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The 4th conference “Nanotechnology and Innovation in the Baltic Sea Region” (NIBS2021) brings together researchers and business stakeholders from the Baltic Sea Region (BSR) as well as parties involved in the cooperation with the BSR partners to share latest achievements in research, innovative solutions and applications in the field of nanotechnology. NIBS2021 focuses on the BSR, where the innovation potential from newest technologies is utmost high.

NIBS2021 provides presentations from companies and research institutions on current trends in the field of nanotechnology. Companies have the opportunity to communicate their success stories and the newest developments in-house. Young scientists present their work and find entrepreneurial opportunities.

The conference is organized by the North German Initiative Nanotechnology Schleswig-Holstein e.V. (NINa SH eV), the Mads Clausen Institute at the University of Southern Denmark, Kaunas University of Technology, Lithuania, and Kiel University, Germany.

Due to the Corona crisis the 4th NIBS conference is held virtually. Using a combination of video conferencing and a virtual meeting platform, we are sure that professional exchange and international networking will be successful even under these challenging conditions.

We wish you much success and exciting encounters at NIBS2021!

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NIBS2021 Technical Digest

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All times are in Central European Summer Time (CEST)



Nano-electrified-by-the-sea: sun, wind, water, plasma

Kostya (Ken) Ostrikov,^{1,2,3} Ronny Brandenburg,² Holger Kersten,³

Franz Faupel,³ and Klaus-Dieter Weltmann²

¹ Queensland University of Technology, Brisbane, Australia

² Leibniz Institute for Plasma Science and Technology, Greifswald, Germany

³ University of Kiel, Germany

This presentation explores diverse interesting opportunities to develop and use plasma-electrified, plasma-catalytic, and hybrid (synergistic) processes to desalinate and recover resources from seawater, create high-performance catalysts and membranes, generate and store sustainable energy, capture and convert CO₂, grow food – thus, contributing to several societal goals of the UN.

These opportunities have become even more important because of the COVID-19 pandemic, which stimulated many people to relocate from crowded large cities and settle in less urbanized seaside areas. The “sea-change” is a global socio-economic trend, which deserves dedicated research activities dealing with water treatment and usage technologies, methods for using sustainable off-shore/on-shore wind energy, solar- and hydropower as well as green chemistry to safe water as a resource. On one hand, some fraction of work must be carried away from centralized labs, and on the other hand, we need to contribute to meeting the persistent practical demands of de-centralized clean energy production and sustainable resource recovery.

We focus on what seaside areas offer plentifully: inexhaustible solar energy, breezy air, salt-laden seawater, ocean (wave and chemical) energy, diverse plant material, sand, rocks, seashells, and myriads of living creatures. This enormous diversity of objects creates an opportunity to use renewable energy powered de-centralized systems to sustainably utilize the many resources the seaside offers. One example of such potentially distributed systems are low-temperature plasma-based and electrochemical reactors that can be made portable, scalable, and also powered by renewable (solar, wind or tidal) energy. Diverse nanomaterials can be processed and converted to the higher-value forms, including advanced functional materials for energy and catalytic applications, high-performance catalysts, activated solutions, etc.

Partial support from the Alexander von Humboldt Foundation through the Humboldt Award Program is kindly appreciated.

Keywords: plasma nanotechnology, seawater, clean energy, resource recovery.

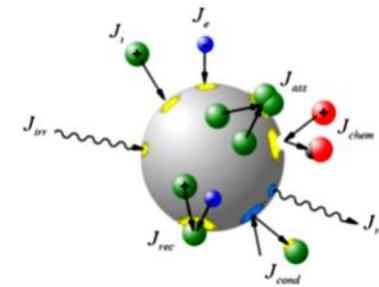
The energy balance of nanoparticles in plasmas

Holger Kersten

PlasmaTechnology, IEAP, University Kiel, Leibnizstr.19, 24098 Kiel, Germany

The origin of particle generation during plasma processing for the formation of nanocomposite materials are (i) the generation of large molecules, mesoscopic clusters and nanoparticles in the plasma bulk by chemically reactive gases, and (ii) the formation and incorporation of nanoparticles at surfaces (target, substrate) by means of plasma-wall interaction (e.g. sputtering, deposition). The plasma process promotes the particle formation by excitation and ionization as well as by dissociation and reaction of the involved species in the gas phase. Typical examples are plasma polymerization and thin film deposition in precursor-containing plasma enhanced chemical vapor deposition (PECVD) processes [1] or sputtering of metal clusters [2].

Determination and understanding of energy fluxes to nano- or micro-particles, which are confined in process plasmas, is highly desirable because the energy balance results in an equilibrium particle temperature which affects the nanoparticle properties [3]. A simple balance model is used to estimate the energy fluxes between plasma and particles on the basis of measured plasma parameters, see Fig.1. Addition of molecular gases to the argon plasma results in additional heating of the particles due to molecule recombination. The several contributions J which affect the particle temperature are discussed in detail. Of special interest are the energy transfer by metastables and the recombination of charge carriers and atoms, respectively, at the particle surface which can result in remarkable heating and subsequent crystallization of nanoparticles in deposition of nanocrystalline thin films [4]. By changing the gas composition, the nanoparticles microstructure can be controlled from mostly amorphous to diamond cubic crystalline.



Material Compounds on Nanoscale: Photo catalysts based on g-C₃N₄ and CD

Wilfried Wunderlich¹, Sergej A. Kulinich²

¹ Material Science Dept., Faculty of Engineering, Tokai University, Japan

² Research Inst. of Science and Technology, Tokai University, Japan

In this overview we will emphasize the important parameter for photocatalysts, the technology of processing and future development of photo catalysts, mainly focussing on water purification. Recently the research proceeded from nano-sized titania particles (fig 1 a) [1, 2] towards nano-composites consisting of mechanical stable skeletons, such as g-C₃N₄. Long-term stability can be achieved, when wrapping the composite with TiO₂ spirals [3]. Laser-ablated Au-particles [4] are now gradually substituted by carbon dots (CD) [5], since in 2004 the ability of tuning their band-gap was discovered [6].

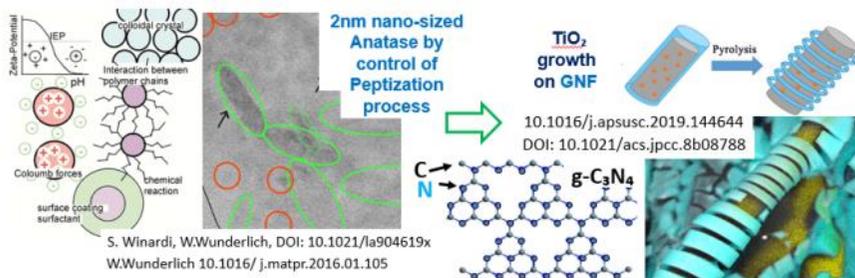


Fig. 1. Development of Photo catalysts from (a) nano-sized TiO₂ to (b) nano-composites

Keywords: photocatalyst, nanocomposite, carbon dots.

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Nano-engineering of III-V semiconductor compounds and metal nanostructures based on electrochemical technologies

Eduard Monaico¹, Ion Tiginyanu^{1,2}

¹National Center for Materials Study and Testing, Technical University of Moldova, Chisinau MD-2004, Republic of Moldova

²Academy of Sciences of Moldova, Chisinau MD-2001, Republic of Moldova

Over the last two decades, it has been demonstrated that electrochemistry is one of the most accessible and cost-effective approaches for nanostructuring semiconductor materials in a controlled fashion. Moreover, taking into account that electrochemical nanostructuring in neutral electrolyte based on NaCl of semiconductor compounds like InP, GaAs, and GaN was demonstrated, we succeeded to make this method also environmentally-friendly [1].

Semiconductor compounds in comparison with Si provide more space for tailored nanofabrication in terms of compositions, bandgaps, mechanisms of the pore growth and new properties with large potential for applications. Besides, semiconductor nanotemplates which properties can be easily controlled by external illumination, applied electric fields, etc. provide wider possibilities for nanofabrication, compared with dielectric nanotemplates. Combining two methods, namely electrochemical etching and electrochemical deposition, one-dimensional nanostructures such as nanowires, nanotubes and their networks have been successfully fabricated. The walls of the porous semiconductor skeleton, exhibiting good electrical conductivity in comparison with the walls of dielectric nanotemplates, create good conditions for uniform nucleation of metal dots [2].

Electrochemistry proved to be an indirect tool for the characterization of semiconductor materials. The non-uniformity of doping during the HVPE-growth of GaN semiconductor crystal was disclosed through electrochemical etching [3], while gold electroplating was used for assessing the conductivity of InP nanostructures fabricated by anodic etching of crystalline substrates.

The report will focus on different aspects of pore growth, transition from the porous semiconductor structures to the formation of semiconductor nanowires, followed by the description of approaches enabling one to fabricate networks of nanowires, nanobelts and nanomembranes as well as technologies for controlled electrochemical deposition of metal nanostructures into porous semiconductor templates.

This work received partial funding from the European Commission under the H2020 grant #810652 'NanoMedTwin' and PostDoc Grant #21.00208.5007.15/PD.

Keywords: porous templates, anodization, nanotubes, nanodots, electroplating.

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Real-time Imaging of Plasmon Propagation on the Nanoscale

Michael Bauer

*Institute of Experimental and Applied Physics, Christian-Albrechts-Universität zu Kiel
<https://www.physik.uni-kiel.de/de/institute/ag-bauer>*

The unique properties of Surface Plasmon Polaritons (SPPs) and Localized Surface Plasmons (LSPs) permit sub-wavelength confinement, light velocity propagation, and highly localized sensing and envision exciting perspectives for combining broadband optics and nanoscale electronics. A profound understanding of plasmonic functionalities relies, however, on suitable experimental means that allow to control, track, and analyze plasmonic excitations in space *and* time at sub- μm lateral and femtosecond temporal resolution.

In my talk I will present examples on how photoemission electron microscopy can be used to analyse 1D and 2D SPP propagation in the frequency and time domain. I will discuss results on SPP supporting nano- to microscale plasmonic operation units [1], [2], on the real-time observation of SPP propagation [2] and SPP-LSP interaction [4], and on the light-control of SPP diffraction using photochromic molecules [5].

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Biometalloid: A New Mesohybrid Superstructure with a Plasmonic readout of its formation and Phase Transformation

Mady Elbahri¹, Ahmed Soliman¹, Duygu Disci²

¹*Nanochemistry and Nanoengineering, School of Chemical Engineering, Department of Chemistry and Materials Science, Aalto University, Kemistintie 1, 00076 Aalto, Finland*

²*Nanochemistry and Nanoengineering Group, Institute for Materials Science, Faculty of Engineering, University of Kiel, Kaiserstrasse 2, 24143 Kiel, Germany*

In contrast to the common wisdom that biofabrication of nanomaterials, which is often boiled down to relatively simple chemistry described by an underestimated oversimplified view, here we set forth the Bioshell concept as a sustainable design of unconventional Biometalloid superstructures of atomistic-molecular silver-based Mesohybrid. Indeed, a deep and complex multidisciplinary knowledge in characterization and simulation techniques were required to unravel the secrets behind these intelligent nanobiosystems from a mechanistic standpoint. Undoubtedly, Biometalloids open up a new vein of research that ranges from a phase-dependent property exhibited by its plasmonic coloration, up to self-adherent biofilms for electrocatalytic applications.

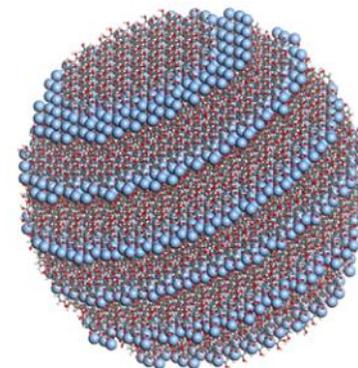


Fig. 1. Monte Carlo based Ab-initio prediction of 10 nm cluster crystal structure of the Ag Biometalloid obtained via polymorph package in Materials Studio

Keywords: *Plasmonic Coloration, Oxygen reduction reaction, organic/inorganic hybrid material, biogenic synthesis, superstructures, phase transformation*

2D plasmonic nanostructures

Sigitas Tamulevičius

*Institute of Materials Science, Kaunas University of Technology, Baršausko st. 59,
Kaunas 51423, Lithuania, Sigitas.Tamulevicius@ktu.lt*

It is known that light can strongly interact with metal nanoparticles exciting collective oscillations of conduction electrons called local surface plasmon resonances (LSPRs). The intensity and width of the LSPR peak is important because it represents the rate of energy loss in the system, which has an impact on various applications in biosensing, photocatalysis, and nanoscale lasers. Single nanoparticles have broad LSPRs due to loss processes associated with radiative damping and depolarization. However, the intrinsic Q-factors of nanoparticles (NPs) can be improved significantly by patterning them in arrays. This enables the NPs to couple via diffraction and oscillate in sync as a collective group, causing each LSPR to narrow. The presentation will include the results of studies of the collective optical behavior of self-assembled noble nanoparticle arrays, their internal photophysical processes, and potential exploration for plasmon nanolasers and biosensors. The optical properties including steady state and ultrafast response of noble metal nanoparticles will be considered including the influence of long range periodicity. The capillarity assisted particle assembly as an efficient method to produce 2D nanostructures will be discussed, paying attention to the technology of dedicated templates and processes of deposition.

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MoS₂ a Two Dimensional Nanomaterial for Next Generation Electronics

Arun Kumar Singh

*Department of Pure & Applied Physics, Guru Ghasidas Vishwavidyalaya, Bilaspur,
Chhattisgarh 495009, India, arunsingh.itbhu@gmail.com*

Two-dimensional (2d) nanomaterials have stimulated a vast amount of research in the fields of condensed matter physics and materials science in the past several years due to their rich and tunable electronic, optical, chemical, mechanical, magnetic and thermal properties. Graphene has received widespread attention since it was first isolated from graphite, has very high charge carrier mobility at room temperature, optically transparency and flexibility making graphene an ideal candidate for a transparent conducting electrode. However, the absence of an energy gap in the electronic band structure limits its practical applications in logic electronic devices. Recently the field has rapidly expanded beyond graphene following the upsurge of semiconducting 2D materials with finite bandgaps such as the transition metal dichalcogenides (TMDCs). Molybdenum disulfide (MoS₂), is n-type TMDC semiconductor with tunable bandgap, is attracting increasing interest for its novel electronic and optoelectronic properties. Tailoring of electronic properties of MoS₂ is essential in order to best performance of its electronic and optoelectronic devices. Here we present some simple and easy technique to tune the electrical properties of MoS₂ nanosheets by doping as well as low-voltage and high-performance dual gated transistors of MoS₂ using the single-layer graphene as source-drain electrodes.

