



### **Book of Abstracts**

19<sup>th</sup> Joint Vacuum Conference

&

30<sup>th</sup> International Scientific Meeting on Vacuum Science and Technique



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1. Preface

Dear Colleagues,

on behalf of the Croatian Vacuum Society, International Program and Organizing Committees, and the Local Organizing Committee we are pleased to welcome you to Podstrana from the 29<sup>th</sup> of September to the 4<sup>th</sup> of October 2024, to attend

19<sup>th</sup> Joint Vacuum Conference & 30<sup>th</sup> International Scientific Meeting on Vacuum Science and Technique.

The JVC-19 conference is the nineteenth edition of a series of successful biannual symposia organized in Middle-European countries by Vacuum Societies of Austria, Croatia, Czech Republic, Hungary, Slovakia, and Slovenia.

The conference participants present their recent scientific results and achievements in the following fields: surface science, vacuum science and technology, thin films and coatings, nanotechnology, plasma science and technology, electronic materials, surface engineering, and biointerfaces. The scientific program of the conference consisted of 4 plenary lectures, 13 invited lectures, 25 oral contributions, and 35 posters.

We hope that this joint conference will show advancements in the field of vacuum and related emerging technologies, reinforce existing networks of scientific interactions, and stimulate the development of new collaborations.

On behalf of the international program and organizing committees, we thank the plenary and invited speakers for accepting our invitation and all participants for their valuable contributions. The organizers would like to thank the Braavos agency for professional governance of all administrative matters, and the management and staff of the Hotel Le Méridien Lav, for their help in the smooth implementation of the meeting.

We are confident that your participation at the JVC-19 conference will enable further successful organization of JVC conferences and a fruitful and challenging experience for all of us.

Zagreb, September 2024.

We wish you all a pleasant stay in Podstrana! With best regards, Nikša Krstulović and Maja Mičetić Chairs of JVC-19 and 30<sup>th</sup> ISMVST



#### 2. Committees

Conference Chairman: Nikša Krstulović, Institute of Physics, Croatia

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Vice Chairman: Maja Mičetić, Rudjer Boskovic Institute, Croatia

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### 4. Program at a glance

	SUNDAY 29th Sept	MONDAY 30th Sept	TUESDAY 1st Oct	WEDNESDAY 2nd Oct	THURSDAY 3rd Oct	FRIDAY 4th Oct
8:45-9:00		Opening of JVC-19				
		Session 1	Session 4	Session 7	Session 9	
9:00-9:45		Ruzic	Óvári	Asensio	Driscoll	
9:45-10:15		Čada	Vašina	Tkadletz	Štrkalj	Meeting 1
10:15-10:35		Čekada	Gostenčnik	Zaplotnik	Šoltýs	
10:35-10:55		Novák	Dodony	Primc	Mašek	
10:55-11:30		Coffee break	Coffee break	Coffee break	Coffee break	
		Session 2	Session 5	Session 8	Session 10	Meeting 2
11:30-12:00		Csík	Noga	Panjan	Šetina Batič	
12:00-12:20		Drobnič	Száraz	Recek	Kutassi	
12:20-12:40		Hajagos-Nagy	Majkić	Kurtishaj Hamzaj	Roy	
12:40-13:00		Novosel	scia systems	Ukropcová	Zver	Closing of JVC-19
13:00-15:00		Lunch	Lunch	Lunch	Lunch	
		Session 3	Session 6		Session 11	
15:00-15:30		Siffalovic	Paramon		Skoko	
15:30-15:50		Kousal	Čurda		Janů	
15:50-16:10		Đerek	Omerzu	Excursion/City tour	Delač	
16:10-16:40		Hahn	Kun	Split sightseeing	Final remarks &	
16:40-17:10		Coffee break	Coffee break	&	coffee break	
17:10-19:00	Registration	Poster session	Poster session	Conference dinner		
19:00-22:00	Welcome drink & welcome dinner	Dinner	Dinner	at konoba Rudine	Dinner	
	wereome unmer					
	r					JVC-19
	Legend:	Plenary	Invited	Oral	Sponsor	19th Joint Vacuum Conference

### 5. Program

### Sunday, 29<sup>th</sup> Sept

17:00	Registration
19:00	Welcome drink & welcome dinner

#### Monday, 30<sup>th</sup> Sept

8:45	Opening of JVC-19	
	Session 1	(chairman: Alenka Vesel)
9:00	David N. Ruzic, Illinois Plasma Institute, USA Controlling the Energy and Species in PVD: HiPIM Plenary	S with Cathode Reversal,
9:45	Martin Čada, Czech Republic HiPIMS with controlled arc ignition: hybrid plasm hard coatings, <b>Invited</b>	a deposition technique for
10:15	Miha Čekada, Slovenia Property variation of hard coatings deposited in in systems, Oral	ndustrial deposition
10:35	<b>Jozef Novák</b> , Slovakia Focused Ion Beam technology as an excellent too three dimensional GaP nanocone onto Single Mod	
10:55	Coffee break	

	Session 2 (chairman: Maja Mičetić)		
11:30	Attila Csík, Hungary		
11.50	Surface coating layers for sensors and biomedical applications, Invited		
	Matej Drobnič, Slovenia		
12:00	Effect of LCO2 on TiAIN hard coating wear behavior at elevated		
	temperatures, <b>Oral</b>		
	Klára Hajagos-Nagy, Hungary		
12:20	Non-equilibrium solubility of Cu in $\alpha$ -Mn structure – microstructure and		
	growth of α-Mn(Cu) films, <b>Oral</b>		
	Nikolina Novosel, Croatia		
12:40	Complex magnetic and transport phenomena in nanocrystalline gadolinium		
	doped manganite CaMnO3, <b>Oral</b>		



13:00 Lunch break

	Session 3 (chairman: Zoltán Száraz)
15:00	<b>Peter Siffalovic</b> , Slovakia In-Situ Techniques for Perovskite Layer Growth Monitoring: X-ray Scattering and Photoluminescence, <b>Invited</b>
15:30	Jaroslav Kousal, Czech Republic Plasma-Assisted Vapour Thermal Deposition for Controlled Deposition of Nanocomposites, <b>Oral</b>
15:50	<b>Vedran Đerek</b> , Croatia Optically driven thin film OECT electrochemical sensor, <b>Oral</b>
16:10	<b>Rainer Hahn</b> , Austria title: Assessing and enhancing fracture toughness of hexagonal diboride coatings, <b>Invited</b>
16:40	Coffee break
17:10	Poster session 1
19:00	Dinner

