATH or MDH in a polymer matrix) with the nanoclayreinforced wire coating, where the montmorillonite replaces a low percentage of the mineral flame retardant. The system boundaries of the study include the following unit processes: nanoclay production, thermoplastic material and mineral flame retardants production, cable coating manufacturing by extrusion and different end of life scenarios (recycling, incineration and landfill disposal).

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## Assessment of Nanoparticle Release and Associated Health Effect of Polymer-Silicon Composites

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The fast advancement in the nanotechnology and manufacturing of engineering nanomaterials has inevitably caused concerns over their impact on environment and human health. Little information is currently available on possible release of nanomaterials or/and nanoparticles (NP) from conventional and novel products and associated health effect. The NEPHH project funded under the EU FP7 programme is aimed to assess the environment and health impact of polymer-silicon nanocomposites in comparison to conventional polymer composites from life cycle perspective. As part of the NEPHH project, this study focused on assessing the possible release of NP during the application stage of conventional and nanoproducts. NP release was monitored during physical processing of polymer-silicon composites, and the toxicity of both the released NP and the raw silica nanomaterials that were used as fillers in nanocomposites was assessed in vitro using human lung epithelial A549 cells.

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## **Rethinking Characterisation: Complexity and Uncertainty in Characterising Nano-objects**

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A conceptual (paradigm) shift is necessary to make real progress in understanding the environmental effects of nano-objects. Questions about characterisation on the nanoscale are often set within the assumptions of higher scale materials and in a reductionist manner. It is often a misconception that a 'full characterisation' of a nanomaterial will enable understanding, prediction and control. A new approach will set questions within the context of complexity, scale variance, time-dimension, ambient conditions and uncertainty.

The nanoparticle's characteristic is a function of the method and instrument that characterised it. In an application the nanoparticle's characteristic is a function of its 'relations', that is, a different scale, ambience and time-slot will mean a different characteristic. At the nanoscale the gap between laboratory and reality will be much bigger than anything we are used to at higher (bulk) scales.

To avoid R&D failures due to the inappropriateness of orthodox characterisation approaches for the particular functionality of a proposed nanotechnology application, nanotechnologists will have to be theoretically creative. In a 'complexity theory' framework nanotechnologists will go beyond 'trial and error' to theoretical approaches; diversify characterisation methods while making them more specific or 'tailor-made' to the particular objective; take novel approaches to the relativity that the characterisation data will have to the method/instrument employed; for meaningful and valid determinations for applications they will take better account of the specific context, ambience and dynamic relations of the material by using multiple methods; and take theoretical account of non-linearities, chaos and uncertainty.

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### **Enhancement of Thermal Conductivity of Materials Using Different Forms of Natural Graphite**

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The quest for improvement of thermal conductivity in every area including aerospace structures is gaining momentum as the modern day technology is embedded with electronics which generate considerable amounts of heat energy. In some other applications, the thermal energy must be absorbed or released at a very fast rate.

Natural graphite is highly anisotropic and has excellent thermal and electrical conductivity in a-direction hence; it is one of the potential candidates to enhance thermal conductivity. This paper encompasses various preparation routes used to enhance thermal conductivity in materials/composites using graphite flakes in its natural and treated forms along with characterization results of thermal conductivity and/ or electrical resistivity where available. Addition of graphite has shown to improve thermal conductivity in some materials by 30-130 times whereas one method of preparation described here shows composites to have thermal conductivity as high as 750 W/m K.

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## Mechanical Stabilisation of Graphene Aerogels by Vulcanization with Pure Sulphur

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Graphene aerogels are very useful materials in those technological applications where high-surface development and good electrical conduction characteristics are required<sup>1</sup>. Such nanostructured materials can be prepared by drying a high concentrated graphene colloid, however the resulting mechanical properties are not enough for technological applications. In order to give strength and to improve other properties, graphene was vulcanized with pure sulphur (at 180°C). In particular, graphene is a very good substrate for chemical functionalization, since its reactivity is comparable with that of other polycyclic aromatic hydrocarbons. The presence of carbon-carbon double bonds (C=C) makes possible radical addition reactions. Sulphur molecules (S<sub>8</sub>) decompose in the molten form, producing linear bi-radicals  $c-S_8 \rightarrow \bullet I-S_8 \bullet$  ( $\lambda$ -transition)<sup>2</sup> which are able to cross-link the graphene-based framework constituting the aerogel. Such a process significantly improves the material mechanical stability (see Figure 1). Scanning electron microscopy (SEM) images of the graphene-sulphur composite (after the vulcanization process) showed such sulphur particles at edges of graphene sheets. To verify the composition of our materials, we carried out energy dispersive spectroscopic (EDS) mapping. Furthermore, differential scanning calorimetry (DSC) was used as a really convenient approach to study this vulcanization process, in fact the complete cross-linking of the graphene structure was evidenced by the disappearance of the pure sulphur phase melting. The amount of residual sulphur in the vulcanized material was evaluated by thermo-gravimetric analysis (TGA) and it corresponded to ca. 30% by weight. A further approach used to investigate the morphology and the structure of vulcanized graphene aerogels has been the large angle X-ray powder diffraction. This material characterization is useful to identify and verify the properties of graphene structures. The large surface area and the high conductivity make

<sup>1</sup> Worsley, M A., Olson, TY., et al., " High Surface Area, sp<sup>2</sup>-Cross-Linked Three-Dimensional Graphene Monoliths", *J. Phys. Chem. Lett*, 2 (2011), 921-925

<sup>2</sup> Eichinger, BE., Wimmer E., et al., " The Structure of Amorphous Sulfur", *Macromol. Symp*, 171 (2001), 45-56



graphene and graphene-sulphur composite excellent candidates for many automotive applications such as electrodes for high performance supercapacitors and for displays, nanoparticles (NGPs) for production of conductive plastic composites, nanofluiding and anti-friction fields.

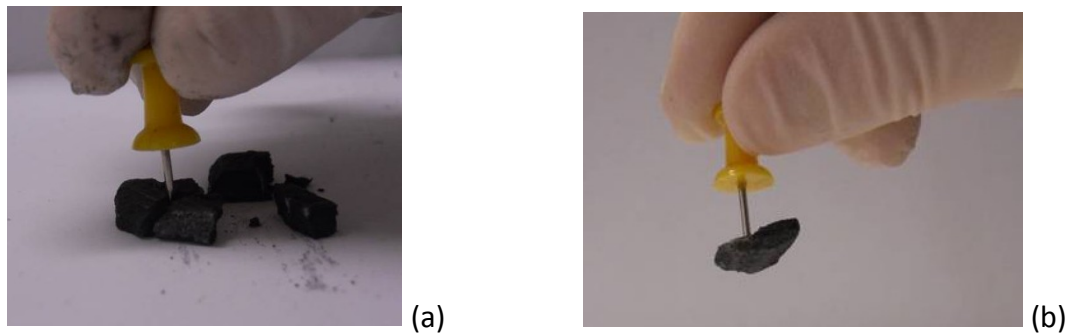


Figure 1. - Graphene aerogel sample before (a) and after (b) the vulcanization treatment.