Keywords: 2D nanomaterials, MoS₂, electrical properties, doping

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Self-assembled copolymer systems studied by NMR and other methods

Jacek Jenczyk, Stefan Jurga

NanoBioMedical Centre, Adam Mickiewicz University, Wszechnicy Piastowskiej 3, 61-614 Poznań, Poland

Block copolymers (BCPs) represent interesting group among synthetic materials since they are well known from their unique self-assembling properties [1]. BCPs self-assembly has drawn the attention of scientists and engineers working in a field of microelectronics and nanolithography due to both periodic nature and nanoscopic dimensions of observed domain structures. It has been well documented that BCP patterning can easily compete with conventional photolithography giving access to sub-10-nanometric feature sizes [2]. The ultimate goal is to develop bottom-up technology based on self-organized organic compounds which on the one hand would be a low cost alternative to the expensive top-down strategies and on the other hand would provide a high quality products comparable with those manufactured utilizing already established methods. Therefore, BCPs based technology requires thorough assessment of domain architecture, domain dimensions and the size of interfacial region. The latter parameter appears to be particularly important in nanolithography however its accurate estimation remains challenging. Throughout the processing stages, quite often the BCPs matrix undergoes selective etching and hence the interface determines eventual feature size resolution and sharpness of attained structural motifs. Although, microscopic and scattering techniques provide complete and reliable picture concerning the domain morphology and its periodicity, they both lack of quantitative data regarding the mentioned transition phase. Therefore, in order to thoroughly monitor the copolymer systems, complementary NMR methods are employed. Accordingly, when investigated spectroscopically, this seemingly simple, two phase BCPs model reveals additional interesting features and more complex structure than expected. Apart from structural studies we also monitored polymer chain dynamics using both, broadband dielectric spectroscopy and NMR relaxometry. Obtained data enabled assessment of: i) the polymer chain reorientations difference between neat polymers and their copolymers, ii) impact of nanoparticles inclusion on polymer dynamics, iii) observation of motions within rigid and mobile amorphous phase within semicrystalline polymers.

Keywords: self-assembling systems, block copolymers, NMR, AFM.

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Nanocomposites Based on Biomimetic Polymers and Semiconductors for Energy Applications

Emerson Coy¹

¹NanoBioMedical Centre, Adam Mickiewicz University, Wszechnicy Piastowskiej 3, 61-614 Poznan, Poland

Light-driven photocatalysis is at the core of global energy concerns, due to its implications for hydrogen production and the imperative need to reducing our fossil fuel dependency. Polydopamine (PDA) is a popular mussel inspired material with large applicability in biomedical and drug delivery fields, which recently has been gathering attention in the fields of catalysis and photocatalysis. This is because it seems to play an unexpected role in enhancing hydrogen production and tuning optical bandgaps of coated semiconductors **Fig 1(a-d)**[1,2]. In this talk, we will show the recent advances towards understanding the role of PDA on the enhanced photocatalytic properties of several semiconductors and we will discuss further strategies and developments in order to optimize coating processes of PDA **Fig 1(e-f)**[3]. Finally, we will outline some of the prospects and opportunities for PDA/semiconductor research in the next few years.

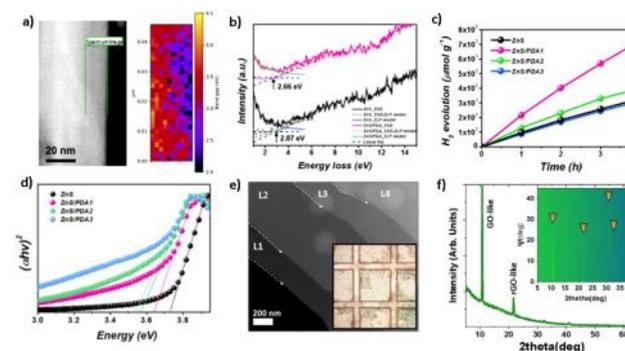


Fig. 1. a) S-TEM image with EELS bandgap map for PDA/ZnS wires. b) bandgap estimation at the interface of PDA/ZnS and bare ZnS. c) Hydrogen evolution of PDA/ZnS at different thicknesses. d) Bandgap changes with PDA thickness. e) Layered structure of PDA 2-like membranes and f) supramolecular structure of the PDA layers.

Keywords: Polydopamine, Semiconductor, Photocatalysis, Nanocomposites

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Acknowledgements

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New developments in the realization of hybrid organic-inorganic nanocomposites for ionizing radiation detectors

Rosana Martinez Turtos¹, Camilla Lønborg Nielsen¹, Brian Julsgaard¹, Peter Balling¹

¹Department of Physics and Astronomy, Ny Munkegade 120, Aarhus, Denmark

Light-emitting semiconductor and insulating nanocrystals have opened a new route to the realization of fast, next generation scintillator-based particle detectors [1] and novel 3D dosimeters [2]. While their nano-scale sizes enable to achieve high optical transparency in large scale sensors even with a considerable refractive index mismatch [3], their dimensions impose severe limits on the amount of energy deposited per nanoparticle [4].

In this talk, we will review the efforts in the direction of developing new scintillator-based particle detectors required for advanced medical imaging scanners, together with new research in the field of 3D reusable dosimetry for particle therapy verification.

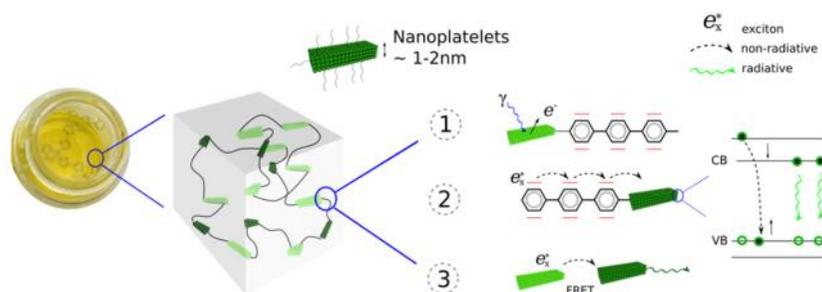


Fig. 1. Fundamental energy conversion and transfer mechanism in hybrid inorganic-organic nanocomposites. 1. High-energy particle conversion to secondary electrons and hot-electron thermalization, 2. Non-radiative energy transfer organic-inorganic and 3. Non-radiative energy transfer inorganic-inorganic.

Keywords: hybrid inorganic-organic nanocomposites, nanocrystal-based radiation detectors, 3D dosimetry.

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New strategies in polymer capacitor dielectric fabrication to meet the needs of power electronics in the green transition

William Greenbank¹, Luciana Tavares¹, Thomas Ebel^{1,2}

¹Department of Mechanical and Electrical Engineering, Centre for Industrial Electronics (CIE), University of Southern Denmark (SDU), Alision 2, DK-6400 Sønderborg, Denmark

²SDU Electrical Engineering, Alision 2, DK-6400 Sønderborg Denmark

Capacitors are the basic building blocks of the electronic world and their optimisation can have direct and tremendous impact in the improvement of the performance of power electronics and, consequently, on the reduction of CO₂ emissions.[1] Polymer capacitors fulfil important demands for these applications, which includes their unique self-healing capability, high breakdown strength, low mass density, inexpensive production, and ease of processing. However, their low volumetric capacitance, limited energy storage densities and poor heat transfer (which keeps the devices operating reliably) are still challenges that limit the use of film capacitors. Therefore, we have developed new fabrication techniques to improve these properties. This includes a novel layer-by-layer fabrication technique that allows the facile printing of thin (sub-micron) polypropylene capacitor dielectrics with tuneable properties through the use of nanocomposite fillers and careful dielectric design.[2]–[4] This new technique allows the tailoring of dielectrics to different specific applications through the selective optimisation of different dielectric properties (dielectric constant, breakdown strength, thermal stability, etc). Furthermore, we are investigating the use of a multilayer film modification procedure that improves heat removal from the dielectric. This work will result in capacitors that are more efficient and reliable both at high temperature and high voltages, meeting the needs of industry and the green transition.

Keywords: polymer capacitors, volumetric capacitance, energy storage density, thermal conductivity, layer-by-layer fabrication, nanocomposites.

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Formulation optimisation of chitosan-based nanoparticles with the aim of efficient protein loading

Lena Valentin, Regina Scherließ

Department of Pharmaceutics and Biopharmaceutics, Kiel University, Grasweg 9a, 24118 Kiel, Germany

Chitosan nanoparticles have a high potential as drug carrier for nasal or pulmonary vaccination. Chitosan with its versatile properties, such as mucoadhesiveness, permeation enhancement and immunogenicity, is well suited for preparing nanoparticles for mucosal vaccination [1]. An efficient protein/antigen loading is key to cost reduction in formulation development and effective therapy. Therefore, this work aims at optimising protein loading efficiency and stability on the nanoparticle suspension by changing the pH, polymer and protein concentration of the formulations.

Preparation of nanoparticles: Nanoparticles (NP) were prepared by ionic gelation, possible through the contrarily charged polymers chitosan (positive) and carboxymethyl-cellulose (CMC) (negative). Chitosan was diluted in acetic acid and the pH was adjusted (3, 3.5, 4, 5, 5.5, 6) with diluted NaOH. Protein solution (Ovalbumin (OVA) in ultrapure water) and chitosan solution were mixed up, before CMC solution was added dropwise. Directly, nanoparticles were formed, resulting in a turbid suspension.

Protein quantification: Ovalbumin concentration in the nanoparticles was quantified indirectly by absorption at 280 nm. For this, the OVA concentrations in the total suspension and in the supernatant (NP were centrifuged before) were measured and the values were subtracted to obtain the protein concentration in the nanoparticles. Nanoparticle suspensions without OVA was used as blank.

Size and zeta potential: Size and zeta potential were measured by dynamic light scattering with the Zetasizer (ZS Nano, Malvern Instruments, UK).

We were able to increase the loading efficiency (LE) immensely through the adjustment of pH value. The results show linear correlation between pH and LE with an increase from 4% to 27%. The results can be explained by the charge of the polymers and OVA. OVA has an isoelectric point (IEP) of 4.5, hence at pH values under its IEP it is positively charged and above negatively charged. Therefore, at higher pH (> 4.5) OVA is negatively charged and can form a complex with chitosan before CMC will be added. The higher pH values lead to less positive charges in total and this results in low stability of the suspension. Therefore, the ratio of 1:1:0.5 chitosan-CMC-OVA and a pH of 5.5 was found out for the best compromise between LE and stability of the nanoparticle suspensions.

Keywords: loading efficiency, pH, stability, zeta potential, ionic gelation

References:

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Investigation of colour representation accuracy of true colour hologram rendering algorithm

Tomas Klinavičius¹, Tomas Tamulevičius^{1,2}

¹Institute of Materials Science of Kaunas University of Technology, K. Baršausko Str. 59, LT-51423 Kaunas, Lithuania

²Department of Physics of Kaunas University of Technology, Studentų Str. 50, LT-51368 Kaunas, Lithuania

Dot matrix security holograms are photonic devices that use diffraction to create colour and other optical effects. Such holograms consist of an array of diffraction gratings that differ from one another in their grating period and in plane orientation. However, ordinary dot matrix holograms only generate spectral colours because their smallest structural unit is a single diffraction grating which can only create a monochromatic spectrum [1]. True colour holograms are also capable of recreating mixed colours [2]. Human colour perception has been thoroughly investigated in the literature. However, due to differences in perception varying from individual to individual, only statistical research has yielded significant results [3].

This work investigates colour representation accuracy of true colour holograms by applying statistical methods and CIE colour system theory in order to form a metric for investigation of colour differences based on human perception.

Keywords: colour, dot matrix hologram, diffraction

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Analysis of ZnO Nanotetrapods Using UV-Vis Spectroscopy

Mindaugas Ilickas^{1,2}, Simas Raèkauskas^{1,2}

¹Department of Physics, Kaunas University of Technology, Lithuania

²Institute of Materials Science of Kaunas University of Technology, Lithuania

This work focuses on investigation of optical properties of ZnO nanotetrapods using UV-Vis spectroscopy. The ZnO material has a wide band gap – $E = 3.37$ eV [1] and offers many applications, especially for the ZnO nanostructure forms. The tetrapod is formed of four rod-shaped structures, also known as "legs," which are interconnected with one another by the central core [2]. The tetrapods have many advantages due to their structure, ensuring a good stability. The work investigates ZnO nanoparticles obtained by rapid oxidation (combustion) synthesis. It was found that more and more nanoparticles precipitate with increasing test periods after conducting UV-Vis spectrometric analysis that different nanostructures – particles, wires, tetrapods – were generated during rapid oxidation (combustion) synthesis. The particle sizes can be reduced by the centrifugation. Increasing centrifugation speed particles can be separated into fractions, with the largest (63 nm) particles sedimented at 1000 rpm and the smallest (52 nm) at 10000 rpm by centrifugation. The outcome of the work may be used for different real world applications, such as versatile UV or gas sensors.

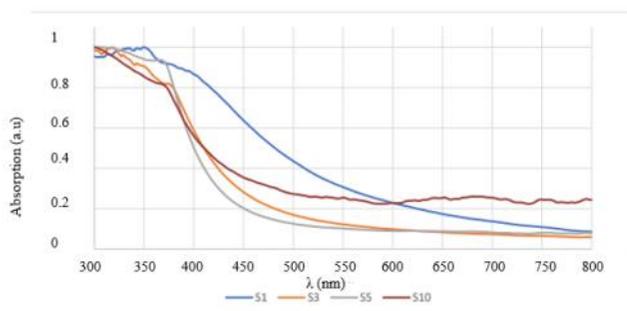


Fig. 1. Dependence of light absorption on centrifugation frequency at different phases 1000 rpm to 10000 rpm (S_1 - S_{10}) of the sedimented fraction

Keywords: nanoparticles, sensors, UV, synthesis

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This research has received funding from the European Regional Development Fund (project No. 01.2.2-LMT-K-718-02-0011) under a grant agreement with the Research Council of Lithuania (LMTLT).