#### Tuesday, 1<sup>st</sup> Oct

	Session 4	(chairman: Karel Mašek)
	László Óvári, University Szeged & Extreme Light Infr	astructure ALPS,
9:00	Hungary	
	2D Boron Carbon Nitride structures: from synthesis t	o application, <b>Plenary</b>
9:45	Petr Vašina, Czech Republic	
5.45	Me-B-C coatings - hard and fracture resistant mater	ial, <b>Invited</b>
	Žan Gostenčnik, Slovenia	
10:15	Refractory high-entropy nitride coatings deposited b	y reactive direct current
	magnetron sputtering, <b>Oral</b>	
10:35	Erzsébet Dodony, Hungary	
10:22	EDIC intensity correction of electron diffraction, Ora	Ι
10:55	Coffee break	



	Session 5 (chairm	an: Robert Peter)
	Pavol Noga, Slovakia	
11:30	Ion beams for high fluence irradiation testing and characterize	ation of
	materials, <b>Invited</b>	
	Zoltán Száraz, Slovakia	
12:00	Ion beam irradiation and analysis of materials for radiation er	nvironments,
	Oral	
	Milena D. Majkić, Serbia	
12:20	Mechanism of nanostructure formation on metal surface by a	n impact of
	slow highly charged ions under arbitrary angle of incidence, <b>O</b>	Iral
	Andrea Schulze, Germany	
12:40	Enhancing Magnetic Sensing & Next-Gen AR Devices - Superic	or Performance
	with Ion Beam Processing ( <b>scia systems</b> – sponsor presentatic	on)
13:00	Lunch	

	Session 6 (chairman: Miran Mozetič,		
15:00	Jordi Sancho Paramon, Croatia		
15.00	Semi-continuous metal films as building blocks for optical coating, <b>Invite</b>		
	Pavel Čurda, Czech Republic		
15:30	ITO nanostructures for label free electro-optical modulated LMR sensing,		
	Oral		
	Aleš Omerzu, Croatia		
15:50	Thin ZnO films prepared by plasma-enhanced atomic layer deposition for		
	enhanced photocatalytic applications, <b>Oral</b>		
	Róbert Kun, Hungary		
16:10	Nanomechanical characterization of solid-state Li-ion battery components,		
	Invited		
16:40	Coffee break		
17:10	Poster session 2		
19:00	Dinner		



Wednesday, 2<sup>nd</sup> Oct

	Session 7	(chairman: Nepal Chandra Roy)
	María C. Asensio, Materials Science Institute c	of Madrid, Spain
9:00	Discovery of advanced materials using artificia	l intelligence methods and
	advanced characterization techniques, Plenary	/
	Michael Tkadletz, Austria	
9:45	Microstructural Design of Cathodic Arc Deposit	ted fcc-TiAIN Coatings: A Path
	Toward Sustainable Hard Coatings, Invited	
10:15	Rok Zaplotnik, Slovenia	
10.15	Novel plasma-supercavitation technology for v	vater cleaning, <b>Oral</b>
	Gregor Primc, Slovenia	
10:35	Combination of advanced oxidation technologi	ies for removal of
	micropollutants, <b>Oral</b>	
10:55	Coffee break	

	Session 8	(chairman: Kinga Kutasi)
	Matjaž Panjan, Slovenia	
11:30	Unraveling plasma self-organization of magnetron s	puttering discharges,
	Invited	
	Nina Recek, Slovenia	
12:00	Upscaling the plasma reactor for the treatment of s	eeds and seed-borne
	toxins in agriculture use, <b>Oral</b>	
	Ardita Kurtishaj Hamzaj, Slovenia	
12:20	Hydrogen interaction and oxide permeation barrier formation in B2-ordered	
	Fe-Al intermetallic alloy, <b>Oral</b>	
12:40 Iveta Ukropcová, Czech Republic		
12.40	3D Printing of Semiconductors using Electron Tweez	ers, <b>Oral</b>
13:00	Lunch	
	Excursion / City tour - Split sightseeing	
15:00	&	
	Conference dinner at konoba Rudine	



Thursday, 3<sup>rd</sup> Oct

	Session 9	(chairman: Miha Čekada)
9:00	Kingdom	
9:00	Challenges and Solutions of Oxides for Future AI H	ardware, <b>Plenary</b>
	Nives Štrkalj, Croatia	
9:45	Nanoscale Ferroelectricity and Electrochemical Dy	namics in Hafnia-Based
	Films, <b>Invited</b>	
	<b>Ján Šoltýs</b> , Slovakia	
10:15	Fabrication of NbTiN superconducting nanowire si	ingle-photon detector,
	Oral	
10:35	Karel Mašek, Czech Republic	
10:35	Structure and growth of cobalt oxide epitaxial laye	ers, <b>Oral</b>
10:55	Coffee break	

	Session 10	(chairman: Slobodan Milošević)
11:30	Barbara Šetina Batič, Slovenia Microbiologically induced corrosion of power Invited	plant steel components,
12:00	<b>Kinga Kutasi</b> , Hungary Alginate-stabilized colloidal ZnO NPs for biom	edical applications, <b>Oral</b>
12:20	<b>Nepal C. Roy</b> , Belgium Atmospheric plasmas for depositing sulfonated plasma-polymerized films for use in liquid contacts, <b>Oral</b>	
12:40	Mark Zver, Slovenia Plasma-generated vacuum-UV radiation for ir Oral	nactivation of viruses in water,
13:00	Lunch	



	Session 11 (chairman: Klára Hajagos-Nagy)	
15:00	Željko Skoko, Croatia	
	Revealing nanoscale mechanism in the jumping crystals, Invited	
	<b>Lucie Janů</b> , Czech Republic	
15:30	Radical-assisted covalent binding of biomolecules to amine plasma	
	polymers, <b>Oral</b>	
	Ida Delač, Croatia	
15:50	Exploring the effects and stability of organic molecule modifications on	
	CVD-synthesized monolayer MoS2, <b>Oral</b>	
16:10	Closing remarks	
16:20	Coffee break	
19:00	Dinner	