## Evaluating the Electrical Discharge Machining (EDM) Parameters with Using Carbon Nanotubes

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Electrical discharge machining (EDM) is one of the most accurate non-traditional manufacturing processes available for creating tiny apertures, complex or simple shapes and geometries within parts and assemblies. Performance of the EDM process is usually evaluated in terms of surface roughness, existence of cracks, voids and recast layer on the surface of product, after machining. Unfortunately, the high heat generated on the electrically discharged material during the EDM process decreases the quality of products. Carbon nanotubes display unexpected strength and unique electrical and thermal properties. Multi-wall carbon nanotubes are therefore on purpose added to the dielectric used in the EDM process to improve its performance when machining the AISI H13 tool steel, by means of copper electrodes. Some EDM parameters such as material removal rate, electrode wear rate, surface roughness and recast layer are here first evaluated, then compared to the outcome of EDM performed without using nanotubes mixed to the dielectric. Independent variables investigated are pulse on time, peak current and interval time. Experimental evidences show that EDM process operated by mixing multi-wall carbon nanotubes within the dielectric looks more efficient, particularly if machining parameters are set at low pulse of energy.

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## **Twin-Screw Extrusion of Multi Walled Carbon Nanotubes Reinforced Polycarbonate Composites: Investigation of Electrical and Mechanical Properties**

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1, 3 and 5 wt.% multi walled carbon nanotubes (MWCNT) reinforced polycarbonate (PC) composites were processed in a twin-screw extruder (L/D=52) with two different screw speeds, throughputs and screw configurations. Extruded strands were characterized for dispersion and measurement of electrical resistivities while the pelletized extrudates were injection molded to produce samples for mechanical and further electrical property measurements. The absolute resistance of the melt was recorded with an online melt resistance setup developed by our group. The volume resistivity of pure PC ( $10^{17}$   $\Omega\cdot\text{m}$ ) was lowered to  $10^4 - 10^5$   $\Omega\cdot\text{m}$  on an injection molded PC-1 wt. % MWCNT composite. 3 wt.% MWCNT incorporated composites showed volume resistivity less than 1  $\Omega\cdot\text{m}$  independent of process conditions. At lower filler contents the volume resistivity of injection molded samples were higher than those observed on the extruded strands and this effect diminished with increasing MWCNT loadings; owing to the loss of CNT network contacts due to shear induced filler orientation and core-skin effects. The quality of dispersion was exceptional for all filler concentrations at any process condition owing to the affinity of MWCNT towards PC due to the lower interfacial energy difference between the reactants and high polarity of PC. The modulus and strength of the composites increased with filler addition, however at 5 wt.% filler loading the strength of the composites processed with lower SMEs was less than that observed on the 1 wt.% MWCNT reinforced PC composite. The elongation of the composites at maximum tensile strength were comparable to that of neat PC except for composites with 5 wt.% MWCNT loading processed with lower SMEs. Composites with identical filler loadings which were processed with higher SMEs showed

higher notched impact strength values principally because of the ability of very well dispersed filler fractions to inhibit crack propagation. The significance of the results obtained in this work stems from the fact that we were able to produce composites with substantial improvements in electrical properties with improvements/without significant loss in mechanical properties. This is one of the few exceptions to results achieved with commercially available MWCNT reinforced polymer composites processed on a large scale.

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## High-Performance Supercapacitor Cells with Activated Carbon/Mwnt Nanocomposite Electrodes

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The purpose of this work was to investigate and improve the performance of supercapacitor cells with carbon-based nanocomposite electrodes. The electrode structure comprised activated carbon (AC), four types of multi-wall nanotubes (MWNTs) and two alternative polymer binders, Polyvinyl alcohol (PVA) or Polyvinylidene fluoride (PVDF). Electrode fabrication involved various stages of mixing and dispersion of the AC powder and carbon nanotubes, rolling and coating of the AC/MWNT/binder paste on an aluminium substrate which also served as current collector. The organic electrolyte utilised was 1M tetraethylammonium tetrafluoroborate (TEABF<sub>4</sub>) fully dissolved in propylene carbonate (PC). All devices were of the electrochemical double layer capacitor (EDLC) type, incorporating four layers of tissue paper as separator material. The surface topography of the so fabricated electrodes was investigated with scanning electrode microscopy (SEM). Overall cell performance was evaluated with a multi-channel potentiostat/galvanostat/impedance analyser. Each supercapacitor cell was subjected to Cyclic Voltammetry (CV) at various scan rates from 0.01 V/s to 1 V/s, Charge-Discharge at a fixed current steps (2 mA) and Electrochemical Impedance Spectroscopy (EIS) with frequency range from 10 mHz to 1 MHz. It was established that an AC-based supercapacitor with 0.15%w/w MWNT content and 30 µm roll-coated, nanocomposite electrodes provided superior energy and power and energy densities while the cells was immersed in the electrolyte; well above those generated by the AC-based EDLC cells.

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## Reinforcement of Poly Ether Sulphones (PES) with Exfoliated Graphene Oxide for Aerospace Applications

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Composite materials have been used for aerospace for some time now and have gained virtually 100% acceptance as the materials of choice. Speciality polymers like poly ether sulphones (PES), poly ether ether ketones (PEEK) , poly ether imides (PEI) are highly preferred materials as plastic matrix due to their superior temperature performance, excellent wear & friction resistance, excellent dimensional accuracy, high tensile strength, high modulus, precise machinability and chemical resistance. In recent years nanoadditives like single and multiwall carbon nanotubes, graphenes and graphene oxides(GO) are finding huge market potential in aerospace and automobile industries. But manufacture related factors such as particle/ matrix interphases, surface activation, mixing process, particle agglomeration, particle size and shape may lead to different property effects. In this research GO/PES composites were prepared by high shear melt blending technique. GO monolayers were exfoliated from natural graphite flake and dispersed homogeneously in PES matrix for the GO content ranging between 0.5 to 2.0 volume percentage with a high shear twin screw batch mixer. These melt blended nanocomposites were injection moulded for mechanical property validation of tensile strength, flexural modulus and impact resistance. Addition of 0.5 volume percentage of GO enhanced the tensile strength and flexural modulus by 40% and 90% respectively. The results show that addition of GO to PES increase mechanical properties due to the formation of continuous network, good dispersion and strong interfacial interactions. The strong interfacial interactions were accounted for the increase in glass transition temperature. Also there was a significant improvement in the impact resistance of the PES/ GO nanocomposite. The injection moulded samples were tested for stealth performance by measuring the electromagnetic shielding property.