Dual-Functional Optical Waveplate Based on Gap-Surface Plasmon Metasurfaces

Ziru Cai¹, Yadong Deng^{1,2}, Cuo Wu², Chao Meng², Yingtao Ding^{*1}, Sergey I. Bozhevolnyi², and Fei Ding^{*2}

¹School of Information and Electronics, Beijing Institute of Technology, Beijing 100081, China

²Centre for Nano Optics, University of Southern Denmark, DK-5230 Odense M, Denmark

Gap-surface plasmon (GSP) metasurfaces have attracted increasing attention and have become an emerging research area due to their planar configurations, high efficiencies, ease of fabrication, and unprecedented capabilities in manipulating reflected fields, thereby allowing one to reduce a set of bulky optical components to a single ultrathin element with multiplexed functionalities¹. Herein, a reflective dual-functional optical waveplate (Fig.1a) based on periodic GSP meta-molecules (Fig.1b) is designed and experimentally demonstrated, exhibiting combined quarter-waveplate and half-waveplate functionalities, i.e., providing linear-to-circular and linear-to-linear polarization conversion simultaneously, at the same operating wavelength for orthogonal linear polarizations. The proof-of-concept fabricated metasurface demonstrates excellent and distinct polarization conversion functionalities with the efficiencies of $\sim 73\%$ and 30% at the design wavelength of 850 nm for the orthogonal incident polarizations, well in agreement with the simulations. Owing to the compactness and excellent performance, the demonstrated reflective dual functional optical waveplate opens yet another avenue for further development of flat polarization optical components with multiplexed functionalities.

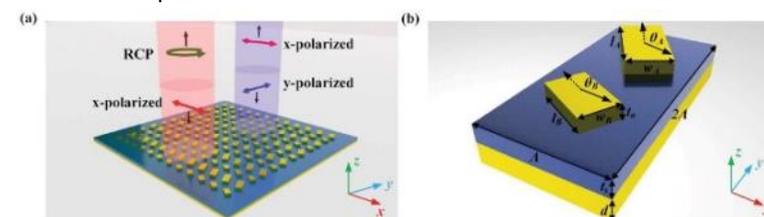


Fig. 1. a) Working principle of the dual-functional optical waveplate. b) Schematic of the meta-molecule unit cell.

Keywords: dual-functional, waveplate, optical range, gap-surface plasmon metasurfaces

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Modification of the electronic properties of the TiO₂(110) surface by sputtering and annealing

Karol Cieřlik¹, D. Wrana¹, K. Szajna¹, W. Betza¹, M. Rogala²,
C. Rodenbächer³, P. Dąbczyński¹, K. Szot^{4,5}, F. Krok¹

¹ Marian Smoluchowski Institute of Physics, Jagiellonian University,
Krakow 30-348, Poland

² Faculty of Physics and Applied Informatics, University of Lodz, Pomorska 149/153,
90-236 Lodz, Poland

³ Institute of Energy and Climate Research (IEK-14), Forschungszentrum Jülich,
52425 Jülich, Germany

⁴ August Chelkowski Institute of Physics, Silesian University, Katowice, Poland

⁵ aixACCT Systems GmbH, 52068, Aachen, Germany

Titanium dioxide (TiO₂) nanoparticles are undoubtedly one of the most studied materials for photocatalytic applications. It comes with no surprise that many different crystallographic forms of TiO₂ have been studied in various shapes and sizes, modified using multiple methods [1]. However, to better develop these applications, the underlying processes of photocatalysis must first be studied and understood. Powders, nanocrystallites, and nanostructures show promising results. However, due to their complexity, they are not best suited for investigations of fundamental processes. On the other hand, studying monocrystal surfaces in ultra-high vacuum comes with many advantages – great control of the experimental conditions, easier to understand the system. This is why these investigations are performed in such conditions.

In order to begin such an investigation, the sample must first be prepared so that it is free of impurities, adsorbates, and is crystalline. The typical way of achieving it is to repeatedly sputter the surface with an ion beam and then anneal it to high temperatures. Only after multiple cycles, the surface is deemed to be fit for experiments. However, the preparation method varies between laboratories – the temperatures, times, fluences and the number of cycles are not set in stone. Typically, such experimental details are omitted in publications. They are considered a means to an end and thus not of significant importance. We show that this initial step significantly changes the electronic properties of TiO₂(110) rutile, that is, work function and conductivity, the properties essential for photocatalysis.

Moreover, we show that using sputtering and annealing, the electronic properties of a TiO₂ crystal can be modified and even tuned to reach desired values [2]. Using local conductivity and Kelvin modes of atomic force microscopy, we show that the rutile surface undergoes substantial changes in properties due to sputtering and annealing. A variation in stoichiometry accompanies these changes, as demonstrated by x-ray photoelectron spectroscopy (XPS) and secondary ions mass spectrometry.

Keywords: titanium dioxide, photocatalysis, work function, conductivity, annealing, sputtering.

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Photocatalytic Deposition: Unconventional Approach for Micro- and Nanoarchitecture

Salih Veziroglu, Josiah Shondo, Tim Tjardts, Tamim B. Sarwar,

Thomas Strunskus, Franz Faupel, Oral Cenk Aktas¹

¹Chair for Multicomponent Materials, Institute of Materials Science, Kiel University,
24143 Kiel, Germany

Micro- and nanostructures specifically noble metals (Au, Ag, and Pt, etc.) have been received outstanding attention during the last decades, because of their unique structural, electronic, and (photo)catalytic properties. In particular, the incorporation of these micro- and nanostructures with metal oxide semiconductors (e.g., TiO₂, ZnO, etc.) has been reported many times for several applications such as sensor, (photo)catalysis, water splitting, self-cleaning, and so on. Generally, researchers focus on the synthesis of micro and nanostructures on the metal-oxide substrate with well-defined size and morphology. However, it is still a challenge to attain good adhesion between micro- and nanostructures and metal oxide surfaces, especially on thin films. Recently, some approaches (seed-mediated growth, etc.) have been demonstrated to improve the adhesion of metallic particles with thin film by using some binder molecules (thiols and silanes, etc.). Here, mostly organic molecules are used for binding metallic micro- and nanostructures with a solid substrate. However, these organic molecules may contaminate the surface and adversely affect the surface properties of the final structure. That is why there is a strong need to prepare stable metallic micro- and nanostructures on metal oxide surfaces without using any organic molecules (binders). Here, we demonstrate a novel photocatalytic deposition approach for preparing metallic and bimetallic Au, Ag, and Pt micro- and nanostructures on TiO₂ thin film surface by UV illumination. Prepared hybrid structures with strong chemical adhesion can be used for wide-range applications such as photocatalytic degradation of organic compounds, self-cleaning and oil-water separation. The photocatalytic deposition technique allows the controlling the geometry, size, and distribution of micro- and nanostructures on thin film by simply changing the deposition solution, photocatalytic activity of metal oxide, UV illumination intensity, and time [1-4].

Keywords: photocatalytic deposition, noble metal, metal oxide, metallic and bimetallic structure, micro- and nanostructure

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A nanoscopic journey through spider silk

Irina Iachina¹, Jacek Fiutowski¹, Yue Jin Oh², Serguei Chiriaev¹,
Horst-Günter Rubahn¹, Fritz Vollrath², and Jonathan R. Brewer³

¹Mads Clausen Institute, University of Southern Denmark, Denmark

²Department of Zoology, University of Oxford, UK

³Department of Biochemistry and Molecular Biology, University of Southern Denmark, Denmark

The remarkable mechanical properties of spider silk have fueled interest in understanding its material properties and developing an artificial material with similar traits[1, 2]. To develop artificial spider silk, it is necessary to understand the complex structure of the natural silk fibers, as the properties of spider silk arise from both the nanoscopic organization of its proteins and the hierarchical structure of the silk. Determining the nanostructures within the protein core is challenging, as the techniques previously used require sample alteration or are limited in resolution[3-6]. In this work, the macro- and nanoscopic structure of pristine dragline (Major Ampullate silk, MAS) and auxiliary spiral (Minor Ampullate silk, MiS) silk fibers from the orb-web weaving spider *Nephila madagascariensis* are examined. Coherent anti-Stokes Raman scattering- and confocal microscopy revealed an outer lipid rich layer of between 600nm-1µm thick. These lipids surround an auto-fluorescent protein core which was divided into two concentric layers in both fiber types. The nanoscopic protein fibrils within pristine spider silk were imaged, for the first time, using scanning helium ion microscopy and confocal Raman microscopy. Fibrils were seen to be arranged parallel to the length of the fiber and were present throughout the protein core. The fibril diameters were between 89-228nm. Combined, our results from these different microscopy techniques, thoroughly characterize the microscopic and nanoscopic structures in these fibers with minimal sample alteration. The knowledge of the silks fibril structure and the methods to measure it presented here will support the development of future superior artificial spider silk.

Keywords: Spider silk, *Nephila*, coherent anti-Stokes Raman scattering microscopy, confocal microscopy, helium ion microscopy, confocal Raman microscopy.

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Mechanical properties of high-temperature Ir_xRe_{1-x} alloys

A K Maddheshiya¹, P S Yadav¹ and R R Yadav¹

¹Department of Physics, University of Allahabad, Prayagraj-211002, India

The Ir_xRe_{1-x} (0.1 ≤ x ≤ 0.7) alloys are hexagonal structured inclusion material used in an on-orbit communication satellite. The characteristic features of high-temperature alloys Ir_xRe_{1-x} (0.1 ≤ x ≤ 0.7) are investigated by the theoretical evaluation of thermophysical and ultrasonic properties at room temperature. Initially, higher-order elastic constants of the alloys are calculated using the simple interaction potential model approach. With the help of this other elastic moduli, elastic stiffness constants and hardness parameters are estimated at room temperature for elastic and mechanical characterization. The obtained results are analysed to explore the inherent properties of Ir_xRe_{1-x} alloys and further helpful for their bulk counterpart.

Keywords: Ultrasonic properties, Elastic constants etc

Potential of novel cadaverine biosensor technology to predict shelf life of chilled yellowfin tuna (*Thunnus albacares*)

Niki Alexi¹, Jeanette Hvam², Birgitte W. Lund³, Lawrence Nsubuga^{2,4}, Roana Melinade, Oliveira Hansen⁴, Kristian Thamsborg⁶, Fabian Lofink⁵, Derek V. Byrne¹, Jørgen J. Leisner⁶

¹Food Quality Perception and Society Group, Department of Food Science, Aarhus University, Agro Food Park 48, 8200 Aarhus N, Denmark

²AminIC ApS, Algade 56, 5500 Middelfart, Denmark

³Danish Technological Institute, DMRI, Gregersensvej 9, 2630, Taastrup, Denmark

⁴SDU NanoSYD, Mads Clausen Institute, University of Southern Denmark, Alsion 2, 6400 Sønderborg, Denmark

⁵Fraunhofer Institute for Silicon Technology, Fraunhoferstrasse 1, 25524 Itzehoe, Germany

⁶Food Safety and Zoonoses, Department of Veterinary and Animal Sciences, University of Copenhagen, Grønnegårdsvej 15, 1870 Copenhagen, Denmark

A considerable amount of fresh fish is wasted due to imprecise shelf life estimations that lead to relatively short “use-by” dates. This study focussed on evaluation of a newly developed micro-cantilever-based sensor measuring cadaverine headspace concentrations, as a rapid and precise measure to estimate within a realistic Denmark retail scenario freshness and shelf life of imported tuna loins. Cadaverine concentrations as measured by Liquid chromatography-tandem mass spectroscopy (LC-MS/MS) and Quality Index Method (QIM) and microbial measurements were used for validation of the novel cadaverine biosensor.

The results indicated that the novel cadaverine biosensor allowed an estimation of freshness and transition of acceptable to unacceptable quality of the tuna steaks comparable to other methods such as LC-MS/MS determination of cadaverine, QIM and determination of microbiological counts.

Keywords: Tuna, Freshness, Shelf life, Sensor technology, Cadaverine, Quality index method

References:

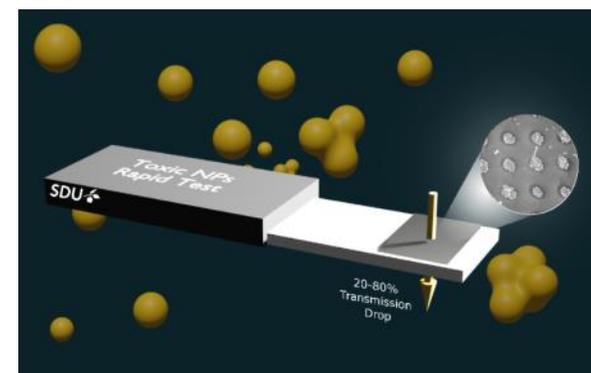
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Rapid Template-Assisted Self-Assembly: A Practical Route to the Fast Assembly of Colloids

Ayoub Laghrissi, Prince Gupta, Horst-Günter Rubahn and Jacek Fiutowski

Mads Clausen Institute, NanoSYD, University of Southern Denmark, Alsion 2, DK-6400 Sønderborg, Denmark

A simple, easy, and quick technique for assembling nanoparticles in topographically designed PDMS molds of nanosized shapes, has great potential in many spectroscopic and sensing tools, paving the way for many applications. Close-packed particle poses rich plasmonic resonances, which enable tailoring the optical response, on both the nano- and the macroscale. Template-assisted self-assembly (TASA) is a method that creates colloidal aggregates with controlled sizes formed by dewetting aqueous dispersions of nanoparticles across surfaces. We present a modified version in which the overall time of the process is less than five minutes, namely, rapid TASA (rTASA), which adds speed and user-friendliness compared to capillary force-assisted nanoparticle assembly (CAPA), where the highest precision in particle positioning is ensured with a high cost of time and critical control of the meniscus shape. Depending on the array pitch distance and the average number of particles per trap, the transmission through the template drops between 20 – 80%, which makes their detection possible using even the most simple spectroscopic solutions. This rapid method could be used as a building block to generate self-assembled systems that may exhibit interesting properties in highly valuable areas, especially in building a fast test for nanoparticle detection.



Epitaxial Pt electrodes for AlScN integration on GaN interfaces

Niklas Wolff¹, Georg Schönweger², Md Redwanul Islam¹, Hermann Kohlstedt²,

Fabian Lofink³, Simon Fichtner^{1,3}, Lorenz Kienle¹

¹Institute for Materials Science, Kiel University, Kaiserstr. 2, 24143 Kiel, Germany

²Institute of Electrical Engineering and Information Engineering, Kiel University, Kaiserstr. 2, 24143 Kiel, Germany

³Fraunhofer ISIT, Fraunhoferstr. 1, 25524 Itzehoe, Germany

Ferroelectric AlScN thin films exhibit a very high spontaneous and piezoelectric polarization [1], a high sheet carrier density and outstanding temperature stability [2] which is intriguing for the next generation of high-power electronics [3]. The integration of high quality epitaxial AlScN thin films in CMOS and III-V technology is one of the recent milestones. In this work, a ferroelectric capacitor based on a Pt/AlScN/Pt/GaN architecture (Fig. 1a) is designed by sputter deposition for demonstration of process compatibility, high structural quality and decent ferroelectric properties.