Monday 30<sup>th</sup> Sept

17:10-19:00

Poster session 1		
P1	Changhwan Choi, Korea	
	ALD HfZrOx and InGaOx Thin Films for Flash Memory Application	
P2	Robert Peter, Croatia	
	Photocatalytic activity of TiO2 thin films embedded with Cu nanoparticles	
	prepared by atomic layer deposition technique	
	Aljaž Drnovšek, Slovenia	
P3	High-Temperature Tribology of Multilayered CrN/(Cr,V)N/VN Protective	
	Coatings	
	Vedran Brusar, Croatia	
P4	Pressure dependent nonlinear spectral broadening of ultrashort laser pulses	
	in hollow-core fiber (HCF)	
	Iva Šrut Rakić, Croatia	
P5	Monolayer MoS2 growth on a pre-patterned substrate as a way to induce	
	strain	
	Andrea Jurov, Slovenia	
P6	Characterization of Alumina Scales on Fe-Al Alloys: Enhancing High-	
	Temperature Hydrogen Permeability Resistance	
P7	Davor Čapeta, Croatia	
F 7	High resolution, compact Raman spectrometer based on CMOS camera	
	Julio Car, Croatia	
P8	Determination of dielectric functions of colloidal silver nanoparticles in LSPR	
	relevant wavelength range	
	Filip Ferenčík, Slovakia	
P9	Effect of Sputtering Power on Low Concentration Impurities in Binary Oxides	
	for Semiconductor Applications: A ToF-ERDA Characterization Study	
	Iva Šarić Janković, Croatia	
P10	Low-energy ion bombardment induced reduction of TiO2 grown by Atomic	
	Layer Deposition	
	Karmen Kapustić, Croatia	
P11	Visualizing intercalation of 2D material using AFM based techniques: MoS2	
	on graphene/Ir(111) case study	
	Nikša Krstulović, Croatia	
P12	UV and solar-driven photocatalysis of organic dyes using ZnO-Ag	
F 12	heterojunction nanoparticles synthesized by one-step laser synthesis in	
	water	
	Karolina Pietrzak, Croatia	
P13	Application of electrochemical impedance spectroscopy in studying the	
	mechanism of metal oxide nanoparticles used in ion-selective electrodes	
	Šárka Zuzjaková, Czech Republic	
P14	High entropy nitrides: High temperature study of Cr–Hf–Mo–Ta–W–N thin	
	films	



	Zsolt Fogarassy, Hungary
P15	Structural investigation of the heat-treated Ti/Al/TiN/Au contact layer on n-
	GaN - role of TiN
	Miklós Serényi, Hungary
P16	On the challenges in reactive HiPIMS co-sputtering of Y and Ti for oxide
	layers
	Béla Pécz, Hungary
P17	Advanced structural characterization of Gallium Oxide by transmission
	electron microscopy

#### Tuesday 1<sup>st</sup> Oct

17:10-19:00

Poster session 2		
P1	Lucija Krce, Croatia	
	PET surfaces treated with atmospheric pressure plasma jet and enhanced	
	with ZnO nanoparticles: characterization and antibacterial activity	
P2	Damir Dominko, Croatia	
	Collective states driven far away from the equilibrium	
_	Miran Mozetič, Slovenia	
P3	Characterization of low-pressure gaseous plasmas by optical emission	
	spectroscopy	
P4	Maja Mičetić, Croatia	
	Synthesis and properties of Ge-based core-shell quantum structures	
P5	Mario Rakić, Croatia	
	Femtosecond engraving computer-generated holograms	
P6	Rafaela Radičić, Croatia	
	Fabricating PET/ZnO composite assisted by RF plasma processing	
P7	Tihomir Car, Croatia	
F7	Silver nanoparticles in SiC matrix	
	Zsolt Fogarassy, Hungary	
P8	Technological applicability of VO2 layers prepared with different	
	approaches	
	Krešimir Salamon, Croatia	
Р9	Grazing incidence high-energy heavy ion irradiation for surface	
	nanopatterning	
P10	Tímea Hegedűs, Hungary	
	Pt nanoparticles on inert h-BN support: investigation of the CO2	
	hydrogenation mechanism on the surface level	
	Ildiko Cora, Hungary	
P11	In situ TEM examination of the monoclinic VO2 thin layer phase	
	transformation to tetragonal VO2	



Dimo Yosifov, Canada
Python Modeling of Beamline Vacuum System
Klára Hajagos-Nagy, Hungary
The effect of impurities on the structure and mechanical properties of
CoCrCuFeNi alloy films
Pranjali Jadhao, Czech Republic
Direct growth and application of 2D single crystals of MXenes
Ivna Kavre Piltaver, Croatia
Enhanced Photocatalytic Activity of ZnO Films with Fe2O3 Intermediate
Layers for Efficient Water Purification
Alenka Vesel, Slovenia
Influence of chitosan-coated urinary catheters on adhesion of urethral cells
Martin Vičar, Czech Republic
New stable ionization vacuum gauge as the transfer standard
Marcell Gajdics, Hungary
GaON thin films prepared by reactive sputtering of a liquid Ga target



6. Abstracts

#### Session 1

#### Plenary

#### Controlling the Energy and Species in PVD: HiPIMS with Cathode Reversal

David N. Ruzic, Zachary Jeckell, Nicholas Connolly, Tag Choi, Dongho Lee, Dren Qerimi

Center for Plasma Materials Interactions, Department of Nuclear, Plasma and Radiological Engineering, University of Illinois at Urbana-Champaign, Urbana IL USA

#### email: druzic@illinois.edu

High Power Impulse Magnetron Sputtering (HiPIMS) has been proven as an ionized physical vapor deposition (iPVD) technique to provide superior film quality and adhesion. The ionization of the target material is done without the use of additional plasma generators through high power densities at the target surface, ultimately increasing the ion flux to the substrate. The incident ions allow for controlled ion bombardment energy leading to superior quality films over that of direct current magnetron sputtering (DCMS). One limiting factor of implementation of HiPIMS in industrial settings is the decreased deposition rates as compared to conventional DCMS. The reduction in deposition rate is believed to be mainly due to the return of ionized target material back to the target surface. The Center for Plasma Material Interactions has developed along with Starfire Industries LLC, a new HiPIMS power supply module which is capable of controlled cathode voltage reversal. The bipolar supply allows for control, both potential and timing, of a positive polarity pulse following the negative polarity main pulse. Timed control of the substrate bias with respect to the HiPIMS pulse, has been proven to provide control of film growth morphology, increase deposition rate and reduce film stress. Most importantly the energy of the ions reaching the substrate can be precisely controlled, even for insulating substrates. This work investigates the use of a controlled positive polarity pulse and increased ionization regions to increase deposition rate while controlling ion bombardment energy without an external substrate bias, retaining the superior film qualities expected from HiPIMS. Time resolved electron energy distribution functions and time-resolved ion energies at the substrate are shown for each species. Furthermore an etching regime is explored where it is possible to etch surface impurities off the substrate milliseconds before desposition all in the same device. By manipulating the pulse conditions, the ratio of target ions to gas ions can also be controlled.