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## Nylon 6,6 Electrospun Fibres Reinforced by Amino Functionalised 1D and 2D Carbon

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Nylon 6,6 electrospun nanocomposites were prepared and reinforced with 0.1, 0.5 and 1wt.% of 1D and 2D carbon. Both carbon nanotubes and graphene were functionalised with amino groups (f-CNT and f-Ge respectively). The morphology and graphitization changes of carbon nanomaterials were evaluated by transmission electron microscopy (TEM) and Raman spectroscopy; functional groups of modified nanomaterials was analysed by infrared spectroscopy. The mechanical response and the crystallinity of the fibres were measured by dynamical mechanical analysis, differential scanning calorimetry and wide angle x-ray diffraction. The storage modulus was improved by 118% for f-CNT and 116% for f-G. The mechanical response of the nanocomposites exhibited different behaviour upon loading of 1D and 2D carbon. The morphology and dispersion of the nanomaterials in the nanofibres was studied by scanning electron microscopy and TEM. This trend is consistent with the crystallinity of the nanofibres. This study showed f-CNT resulted in better mechanical properties at the lowest loading. On the other hand f-Ge showed improved reinforcing effect by increasing the filler loading. The two-dimensional structure of graphene was also an important factor for the higher crystallinity in the electrospun nanofibres.

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## Efficient Optical Resolution of Amino Acid by Alanine Racemase Chiral Analogue supported on Mesoporous Carbon

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Optically pure D-amino acids are industrially important chiral building blocks for the synthesis of pharmaceuticals, food ingredients, and drug intermediates. Chemoenzymatic dynamic kinetic-resolution processes have recently been developed for deracemization of amino acids. S-ARCA would be a good candidate for the selective adsorption of D amino acid through the imine formation reaction. The organic phase containing S-ARCA adsorbent, TPPC or Ionic Liquid (as a phase transfer catalyst) in MC were coated on the surfaces of mesoporous carbon C-SBA-15(CMK). The aqueous solution of racemic D/L-amino acid and NaOH were added to the carbon support coated with ARCA. The D/L ratios on ARCA and in solution were determined with increasing reaction time. S-ARCA has a unique property for the selective adsorption of D- amino acid (up to 90% selectivity) in the racemic mixture. The fixed bed reactor containing ARCA/carbon support was also adopted successfully for the selective separation of amino acid.

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### **Macro Material Models for Nano-Materials**

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As soon as a given nanomaterial becomes feasible and interesting for industrial use the question of the material models (laws) are posed. This is directly related to the design procedure which involves extensive physical modeling of the behavior of the component made of such Nano-composite, often leading to FE analyses.

The material models used in conjunction with FE codes (in particular for non-linear or composite models) are often of macro nature in the sense that they represent the material behavior under a given laboratory test (e.g. tension, compression or shear) where measurements are made similarly to metallic or homogeneous materials via strain and force captors. These models do not necessarily represent the Nano-scale behavior of the material since they are concerned essentially with the homogenization of the material response. This poses the problem of exploring the material property sensitivities to Nano-scale component variations since Nano-scale tests cannot be implemented immediately into macro scale material models. Macro level tests are again necessary for each Nano-component sensitivity which makes the process lengthy and inefficient.

In this presentation we shall treat the topic of defining material models for nano-materials which could be implemented in FE analysis codes with much reduced effort. The method is based on response decomposition techniques (borrowed from data mining) and data fusion procedures (taken from machine learning techniques). Little if any reformulation of the material models are necessary. Examples of applications will be provided.

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## Modeling of Short Fiber Reinforced Injection Moulded Composite

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A micromechanics based finite element model (FEM) is developed to facilitate the design of a new production quality fiber reinforced plastic injection molded part. The composite part under study is composed of a polyetheretherketone (PEEK) matrix reinforced with 30% by volume fraction of short carbon fibers. The constitutive material models are obtained by using micromechanics based homogenization theories. The analysis is carried out by successfully coupling two commercial codes, Moldflow and ANSYS. Moldflow software is used to predict the fiber orientation by considering the flow kinetics and molding parameters. Material models are inputted into the commercial software ANSYS as per the predicted fiber orientation and the structural analysis is carried out. Thus in the present approach a coupling between two commercial codes namely Moldflow and ANSYS has been established to enable the analysis of the short fiber reinforced injection moulded composite parts. The loaddeflection curve is obtained based on three constitutive material model namely an isotropy, transversely isotropy and orthotropy. Average values of the predicted quantities are compared to experimental results, obtaining a good correlation. In this manner, the coupled Moldflow- ANSYS model successfully predicts the load deflection curve of a composite injection molded part.

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## Preparation, Characterization and Fe-Simulation of the Reinforcement of Polycaprolactone with Pegylated Silica Nanoparticles

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We recently published the preparation and characterization of polycaprolactone (PCL) nanocomposites with a 45% increased modulus reinforced with only 4 wt% PEGylated silica (polyethylene-glycol/SiO<sub>2</sub>) nanoparticles obtained by melt-extrusion [1]. The achieved reinforcement is related to an excellent dispersion of the nanoparticles due to the polyethylene-glycol graft of the nanoparticles which was obtained by a simple one-pot synthesis. X-ray photoelectron spectroscopy (XPS) and infrared spectroscopy (FTIR) analyses identified the location of the PEG at the PCL/silica interface. However, the extension of the interface could not be resolved. In an attempt to describe the effect of the interface on the reinforcement we applied several analytical micromechanical models. Models considering core-shell systems fitted the experimental data well and gave estimations of the modulus and extension of the interphase. However, different sets of parameters gave equally good representations. In an alternative approach, 3D representative volume elements (RVE) of the composite with spherical nanoparticles including the shell were built-up from the morphological data to carry out computational micromechanics based on finite elements (FE). The interphase was modeled in the RVE. Both approaches demonstrated the need of an interphase extension of roughly twice the radius of the particle. The FEM approach estimates the interface-modulus much higher than the analytical models.

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## Failure Prediction of Composites under Transverse Compression by Discrete Element Method

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A numerical model based on a two dimensional discrete element method (DEM) has been used to predict the failure of unidirectional composite subjected to transverse compression loading. A representative volume element (RVE) with periodic boundary conditions applied onto surfaces to ensure the continuity between neighbours RVE was conducted. Hexagonal particles arrangement with randomly distributed fibres was used. The matrix/fibres interfaces were characterised by a contact softening model and the plastic deformation of matrix was governed by the Mohr-Coulomb criterion. This has been conducted in DEM using smooth joint model. Different matrix/fibre interfaces strength were compared. Experimental test results compared to DEM. Good agreements were observed between numerical simulation and those obtained experimentally.

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## Primitive Path Network, Structure and Dynamics of SWCNT/ Polymer Nanocomposites

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We investigate the entanglements, structure and dynamics of monodisperse polymer melts in the presence of a single wall carbon nanotube (SWCNT) in comparison to inclusion-free polymer melts by molecular dynamics simulations. The SWCNT has an infinite aspect ratio and radius smaller than the polymer radius of gyration. In the presence of SWCNT with or without attractive interactions, the contour length of the primitive path increases indicating more entanglements. We also find that the overall configuration, as characterized by the radius of gyration, is not perturbed by either the interaction energy between the polymer and the SWCNT or by variations in the SWCNT radius. We also find that there is a large heterogeneity in the polymer dynamics of the polymer melts with a SWCNT due to the polymers in contact with the SWCNT.

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## Prediction of Energy Absorption Characteristics of Carbon Nanotube/Epoxy Nanocomposites

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Carbon fibre reinforced plastic (CFRP) laminates are commonly used in aircraft industry due to their high specific stiffness and high specific strength. Unfortunately they frequently behave as brittle materials when exposed to various types of impact loading as they cannot absorb enough energy.