The designed capacitor structure was analyzed with respect to its micro- and nanostructure and its ferroelectric characteristics in dependence of AlScN film thickness. Transmission electron microscopy studies and X-ray diffraction high-resolution reciprocal space mapping demonstrate the epitaxial [211]Pt/[10-10]GaN growth of a 12.5 nm thin bottom Pt electrode (FWHM = 0.107°) onto a GaN buffer promoting epitaxy for AlScN films. Electrical PUND measurements demonstrate ferroelectric switching for 20 nm AlScN films (Fig. 1b).

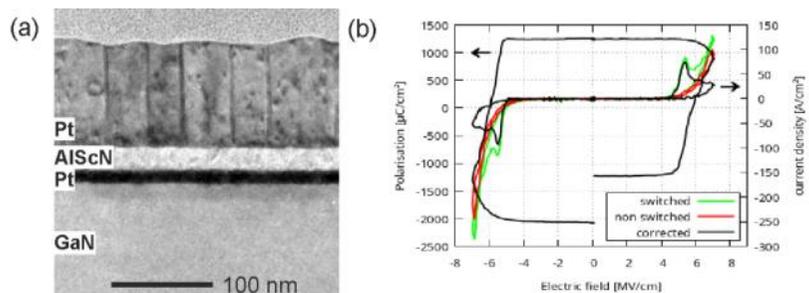


Fig. 1. (a) TEM image of the cross-section through a Pt/AlScN/Pt/GaN capacitor with 12.5 nm Pt and 20 nm AlScN. (b) Positive-Up-Negative-Down (PUND) correction for 20 nm AlScN, measured with a triangular signal at 3.3 kHz.

Keywords: Ferroelectric, Semiconductor technology, III-N, Thin film

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Novel electrochemical approach for the fabrication of free-standing perforated Au nanomembranes in two steps

Eduard Monaco¹, Elena Monaco¹, Veaceslav Ursaki^{1,2}, Ion Tiginyanu^{1,2}

¹National Center for Materials Study and Testing, Technical University of Moldova, Chisinau MD-2004, Republic of Moldova

²Academy of Sciences of Moldova, Chisinau MD-2001, Republic of Moldova

We propose a room-temperature two-step cost-effective electrochemical technology for the preparation of free-standing Au nanomembranes. A thin Au film with thickness less than 100 nm was deposited by pulsed electroplating on a GaAs substrate in the first step [1], followed by anodization in the second technological step to introduce porosity into the GaAs substrate underneath the Au film [2]. At optimized parameters of anodization, detachment of the film from the substrate occurs. The gold film consists of a monolayer of Au nanoparticles 20-30 nm [3]. The flexibility and stretchability, along with possibilities to transfer the prepared nanomembranes to various substrates are expected to be prospective for new optical, plasmonic and electronic applications.

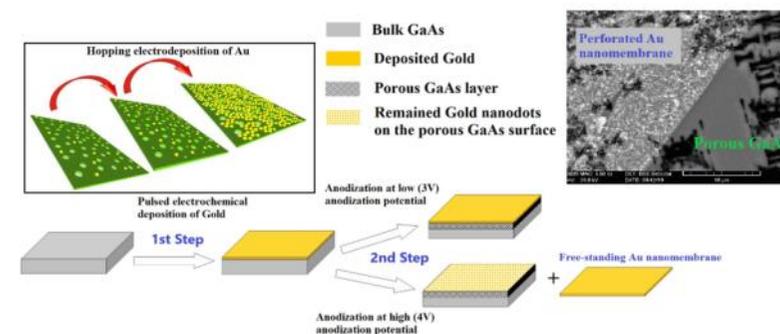


Fig. 1. Schematic representation of the technological route for the fabrication of Au nanomembrane on a porous semiconductor substrate with possibilities to be transferred to another substrate. Inset is the illustration of the mechanism of hopping electrodeposition of a monolayer of Au nanodots and SEM image of an Au nanomembrane prepared by electroplating with pulse duration of 300 us.

Keywords: gold nanomembrane, pulsed electrochemical deposition, anodization, porous GaAs.

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From nano to micro: PowderMEMS technology for innovative microsystems

Axel Müller-Groeling¹

¹Fraunhofer Institute for Silicon Technology, Fraunhoferstrasse 1, 25524 Itzehoe, Germany

The novel fabrication technique “PowderMEMS” developed at Fraunhofer ISIT enables the integration on substrate-level of functional three-dimensional microstructures from a multitude of materials. [1] The process uses nanometer-thin films deposited by atomic layer deposition to solidify particles in pre-formed cavities, Fig. 1a).

The generic technique yields high innovation potential for microtechnology due to the numerous parameters like magnetism [2], porosity, and inner surface which can be used to realize new microsystems, Fig. 1b). Accordingly, application examples of the PowderMEMS-technology will be given for magnetic and fluidic systems.

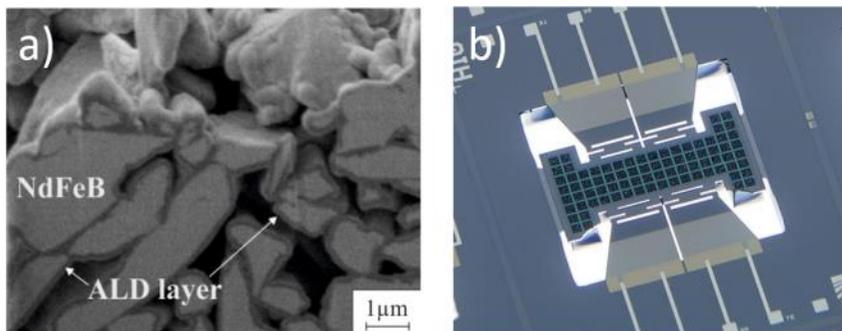


Fig. 1. a) Cross-section of a PowderMEMS agglomerate consisting of NdFeB particles solidified by a layer of 75 nm Al₂O₃. b) PowderMEMS micromagnetic structures integrated in an energy harvesting device [3].

Keywords: Powder-based materials, functional microstructures, MEMS, energy harvesting, biosensors

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From μ to nano: Ferroelectric AlScN for adaptive and neuromorphic devices

Georg Schönweger¹, Tom-Niklas Kreutzer², Md Redwanul Islam³, Adrian Petrau¹,
Niklas Wolff³, Lorenz Kienle³, Fabian Lofink², Hermann Kohlstedt¹, Simon Fichtner^{2,3}

¹Institute of Electrical Engineering and Information Engineering, Kiel University, Kaiserstr. 2, 24143 Kiel, Germany

²Fraunhofer ISIT, Fraunhoferstr. 1, 25524 Itzehoe, Germany

³Institute for Material Science, Kiel University, Kaiserstr. 2, 24143 Kiel, Germany

The discovery of ferroelectricity in AlScN [1] has opened parameter regions that were previously inaccessible with alternative ferroelectric thin films [Fig. 1(a)]. Remanent polarization in excess of 100 $\mu\text{C}/\text{cm}^2$, coercive fields that are systematically adjustable by more than 2MV/cm and excellent compatibility promise to greatly advance the commencing integration of ferroelectric functionality to micro- and nanoelectronics, e.g. for neuromorphic devices.

The eventual success of ferroelectric AlScN will greatly depend on the possibility to scale the material from μm film thicknesses – were its ferroelectric properties first emerged – towards the nm range. This will allow the integration of AlScN into current semiconductor technology nodes and switching voltages that are readily available on integrated circuits. Here, we give a comprehensive overview of our recent progress in scaling ferroelectric AlScN films to below 20 nm film thicknesses and to single digit switching voltages [Fig. 1 (b)]. Thereby, on both Si and GaN substrates, we can confirm a virtually constant coercive field and large remnant polarization in films with thickness between 1 μm and 15 nm – a behaviour that is far from self-evident in ferroelectrics. With this development, the integration of ferroelectric AlScN films to both CMOS and III-N technology has passed an important milestone that will allow the fabrication of first demonstrator devices to commence.

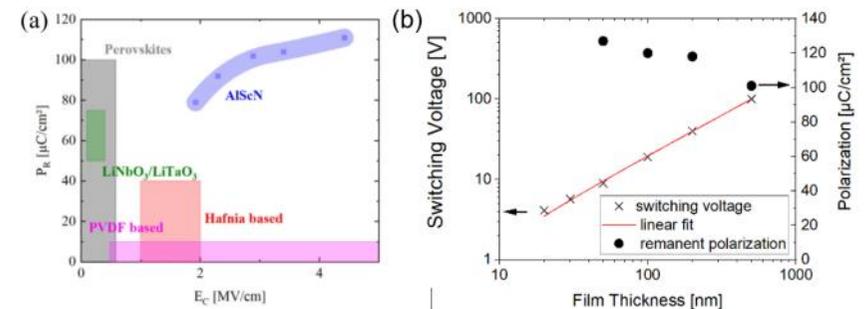


Fig. 1. (a) Remanent polarization and coercive field of AlScN thin films compared to the predominant classes of thin film ferroelectrics. (b) Ferroelectric switching voltage and remanent polarization over film thickness for Al_{0.70}Sc_{0.30}N films deposited on molybdenized Si wafers.

Keywords: Ferroelectric, Semiconductor technology, III-N, Thin film

References:

1. Fichtner et al., *JAP* **125**, 114103 (2019).

NanoFrazor Lithography for advanced 2D & 3D nanodevices

Zhengming Wu, T.S. Kulmala, Nils Goedecke

Heidelberg Instruments Nano, Technoparkstrasse 1, 8005 Zürich, Switzerland

Thermal scanning probe lithography (t-SPL) uses a heatable ultra-sharp tip for patterning thermal resists and simultaneous inspection of the nanostructures being patterned [1]. The technology has proven its value as an enabler of new kinds of ultra-high resolution nanodevices [2] as well as for improving the performance of existing device concepts [3]. Therefore, t-SPL offers the first true alternative or a complementary extension to other mask-less nanolithography methods such as electron beam lithography (EBL). The range of applications for t-SPL is very broad [1] spanning from ultra-high resolution 2D and 3D patterning to chemical and physical modification of matter at the nanoscale. Nanometer-precise markerless overlay and non-invasiveness to sensitive materials are among the key strengths of the technology. However, while patterning at below 10 nm resolution is achieved, significantly increasing the patterning speed at the expense of resolution is not feasible by using the heated tip alone. Towards this end, an integrated laser write head for direct laser sublimation (DLS) of the thermal resist has been introduced for significantly faster patterning of micrometer to millimeter-scale features [4]. Remarkably, the areas patterned by the tip and the laser are seamlessly stitched together and both processes work on the same resist material.

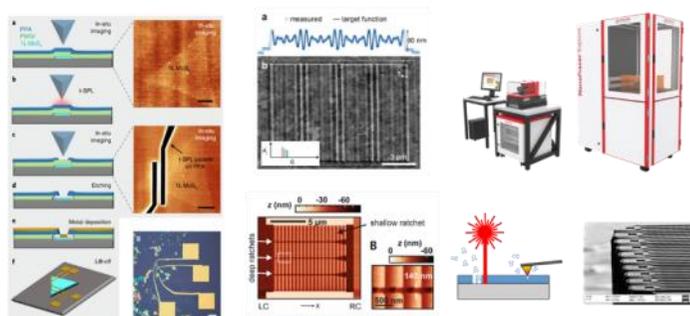


Fig. 1. Left: A typical application of NanoFrazor lithography is the fabrication of high-quality metal contacts using lift-off on sensitive materials without markers and without damage [3]. Middle: Grayscale lithography with sub-nm precision enable new applications in photonics [5] and nanofluidics [2]. Right: Top NanoFrazor Scholar and NanoFrazor Explore. Bottom left: mix & match lithography with direct laser and multi-tip array.

Keywords: grayscale lithography, nanofabrication, 2D material, nano-optics, nano-fluidic, photonics, Laser, magnetic spin wave, nanoparticle assemble,

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Coordinated Development of Tubes and Optics: New possibilities for X-ray Sources

Jörg Wiesmann, Jürgen Graf, Frank Hertlein, Moritz Schlie, Paul Radcliffe,

Carsten Michaelsen

Incoatec GmbH, Max-Planck-Straße 2, 21502 Geesthacht, Germany

As a specialist in multilayer optics, we at Incoatec have a long tradition of providing solutions that are driven by customer needs. Consequently, in 2006 we introduced our multilayer-based microfocus source $\text{I}\mu\text{S}$ and entered the crystallography market. We learnt that our optics can show their full strength as a part of a microfocus source, if the tube is matched to the optics. Based on this fact, we started developing X-ray tubes in-house in 2011. We strived to find the best combination of optics and tube for specific applications in small and macromolecular structure analysis.

This allowed us to launch new solutions such as the $\text{I}\mu\text{S}3.0$ and the $\text{I}\mu\text{S}$ DIAMOND, which offers a flux density of more than $5 \cdot 10^{10}$ ph/s/mm² in a spot of less than 100 μm . This very high flux density was achieved with a low power air cooled tube that is maintenance free during its typical lifetime of more than 6 years.

In our talk, we will summarize the key parameters for combining multilayer optics and microfocus tubes to achieve collimated or focused X-ray sources with high brilliance. The main part of the talk will explain the application-dependent design and capabilities of our newly developed custom metal-ceramic tubes and how to match them with our advanced multilayer optics.

Stand-alone custom tubes and optics made possible by the advanced knowledge of the two centerpieces of the source will also be covered. Applications include crystallography, nanotechnology, thin film research, or as a tool at synchrotrons during downtime or construction periods.

Keywords: X-ray optics, multilayer, magnetron sputtering, diffractometry, X-ray tubes, X-ray analytics

Optical Multiplex Biosensors

Martina Gerken

*Institute of Electrical Engineering and Information Engineering, Kiel University, Kaiserstr. 2,
24143 Kiel, Germany*

Point-of-care screening of multiple molecular biomarkers from a single sample is an essential next step in early disease diagnosis as well as infectious disease identification. Here, two approaches for optical multiplex biomarker detection are discussed. The first approach is based on fluorescence detection of fluorophore-labelled biomarkers. We develop a miniaturized optical detection system based on integration of organic light emitting diodes (OLEDs) and organic photodetectors (OPDs) on flexible substrates [1,2]. The second approach targets label-free detection of biomarkers based on nanostructured surfaces and specific surface functionalization. We investigate the use of photonic crystal waveguides near waveguide cut off [3]. We discuss the fabrication of design variations of nanostructured waveguides based on a combined process of nanoimprint lithography and ion-beam etching [4]. Finally, we demonstrate a new photonic-crystal readout scheme based on multi-pinhole interferometry [5]. The advantages and challenges of the two approaches are compared. The fluorescence-based approach has the potential for a fully-integrated stand-alone test chip, but at the cost of a more complex and more expensive chip. The label-free approach is based on the combination of a simple microfluidic chip with a separate readout system. This reduces the cost per test, but requires the reusable readout system.