Invited

## HiPIMS with controlled arc ignition: hybrid plasma deposition technique for hard coatings

#### <u>Martin Čada<sup>1</sup></u>, Iryna Naiko<sup>1</sup>, Alina Ostapenko<sup>1</sup>, Ivana Venkrbcová<sup>1</sup>, Zdeněk Hubička<sup>1</sup>, Pavel Mareš<sup>2</sup>, Jiří Vyskočil<sup>2</sup>

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Diamond-like carbon (DLC) thin films are in demand in modern industry as a hard coating for machining knives, drilling or pressing tools. Since DLC films are characterized by low friction they can be effectively applied on surfaces suffering from wear e.g. in combustion motors. Tetrahedral amorphous carbon (ta-C) prepared by cathodic arc physical vapor deposition (PVD) method from pure carbon target is promising candidate characterized by hardness higher than 60 GPa, high deposition rate but with surface roughness around 1  $\mu$ m requiring a subsequent polishing of deposited thin films. Recently, one has been demonstrated that High Power Impulse Magnetron Sputtering (HiPIMS) operated in a mixed sputtering/cathode arc mode can produce ta-C films with a sp3 bond content of 80% [1]. To control the regularity, duration and timing of the cathode arc during the HiPIMS pulse, the secondary high-voltage negative pulse with defined phase shift was applied on the cathode [2]. For better understanding of this hybrid HiPIMS deposition process, the time-resolved ion composition and ion flux on substrate was investigated using energy-resolved mass spectrometer and Sobolewski probe. Four different plasma regimes were identified in dependence on the highvoltage pulse delay from start of HiPIMS pulse: 1) pure HiPIMS with IEDFs demonstrating presence of population of C++ ions for a significantly longer time, 2) activated HiPIMS, 3) hyper HiPIMS and 4) HiC ARC. The total ion flux on the substrate proved that cathodic arc ignition increases the ion flux on the substrate by factor 2 in comparison with HiPIMS without arc ignition. The obtained results also showed that controlled ignition of the cathodic arc near the end of the HiPIMS pulse generates short-living argon and carbon ion fluxes whilst randomly ignited cathodic arc produces ion population bombarding a substrate for more than 100 µs.

[1] R. Ganesan et al. J. Phys. D: Appl. Phys. 48 (2015) 442001

[2] J. Vyskočil et al. Surf. Coat Technol. 446 (2022) 128765



Oral

#### Property variation of hard coatings deposited in industrial deposition systems

#### Miha Čekada<sup>1</sup>, Aljaž Drnovšek<sup>1</sup>, Matej Drobnič<sup>1</sup>

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In a lab-scale PVD system, the relationship between deposition parameters and coating properties is relatively straight-forward. In an industrial deposition system, on the other hand, there are several additional points which need to be considered. This includes possible gradients in vertical and radial axes, rotation mode dependence and shading effects. Though the deposition system producer guarantees the required coating quality within the specified chamber volume, these (relatively minor) variations can have an impact in specific tooling applications.

In this work we used both standard samples (polished disks) as well as simple cutting tools (drills) to evaluate the above mentioned dependences. We mounted these samples/tools at different rotation modes within the same batch, or at different heights in the chamber. While some batches were purpose-designed for this work, many were standard batches containing tools from our partners; this ensured the relevance of the results in a real industrial environment. We evaluated the coating thickness, the surface topography and the growth defect density in all these cases. The majority of work was conducted using cathodic arc evaporation, while for reference, magnetron sputtering was used too.

Unsurprisingly, the mounting rotation mode yielded the most expressed differences between the samples. However, a non-negligible gradient in roughness was found in the vertical direction. Finally, selected effects on reground tools will be shown where a consecutive sputtering of several coatings severely increases both the thickness and the roughness, which can severely deteriorate the tribological properties of the surface.



### Focused Ion Beam technology as an excellent tool for integration of the three dimensional GaP nanocone onto Single Mode Optical Fibre

<u>J. Novák<sup>1</sup></u>, A. Rosová<sup>1</sup>, S. Hasenöhrl<sup>1</sup>, M. Sojková<sup>1</sup>, J. Kováč <sup>2</sup>jr., J. Kováč <sup>2</sup>, I. Lettrichová<sup>3</sup>, D. Pudiš<sup>3</sup>

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<sup>3</sup> Department of Physics, University of Žilina, Univerzitná 1, 01026 Žilina, Slovakia email: <u>eleknova@savba.sk</u>

Fiber-optic nanosensors (OFN) with nanoscale dimensions are capable of sensing intracellular/ inter-cellular physiological and biological parameters in submicron environments. Basically, there are three main groups of configurations in which all the different types of optical fibers can be exploited as biosensors: through direct excitation of their end face, through the exposition of their core on their longitudinal dimension or through the resonant coupling of internal gratings.

Focused ion beam (FIB) method was applied for separation of individual NCs from the sample. The typical device shape is square-like with side size close to 50  $\mu$ m with NC in the middle. To prevent contamination of nanocone surface by aggressive waste produced during the cutting process, it is necessary make such cutting far from the nanocone: To our best experience such sufficient safety distance is higher than 10 micrometers. Crucial point of this experiment is backside thinning and polishing of GaP substrate under final thickness of 15 micrometers. Nanocone chip was after separation from the substrate transported to the front facet of the single-mode optical fiber (SMOF). This operation has to be realized extremely carefully. The front facet of the fiber is charged by electrons and scanned image is partially deformed. In this case the exact navigation of chip (or exact navigation of nanocone) on the precise position is hindered. Chip after landing on the fiber core is bonded to the fiber facet by platinum. Finally, the GaP nanocone was illuminated through the optical fiber and its optical picture was studied by near field scanning optical microscope (NSOM). We used a non-contact mode of NSOM technique where the tapered fiber tip is performing the in-plane movement over the sample without touching the sample surface. In summary, we studied preparation and properties of nanocone probes consisted from GaP nanocone integrated on central part of single mode fiber.