We believe that the incorporation of a nanocomposite coating onto the laminate surface can help to enhance overall energy absorption characteristics of the laminates. This is assumed to result (1) from the large interfacial area of nanoparticles, which becomes potentially available for fracture propagation, and (2) from an enhanced nonlinear deformation of the nanocomposite matrix due to the presence of nanoparticles.

Hence, this project is focussed on developing a surface coating composed of epoxy reinforced with a low weight percentage of carbon nanotubes (CNT) reinforced epoxy to improve the impact performance of the CFRP laminates. The ultimate objective of our research project is to prepare and characterise an impact resistant CNT/epoxy coating, and then predict its effect on the impact response of CFRP laminates using multiscale modelling validated by experiments. This study is part of our project, and it is focused on the prediction of energy absorption characteristics of CNT/epoxy nanocomposites across different strain rates. In particular, the emphasis is on the influence of aligned CNTs on the compressive response of the nanocomposite at strain rates ranging from quasi-static to impact rates. For this, a rate-dependent constitutive model for epoxy is implemented into a finite element (FE) framework, and combined with the representative volume element (RVE) and numerical homogenisation. FE predictions of nanocomposite response for different rates are compared with unfilled epoxy, to demonstrate the effect of CNTs (aspect ratio, volume fraction) on energy absorption characteristics of CNT/epoxy nanocomposites.

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## Multi-Scale Modeling of the Progressive Damage in Cross-Ply Laminates Under Thermal and Mechanical Loading

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The progressive damage in cross-ply laminates was modeled by discrete element method (DEM). A particle radius expansion method was used to account for thermal loading applied to cross-ply laminates in which nominal fibers were introduced in the 0° plies so as to achieve the anisotropic thermal expansion behaviors. A series of convergence and validation tests of both mechanical and thermal properties of the 0° plies with nominal fibers have been carried out in order to validate the method. The DEM results of interfacial stress distribution of cross-ply laminates under pure thermal loading and under coupled thermal/mechanical loading were compared with other theoretical predictions. Microstructure of 90° plies was also studied by the DEM model. Transverse cracking which was formed by the coalescence of micro cracks in matrix and at fiber/matrix interface has been observed in the modeling results together with the ply-ply delaminations. It was found that the DEM model can predict not only the stress distribution but also the progressive damage initialized from the constituent failure due to its multi scale nature.

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### Tannin Based Flax Fibre Reinforced Composites for Structural Applications

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The production cost and safety requirements limit the use of electric vehicles for decades. The ECOSHELL project, sponsored by EU, aims to use sustainable composite in order to develop the structure components of electric small vehicles benefiting from reduced weight and environment advantages. (see Figure 1(a)). Flax reinforced mimosa tannin-based composite was developed and initially investigated by authors. Flax has been widely used as bio-reinforcement due to its cheap cost and good mechanical properties. Tannin resin is also environmentally friendly, making it an ideal matrix candidate in composites. The flax/tannin composite, a new composite type, were studied on the basis of a research cycle (Figure 1(b)). Nanotechnology may be adopted in future to improve the performances.

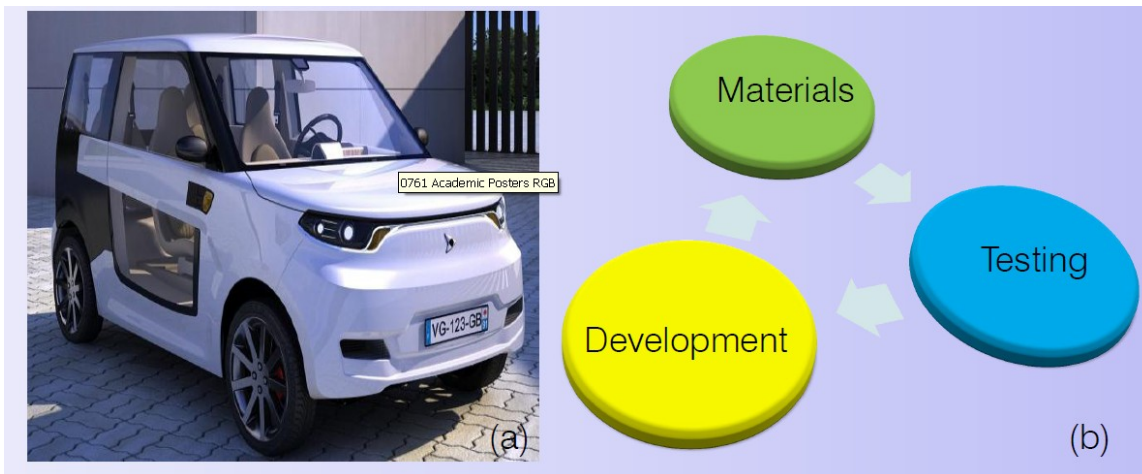


Figure 1. (a) Proposed superlight electric vehicle; (b) research cycle of flax/tannin composites.

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## **Graft Copolymerization of Synthetic Polymers onto Plant Fibers for the Development of Polymer Composites with Improved Mechanical Properties**

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Plant fiber reinforced polymer composites have received much attention because of their lightweight, nonabrasive, combustible, nontoxic, low cost and biodegradable properties. The hydrophilic nature of plant fibers often results in poor compatibility with hydrophobic polymer matrices. So in order to develop composites with better mechanical properties, it becomes necessary to modify the surface of plant fibers for better binding between fibers and matrix. Surface modification of the plant fibers through various chemical treatments can clean the fiber surface, chemically modify the surface, stop the moisture absorption process, and increase the surface roughness. Most of the chemical treatments involve mercerization, silylation, acetylation, benzoylation, peroxide, maleated coupling agents, isocyanate treatment and graft copolymerization. Among the various chemical treatments, graft copolymerization of synthetic polymers onto plant fibers is the best method for surface modification of plant fibers. Surface of plant fibers becomes rough during grafting, which results in better binding between fibers and matrix. Graft copolymerization of synthetic polymers onto various plant fibers has been discussed in this paper. Effect of surface modification of plant fibers on the properties of fibers and fiber reinforced polymer composites has also been discussed.

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### Conductive Adhesives with Self-Organized Silver Particles

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Conductive adhesives are getting more and more in focus for various kinds of microelectronics uses, photovoltaic applications and printable RFID antenna structures. In the last years, there were several developments to enable the application of the mostly silver filled epoxy or acrylate based systems with different kinds of processing like inkjet-, offset-printing or sintering. In all cases, there is the technical need to lower the filler content to decrease the viscosity and the pressure to finally decrease the costs for the highly expensive silver filled adhesives. Common available adhesives are filled with about 70 weight percent of silver particles. The shape of the particles was the main topic investigated to decrease the silver amount and resulted e.g. in silver flakes with a highly irregular structure.

Our approach to decrease the silver content with at the same time similar conductivity was to use nano-sized silver particles with different modified surfaces to obtain self-organized distribution of the silver particles in the liquid adhesive film followed by conventional curing techniques like temperature or UV curing. Through the self-organization, a bridging of the particles should be achieved which gives a similar conductivity as in highly filled commercial adhesives.

