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# IMech /OLEM/ Application to Hop On Facility call

The Institute of Mechanics (IMech) is a leading center in the Bulgarian Academy of Sciences for fundamental and applied research in mechanics. The IMech staff consists of 90 researchers and PhD students working in six main areas: micro & nanomechanics, mechatronics, solid mechanics, fluid mechanics, biomechanics, modeling and simulations in mechanics.

The Open Laboratory on Experimental Micro & Nano Mechanics (OLEM), which apply for the Hop on Facility call, is a unit of the Institute of Mechanics, founded in 2010 under the Research Infrastructure Project, supported by the BGNSF, Bulgaria. The OLEM is a Key Laboratory for research and training in the field of surface nanomechanical characterization and the development of novel nanomaterials / https://www.imbm.bas.bg/index.php/en_US/olem /

OLEM operates in six Laboratories of 200 $m^2$ space. The Labs are well equipped with advanced facilities for Nanomechanical Testing; Micromechanics &Tribology; Thermal & Electrical Conductivity; Rheology & Thermal Analyses; Polymer Nanocomposite Processing; and 3D Printing / https://www.imbm.bas.bg/index.php/en_US/equipment-5 /

OLEM is a part of the Centre of Competence "MiRacle" (coordinated by IMech-BAS), and participate in the Centre of Excellence on Mechatronics and Clean Technologies in Bulgaria.

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## OLEM EXPERTISE, FACILITIES AND CASE STUDIES

### Topics I. Advanced Nanomechanical Characterization of Surfaces and Interfaces
***(coatings, layers, multilayered devices, bio- and - nanostructured surfaces, etc*.)**

The adhesion is of significant importance for the properties and performance of coatings, films, multilayered electronic devices, etc. We investigate nanomechanical properties of the surfaces and interfaces, such as adhesion of a monolayer to the substrate; adhesion between multilayers, interfacial adhesion at boundary, nanohardness and elasticity, mapping of nanomechanical properties, topology and homogeneity, micro- and nanostructure of the surface, etc. Our expertize is on different materials, starting from soft polymeric films and biomaterials; nanostructured hard and super hard coatings; multilayered electronic devices; nanocomposite materials incorporating nanofillers (graphene and other 2D materials, carbon nanotubes, nanoparticles). Variety of methodologies are used for this study, such as: dynamic and quasi static nanoindentation, nanoscratch and nanowear, XPM mapping, SPM and AFM surface scanning, etc.

**Research Facilities**: *Triboindenter Hysitron T980 with integrated SPM and XPM* (Bruker); and *Nanoindenter with inline AFM* (Bruker) and *Micromechanics and Tribology Testing UMT-2* (Bruker).

CASE STUDIES:

**(i) Study on the adhesion of a monolayer to the substrate; Multilayered adhesion in novel electronic devices**:

Recently, the incorporation of graphene (GR) and hexagonal boron nitride (hBN) monolayers within the multilayered stacks is the main advantages of graphene nanoelectronics, due to the unique structure and outstanding electronic properties of the 2D nanomaterials. We have studied novel $Al_2O_3$/GR/$SiO_2$/Si and $Al_2O_3$/hBN/GR/$SiO_2$/Si multilayered devices in order to understand the effects of hBN/GR bilayer deposited on $SiO_2$ for enhancing the adhesion of a monolayer Graphene [1]. The effect of annealing as a processing step is studied and optimised. The scratch-adhesion test was adopted using nanoscratch / microscratch sliding under a linearly increased load, combined with SPM or AFM visualization. The methodology is applied with both the Nanoindenter with inline AFM and the Triboindenter Hysitron T980, depending on the thickness and the hardness on the investigated mono- or bilayer. The failure of different layers in the multilayered stacks was determined by the critical load producing a sudden change in the coefficient of friction or a jump in the contact acoustic emission signal during the scratch test. This scratch-adhesion test methodology is proven at OLEM, as a suitable fast technique for control on the adhesion between layers in the multilayered stecks, as well as for a quality control on the fabrication process and the operational performances of novel micro/nano devices.

**(ii) Quasi-static nanoindentation and Dynamic nanoindentation combined with XPS-accelerated Property Mapping for the analysis of surfaces:**

We have investigated the reinforcing effects of graphene nanoplatelets and multiwall carbon nanotubes in the polymer matrix by evaluation of nanohardness, elastic modulus and their mapping on the surfaces using these test methodologies [2]. Correlative SPM microscopy and the XPM mechanical property mapping allow us also to study nanoscale surface homogeneity, boundary layers and interfaces of dual-phase and multi-phase materials, stress relaxation and creep behavior.