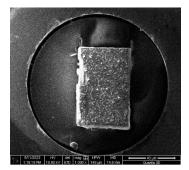


Fig.1 SEM top view on GaP nanocone chip onto top of SMOF (ion view)

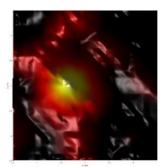


Fig.2 NSOM view on GaP nanocone deposited on SMOF – top view. Illumination is connected from the bottom side through the SMOF



#### Session 2

Invited

#### Surface coating layers for sensors and biomedical applications

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Kálmán Vad<sup>1</sup>

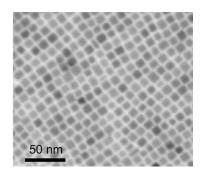
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By taking advantage of the opportunities provided by thin layers with the right thickness, composition and structure, we can reduce the size of the technological devices around us (e.g. sensors, portable computers, mobile phones). Thin layers can also be grouped in terms of their functionality. There can be layers providing chemical/physical protection on the surface of devices, such as diffusion barriers, layers that increase biocompatibility, and active layers that provide some physical response to external influences in sensors or light-emitting devices.

In this work, we report on the study of surface layers of dental implants that increase

biocompatibility, and of a luminescent perovskite thin layer which is suitable for detecting radioactivity. First, the basis of the layer covering the surface of implants is a CaSi-ceramic powder. Using this powder, we investigated the possibilities of forming nanostructured coating layers on titanium substrates in order to significantly reduce the ossification time of the implants after implantation [1]. Secondly, the chemical synthesis of inorganic perovskite nanocrystals and the constructions of thin layers from them were performed. The subsequent irradiation of the thin layers was carried out with radiation sources and a particle accelerator beam. The aim of our work was to collect data on the potential applications of



Scanning Transmission Electron Microscope (STEM) image from the prepared CsPbBr3 nanocrystals.

perovskite thin films as particle detectors, which are currently not available in the literature [2].

[1] Cs. Buga et al.: Colloids and Surfaces B: Biointerfaces 202 (2021) 111699.

Acknowledgements: This work was carried out in the frame of the projects TKP2021-NKTA-42 and 2019-2.1.7 ERANET-2021-00021 financed by the National Research, Development and Innovation Fund of the Ministry for Innovation and Technology, Hungary.

<sup>[2]</sup> M. Hunyadi et al.: Advanced Functional Materials 32/48 (2022) 2206645.



Effect of LCO<sub>2</sub> on TiAIN hard coating wear behavior at elevated temperatures

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PVD hard coatings are widely used to improve the wear and friction properties of cutting tools. These coatings significantly extend the tool life, thereby reducing total machining time and lowering production costs. Wear has a key impact on tool life and can be reduced by using cooling and lubrication technologies. Conventional polluting cooling fluids such as oil or emulsion can be replaced using a novel lubricating liquid carbon dioxide (LCO<sub>2</sub>) approach. This method not only promotes a cleaner machining process but also enhances productivity.

To simulate the conditions of use of a TiAlN-coated cutting tool with the addition of LCO<sub>2</sub>, the friction and wear behavior were studied in a laboratory-scale experiment using a high-temperature ball-on-disc tribometer. We used a TiAlN-coated disc, deposited by the cathodic arc evaporation and an uncoated ball, both made of WC-Co. The tests were performed in air, N<sub>2</sub> and CO<sub>2</sub> atmospheres at room temperature, 200 °C, 500 °C and 700 °C. Subsequently, at the same temperature conditions, we also performed experiments with direct addition of LCO<sub>2</sub> to the contact zone in a CO<sub>2</sub> atmosphere. Wear analysis on ball and disk were performed using a confocal optical microscope and a profilometer, detailed characterization of the wear was obtained using SEM and FIB analyses.

Research results show that the TiAlN coating has the highest wear rate at room temperature conditions, regardless of the atmosphere, with the wear rate decreasing with temperature. The optimal temperature for the TiAlN coating in our case was around 500 °C in all atmospheres. However, when LCO<sub>2</sub> was introduced into contact, wear was the highest at this temperature. In all LCO<sub>2</sub> tribological tests, we also observe increased friction coefficient fluctuations, which may originate from the formation of dry ice particles at the nozzle outlet and non-uniform flow of LCO<sub>2</sub>.



## Non-equilibrium solubility of Cu in $\alpha$ -Mn structure – microstructure and growth of $\alpha$ -Mn(Cu) films

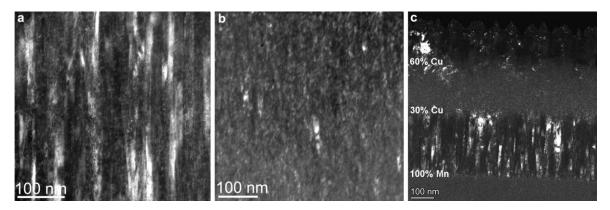
#### Hajagos-Nagy, Klára; Czigány, Zsolt; Misják, Fanni; Radnóczi, György

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The stable form of Mn at room temperature,  $\alpha$ -Mn, is considered problematic due to its brittleness, even though the  $\alpha$ -Mn structure contains many possibilities of use, such as para-, antiferro- and weakly ferromagnetic behaviour, anomalous Hall effect and high pressure (220 GPa) stability. The usability of  $\alpha$ -Mn can presumably be expanded by alloying. Cu does not dissolve in it under equilibrium conditions, however, in our previous work we produced an  $\alpha$ -Mn(Cu) solid solution under non-equilibrium conditions - by DC magnetron sputtering. In this research, we investigated the non-equilibrium solubility limit of Cu in  $\alpha$ -Mn, the growth and microstructure of  $\alpha$ -Mn(Cu) layers.