All used formulations were based on commercially available raw materials. The influence of the surface modification on the strength of the interaction between differently modified silver particles in the liquid adhesive was simulated with Dissipative Particle Dynamics (DPD). Surface modification was verified with surface enhanced Raman spectroscopy (SERS) and X-ray photoelectron spectroscopy (XPS). The distribution of the silver particles in the cured adhesive film was investigated with scanning and transmission electron microscopy (SEM, TEM).

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## Photoluminescence Properties of Gadolinium Oxide Nanophosphor

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Rare earth oxides have been extensively investigated due to their fascinating properties such as enhanced luminescence efficiency; lower lasing threshold, high-performance luminescent devices, drug-carrying vehicle, contrast agent in Magnetic Resonance Imaging (MRI), up-conversion materials, catalysts, and time-resolved fluorescence (TRF) labels for biological detection etc. The solution combustion process involves a self-propagating, gas producing, exothermic redox reaction between the corresponding metal nitrate and a suitable organic fuel that results in the desired products. Gadolinium oxide ( $Gd_2O_3$ ) is a  $Ln_2O_3$ -type oxide.  $Gd_2O_3$  nanocrystalline phosphor was synthesized by solution combustion method by using citric acid and urea fuels. The final product was examined by well characterized techniques such as powder X-ray diffraction pattern (PXRD), UV-Visible, photoluminescence (PL) measurements etc. The citric acid and urea readily gives pure cubic phase of  $Gd_2O_3$ . The average particle size of the as-formed and calcined  $Gd_2O_3$  nanophosphor was estimated using Scherrer's formula found to varies from 42 to 80 nm. The UV-Visible absorption peaks observed at 243 and 305 nm. Further the band gap energy of as-formed and calcined  $Gd_2O_3$  were estimated by Wood and Tauc relation and it is found to be 5.3 and 5.5 eV. A broad PL emission band at  $\sim 375$  nm was observed in all samples excavated at 270 nm. This emission band may be attributed to recombination of a delocalized electron close to the conduction band with a single charged state of surface oxygen vacancy, according to Wang's proposal. These studies reveal that  $Gd_2O_3$  is a versatile material with high application potential in several technological fields (photonics).

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## Purification of Nanoparticles by Hollow Fiber Diafiltration

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Hollow Fiber Diafiltration (Hollow Fiber Tangential Flow Filtration) is an efficient and rapid alternative to traditional methods of nanoparticle purification such as ultracentrifugation, stirred cell filtration, dialysis or chromatography. Hollow Fiber Diafiltration can be used to purify a wide range of nanoparticles including liposomes, colloids, magnetic particles and nanotubes.<sup>1,2</sup> Hollow Fiber Diafiltration is a membrane based method where pore size determines the retention or transmission of solution components. It is a flow process where the sample is gently circulated through a tubular membrane. With controlled replacement of the permeate or (dialysate), pure nanoparticles can be attained. Hollow Fiber Diafiltration can be directly scaled up from R&D volumes to production. By adding more membrane fibers and maintaining the operating parameters, large volumes can be processed in the same time with the same pressure, and flow dynamics as bench-scale volumes.

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## Recent Advances in Nanocomposites for Waste Water Treatment

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Waste water studies have always been a thrust area of research due to ever growing pollution and scarcity of the tools to combat this challenge at domestic and industrial level. The most important features that renders the pollutants hazardous in waste water includes non-biodegradability, lack of large scale remediation and recovery processes. This has led to a widespread environmental endanger accompanied with serious and adverse health problems such as renal disturbances, lung insufficiency, bone lesions, cancer and hypertension in humans. At present, there is limited and old fashioned technologies that can effectively and efficiently remove the organic/inorganic contaminants from the waste water. Nanotechnology is a new and novel tool that has offered a domain for waste water research. For the unique and fascinating properties of nanomaterials and nanocomposites such as an ability to attract the pollutants at lowest concentrations in waste water have been identified as a smart material. The nanocomposites can be applied as such and also to the existing technologies for the water research. There is a huge demand for the investigation into the application of nanocomposites to water research and hence it opens one of the most desirable fields of research and development.

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## Silver Nanocluster/Silica Composite Coatings Obtained by Sputtering for Antibacterial Applications

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The antibacterial activity of the silver has long been known and well documented. An antibacterial behaviour could be provided on different materials by means of several techniques, e.g. sol-gel, CVD, absorption on zeolites, ion exchange on bioglasses.

The aim of this work is the preparation and characterization of a new, chemically/thermally stable and low-cost silver nanocluster/silica composite coating. These antibacterial thin films are deposited by the RF co-sputtering of silica and silver [1,2]. The technique is extremely versatile, in fact it allows the deposition on various substrates (glasses, ceramics, polymers and metals), and the tailoring of silver content/antibacterial activity to match different needs for several applications (e.g. agricultural/food industry, biomedical implants, parts of mobile telephones, personnel protective systems, space structures).

The antibacterial effect of coated samples has been evaluated in different experimental conditions in accordance to National Committee for Clinical Laboratory Standards (NCCLS), using a standard bacterial strain (*Staphylococcus aureus* ATCC 29213). The antimicrobial properties are maintained also after gamma ray and EtO sterilization processes; this is a promising result about the possible use of these coatings for applications requiring sterilization procedures, as in the biomedical field. Tape resistance (ASTM D3359-97) and scratch resistance tests demonstrate a good adhesion and mechanical properties of the coatings.

This work was funded by the project "NABLA-Nanostructured Anti-Bacterial LAYers" from Regione Piemonte, Italy and by the project "NASLA- Nanostructured Anti-Septical coatings" from the European Union's Seventh Framework Programme.

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## Potential of Silicon Nanowire Structure as Nanoscale Piezoresistor in Mechanical Sensors

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This paper presents the design of a single square millimeter 3-axial accelerometer for bio-mechanics measurements that exploit the potential of silicon nanowires structures as nanoscale piezoresistors. The main requirements of this application are miniaturization and high measurement accuracy. Nanowires as nanoscale piezoresistive devices have been chosen as sensing element, due to their high sensitivity and miniaturization achievable. By exploiting the electro-mechanical features of nanowires as nanoscale piezoresistors, the nominal sensor sensitivity is overall boosted by more than 30 times. This approach allows significant higher accuracy and resolution with smaller sensing element in comparison with conventional devices without the need of signal amplification.

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## New Active Control Nano-System to Use in Composites Structure

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A new development is proposed, which is based on nano-sensors and active devices. They are installed into the material structure. The information from the sensors is transmitted by optical fibers, to a radio transmitter, equally installed into the material. An external receptor picks up those data and transmits them to an external device. This external device presents/displays all the information across an interface GUI, in real time, to the supervisor. The supervisor can then see what is happening in the material, in real time. Additionally alarms can be programmed, by the supervisor. All the devices and software are in develop in our laboratories. We think that this development will be used by the industry of materials, and that gradually, it will have other applications in the transport area (like new vehicles, wagons of train and metro, etc.).