**(iii) Nanoindentation, Nanoscratch, and Scanning Wear of Soft, Hard and Super hard Thin Films:**

Thin films find important applications in microelectronics, optics and optical coatings, MEMS devices, hard and corrosion resistant coatings, photovoltaics, shape memory alloys. We apply these in-situ testing techniques for quantitative material properties characterization, including hardness, modulus and stiffness; depth profiling, quantification of Graphene monolayer delamination or breakthrough, nanotribology, friction, wear resistance and wear failure. The in-situ SPM imaging couples nanomechanical and nanotribological characterization with high-resolution imaging for qualitative surface analysis and surface roughness [1,3,4].

**(iv) Conductive Nanoindentation**.

We apply NanoECR (Electrical Contact Resistance) measurements for *in-situ* electrical and nanomechanical measurements of thin conductive layers and films. The combination of electrical contact resistance with nanoindentation allows a precise study of thin film fracture, dislocation, deformation, fatigue, contact resistance, tunneling effects, piezoelectric response, phase transformations, adhesion investigations, and more [1,2].

**(v) Tribology, wear and lubricant properties at micro and nanoscale**:

Polymer nanocomposites incorporating various amount of reinforced fillers of Graphene and MWCNTs are studied for tribology applications. The coefficient of friction (COF) from scratch and wear experiments of the 3D printed elements was determined. The SEM images revealed two types of failure mechanisms over the worn tracks depending on the carbon nanofiller in the composite die. Dry sliding reciprocating wear tests disclosed much lower values of COF for the monofiller and bifiller compounds regarding the unreinforced matrix polymer, due to the self-lubricating effect of GNP and MWCNT fillers. [5]

## Topics II. Graphene - Polymer Research and Applications

**Research Facilities**: *Processing of polymer nanocomposites* by tween screw extruder Process 11, Single screw filament extruder; Ultrasonic bath, US horn, Spin coater; Thermal analyses by *DSC Q20* and *TGA Q50 (TA Instruments);* Laser flash thermal conductivity and diffusivity - *LFA 467 Hyper Flash (NETZSCH); Keithley 6517B electrometer; Tabletop SEM HIROX SH 4000; 3D printer X400 PRO German RepRap;*

CASE STUDIES:

(i) **Multifunctional polymer nanocomposites based on biopolymer doped with graphene nanoplatelets, carbon nanotubes and hybrid fillers** are developed and characterized for 3D printing applications within the H2020-734164 Graphene 3D project and H2020-Graphene Flagship. The effects of intrinsic characteristics of the carbon nanofillers (type, size, shape, aspect ratio, surface area, functionalization) on the degree of dispersion and microstructure of nanocomposites are investigated and related to the mechanical reinforcement, electrical and thermal conductivity, and electromagnetic shielding efficiency. The percolation threshold, interfacial interactions, and microstructural features are estimated as the essential nanostructure parameters which control the reinforcement and multifunctionality of nanocomposites. The main parameters for the design of multifunctional nanocomposites for 3D printing applications are proposed. As a result, a novel composite filament for 3D printing (FDM) is fabricated having robust mechanical, electrical, electromagnetic and thermal properties, competitive to the benchmark filaments [6].

(ii) **Effective 3D printed microwave absorbers**: The design concept of effective microwave absorbers based on 3D-printable nanocarbon composites with filler content above the percolation threshold is proposed. The electromagnetic properties of the printed pyramidal regular structures were experimentally investigated and numerically simulated in 12–18 GHz (Ku-band) and 26-37 GHz (Ka-band) frequency ranges. Tested components demonstrate remarkable shielding efficiency ($> 20$ dB) within whole Ku- and Ka-bands and are suitable for practical application related to effective absorption of microwave radiation. The production of 3D-printable materials with controlled and predicted losses offers the possibility for miniaturization of 3D printed microwave components, such as absorbers and loads [7]

(iii) **3D printed carbon-based phantom, which mimics the thermal and electrical properties of the human skin and the abdominal muscle to measure the heating induced by electro-medical fields**: The progress of technology and science has enabled new tools for the diagnosis and treatment of diseases based on electromagnetic (EM) fields. Radiofrequency (RF) EM fields also find application in aesthetic medicine treatments, e.g. for the removal of stretch marks and scars. The employment of EM fields in such treatments is regulated by national and supranational legislation, which limit the Specific Absorption Rate (SAR) below 0.4 W/kg and the temperature increase below 1°C. In this work we propose the design and fabrication of a 3D printed carbon-based phantom, highly customizable, which mimics the thermal and electrical properties of the three layers of the skin and the abdominal muscle to measure the heating induced by electro-medical fields. The phantom was fabricated by 3D printing (FDM) technique using filaments based on the biopolymer PLA filled with different concentrations of Graphene and Carbon Nanotubes. The platform is designed to tune the thermal and electrical conductivity of the deposited layers according to the skin and muscle layers properties. Each layer has inserted thermocouples to measure the heating induced by electro-medical fields. This work paves the way to the development of reliable phantoms of human tissues by means of innovative materials for the thorough investigation of the EM fields effect on human bodies. [8].