Keywords: *photonic crystal, biosensor, organic optoelectronics.*

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Capability of ACQUANDAS fabrication technology for minimal-invasive implants

Rodrigo Lima de Miranda, Christoph Bechtold

ACQUANDAS GmbH, Kiel, Germany

Today, most Nitinol implants or instruments for minimal-invasive procedures are manufactured by a combination of conventional metal fabrication steps, e.g. melting, extrusion, cold working, etc., and are subsequently structured by high accuracy laser cutting. This combination has been proven to be very successful; however, there are limitations to this fabrication route, e.g. in respect to the fabrication of more complex geometries, miniaturization, the combination of different materials or the integration of further functionality.

ACQUANDAS fabrication technology uses a series of microsystem technology processes, which allows producing Nitinol components with high resolution and excellent mechanical properties, unmatched freedom of design and the possibility to add bio-electrical features.

The capabilities of ACQUANDAS technology will be presented, as well as prototypes for specific applications within the endovascular field.

Microfluidic artificial eye with AI for COVID-19 testing

Dagmar Schneider¹, Tobias Lange, Michele Jamrozik²

¹nandatec GmbH, IZET Innovation Center, Fraunhoferstr. 3, 25524 Itzehoe, Germany

Replacing the replacement model is an ongoing topic with regard to regulatory aspects and faster test and drug development especially during the COVID-19 pandemic. High costs of animal laboratories, ethical issues but also low transferability of the results to humans are obstacles during test and drug developments.

Allergies and recurrent conjunctivitis are known complications associated with contact lenses. Manufacturers try to overcome this by developing antimicrobial nanomaterial surface modifications for contact lenses [1]. Although challenging, the extension of artificial intelligence to microfluidic models could provide a new powerful tool also with regard to possible diagnosis and therapy of infectious diseases like SARS-CoV-2. Therefore a microfluidic artificial eye with stem cell response monitoring is established [2]. Based on patient data the human cornea is simulated by an artificial intelligence. Different shapes, sizes or corneal variations are integrated in the following 3D-printing step. Within the microfluidic eye model early inflammatory cytokine release, pathogen concentration and MAP-kinase activation is analysed via quick test. First results are determined after 4 hours. For longer experiments a stand-alone cell box is established for easy, cost-effective use in laboratories without an established cell culture. The developed eye model enables enhanced results and is very likely to provide earlier diagnosis and treatment of ophthalmologic diseases.

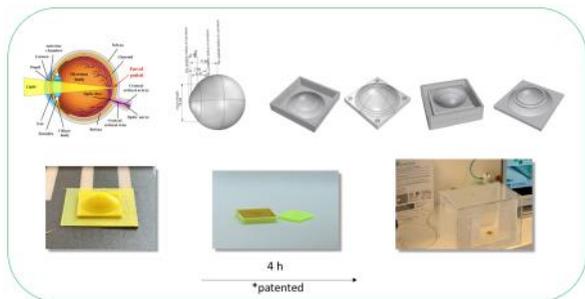


Fig. 1. Digitalisation in Ophthalmology: from computer simulations of patient data to 3D-printed artificial eye for inflammation and allergy testing of nanomaterial-modified contact lenses in 4 hours.

Keywords: animal replacement model, artificial eye, artificial intelligence infectious disease, allergy, stem cells, nano-surface modifications

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Detecting of Aerosols with SARS-CoV-2 Viruses

Udo Riss

DRE-Dr. Riss Ellipsometerbau GmbH, Feldstr. 14, 23909 Ratzeburg, Germany

Aerosols with SARS-CoV-2 viruses have a shell of surfactants (surface active agents). The production of aerosols is started after an infection of the lung. We measured in a proof of principle study at Gesundheitsamt Berlin-Neukölln the quantity of aerosols on a chip area of about 16 mm² and found virus quantities between 0 and 1 million viruses for the invited contact persons. From this we calculated the total virus load per breathe out (0.5 liter) to be between 0 and 122 million viruses.

The technology we used is called Terahertz phase coupling detection. Basis for the technology is a semiconductor chip which is covered with monoclonal anti- corona antibodies. On top of this antibody layer we created a “virtual antigen”, that is catching SARS-CoV-2 viruses in aerosols and brings them to the real antibodies. Then we are measuring the layer thickness produced by the viruses. Because of optical diffraction we always measure thicknesses that are thinner than a real virus, in our case thicknesses between 10 fm und 100 pm. Fig. 1 shows the prototype-detector.

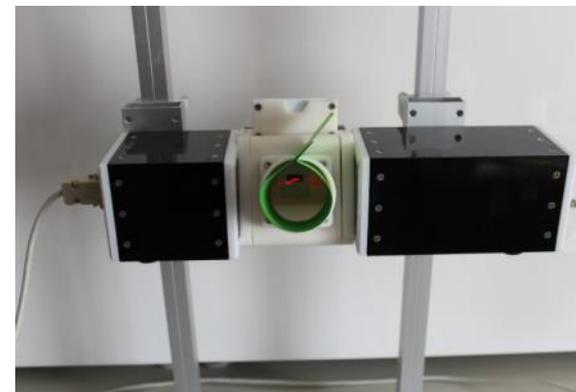


Fig. 1. Terahertz Phase Coupling Detector (prototype)

In contrast to other Terahertz - technologies for virus detection like time domain terahertz spectroscopy we are working with longitudinal THZ-waves instead of transversal THZ waves. This increases sensitivity and specificity significantly by several decades. The measurement time is only some seconds.

The technology is applied for patent.

Keywords: Terahertz phase coupling, virtual antigen.

Synthesis, Properties and Applications of Nanocrystals in Materials and Life Sciences

Horst Weller

Universität Hamburg, Institut für Physikalische Chemie &

Fraunhofer Zentrum für Angewandte Nanotechnologie

Although colloidal nanocrystals of many different materials can be synthesized in high quality in respect of size, shape and crystallinity, our understanding of their formation and the involved chemical reactions is still rather poor. We will present detailed studies on nucleation and growth as well as ion exchange processes in nanocrystals. These include mass spectrometric, optical, electron microscopic and x-ray synchrotron experiments.

Almost all applications of nanocrystals require the control of surface properties in respect of solubility, miscibility, biocompatibility, passivation of surface states as well as electronic and magnetic interaction with the environment. We will show various examples for ligand exchange and encapsulation of quantum dots, plasmonic and magnetic nanocrystals and will report on applications as high-performance ceramics, for display and lighting, electrocatalysis as well as for biolabeling and drug delivery.

CheckNano: nanoparticle detection and sizing

Jacek Fiutowski¹, Prince Gupta¹, Ayoub Laghrissi¹, Jost Adam², Jonas Beermann³, Torgom Yezekan³, Holger Rehmann⁴, Antje Labes⁴, Horst-Günter Rubahn¹

¹Mads Clausen Institute (MCI), NanoSYD, SDU, Alsion 2, Sønderborg, Denmark

²Centre for Industrial Mechanics (CIM), SDU, Alsion 2, Sønderborg, Denmark

³Center for Nano Optics, MCI, SDU, Campusvej 55, Odense, Denmark

⁴Flensburg University of Applied Sciences, Department Energy and Biotechnology, Kanzleistraße 91-93, Flensburg, Germany

Today, nanoparticles are used in various applications to radically improve products, from the cosmetics and food industries to medical technology and pharmaceuticals. However, nanoparticles may also end up taking their toll on our health by penetrating cell barriers. Detailed studies of recent years for silver nanoparticles have identified the underlying mechanism and determined this critical size. Knowledge of the size and shape of nanoparticles is therefore essential for their safe application and forms the basis of an innovative fast test in this project.

The project "CheckNano - Nanosafety Fast Test" brings together partners from Germany and Denmark to develop a prototype for a fast test to identify potentially toxic nanoparticles. The project combines interdisciplinary competencies from nanotechnology (characterization and structuring), biotechnology, nutrition, and sensor technology in the German-Danish border region. Nano-filters and microfilters pre-select the particles, followed by an exact optical sizing and fine filtration. The methods selected are aimed at silver nanoparticles as model particles as they are the most commonly used nanoparticles in consumer products due to their antibacterial properties. However, little is known about the behaviour and modification of such particles in food. Since silver nanoparticles can be synthesized and physically characterized in defined quantities, they are well suited to optimize the fast test for particles of different sizes, shapes and surfaces and to investigate the influence of real media as they occur in foodstuffs.

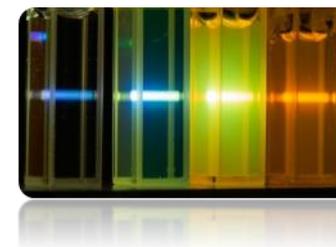


Fig. 1. Ag Silver nanoparticles (50 – 200 nm) illuminated by a white light beam.

Keywords: nanoparticles, detection, assembly, spectroscopy

Fully Automatic Colloid Maker: Nanoparticles on Demand

Tobias Bessel, Stephan Barcikowski, Friedrich Waag

Technical Chemistry I, Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg, Universitaetsstrasse 7, 45141 Essen, Germany

Nanometer-scaled objects have fascinated scientists for decades. In the transition region from molecules to solids, interesting physical properties emerge like surface plasmon resonance or single-domain magnetism. Besides extended physical understanding of states of matter, such phenomena enable advanced applications in the fields of analytics and electronics. The specific nanosize itself that also applies to many biological structures already enables the use of nanoparticles in biomedicine, e.g. drug delivery or cell labelling. In addition, the simple geometric effect of the high specific surface area of nanoparticles provides high potential for applications like the heterogeneous catalysis.

Some nanoparticle materials, e.g. noble metals and simple oxides, already made the leap from research to application. R&D now concentrates on complex materials, such as alloy nanoparticles, and on alternative synthesis methods that make such complex materials easier and “greener” to access. This is necessary to ensure the transfer from research to application in the future.

Our compact and fully automatic machine (Fig. 1a) produces colloidal nanoparticles quickly, easily and reliably. Different nanoparticle materials can be selected by simply replacing a capsule (Fig 1b). Besides common materials, the machine can process practically any solid material into nanoparticles, including complex alloys and ceramics, without additional effort. This is made possible by the method of laser synthesis of colloids [1]. It also allows the use of various dispersants and stabilizers in the synthesis offering an almost infinite range of colloids to explore. The absence of ligands and reducing agents makes the synthesis particularly sustainable and thus makes our machine the preferred source for colloidal nanoparticles in a “green” future.

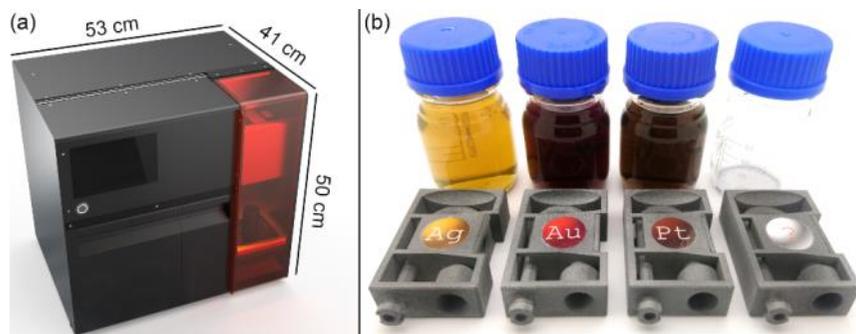


Fig. 1. (a) Fully automatic colloid maker and (b) exemplary colloids and related capsules.

Keywords: suspensions.

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Tetrapods based Smart Materials for Advanced Technologies

Yogendra Kumar Mishra

Smart Materials Group, University of Southern Denmark, Alsion 2, 6400 Sønderborg, Denmark

Considering the size dependent utilization complexities of nanoscopic dimensions towards real applications, the focus of nanomaterials community is merging to three-dimensional (3D) form of materials which are built out interconnected nanostructures [1]. This talk will briefly introduce the importance of complex shaped nanostructures towards smart 3D nanomaterials structuring. A simple flame based single step approach was developed for synthesizing zinc oxide tetrapods which demonstrated many applications in different technologies. These tetrapods have been used as building blocks to construct highly porous interconnected 3D nanonetworks in form of flexible ceramics which offer further new application avenues. Additionally, these 3D networks have been utilized as sacrificial templates to develop hollow tetrapodal 3D networks from almost any desired material, carbons, nitrides, oxides, polymers, hydrogels, etc. The sacrificial template-based strategy offers new and unique opportunities in the direction of 3D nanomaterials engineering and accordingly advanced technological applications. Some examples of 3D nanomaterials engineering will be demonstrated alongwith their applications [1-10]. The scopes of 3D nanostructuring based smart materials in sensing, optoelectronics, energy, and biomedical engineering will be briefly highlighted in the talk.

Keywords: Smart materials, Tetrapods, Hybrid nanomaterials, Advanced Technologies

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Business Development Forum: From Bright Ideas to Business

Introduction to the forum

Company Pitches

NanoCuvette™ – From cleanroom prototypes to roll-out in 22 countries with Avantor/VWR, *Emil Højlund-Nilesen*

Co-development and commercialization of Freshness and Expiration date sensor for meat and fish, *Jeanette Hvam*

Self-Assembled Metasurfaces for Functional Nanophotonics, *Tomas Tamulevičius*

Atomic Layer Nanoprinting, *Maksym Plakhotnyuk*

Business evolution: from obstacles to support



PowderMEMS - A novel fabrication technology for innovative microsystems

Björn Gojdka

Fraunhofer Institute for Silicon Technology ISIT, Fraunhoferstrasse 1, 25524 Itzehoe, Germany

At ISIT, an innovative technological process has been developed that allows the integration of almost any material on planar substrates in order to form three-dimensional functional microstructures. The technology is based on the agglomeration of μm -sized powder (particles) by nanometer-thin layers formed with atomic layer deposition (ALD). Similar to the fabrication of ceramic materials, a mold, in this case microcavities in a silicon substrate, is first filled with loose powder. However, instead of sintering the particles under high pressure at 800°C to 1400°C , the substrate with the loose powder is subjected to an ALD process at much lower temperatures, Fig. 1.