We considered this research project as very important, because it reunites in its development, several basic technologies: electronics, communications, software and new materials, all very interesting for the European industry. The CSIC (Spanish Researcher Center) is involved in the development of nano-sensors, specialized in detecting defects, difficulties or problems in structures of composed materials. These materials are being used and are of importance in applications where a high degree of security is necessary. Some fields in the system usage are aeronautical and military applications whit a necessary high security degree.

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## Composite Material Pedestrian Bridge for the Port of Bilbao

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Composite materials present advantages in comparison to traditional ones, steel and concrete, in civil works construction: lower weight, higher corrosion resistance (especially in the marine environment), and ease of installation. On the other hand, fabrication costs are generally higher being this the reason why this technology is not widely used.

This work illustrates the process followed for the design, fabrication and installation of a composite material pedestrian bridge in the Port of Bilbao (Northern Spain).

Two material choices were studied. In order to reduce the price of the bridge, the use of low cost materials was considered, therefore polyester resin was selected as the polymeric matrix, and glass fibres as reinforcement.

Currently in the market there is high availability of carbon nanoparticles: carbon nanotubes (CNT) and carbon nanofibres (CNF), so it was decided to add this kind of nanoparticles to the reference material with the objective of improving its mechanical properties. The main challenge was to transfer the CNT and CNF excellent properties to the polymeric matrix. This requires dispersing the nanoreinforcements as individual particles in the polymeric matrix to avoid agglomerates. For this reason, an advanced high shear forces dispersion technique (called "three roll mills") was studied and implemented. Also surface functionalization of the nanoreinforcements by chemical treatment was carried out.

Herein, a comparison is performed between both materials studied, the explanation of the employment of the reference material (without nanoreinforcement) as the one used in the fabrication of the pedestrian bridge is justified and, finally, the main characteristics of the final design of the structural element are described.

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## Using Artificial Neural Networks to Model Extrusion Processes for the Manufacturing of Polymeric Micro-Tubes

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In this paper a methodology and an application example are presented aiming to show how Artificial Neural Networks (ANNs) can be used to model manufacturing processes when processes' mathematical models are missing or are not applicable e.g. due to the micro- & nano-scaling, due to non-conventional processes, etc. Besides the ANNs methodology, a Software System developed will be presented that is used to create ANNs models for micro & nano manufacturing processes and also a specific application case example concerning the modeling of extrusion processes for the production of polymeric micro-tubes. ANNs models are capable of modeling manufacturing processes as far as adequate experimental and/or historical data of processes' inputs & outputs are available for their training. The POLYTUBES ANNs models have been trained and tested with experimental data records of process' inputs and outputs concerning a micro-extrusion process of polymeric micro-tubes for several materials such as: COC, PC, PET, PETG, PP and PVDF. The ANN model of the extrusion application example has three inputs and nine outputs. The inputs are: the tube's inner & outer diameters, and the material density. The model outputs are 9 process parameters, which correspond to the specific inputs e.g. process temperature, melt density, die inner & outer diameters, extrusion pressure, draw speed (m/min), etc. The training of the ANN model was completed, when the errors for the model's outputs, which expressed the difference between the training target values and the ANNs outputs, were minimized to acceptable levels. After the training, the micro-extrusion ANN is capable to simulate the extrusion process and can be used to calculate model's outputs, which are the process parameters for any new set of inputs. By this way a satisfactory functional approximation of the whole process is achieved. This research work has been supported by the FP7 NMP project POLYTUBES.

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## Hierarchical Polymer Nanocomposites for Automotive Applications

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Since the late 1980s, much research has been conducted in the field of nanocomposites. The real and profitable industrial applications, however are more recent, and have increased in the last decade. In particular, in the automotive and packaging sectors, the evolution of this research is evident. These sectors represent 80% of the total consumption of nanocomposites.

Nanocomposites with a polymer-matrix are a new promising asset for promote less polluting and eco-friendly cars. Indeed, as an ecological goal, car manufacturers aim to reduce the fuel's consumption and CO<sub>2</sub> emissions of their vehicles to allow manufacturing of lightweight cars. Polymer nanocomposites bring a new solution for this problem. Less than 5% of nano-reinforcements can significant improve the properties of a neat polymer. Further advantages are low cost of production, significant decrease of weight and ease of manufacturing.

In this work, we have reviewed existing and commercialised automotive components for interior and exterior applications, mechanical parts, and fuel system components. The review investigation focuses on uses and improved properties of polyamide, polypropylene and thermoplastic olefin nanocomposites. The most used nano-reinforcements, natural or modified clay such as mica, or montmorillonite, are also covered.

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## Expression of CIRCADIAN GENES in Different Rat Tissues is Sensitive Marker of In Vivo Silver Nanoparticles Action

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Circadian factors PER1, PER2, ARNTL and CLOCK are important molecular components of biological clock system and play a fundamental role in the metabolism at both the behavioral and molecular levels and potentially have great importance for understanding metabolic health and disease, because disturbance the circadian processes lead to developing of different pathology. The antibacterial effect of silver nanoparticles has resulted in their extensive application in health, electronics, home products, and for water disinfection, but little is yet known about their toxicity. These nanoparticles induce blood-brain barrier destruction, astrocyte swelling, cause degeneration of neurons and impair neurodevelopment as well as embryonic development. We studied the expression of genes encoded the key molecular components of circadian clock system in different rat organs after intratracheally instilled silver nanoparticles which quite rapidly translocate from the lungs into the blood stream and accumulate in different tissues. We have shown that silver nanoparticles significantly affect the expression levels of PER1, PER2, ARNTL and CLOCK mRNA in different rat tissues in time-dependent and tissue-specific manner. High level of PER1, ARNTL and CLOCK mRNA expression was observed in the lung on the 1<sup>st</sup>, 3<sup>rd</sup> and 14<sup>th</sup> day after treatment of rats with silver nanoparticles. At the same time, the expression level of PER1 mRNA in the brain and liver increases predominantly on the 1<sup>st</sup> and 14<sup>th</sup> day but decreases in the testis. Significant increase of the expression level of PER2 and ARNTL mRNA was detected only in the brain of treated by silver nanoparticles rats. Besides that, intratracheally instilled silver nanoparticles significantly reduced the expression levels of CLOCK mRNA in the brain, heart and kidney. No significant changes in the expression level of PER2 mRNA were found in the lung, liver, heart and testis, except kidney where this mRNA expression decreases on the 3<sup>rd</sup> and 14<sup>th</sup> day after treatment of rats with silver nanoparticles. It was also shown that expression level of PFKFB4, a key enzyme of glycolysis regulation, gradually reduces in the brain from 1<sup>st</sup> to 14<sup>th</sup> day being up to 4 fold less on 14<sup>th</sup> day after treatment of animals with silver nanoparticles.

Thus, the intratracheally instilled silver nanoparticles significantly affect the expression of PER1, PER2, ARNTL, and CLOCK genes which are an important molecular component of circadian clock system. This is because a disruption of the circadian processes leads to a development of various pathologic processes. The results of this study clearly demonstrate that circadian genes could be a sensitive test for detection of silver nanoparticles toxic action and suggest that more caution is needed in biomedical applications of silver nanoparticles as well as higher level of safety in silver nanoparticles production industry.