(iv) **Thermo-Electric Properties of Polymers with Carbon-Based Nanofillers:** Overheating effect is a crucial issue in different fields and particularly in high power electronics. Polymer composites filled with high thermal and electrical conductivity nanofillers show enhanced thermo-electric properties which encourage their use in heat transfer applications. The present work deals with the experimental evaluation and model simulations of the thermophysical properties of nanocomposites incorporating carbonaceous nanofillers. Thermal conductivity and diffusivity, as well as specific heat capacity, are measured in wide temperature range. The best thermal performances are observed for nanocomposites including graphenebased particles rather than carbon nanotubes that are better performing from an electrical point of view. Simulation studies were performed for predicting the thermal response of such materials in the face of an electric heating or a direct heat flux. Thanks to the numerical analysis working temperatures can be tailored to be lower than that the glass transition reducing the risk of material degradation in case of their use in heat transfer applications [9,10].

(iv) **Safety issues: Study on the release of graphene and carbon nanotubes from biopolymer-based nanocomposite films**. Incorporation of graphene and carbon nanotubes in biopolymeric films enhanced gas-barrier properties, mechanical strength, thermal properties and add functionality, hence the nanocomposite films are rapidly becoming a commercial reality. However, detailed safety studies are required in order to understand if graphene and carbon nanotubes can migrate into food from the films and what is the potential hazard. We have performed such study, as varying time-temperature conditions and food simulants, according to the European Standard EN 13130-1:2004 (EN 2004). The laser diffraction analysis, TEM, SEM, swelling tests and differential scanning calorimetry have been used to analyze the films and the released migrants. It was observed that graphene nanoplates of about 100 – 1000 nm in length and a few nanometres in thickness are indeed released from the investigated PLA composite film under rather extreme migration conditions of 90 °C for 4 hours in the three food simulants: ethanol, acetic acid and oil. In contrast, the fibrous MWCNTs formed entangled networks on the film surfaces as the PLA polymer matrix dissolve, which prevent their release as single nanoparticles into the food simulants. The total amount of released migrants is below overall migration limits. Nevertheless, the release of graphene from the PLA- film into foodstuff has to be taken into account at high temperature processing of packaged food in order to predict the risk from graphene in the food chain over long term exposure [11,12]

## Topics III. Rheological Study and Simulations of Optimization of 3D printing Process

**Research Facilities**: *Rheometer AR G2 (TA Instruments)*; *3D printer X400 PRO German RepRap; Tabletop SEM HIROX SH 4000*.

CASE STUDY:

**Rheology as a powerful tool for optimization of 3D printing process:**

The 3D printing by material's extrusion (FDM), is among the most widely employed Additive Manufacturing technologies for the production of parts with complex geometries for specific uses. The control of materials' rheology may provide a flexible manufacturing route to fabricating 3D-printed parts with a good resolution. Our study discussed rheological properties related to the microstructure of polymer nanocomposites filled with GNP and MWCNT to simulate the 3D printing process. The alignment and slip of graphene nanoplatelets in the shear-thinning flow govern the printability of nanocomposites at high filler contents. In contrast, a strong thickening effects of entangled nanotubes was observed, which restrict printing. The flow in the nozzle tube of a 3D printer was simulated by an analytical model and the fluid dynamics modeling. An "elastic turbulence" phenomenon, as a plug flow of nanocomposite melt is observed, which cause clogging in the 3D printing nozzle. The main rheological characteristics and numerical parameters that control the printability of nanocomposite filament are determined. These findings will contribute to the development and optimization of novel printable nanocomposite materials with anisotropic fillers. Experimental and modeling parameters are explored in designing printable hybrid polymer nanocomposites with added functionality [13,14].

## OLEM EXPERIENCE IN EU PROJECTS:

OLEM, IMech-BAS has experience as partner in many European projects supported by FP7 and H2020 (see https://www.imbm.bas.bg/index.php/en_US/projects-7). We are partner of the largest project H2020-FET- Graphene Flagship (Core 1, 2 and 3 phases).
OLEM (IMech) is the coordinator of the H2020-MSCA-RISE-734164 Graphene 3D, successfully completed at the end of 2022, with partners from Europe, China and Brazil (http://graphene3d.imbm.bas.bg/).

OLEM was founded by a national Research Infrastructure Project in 2010 and we are currently a partner in two Centers of Excellence in Bulgaria. OLEM has coordinated several bilateral projects with research groups from Italy and China, supported by the Bulgarian Academy of Sciences. Our research is funded by many projects of the National Scientific Fund of Bulgaria.

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