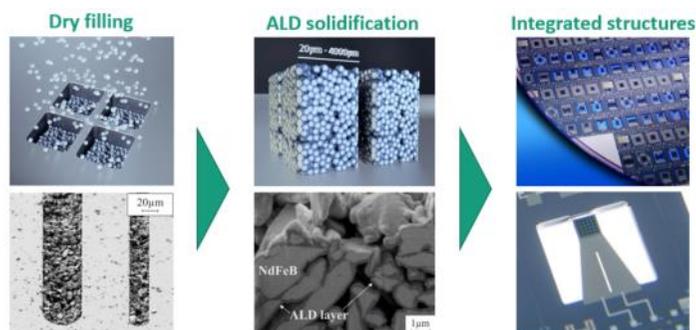


Fig. 1. Novel PowderMEMS fabrication process for wafer-level integrated 3D-microstructures.

Thanks to the extreme penetration depth of the ALD layer that forms, the particles are bonded together over the entire depth of the microcavities (up to $700\ \mu\text{m}$) by a layer only $75\ \text{nm}$ thick to form firmly connected, porous 3D bodies. These are shrinkage-free, mechanically stable and thermally resistant. Structure dimensions between $50\ \mu\text{m}$ and several mm can be realized with high precision. Thanks to the almost perfect encapsulation by the ALD layer, the individual particles within a porous 3D body are excellently protected from external influences. Substrates with embedded porous 3D bodies survive standard MEMS and IC manufacturing processes up to 400°C without damage, so that extensive post-processing in a clean room is possible. The potential applications are extraordinarily diverse. Applications considered so far include integrated permanent magnets made from NdFeB powder. Strong magnetic fields in a very small space are of interest for many MEMS actuators and sensors. The achieved porosity of the structures is of interest for microfluidic applications.

Keywords: Atomic layer deposition, functional microstructures, wafer-level integration

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IR photodetector based on the GaAs nanowire

Eduard Monaico¹, Elena Monaico¹, Veaceslav Ursaki^{1,2}, Ion Tiginyanu^{1,2}

¹National Center for Materials Study and Testing, Technical University of Moldova, Chisinau MD-2004, Republic of Moldova

²Academy of Sciences of Moldova, Chisinau MD-2001, Republic of Moldova

Herein, we report the manufacture of IR photodetector based on GaAs nanowire with good sensitivity and dynamic characteristics prepared by a cost-effective electrochemical etching of GaAs wafer, which does not require sophisticated and expensive equipment [1,2]. The electrochemical etching being performed at room temperature in 1M HNO_3 electrolyte. A special design of contacts was applied via laser beam lithography on selected nanowires. The deposition of Cr/Au ohmic contacts at the ends of the GaAs nanowire ensures the operation of the photodetector in photoconductor mode. The analysis of the photocurrent build-up and relaxation for a photodetector produced on nanowires with different diameters shows that the measured photoresponse at the voltage of 5V increases from $50\ \text{mA/W}$ to $100\ \text{mA/W}$ with the increase of the nanowire diameter from $200\ \text{nm}$ to $400\ \text{nm}$. It should be noted that, because the photodetector operates in the photoconductor mode, the photocurrent increases linearly with increasing polarization. Thus, at a polarization voltage of 15V the photoresponse is higher than $100\ \text{mA/W}$ for all three manufactured photodetectors.

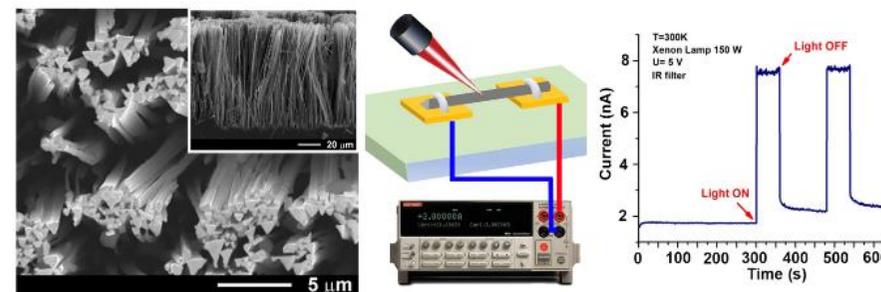


Fig. 1. SEM image in cross-section of GaAs nanowires. Schematic representation of elaborated photodetector. Photocurrent build-up and relaxation of the photodetector measured for an IR illumination density of $800\ \text{mW/cm}^2$.

Keywords: porous GaAs; semiconductor nanowires; photocurrent; anodization.

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Triboelectric energy harvester for scavenging mechanical energy from automobile pedals

Pramila Viswanathan^a, Prabavathi Munirathinam^b, Arunkumar Chandrasekhar^c

^{a, b, c} Nano sensors and Nano energy Lab, Department of Sensors and Biomedical Technology, School of Electronics Engineering, Vellore Institute of Technology, Vellore-632014, Tamilnadu, India

Recent days energy crisis is the major issue due to the oblivious usage of the resources. Most of the time we waste the reliable energy rather than utilizing them efficiently. Triboelectric Nano generator is an energy harvesting device that converts the external Physical motion (mechanical energy) into electric energy by the coupled effect of Triboelectric and Electrostatic induction [1]. This paper discusses about the effective way of exploiting mechanical energy generated from the car's Clutch, brake and Accelerator motion. The TENG will be fixed on the clutch, brake and accelerator which is often pressed by the driver, and electrical energy is harvested efficiently from this physical motion. This energy can further be utilized for self-powered applications [2] such as sleep alert and light up the smart toy which is fixed on car dashboard. Furthermore, this device exhibits long term stability, durability and it's reliable. This device has a capacity to light up a greater number of LEDs [3][4].

Keywords: triboelectric, electrostatic induction, energy harvester, self-powered, stable.

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Influence of the Deposition Duration on the Properties of Vertical Graphene Nanosheets Synthesized by Microwave Plasma Enhanced Chemical Vapour Deposition Technique

Erika Rajackaitė¹, Domantas Peckus¹, Rimantas Gudaitis¹,

Tomas Tamulevičius^{1,2}, Darūnas Međkinis¹,

Sigitas Tamulevičius^{1,2}

¹Institute of Materials Science of Kaunas University of Technology, K. Baršausko St. 59, LT-51423 Kaunas, Lithuania

²Department of Physics, Kaunas University of Technology, Studentø St. 50, LT-51368 Kaunas, Lithuania

Worldwide research attempts on graphene have been implemented for seventeen years with significant breakthroughs and still continue aiming for commercialization at large scales [1,2]. In our studies, vertical graphene nanosheets (VGN) of various heights and densities were synthesised on fused silica substrates by microwave plasma enhanced chemical vapour deposition technique at different growth durations to follow the evolution of VGN properties on the deposition time. The methodology to evaluate the quality of VGN layers with transient absorption spectroscopy (TAS) was elaborated and compared with the results of scanning electron microscopy (**Fig. 1 a**), atomic force microscopy, Raman scattering spectroscopy. A clear correlation between the defectiveness and disorder of VGN layers defined by Raman scattering spectroscopy (**Fig. 1 b**) and the TAS results (**Fig. 1 c**) illustrates that TAS ultrafast charge carrier relaxation dynamics can give meaningful information about the quality of VGN.

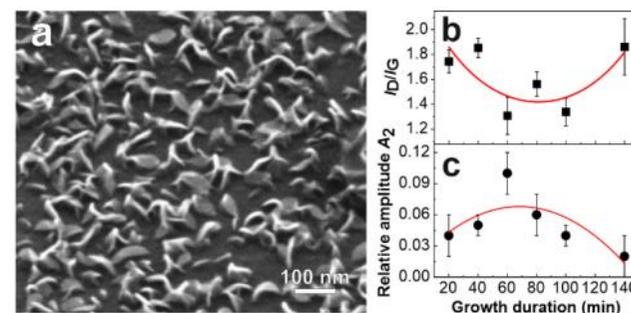


Fig. 1. a) SEM micrograph of VGN grown for 100 min, b) I_D/I_G and c) relative amplitude A_2 dependences on the growth duration.

Keywords: vertical graphene nanosheets, microwave plasma-enhanced chemical vapour deposition, defectiveness of graphene, transient absorption spectroscopy, Raman scattering spectroscopy.

References:

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Impact of the optical effects on the DUV exposition on planar and structured substrates

Agnieszka Zawadzka¹, Regina Paszkiewicz

Faculty of Microsystem Electronics and Photonics, Wrocław University of Science Technology, Janiszewskiego 11/17 50-370 Wrocław, Poland

¹e-mail: agnieszka.zawadzka@pwr.edu.pl

The novel micro- and nanoelectronics technology involves fabrication of structures on non-planar surfaces to increase the spatial density of elements. For optical lithography, that is the most popular lithographic technique, it is a significant challenge.

The aim of this work is to demonstrate the differences in the DUV exposition process for planar and structured substrates. The electric field intensity distribution in the resist layer during the photolithography exposition for different shapes of the substrate will be analysed. The influence of substrate shape on the optical effects like reflection, diffraction, and interference on the shape of the exposed area was studied. The simulations, based on Maxwell's equations were performed using the RF module of COMSOL Multiphysics software. Additionally, the experimental and numerical results of the impact of slits width in the mask on the final profile of the resist structures will be shown and discussed.

Keywords: diffraction, electric field distribution, lithography simulations, reflection, UV exposition.

Acknowledgements:

This work was co-financed by the National Centre for Research and Development grants TECHMATSTRATEG No.1/346922/4/NCBR/2017, Polish National Agency for Academic Exchange under the contract PPN/BIL/2018/1/00137 and Wrocław University of Science and Technology subsidy. This work was accomplished thanks to the product indicators and result indicators achieved within the projects co-financed by the European Union within the European Regional Development Fund, through a grant from the Innovative Economy (POIG.01.01.02-00-008/08-05) and by the National Centre for Research and Development through the Applied Research Program Grant No. 178782 and Grant LIDER No. 027/533/L-5/13/NCBR/20141.

Cation Exchange Strategy for the Synthesis of Symmetry-broken Lateral Heterostructure Nanocrystals

Ranit Ram^{1*}, Rajeev Kumar Rai¹, Ahin Roy¹, N. Ravishankar¹

¹Materials Research Centre, Indian Institute of Science, Bangalore, India, 560012, chemistry1729@gmail.com

Synthesis and applications of colloidal nanocrystals have played a pivotal role in both fundamental research and in technological applications over the last few decades.¹ The morphology control of such nanomaterials is necessary to tailor their optical, electronic, magnetic and catalytic properties.¹ The direct synthesis of symmetry-broken nanocrystal (NC) and their alloys or heterostructures with the compositional control along with their uniform shape and size is often complex. Herein, we have demonstrated a post-synthetic strategy for the synthesis of symmetry-broken nanocrystal and engineering the corresponding lateral heterostructures using Cu_{2-x}Se nanosheets as template nanocrystal.

Cu_{2-x}Se nanosheets has been used as a starting template to obtain the metal selenide heterostructures via cation exchange (CE) at room temperature. Cu_{2-x}Se nanosheets has been synthesized via a hydrothermal method.² Next, partial CE has been performed where Cu_{2-x}Se sheets are the parent crystals and Hg²⁺ are the guest cations. This post-synthetic reaction had performed for 6h. to 18h. of reaction time using Cu_{2-x}Se/Hg²⁺ 2:1 molar concentration. The TEM study for different reaction time of Cu_{2-x}Se nanosheets have revealed that the Cu_{2-x}Se nanosheets has formed through the formation of CuO and Cu₂O as intermediate products.

From STEM-EDS, it has been revealed that for 12 h. of cation exchange reaction, it forms Cu_{2-x}Se/HgSe Janus-like heterostructure. While, after 18 h. reaction, it forms Cu_{2-x}Se@HgSe core@shell architecture. It can also be concluded that the cation exchange has been taken place from edges of the Cu_{2-x}Se nanosheet to the center of the sheets.

Keywords: nanocrystal, cation exchange, heterostructure.

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Sensitivity and Stability of Gas Sensor Based on SnO₂ Nanowires Decorated with Ga₂O₃

Maciej Krawczyk¹, Patrycja Suchorska-Woźniak¹, Ryszard Korbutowicz¹, Helena Teterycz¹

¹Faculty of Microsystem Electronics and Photonics, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Wrocław University of Science and Technology, Poland

Extremely large specific surface area of nanowires enables development of gas sensitive structures which consist of atoms present almost exclusively on their surface. Therefore, the most of the chemically active sites of such structures are exposed to the surrounding atmosphere, and hence the use of nanowires is expected to improve parameters of chemoresistive gas sensors, such as sensitivity, or response and recovery times [1].

The focus of recent scientific papers on chemoresistive gas sensors is mainly on improvement of the sensitivity, and reduction of the power consumption by lowering the operation temperature [2,3]. However, it seems that the problem of stability and repeatability of such sensors is marginalized. The aim of this poster is to address this issue and to present preliminary results of comparison of sensitivity and stability of a nanowires-based sensor at the operation temperatures from 150 °C to 650 °C.

In this research, metal oxide nanowires were synthesized on silicon substrate via thermal method at atmospheric pressure using Sn-Ga alloy containing 1% wt. of Ga as the precursor. The nanowires were transferred to the sensor Au electrodes using PVP-containing glue and a "lift-off" method. The sensor stability, sensitivity, and repeatability of its response to and atmosphere containing 100 ppm of isopropanol was investigated over several consecutive measurements. The sensor was heated using a Pt heater incorporated into the sensor structure and the measurements were done at a constant operation temperature.

It was found out that the sensor peak sensitivity to isopropanol is, approximately, at 350 °C, however, other analysed parameters, namely stability, repeatability of the response, and response and recovery times are greatly improved at temperature above, approximately, 500 °C.

Keywords: chemical gas sensors, nanomaterials, nanowires, metal oxide, thermal synthesis

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Medical Microtechnology: a new Danish-German Master-Program

Klein, Stephan¹, Rubahn, Horst-Günter², Buzug, Thorsten³, Kern, Benjamin¹, Venker, Silke¹, Kjelstrup-Hansen, Jakob², Gräfe, Ksenija³

¹Technische Hochschule Lübeck, Moenkhofer Weg 239, 23562 Luebeck, Germany

²Syddansk Universitet, Aلسion 2, 6400 Sonderborg, Denmark

³Universität zu Lübeck, Ratzeburger Allee 160, 23562 Luebeck, Germany

A new Master-Program for graduate students has been developed and introduced by the three universities Technische Hochschule Lübeck, Universität zu Lübeck (both located in Lübeck, Germany) and the University of Southern Denmark, with its campus in Sønderborg, Denmark.

The program (see Fig. 1) brings together competences in the field of micro- and nanotechnologies (Sønderborg) as well as medical technology (Lübeck) and is offered for students of all nations. First enrolment will be possible in fall 2021. Information can be found at [1]. The students stay for one semester in each of the two cities. After the 1st year, they work on a 24 ECTS-points research project which has to be presented at Lübeck's student's conference [2]. The program is finished after a thesis-project of 26 points and a final oral examination.