The non-equilibrium solubility was investigated on a 50 nm thick combinatorial sample grown by DC magnetron sputtering, where the composition varied linearly between pure Mn and 50 at% Cu content. Electron diffraction patterns showed that  $\alpha$ -Mn can dissolve up to 30 at% Cu. Film growth was examined on 0.5-1 µm thick films, comparing discrete and variable composition (gradient) layers. At 10 at% Cu content a single-phase  $\alpha$ -Mn(Cu) solid solution layer grows with a columnar structure (fig. a). As the Cu content increases, an amorphous minority phase appears and hinders the growth of the  $\alpha$ -Mn(Cu) grains (fig. b). Examination of gradient films showed that the columnar structure - characteristic of single-phase growth - can be maintained until the crystalline phase ceases (fig. c), which is favourable for many applications.



Microstructure of Mn-Cu films with Cu content of 10 at% (a), 20 at% (b) and 0-60 at% (c) on dark field TEM images.

This work is supported by the project VEKOP-2.3.3-15-2016-00002 of the European Structural and Investment Funds and the Hungarian Academy of Sciences, grant number K143216.



## Complex magnetic and transport phenomena in nanocrystalline gadolinium doped manganite CaMnO<sub>3</sub>

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Manganese oxides or manganites are widely known for their colossal magnetoresistance (CMR) effect. CMR is a metal-insulator transition caused by a magnetic field at the temperature close to the Curie point, and characterized by a drop of the resistivity by many orders of magnitude. Besides tuneable conductivity, manganites show complex magnetic behaviour with different (simultaneous) types of magnetic ordering, charge ordering, unconventional mechanisms of electrical conductivity, phase separation, memory, and ageing effect. These properties arise from the strong coupling of spin, charge, lattice, and orbital degrees of freedom, and are highly dependent on the chemical composition of manganites, temperature, and applied magnetic field. Despite several decades of intensive research, complex interactions between electrons in manganites are still not well understood, and further investigation is needed.

Calcium manganite CaMnO<sub>3</sub> is known to be an antiferromagnetic insulator. This work focuses on poorly investigated gadolinium-doped calcium manganite Ca<sub>1-x</sub>Gd<sub>x</sub>MnO<sub>3</sub>. Nanocrystalline samples with x = 0.05, 0.1, 0.15, 0.2 were synthesized by a modified glycine-nitrate procedure followed by calcination and sintering at high temperatures. We performed detailed measurements of transport and magnetic properties of the prepared samples in a wide temperature range and high magnetic fields. We found that gadolinium doping causes suppression of the antiferromagnetic order giving rise to new magnetic phases and phase separation, as well as the strong drop of resistivity and appearance of CMR effect.



#### Invited

#### In-Situ Techniques for Perovskite Layer Growth Monitoring: X-ray Scattering and Photoluminescence

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Metal halide perovskites, a class of hybrid organic-inorganic materials, offer exceptional optoelectronic properties, making them prime candidates for next-generation solar cells and radiation detectors. This review traces the evolution of perovskite thin film fabrication from traditional liquid-deposition methods to vacuum co-deposition and cutting-edge chemical vapor deposition techniques. A key focus is on *in-situ* characterization techniques that can enhance thin film performance by reducing defects and improving crystallinity. In particular, we highlight the combined power of *in-situ* grazing-incidence wide-angle X-ray scattering (GIWAXS) and photoluminescence (PL) in probing both structural and optical properties. Through real-world examples, such as perovskite solar cells, we demonstrate how this tandem approach can drive advancements in perovskite-based devices.



#### Plasma-Assisted Vapour Thermal Deposition for Controlled Deposition of Nanocomposites

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Plasma polymerization, or plasma-enhanced chemical vapour deposition (PECVD), is widely used for depositing organic thin films. Despite its popularity, it often results in films with limited molecular complexity. This is partly because it relies on relatively low molar mass precursors that can be vaporized into the gas phase. Plasma-assisted vapour thermal deposition (PAVTD) was developed to address this limitation.

In PAVTD, a solid polymer is heated in a crucible, causing thermal degradation (evaporation) and in-situ formation of oligomers heavier than typical PECVD precursors ( $10^2$ - $10^3$  g.mol<sup>-1</sup>). These oligomers are subsequently re-polymerized in RF plasma, resulting in films that can exhibit properties characteristic of classical polymer physics and chemistry, which is uncommon for plasma polymers. This method enables fine-tuning properties such as biodegradability and hydrolyzability, as demonstrated in polylactic acid-based films [1,2].

PAVTD effectively bridges the gap between the composition and properties of classical and plasma polymers. However, its batch processing presents challenges in terms of deposition duration and stability. To overcome these challenges, continuous-PAVTD was developed, employing standard FDM 3D printing filaments. This advancement improves stability and increases deposition rates (up to several nm/s), making PAVTD a more practical deposition method and a valuable tool for exploring plasma polymerization processes. Moreover, PAVTD can be integrated with other deposition techniques. This versatility of PAVTD was demonstrated by the preparation of polymer-metal nanocomposites with PLA-like matrix using two different filler fabrication methods: magnetron sputtering and gas aggregation source of nanoparticles. The precise control achievable with PAVTD was utilized to produce nanocomposite-based Bragg's multilayer reflectors.

Acknowledgements: This work was supported by the grants GA22-21007S of the Czech Science Foundation and GAUK 411822 of the Grant Agency of Charles University.

<sup>[1]</sup> Krtouš et al., Materials, 2021, vol. 14, pp. 459.

<sup>[2]</sup> Krtouš et al., Surface and Coatings Technology, 2021, vol. 421, 127402



#### Optically driven thin film OECT electrochemical sensor

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Thin film electrochemical transistors (OECTs) made from poly(3,4organic ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) are extensively utilized in highly sensitive biosensors. With suitable gate or channel functionalization, they are effective for bio-sensing and bioelectronic readout applications. For implantable sensor applications, these devices should ideally function fully wirelessly, with wireless power supply, gating, and readout capabilities. Moreover, they should be manufactured on flexible and stretchable substrates to conform well to various tissue shapes. We present methods for the wireless powering and electrical readout of implanted electrochemical sensors based on OECT devices. We will demonstrate our method for wirelessly gating OECTs using a gate electrode modified with an organic, capacitively coupled, photovoltaic-like stack. This stack consists of a vertical tandem of metal-free phthalocyanine (H2PC) and N,N'-dimethyl perylenetetracarboxylic diimide (PTCDI), deposited by thermal vacuum evaporation and enhanced with PEDOT:PSS to increase electrode capacitance. Additionally, we will present external readout strategies that do not depend on complex implanted electronic components.