## **An Overview of Manufacturing Techniques for Polymer Composite Components and Products for Mass Production**

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Polymer composite components in automotive products are gaining importance, due to their availability and related cost reduction. Moreover, they offer additional benefits such as increased thermal, impact, aesthetics and structural properties while keeping the component weight at a minimum. To exploit these material characteristics, manufacturing techniques have recently been developed to fabricate composite materials to suit the specific applications.

The most common manufacturing techniques for polymer composite including injection, backwards injection, compression and resin transfer moulding. Additionally, other employed techniques include layered thin film technologies such as extrusion, blow forming, calendaring and laminating. While these manufacturing techniques are already widely used for mass production of polymer components, their usage for polymer composites components is still facing major challenges. Main difficulties lay in restricted ability of homogeneous dispersion of the filler material in polymer matrix and filler/matrix de-bonding in complex structures.

Therefore, this study is aims to review the current state-of-the-art in manufacturing and fabrication methods for complex polymer composite components. Special emphasis will be given on the future prospects and the key problems and challenges involved in the mass production of complex polymer nanocomposite products for automotive applications.

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## Mechanical Performance of Foam Glass with Nanoscale Structural Elements

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The paper examines the process of glassceramic foam material strengthening. This material is manufactured of semiprocessed product – the quenched cullet synthesized at relatively low temperatures (< 950 °C). It is already at this stage when the quenched cullet contains glass phase in a noncrystalline state. It ensures the pyroplastic state of the material at the stage of repeated thermal treatment (sponging) of the quenched cullet. The foam materials were produced according to the two-stage technology developed by the authors. Such crystal and noncrystalline silica-containing rocks as silica sand, diatomite, flint, silica clay, and perlite were sampled as a feedstock. The samples of foam material were obtained with the content of residual crystalline material from 5 to 20 % by volume. It was established that the strength of experimental samples increased from 1.8 to 3 MPa as the crystalline phase particle size decreased from 1,000 to 300 nm. The quenched cullet is a noncrystalline matrix containing residual silica particles with the size up to 200 nm. Strengthening of foam material is related to two processes: 1) decreasing of the particle size in residual crystalline material; 2) formation of nanoscale spheroids in the noncrystalline matrix of glass foam material. In the course of the first process – the change of the crystal size the strength of 3 MPa is reached, and at the introduction of the second process a theoretical strength of 5 MPa can be attained. The major energy dissipation at the deformation is performed by the areas with densely packed globules, with the volume content of which can amount to 95% in the material. For the first time a new type of strengthening is observed in the noncrystalline material, that is the self-organization of spheroids in the body of noncrystalline matrix.

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## **Recent Advances in Structural Nano-Composites for Sustainable Construction Industry: Applications, Opportunities and Challenges**

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Nanotechnology has a huge potential in the field of construction and building materials. In particular, manufactured nanomaterials can improve vital characteristics of construction materials such as strength, durability, energy savings and lightweight besides endowing useful properties. It also follows that one of the most promising areas in the field of nanotechnology relates to the replication of natural systems such as self-cleaning, antifogging, self-healing and structural health monitoring.

In this work, we review the current state-of-the-art of nanomaterial for the construction industry that offers significant improvement to conventional materials. The review covers vital nanocomposites applications such as nano insulation materials, concrete and mortar nanoproducts, sandwich impact barriers, nanosensors and air pollution reduction/control in the construction industry.

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### **Wound-Dressing Materials with Antibacterial Activity from Electrospun Polyurethane-Dextran Nanofiber Mats Containing Ciprofloxacin HCl**

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An antibacterial electrospun scaffold was prepared by physically blending Dextran and Polyurethane (PU) with Ciprofloxacin HCl (CipHCl). Dextran is a versatile biomacromolecule for preparing nanofibrous electrospun membranes by blending with either water-soluble bioactive agents or hydrophobic biodegradable polymers for biomedical applications. Thus obtained composed mat was characterized with various methods. The interaction between fibroblasts and the PU-dextran and PU-dextran-drug scaffolds such as viability, proliferation, and attachment were characterized. The results indicated that cells interacted favorably with the scaffold. Moreover, the composite mat showed good bactericidal activity against both of gram+ve and gram-ve bacteria. Our results suggest that this could be an ideal biomaterial for wound dressing applications.

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## **Structural and nitrogen gas adsorption-desorption study of Bragg grating waveguide fabricated on porous silicon nanostructure**

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Fabrication and structural analysis of Bragg grating waveguide on porous silicon were presented in this work. The Bragg grating structure was directly etched on porous silicon. The multilayer structure of porous silicon with alternating values of thickness and refractive index were determined. Nitrogen gas adsorption-desorption is a non-destructive method to determine the fabricated Bragg grating's interaction with gases. It is known that porous silicon is a high potential in-situ gas sensor due to the strong tube capillary effect and enormous surface area for molecular binding and detection. Thus, specific surface area and porosity of fabricated Bragg grating are also in the scope of this study. Good agreement of pore size distribution and multilayer thickness/refractive index achieved from adsorption-desorption technique and ellipsometer measurement respectively when comparing with estimations from field emission scanning electron microscopy. This Bragg grating waveguide has a big potential as gas sensor in many areas such as aerospace and in industrial particularly.

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## Development of an Active Catheter System using IPMC for *In-vivo* Inspection

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Active materials are nowadays utilized successfully for interventional diagnosis and therapy for their biocompatibility, suitability for smooth and safe operation. Active catheter is one such mechanism where active materials such as shape memory alloys (SMA), electroactive polymers (EAPs) and piezoelectric materials (PZT) are successfully used over the few years. Electroactive polymers (EAPs) exhibit properties most closely matching those of natural muscles like low density, short response time, resilience and large actuation strains. Among various types of EAPs, ionic polymer metal composites (IPMCs) are especially more prominent material to be used in the active catheter system because of their large deformation in the presence of very low input voltage (1-2 V).

An active variable length optical catheter system of ionic polymer metal composite (IPMC) actuator is developed for medical applications i.e., oral or inside the body imaging. Ionic polymer metal composites (IPMCs) are fabricated first using non-precious metal silver (Ag) as surface electrode while Nafion-117 as the base polymer following the chemical decomposition method. The IPMC actuator in catheter generates a transverse scanning movement with low input voltage (maximum 1.2V). Low driving voltage ensures safety for clinical application inside the body parts. The catheter design takes into account of the forward and backward movement of scanning mechanism and captures scanning image covering wide area. The active length of the IPMC actuator varies with input voltage thus can cover wide range of working area with same input voltage. Apart from oral and dental cavity inspection, the low cost Ag-IPMC mechanism has the potential applications in endoscopes for imaging of internal human tissues, blood vessel etc.