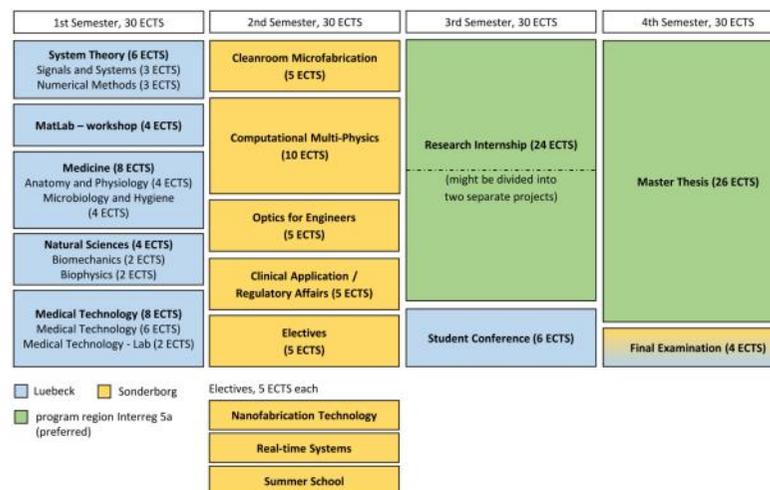


Fig. 1. Structure of the program Medical Microtechnology

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Acknowledgment

The program development is part of the MicroMedTech project that is financed by Interreg Deutschland-Danmark with funds from European Regional Development Fund.

Porous Anodized Aluminium Oxide Thickness Determination during Anodization Process

Audrinė Jurkevičiūtė^{1,2}, Raimonds Poplauskis¹, Aleksandrs Dutovs¹, Juris Prikulis¹

¹Institute of Chemical Physics, University of Latvia, Jelgavas st. 1, LV-1004, Riga, Latvia

²Institute of Materials Science of Kaunas University of Technology, K. Baršausko st. 59, LT-51423, Kaunas, Lithuania

ausrine.jurkeviciute@lu.lv

Porous anodized aluminium oxide (PAAO) is a spontaneously periodically ordered material with pores fabricated using electrochemical methods. To obtain a better uniformity of PAAO, the anodization is carried out in two steps: first anodization is longer, while second anodization depends on the desired thickness of the final structure.

In this work, aluminium substrate was anodized in oxalic acid for 1 hour. Every 2 s reflection spectra were recorded using ThorLabs SLS201L/M light source, Ocean Optics USB4000 spectrometer, and Ocean Optics *SpectraSuite* software. Spectra analysis and fitting was carried out using *Octave* freeware and propagation and matching matrices formalism.

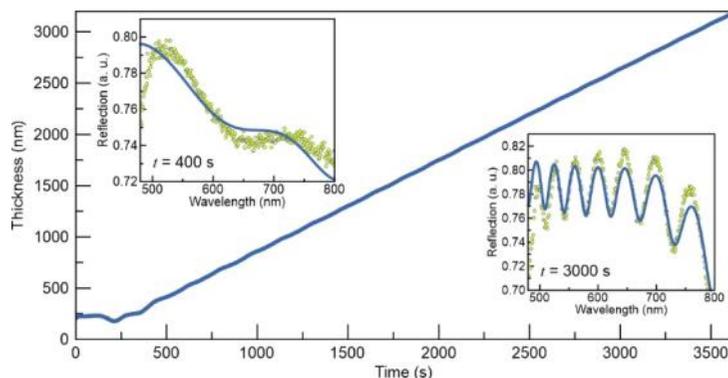


Fig. 1. PAAO thickness dependence on anodization time. Insets show the measured and fitted reflection spectra at 400 s and 3000 s.

From the linear part of thickness dependence on anodization time graph (Fig. 1), PAAO growth rate of around 0.9 nm/s was obtained. Knowing this rate helps to predict the required duration for second anodization.

Keywords: PAAO, thickness, reflection, fitting, anodization.

Acknowledgements

Research is supported by European Regional Development Fund postdoctoral project "Patterned hybrid multilayer films for optical sensors" (No. 1.1.1.2/VIAA/4/20/615).

Highly enhanced electron field emission and magnetic properties for FM metal embedded thin SiO₂ matrix:

Role of electronic structure and other parameters

Santanu Ghosh, Debalaya Sarker, Hardeep Kumar, Saswata Bhattacharya, Pankaj Srivastava

Department of Physics, Indian Institute of Technology Delhi, New Delhi-16, India

We present here following important functionalities of magnetic nanoparticle (Ni and FeCo) embedded nanoparticles (NPs) in thin silica matrix: (i) field emission (FE), (ii) anomalous Hall effect & tunneling magnetoresistance (TMR) (iii) perpendicular magnetic anisotropy and (iii) exchange bias. FE measurements were carried out in an indigenously developed high vacuum diode set up. The salient results obtained can be summarized as (i) appreciable increase in FE current density with high mechanical durability in metal Ni and FeCo nanoparticles embedded in silica: promising planar emitter for future flat-displays. The enhanced FE characteristics of these planar emitters are understood from a combined experimental and first-principles based calculations study. In the second part we report a novel way to trigger exchange bias and TMR in the FeCo-SiO₂ and nanocomposite films under intense electronic excitation. An anomalous behaviour in magnetic anisotropy and Hall effect in Ni-SiO₂ nanocomposite films are also reported and explained on the basis of DFT calculation.

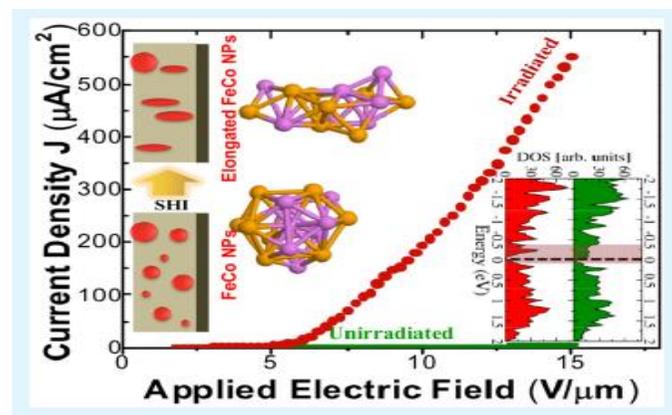


Fig. 1. Highly enhanced field emission current density in elongated FeCo NPs embedded in thin silica matrix

Keywords: Embedded metal nanoparticles, Field emission, Magnetic anisotropy, Exchange bias, Anomalous Hall effect

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Computational Studies for Understanding Nanoparticles Interactions with Lipid Membrane Systems

Sridhar Hariharaputran¹

¹ Faculty of Mathematics and Natural Sciences, Department of Physics, Kaunas University of Technology, Studentø str. 50, LT-51368 Kaunas, Lithuania

The work focuses on the interaction of TiO₂ nanoparticles with heterogeneous lipid membrane systems based on the studies of antimicrobial effect of TiO₂ nanoparticles on gram-negative bacteria and based on our ongoing study, results and previously presented, published studies. TiO₂ due its nature is an attractive antimicrobial compound [1,2,3,4,5,6]. Work involves computational methods to detect the molecular properties using VeloxChem [7], Whirlpool [8] for generating input for Dalton program for QM/MM calculations and using CHARMM-GUI web-based platform, AMBER, GROMACS, GAUSSIAN software packages. Earlier we designed different membrane systems and performed molecular dynamics simulations to analyse the interactions of probe with the membrane. And as predicted probe inserted into the membrane and presented good results. And here we will present the results generated based on the interaction studies of TiO₂ nano particles.

Keywords: TiO₂, nanoparticles, MD simulations, bacteria, modelling, QM/MM

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Fabrication of the plasmonic structure based on “bottle-neck” free porous silicon with embedded silver nanoparticles

Nadzeya Khinevich^{1,3*}, Mindaugas Juodėnas¹, Asta Tamuleviėiene^{1,2}, Hanna Bandarenka^{3,4} and Sigitas Tamuleviėius^{1,2}

¹Institute of Materials Science, Kaunas University of Technology, 59 K. Baršausko St., 51423 Kaunas, Lithuania

²Department of Physics, Kaunas University of Technology, 50 Studentų St., 51368 Kaunas, Lithuania

³Applied Plasmonics Laboratory, Belarusian State University of Informatics and Radioelectronics, 6 Brovka St., 220013 Minsk, Belarus

⁴The Polytechnic School, Ira A. Fulton Schools of Engineering, Arizona State University, 6075 S. Innovation Way West, Mesa, 85212, AZ, USA

Plasmonic nanoparticle arrays and nanoantennas have shown their ability to strongly concentrate and increase the intensity of local electromagnetic fields in the visible light range [1]. Metal–semiconductor interacting systems that enhance the optical response of their individual components have shown their potential application in linear and nonlinear optical devices [2].

The “bottleneck”-free porous silicon (PS) substrate has a great potential as a template, which provides a precise definition of shape, size, and spatial location of different nanostructures for the development of radically new metamaterials such as high-sensitive SERS-active porous silicon-based substrates, high-efficiency solar cells, ultrafast optical switches, etc.

In our work, we present a method of the “bottleneck”-free PS substrate fabrication for silver nanoparticle array formation. The PS morphology and optical properties before and after the pore opening process, including the determination of thickness, pore size, and pore density of PS layers, its surface wettability, and reflectivity have been studied. The PS samples were fabricated by electrochemical anodization of monocrystalline silicon wafer in 5 – 20 wt.% hydrofluoric acid (HF) solution at a current density in the range of 20 – 200 mA/cm² and followed by the pore opening process that was done by reactive ion etching (RIE). The RIE-etched PS sample formed at 10 wt.% HF solution was shown to be a favourable template for the capillary assisted particle assembly (CAPA) deposition of the highly ordered array of the 62 nm size silver nanoparticles divided by silicon walls with the filling factor of ~85%. The final structure is expected to possess prominent plasmonic properties (an extremely intensive electromagnetic field in the spots of silver nanoparticle and silicon wall contact, due to plasmonic coupling between metallic nanoparticles and semiconductor) supported by the modelling results and present the object of our future study.

Keywords: porous silicon, nanoparticle array, electrochemical etching, parasitic layer, reactive ion etching.

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Nanoscale gradient copolymer thin films enabled by initiated chemical vapor deposition (iCVD)

Stefan Schröder¹, Alexander M. Hinz^{1,2}, Oleksandr Polonskyi^{1,3}, Thomas Strunskus¹, Franz Faupel¹

¹Kiel University, Institute for Materials Science, Kaiserstraße 2, 24143 Kiel, Germany

²now at: Fraunhofer FEP, Winterbergstraße 28, 01277 Dresden, Germany

³now at: UCSB, Santa Barbara CA 93106-5080, USA

Nature is filled with a great variety of organic gradient structures. A possible approach to recreate these materials are gradient copolymers. In this study the deposition of such gradient copolymers as thin films via initiated chemical vapor deposition (iCVD) is reported [1]. Due to its solvent-free nature and mild deposition conditions, the iCVD process enables the fabrication of high-quality polymer thin films on large-area substrates, complex geometries as well as temperature-sensitive substrates [2-4]. Furthermore, it enables the unique combination of comonomers, which typically lack a common solvent. To transfer the gradient copolymer film approach to the lower nanoscale via iCVD, a detailed insight and control of the underlying reaction kinetics are required. This is approached by a novel in-situ quadrupole mass spectrometry (QMS) extension for iCVD with supporting ab initio/density functional theory (DFT) calculations, which was developed in this study [5]. It facilitates a deeper insight into the underlying reaction kinetics and a better control over the vapor phase composition. This enables the deposition of gradient copolymer films at film thicknesses below 30 nm. These nanomaterials exhibit completely new chemical and physical properties that cannot be obtained with current materials. They therefore pave the way for a wide range of new applications or enable improvement of current applications as presented by some examples.

Keywords: *Initiated chemical vapor deposition, Polymer thin films, Gradient copolymers, Mass spectrometry.*

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Deposition of photoswitchable polymer thin films via initiated chemical vapor deposition (iCVD)

Maximilian H. Burk¹, Stefan Schröder¹, Lukas Pott¹, Stefan Rehders¹, Thomas Strunskus¹, Rainer Herges² and Franz Faupel¹

¹Faculty of Engineering, Christian-Albrechts-University, Kaiserstr. 2, 24143 Kiel, Germany

²Otto-Diels-Institute for Organic Chemistry, Christian-Albrechts-University, 24118 Kiel, Germany

Initiated chemical vapor deposition (iCVD) is a solvent-free, cost efficient technique to synthesize highly conformal organic thin films directly from the vapor phase. Due to generally mild reaction conditions the individual functionalities of the used monomers are preserved, enabling the homogeneous coating of almost every kind of substrate without the risk of solvent interference. This allows for example the tailored synthesis of super hydrophobic fluoropolymers with application as electret layers as well as the highly innovative deposition of gradient layers with superb adhesion properties. Moreover, iCVD is highly interesting in the field of biocompatible coatings and controlled drug release, for example in the context of bone tissue engineering where biodegradable substrates are coated with hydrogel thin films. [1-3]

When it comes to the design of novel functional coatings, smart surfaces must be based on the ability to perform controlled, reversible transitions upon application of an external stimulus.