#### Invited

#### Assessing and enhancing fracture toughness of hexagonal diboride coatings

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Coherently grown nanolayered PVD thin films, referred to as superlattice thin films, are known for their superior hardness compared to monolithically grown constituents. By employing in-situ micromechanical cantilever bending tests, we have shown that the fracture toughness also shows a significant bilayer period-dependent behavior. While mechanisms based on dislocation activity explain the hardness peak, the linear elastic deformation of the microcantilever during the fracture experiments suggests that these explanations are not directly applicable to describe the enhancement in fracture toughness. Consequently, an underlying, bilayer-period-dependent intrinsic property is responsible for this behavior.

This contribution presents an overview of the ceramic superlattice systems deposited so far, their increase in hardness and fracture toughness, and a mechanistic consideration of the increase in these properties. For this purpose, we selectively deposited systems, which differ in the two decisive factors for increasing the mechanical properties, the shear modulus difference  $\Delta G$  and the difference in the lattice parameters  $\Delta a$ . While our TiB<sub>2</sub>-WB<sub>2</sub> systems show a pronounced difference in G, their lattice parameter is very similar, contrary to our TiB<sub>2</sub>-ZrB<sub>2</sub> systems, having similar elastic properties while showing a pronounced difference in their crystallographic properties. We unravel the active mechanisms responsible for the respective increases in H and K<sub>IC</sub> by performing the same micromechanical tests on these novel diboride coatings. Specifically, the TiB<sub>2</sub>-ZrB<sub>2</sub> coatings showed an increase in K<sub>IC</sub> up to 3.7 MPa·m<sup>0.5</sup>, while the TiB<sub>2</sub>-WB<sub>2</sub> system experienced an increase in hardness to 45.5 GPa, both without a significant increase of the other mechanical properties. These results and investigations, especially the systematic design of the study, allow a targeted development of protective coatings.



#### Session 4

#### Plenary

#### 2D Boron Carbon Nitride structures: from synthesis to application

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Graphene is a well-known 2D semimetal with many attractive properties, while hexagonal boron nitride (h-BN) is its structural counterpart. The different electronegativities of its constituting B and N atoms, however, result in a large bandgap (~6 eV), which makes this stiff and inert material an ideal insulating element of nano-heterostructures. Moreover, periodically undulating h-BN monolayers are good templates. The combination of all three elements in the same lattice has attracted a lot of attention in order to tailor material properties, such as band gap. However, there is a strong tendency for phase separation into graphene and h-BN, which makes the synthesis very difficult. We started our investigations with corrugated h-BN nanomesh monolayers and proved that applying surface alloys as substrates it is possible tune its structural parameters. Besides, the enhancement of the local work function nanopatterning has also been achieved, which is highly relevant for noninvasive surface templating, a promising route towards designing large-scale artificial atomic and molecular nanostructures with tailored chemical, quantum or spin functionalities. As a next step, we prepared metal nanoparticles on h-BN to obtain model catalysts and studied thermal processes with and without adsorbed molecules. Examples for catalytic studies related to the transformation of ethanol and acetaldehyde will be presented. In an attempt to synthetize ordered BCN monolayers on metals, the decomposition of benzene borazine mixtures with different ratios has been studied. It turned out the surface BCN stoichiometry is very different from the gas phase, but at proper conditions intimately mixed well ordered 2D layers were obtained on Rh(111). The applied methods span core level and angle resolved valence band photoemission (momentum microscopy), other electron and ion spectroscopic methods, STM and DFT calculations.



#### Invited

#### Me-B-C coatings - hard and fracture resistant material

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Binary borides and binary carbides have been known for many decades. Their outstanding mechanical, electrical and thermal properties made them indispensable in the industry, either in bulk or as thin films. The idea of combining these systems into a ternary X-B-C system naturally evolved. Such systems in the form of bulk have been investigated since the 1950s. The X has been from all of the s, p d and f blocks of the periodic table of the elements. Different structures were found. The orthorhombic Mo<sub>2</sub>BC phase, being the first with the X<sub>2</sub>BC stoichiometry, was described by Jeitschko in 1963 [1]. This structure attracted the interest for its superconducting properties. Sixty years later, this is still the only synthesised X<sub>2</sub>BC phase.

A renaissance of the X<sub>2</sub>BC phases, this time in the form of the thin films, has begun in 2009 when the Mo<sub>2</sub>BC phase was prepared by direct current magnetron sputtering [2]. This phase was no longer studied for its superconducting properties, in which other materials superseded it, but for its highly unusual combination of high hardness and moderate ductility. Theoretical studies describing other crystalline ternary X<sub>2</sub>BC phases predicting the thermodynamic possibility of their preparation together with even better mechanical properties followed [3]. Since then, several different systems from these predictions have been studied, including W-B-C, Nb-B-C and Ta-B-C. No definitive proof of the existence of any X<sub>2</sub>BC phase apart from the original Mo<sub>2</sub>BC phase was found. This contribution will cruise through the ups and downs of this research in the last decade. It will be shown that thin films from these systems can have interesting mechanical and thermal properties even without the formation of the desired and elusive X<sub>2</sub>BC phase. It will be discussed why these phases won't form even under energetically very harsh conditions such as HiPIMS. We will also sketch the possibilities for the future directions of the studies of the X<sub>2</sub>BCs.