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## Relationship between the Morphology and Physico-Mechanical Properties of Polyethylene/Clay Nanocomposites

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Rheology, morphology and thermal behavior of HDPE/Clay nanocomposites were studied. The mechanical properties of these materials including tensile and creep behaviors were also taken into account. Different PE-g-MA samples with different MA contents and different rheological properties were laboratory synthesized and used as compatibilizer of PE and Clay. The results of X-ray diffraction in conjunction with the results of transmission electron microscopy (TEM) analysis indicated that, increasing in degree of grafted MA increases the penetration of PE chains onto the clay interlayer leading to intercalation and exfoliation. The intercalated and especially exfoliated nanocomposites exhibited higher viscosity and elasticity in particular at low frequency ranges, showing the formation of three dimensional networks with high interfacial interaction. The presence of such a network was evidenced by  $\tan\delta$  studies where the pseudo-solid like behavior was observed for exfoliated nanocomposites. From these results it was demonstrated that the linear viscoelastic properties of the nanocomposites have a reliable sensitivity to the extent of clay dispersion and they can be used as indirect method in the prediction of the morphology and therefore thermal and mechanical behavior of the nanocomposites. Incorporation of clay decreased the onset temperature of degradation due to the Hofmann elimination reaction, but increased remarkably the mid-point of the degradation temperature.

Our laboratory synthesized intercalated nanocomposites displayed higher thermal stability than those of exfoliated samples. This was attributed to the barrier effect of clay layers to oxygen and volatile products, during the degradation of part of polymer chains which was intercalated in clay interlayer. Interestingly the results showed that the effect of Hofmann elimination reaction which decreases the onset temperature degradation of modified clay nanocomposites, can effectively be eliminated using a proper antioxidant in the mixing process of nanocomposites. The results showed that, incorporation of clay increases the young modulus and yield stress but decreases the elongation at break. In agreement to the results of thermal behavior, intercalated nanocomposites showed higher increase in properties than those of exfoliated. The PE nanocomposites showed lower creep strain and higher creep recovery than those of unfilled matrixes.

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## Manufacturing of Nanophase Integrated RTM Manufactured Composite Structures

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After decades-long endeavors regarding the manufacturing and characterization of nanophase incorporated composites, engineers and scientists are eager to see physical implementation of this technology on industrial scale. There are a number of studies and methods in literature to incorporate nanophase structures into polymeric composites to create hierarchical composites. Nevertheless, incorporating nano structures into conventional polymeric composites at large scale still remains as a challenge ahead. Therefore, novel approaches are urgently required to circumvent associated up-scaling issues. In this direction, we have attempted to incorporate carbon nanotubes into fiber glass reinforced composites structures manufactured by Resin Transfer Molding (RTM) method through electro-spraying and electro-spinning processes. In this work, we present the preliminary results of this research focusing on composite manufacturing and the mechanical performance and properties of produced samples.

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### **Título: PCL nanocomposite prepared by conventional and microwave assisted “in situ” polymerization**

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Poly( $\epsilon$ -caprolactone) (PCL) is a synthetic semicrystalline aliphatic polyester that is one of the most promising biodegradable polymers currently available on the market [1]. However the number of applications has often been limited by poor mechanical properties and low melting temperature. The PCL performance can be greatly enhanced with the addition of nanometer-size inorganic fillers. Mesoporous silica like MCM-41 can improve mechanical properties of polymeric matrices [2]. Besides, the structure of this material allows encapsulation and controlled release of molecules.

In the presented work, PCL/MCM-41 with and without EPTES functionalization nanocomposites (1 and 5 wt%) were prepared by conventional and microwave assisted “in-situ” polymerization. Monomer conversion and molecular weight development with reaction time were studied using  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  as catalyst to start the ring opening polymerization. The addition of catalyst was performed by two methods: a) catalyst was added in the reaction medium together with monomer and filler; b) catalyst was added by incipient wetness impregnation to MCM-41 and EPTES/MCM-41. Films of the composites were then prepared by solution blending using THF as solvent. The composites obtained were characterized by TEM, TGA, DSC, NMR, AFM and GPC. The permeability to oxygen and carbon dioxide were measured in a homemade device.

The “in situ” polymerization method produces composites with good dispersion of MCM-41 with or without EPTES functionalization in the polymeric matrix as showed by TEM micrographs. DSC and TGA results showed that the degradation temperature of the composites was up to 65°C higher than the polymeric matrix decomposition temperature. On the other hand, AFM results of the synthesized materials and films showed an improvement of the Young modulus of the matrix when 1% of additive is incorporated.

As conclusion, PCL composites prepared by the two methods presented similar properties but the microwaves synthesis required only 40 min reaction time. PCL/MCM-41 prepared nanocomposites present good thermal and mechanical properties due to the high degree of dispersion of the fillers achieved.

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## Evolution of dispersion properties during the delamination of nanoclays

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Nanoclay dispersions or “nanocomposites” are achieved by the dispersion of intercalated or exfoliated clay platelets in a polymer melt or monomer prior to polymerisation. Polymer-based clay nanocomposites are usually divided into three categories (Fu & Qutubuddin, 2001, Zhu and Xanthos, 2004; Kotsillkova, 2007):

In *conventional composites*, the clay acts as a filler material to the polymer.

*Intercalated nanocomposites* are formed when a small amount of polymer moves into the gallery spacing between the clay platelets to promote the swelling of the clay platelets.

*Exfoliated nanocomposites* are clay platelets fully delaminated and dispersed in a continuous polymer matrix.

Intercalated and exfoliated nanocomposites have shown significant improvements in terms of product properties even at low clay concentrations. These include:

- thermal stability (Zhu and Wilkie, 2000; Wang et al, 2002),
- barrier properties (Messersmith et al, 1995; Bharadwaj, 2001),
- tensile strength (Chen et al, 2000) and
- increased elongation at break (Ma et al, 2001).

This is because when the filler is uniformly distributed throughout the polymer matrix and both components are made chemically compatible through the modification of the surface properties of the clay mineral, a larger surface area contact is achieved.

The study of the intercalation and exfoliation processes has presented challenges in terms of choosing appropriate experimental techniques to monitor these processes. X-Ray diffraction technique has typically been used to determine the inter-gallery spacing and hence has been found useful in monitoring the intercalation process. There has been limited success in obtaining reliable and meaningful particle size data. Dispersion rheology, on the other hand, has proved to be a promising tool as this is a dispersion property that significantly varies throughout the process. Electron microscopy (TEM, SEM) have provided additional information complementing the above.

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## Synthesis and characterisations of organophile montmorillonite – copolymer (st-thf) nanocomposite

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Nanocomposites, are mixed materials synthesized from one or many polymers blended in optimized percentages and organophilic clay. The objective of this study is to use the organophilic clay in the presence of two monomers to obtain a copolytetrahydrofuran-styrene nanocomposite by in situ polymerization.

Organophilic montmorillonite was prepared by ion exchange between Na<sup>+</sup> ions in the clay hexadecyltrimethylammonium bromide cations in an aqueous medium. The organophilic MMT particles were easily dispersed and swollen in two monomers.

The structure of obtained modifier was investigated by proton nuclear magnetic resonance (RMN1H), infrared spectroscopy. The exfoliating structure of nanocomposite was probed by X-ray diffraction and transmission electron microscopy. The Thermal stability were also investigated with thermogravimetric analyzer (TGA), comparing with pure polystyrene, the nanocomposite showed much higher decomposition.

temperature. Introduction

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