Recently we were first to demonstrate the utilization of a solid chromophoric compound within the iCVD process which lead to the synthesis of photoswitchable co-polymer thin films. [4] Photoswitchable molecules are able to undergo a reversible transition upon illumination with monochromatic light which can lead to a change in absorption, dipole moment or geometry. Due to the underlying nature of iCVD it was possible to combine a highly hydrophilic monomer with a rather hydrophobic diazocine (a bridged azobenzene), without the risk of phase separation or unwanted side reactions. Upon illumination with blue light it was possible to induce photoisomerization of the incorporated photoswitch, leading to a reversible change in color as well as expansion/contraction of the thin film. Using iCVD we show the deposition of photoswitchable patterned coatings on glass, the deposition on flexible substrates and the creation of functional surfaces that can be written on with a conventional blue laser pointer. [5]

Keywords: *Polymers, thin films, chromophores, photoswitching, chemical engineering*

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Optimization of 2D-Like Polydopamine membranes from the Air/Water Interphase: towards morphological and hydrophilic properties improvement

J. Szewczyk¹, M. Pochylski², K. Siuzdak³, D. Aguilar-Ferrer¹, M. Kempniński², I. Iatsunskiy¹, J. Gapiński², E. Coy¹

¹ NanoBioMedical Centre, Adam Mickiewicz University, Wszechnicy Piastowskiej 3, 61-614 Poznań, Poland

² Faculty of Physics, Adam Mickiewicz University, ul. Uniwersytetu Poznańskiego 2, 61-614 Poznań, Poland

³ Centre for Plasma and Laser Engineering, The Szewalski Institute of Fluid-Flow Machinery, Fiszera 14 Str., 80-231 Gdansk, Poland

Polydopamine (PDA) is a relatively new biomimetic polymer that made a sizable impact in various fields, i.e. photocatalysis [1]. PDA can be synthesized by an oxidative polymerization of dopamine hydrochloride induced by alkaline pH. During this process, three types of PDA can be formed and distinguished, among others the membrane at the air/water interphase. Importantly, we recently showed a previously unknown, 2D-like arrangement of these membranes [2]. To this date, there is no systematic and comprehensive study towards production, properties and possible application of such materials. Therefore, in our study, an in-situ experimental spectroscopic reflectometry (SR) setup was designed to investigate this issue and allow further control of the growth of PDA membranes under the influence of differential factors (**Fig.1**). Morphological properties of the final membranes (thickness, roughness) were investigated by Atomic Force Microscopy. Moreover, structural studies have been carried out, namely Raman spectroscopy and X-ray diffraction. The hydrophilic properties were determined with water contact angle measurements. These tests made it possible to optimize the morphological and hydrophilic properties of the membranes.

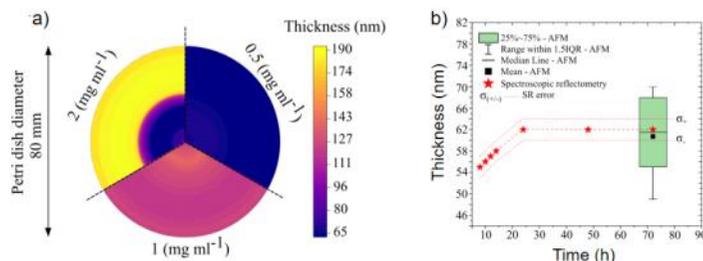


Fig. 1. a) Map of the thickness of the membranes executed by SR method. b) A chart of the thickness growth during polymerization, executed with SR and AFM methods

Keywords: Polydopamine, membranes, optimization, morphology, hydrophilicity

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Improving Photocatalytic Performance of TiO₂ Thin Film by Modification with Metallic and Oxide Nanostructures

Salih Veziroglu, Josiah Shondo, Tim Tjardts, Tamim B. Sarwar,

Thomas Strunskus, Franz Faupel, Oral Cenk Aktas¹

¹Chair for Multicomponent Materials, Institute of Materials Science, Kiel University, 24143 Kiel, Germany

Titanium oxide (TiO₂) is one of the most commonly used photocatalytic materials for various applications such as environmental remediation, water splitting, self-cleaning due to its high activity, low cost, high chemical, and physical stability. However, the photocatalytic activity of TiO₂ is limited by the wide energy of the bandgap, low quantum efficiency, and rapid recombination of photogenerated charge carriers (electrons and holes). During the last decades, numerous approaches, such as tailoring the morphology (nanoparticles, thin film, etc.), combining with metal, noble metal, and metal oxide micro/nanostructures, have been demonstrated to enhance the photocatalytic activity of TiO₂. However, it is still a major challenge to find the best photocatalytic combination for specific applications. Recent studies have been revealed that particle size plays a considerable role in the photocatalytic activity of TiO₂. Reducing the particle size (increasing active surface area) indicates a higher photocatalytic activity. Nevertheless, the use of photocatalytic nanoparticles in continuous flow systems (such as water remediation, water splitting, etc.) has some practical limitations such as reusing and splitting them up from the reaction media. Hence, the use of robust and stable thin film photocatalysts becomes more suitable rather than nanoparticles systems for practical applications. Nevertheless, thin films are restricted by low surface area in contrast to nanoparticles and they show extremely limited photocatalytic activity. Here we present some case studies on enhancing the photocatalytic performance of TiO₂ thin film by modification with metallic [1-3] and oxide [4-5] nanostructures.

Keywords: photocatalysis, thin film, noble metal, metal oxide, nanostructure

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Diagnostics on an atmospheric pressure DC glow microplasma discharge intended for in situ TEM studies

Luka Hansen¹, Niklas Kohlmann², Ulrich Schürmann², Lorenz Kienle², Holger Kersten¹

¹Institute of Experimental and Applied Physics, Kiel University, Germany

²Institute of Materials Science, Kiel University, Germany

One of the most discussed topics in plasma technology is plasma surface interaction due to its relevance for the production and modification of micro- or even nano-structured surfaces. The challenging nature of observing plasma surface processes in real time with nanoscale resolution results in state of the art analysis mostly being limited to the separation of plasma processing and surface analytics. A microplasma cell based on the proof of principle experiments by Tai *et al.* [1] with dimensions suited for TEM integration is being developed, enabling in situ studies of plasma surface interaction in real time.

The two main challenges during the development of the microplasma discharge cell are to generate a stable atmospheric pressure DC glow discharge and to vacuum-proof encapsulate it for in situ operations. While the second task has to be investigated in future extensive leakage testing prior to introducing the cell into the TEM, the generation and operation of a stable DC glow microplasma is already successful.

Conventional (electrical and optical) as well as non-conventional (calorimetry [2]) plasma diagnostics have been applied to the DC glow discharge to ensure its stability and reproducibility and to provide enough insight to understand the observable plasma induced surface changes. The electrical diagnostics show current voltage curves expected for a DC glow discharge with clear differences depending on e.g., working gas or electrode distance. Optical imaging further indicates the stable plasma operation in the glow regime.

Calorimetric measurements using passive thermal probes (PTPs) [3] determine the energy flux from the plasma to the electrode surface. The measured integral energy flux consists of various components, which combination is responsible for the plasma induced surface changes. Depending on the polarity of the to the electrodes applied voltage and the working gas different energy fluxes to the surfaces have been measured. These energy fluxes correspond to the surface changes observed ex situ via SEM and TEM. Furthermore, using a simple model approach enables the determination of effective secondary electron emission (SEE) coefficients.

Keywords: plasma surface interaction, plasma diagnostics, in situ TEM, atmospheric pressure plasma, DC glow discharge

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Investigation of ZnO nanobrushes fabricated by H₂-C₂H₂ plasma etching

Niklas Kohlmann¹, Luka Hansen², Cristian Lupan³, Ulrich Schürmann¹, Armin Reimers¹, Fabian Schütt¹, Rainer Adelung¹, Holger Kersten², Lorenz Kienle¹

¹Institute for Materials Science, Kiel University, Germany

²Institute of Experimental and Applied Physics, Kiel University, Germany

³Center for Nanotechnology and Nanosensors, Department of Microelectronics and Biomedical Engineering, Technical University of Moldova, Moldova

In order to tailor functional materials on the nanoscale to their diverse applications precise control over their properties is necessary. Aside from the atomic structure itself the morphology is of great importance. Morphologies that offer large surface to volume ratios are especially important for catalysis or sensing applications. We report on a plasma etching procedure leading to the formation of ZnO nanobrushes with high surface area to volume ratios (SA:V). The nanobrushes are formed by H₂-C₂H₂ plasma treatment of ZnO microtetrapods [1]. Detailed structural investigations using SEM and TEM reveal the formation of crystalline ZnO nanowire arrays atop tetrapod arms in a preferential etching process. Relative etch rates of 11 nm/min are found for 1% C₂H₂ admixture leading to nanowire lengths of a few 100nm for etching times of 30-60 min, see Fig.1 left. Nanobrushes show an increase in SA:V of up to 13 times compared to pristine ZnO tetrapod arms. Thus, single nanobrush gas sensor devices show excellent H₂ sensing characteristics (Fig.1 right).

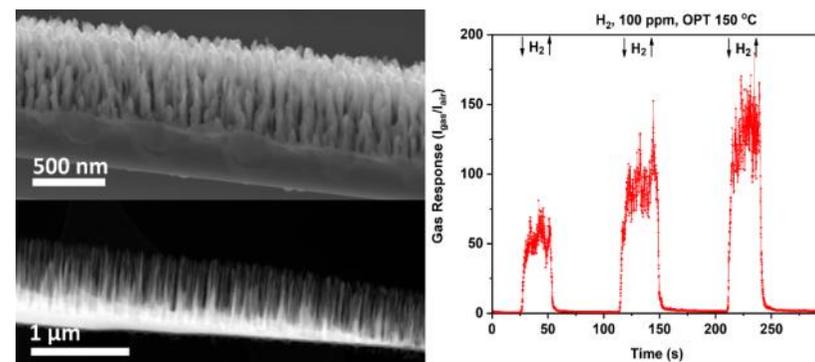


Fig. 1. Left, SEM (top) and TEM (bottom) micrographs of ZnO tetrapod arms after plasma etching with clearly visible nanowire arrays. Right, gas response of a single nanobrush sensor device to 100 ppm hydrogen showing excellent sensitivity as well as response and recovery times.

Keywords: nano-on-micro, nanobrush, plasma, etching, H₂ nanosensor, TEM

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Infrared-spectrometric Monitoring of the Growth and Surface Treatment of a-C:H nanoparticles in a low-pressure plasma

Oguz Han Asnaz¹, Niklas Kohlmann², Hauke Folger¹,
Franko Greiner¹, Jan Benedikt¹

¹ Institute of Experimental and Applied Physics, Kiel University, Germany

² Faculty of Engineering, Kiel University, Germany

Fine control over the particle's surface properties as well as the bulk crystallinity is required in many applications of nanoparticles to adjust their physical, mechanical, electrical, and optical properties. In this contribution, a capacitively coupled low-pressure plasma is used for generation, confinement, and treatment of hydrogenated amorphous carbon (a-C:H) nanoparticles. Particles reach a size of about 500 nm after 90 seconds of growth when using a mixture of argon and acetylene (C₂H₂) at low plasma power. The charged particles are held in the electric field of the plasma sheath and can be confined easily for multiple hours. After their generation, the particles are treated with hydrogen and deuterium to passivate their surface. Both the growth process and the particle treatment is monitored in-situ using Fourier-transform infrared spectroscopy (FTIR) over the course of 45 minutes. A multi-pass setup with 24 passes through the plasma chamber is used to maximize the absorption signal. This measurement allows insight into the chemical bonds on the particle surface as well as in the bulk material. Additionally, using the electrostatic particle extractor system (EPEX) developed in our group [1], particle samples are extracted at multiple moments during the treatment for further SEM analysis with negligible disturbance of the plasma

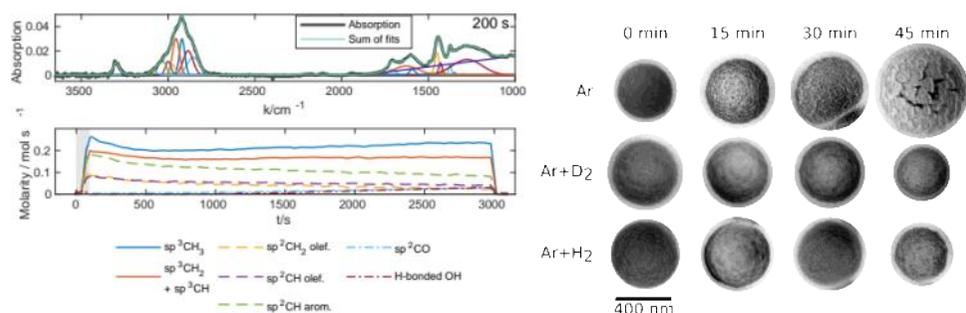


Fig. 1. Left: Decomposition of an infrared absorption spectrum of nanoparticles after 200 s of treatment, analyzed using Gaussian peak fits (top) and the time evolution of these features during the treatment with Ar (bottom). Right: SEM images of collected nanoparticles after 0, 15, 30, and 45 minutes of treatment from left to right.

Keywords: nanoparticles, surface treatment, infrared spectroscopy.

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In situ infrared absorption analysis of silicon nanoparticles generated in atmospheric plasma jets

Maren Dworschak¹, Jessica Ruhnke¹, Niklas Kohlmann², Jan Benedikt¹

¹Institute of Experimental and Applied Physics, Kiel University, Germany

² Faculty of Engineering, Kiel University, Germany

Cold atmospheric plasmas are highly reactive systems and can be used to dissociate precursor gases where reactive species are used for surface treatment or in the generation of nanoparticles and nanostructured thin films. In this contribution, we report on different rf-driven capacitively coupled atmospheric plasma jets with a dielectric on the electrodes. These jets can be operated at large powers and elevated gas temperature. Their differences are in the inner diameter of the plasma channel, electrode geometry (parallel electrode or HelixJet [1]) and in the type of gas injection. A gas mixture of noble gases with silane is used to generate silicon nanoparticles with precise size control from ultra-small (<2 nm) particles up to the range of several tens of nanometres. The particle size is determined with a 1 nm scanning mobility particle sizer (SMPS) and with SEM and TEM measurements. Different reactive gas admixtures are used to modify the surface passivation of the generated nanoparticles. The changes of particle passivation and the silane consumption are monitored by in situ Fourier-transform infrared absorption spectroscopy (FTIR). The measurements of the nanoparticles are performed in reflection mode on a metal-coated silicon wafer that is positively biased to enhance the nanoparticle deposition yield. Additionally, measurements of collected particles performed at different times after the deposition are used to monitor the rate of particle oxidation as a function of changes in reactive gas admixture. Finally, a fly-through annealing step with temperatures up to 1100°C has been integrated between the plasma jets and SMPS or FTIR diagnostics to reach higher crystallinity and first results will be reported here.

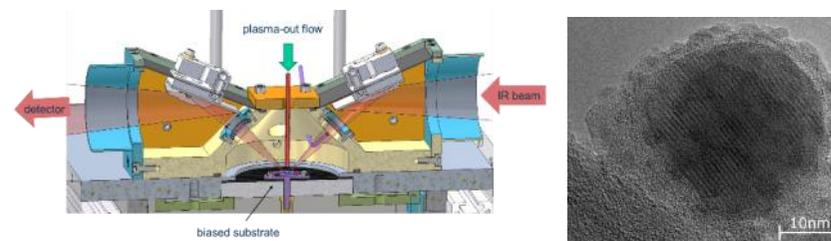


Fig. 1. Left: Set up for in situ Fourier-transform infrared absorption spectroscopy in reflective mode. Right: Crystalline silicon nanoparticle generated after the annealing process.

Keywords: in situ, IR spectroscopy, atmospheric plasmas

References:

1. J. Schäfer et al., *Plasma Process. Polym.* **17** (2020)