<sup>[1]</sup> W. Jeitschko, H. Nowotny, F. Benesovsky, Monatshefte für Chemie und verwandte Teile anderer Wissenschaften 94 (1963) 565–568

<sup>[2]</sup> J. Emmerlich, D. Music, M. Braun, P. Fayek, F. Munnik, J. M Schneider, J. Phys. D: Appl. Phys. 42 (2009) 185406
[3] H. Bolvardi, J. Emmerlich, M. to Baben, D. Music, J. von Appen, R. Dronskowski, J.M. Schneider, J. Phys.: Condens. Matter 25 (2013) 045501



## Refractory high-entropy nitride coatings deposited by reactive direct current magnetron sputtering

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Since its introduction in 2004, high-entropy materials have been extensively researched. These materials have remarkable properties which lead to a wide range of applications. Highentropy nitride coatings have gained attention as potential candidates for protective coatings due to their superior mechanical and thermal properties. Especially promising are coatings that contain refractory elements, due to their strong bonding with nitrogen. In our work, highentropy nitride coatings containing six different refractory elements were deposited using reactive direct current magnetron sputtering with the nitrogen flow ratio  $f_N$  ( $f_N = N_2/(Ar + N_2)$ ) ranging from 0 to 50 % at constant total gas flow. The aim was to study the effect of nitrogen concentration on microstructure and crystal structure, as well as mechanical and corrosion properties. To analyze the microstructure and crystal structure, we employed X-ray diffraction (XRD), atomic force microscopy (AFM) and scanning electron microscopy (SEM). For assessing mechanical properties, we utilized nanoindentation and profilometry techniques. The corrosion resistance of the coatings was evaluated through potentiodynamic polarization measurements. Nitrogen concentration increased with the nitrogen flow ratio non-linearly. Results from X-ray diffraction (XRD) show an amorphous crystal structure for the coating without nitrogen and the fcc crystal structure for nitride coatings. Scanning electron microscopy revealed a columnar cross-section morphology. Hardness and reduced elastic modulus peaked at  $f_N = 40\%$ . Coatings exhibit an increased resistance against corrosion.



#### EDIC intensity correction of electron diffraction

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With advancements in nanotechnology, understanding materials at the atomic level is crucial. Transmission electron microscopy (TEM) stands out among several methods for this purpose. High-resolution TEM (HRTEM) images and electron diffraction (ED) patterns are key in determining material structures but require corrections due to the microscope's transfer function and the Ewald sphere-reciprocal lattice distance.

The structure factors ( $F_{hkl}$ ) of a crystal reveal its atomic arrangement, but the detected diffracted intensity ( $I_{hkl}$ ) is not directly proportional to the square of the structure factors ( $F_{hkl}^2$ ) due to the Ewald sphere-reciprocal lattice distance. In 1992, J.M. Cowley established the fundamentals of Ewald correction [1], defining the relationship between experimental intensity and structure factors.

Expanding on Cowley's work, we defined a correction function to derive  $|F_{hkl}|^2$ -proportional  $I_{hkl}$  values from experimental intensities [2]. This corrected dataset is suitable for accurate crystal structure determination. We also extended this correction method to misoriented crystals. Manual application of these corrections is time-consuming, especially for crystals with large unit cells and hundreds of reflections.

To address this, we developed a computer program (EDIC) to automate the correction process. The program allows rapid intensity correction of experimental ED patterns, enabling the determination of unknown structures from experimental intensity datasets ( $I_{hkl}$ ) with improved accuracy.

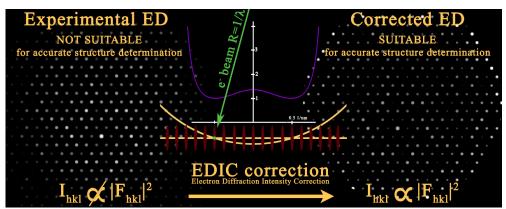


Figure 1. Concept of EDIC correction for a misaligned crystal. Our method recovers the theoretical  $I_{hkl} \propto |F_{hkl}^2|$  intensities from experimental  $I_{hkl}$  intensities determined by the relationship between the elongated diffracted intensities and the Ewald sphere

[1] Cowley, J.M. (1992) In "Electron Diffraction Techniques", J.M. Cowley, ed., Oxford Univ.Press, Oxford Vol. 1.
 [2] E. Dodony, I. Dódony, Gy. Sáfrán, EDIC intensity correction of electron diffraction, Micron, (2024)



#### Invited

Ion beams for high fluence irradiation testing and characterization of materials

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Ion beams are invaluable tools for materials qualification and testing across diverse applications, including nuclear power, aerospace, and electronics. In the nuclear industry, the traditionally lengthy process of materials qualification can be accelerated by using ion accelerators to evaluate irradiation damage in newly developed structural as well as functional materials or electronic devices. The only drawback in comparison with neutron irradiation using research reactors is the very low thickness of the damaged layer. The typical irradiation experiment comprises one ion implantation step, which modifies around 1-3 micrometers of the sample, resulting in a graded layer and damage profile. To overcome this limitation, we employ a multi-step approach that produces a much thicker (up to 70  $\mu$ m) and homogeneously irradiated layer. This method enables subsequent micromechanical testing and assessment of engineering-relevant material properties, providing a more comprehensive evaluation of the material's performance under irradiation.

On the other hand, ion beam analysis offers high sensitivity materials analysis, providing detailed isotope-resolved information on the elemental composition, detection and quantification of trace impurities, including light elements down to hydrogen at ppm concentrations. The latter gains importance with the spread of hydrogen production, transport and storage systems as well as lithium batteries, where the understanding of diffusion and migration of these elements is of critical importance. It can also assess the crystallinity of the sample, or, the degree of damage/amorphization, which is particularly useful in semiconductor processing.



Ion beam irradiation and analysis of materials for radiation environments

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Ion irradiation is widely used as a screening tool for the development and qualification of innovative materials in irradiation environments. Over the past decades, it has been applied in fission and fusion reactor materials research, accelerating exposure and simulating space radiation conditions. Ion irradiation has proven to be an effective surrogate for neutron irradiation, allowing for the study of fundamental radiation effects on materials. Irradiationinduced defects and transmutation-produced impurity atoms can increase the strength and hardness of materials. However, this enhancement often comes with irradiation embrittlement and a deterioration of mechanical properties, such as ductility, fracture toughness, and dimensional stability.

With ion irradiation, the localized damage within a shallow surface necessitates smallscale mechanical testing, making nanoindentation a critical method. Nanoindentation provides convenient measurement of indentation hardness as a function of penetration depth on a nanoscale. The combination of ion irradiation and nanoindentation has been extensively used as a screening method for assessing irradiation-induced hardening in structural materials.

Accelerated ion beams are also utilized for materials characterization. Ion beam analysis (IBA) techniques enable the acquisition of compositional depth profiles of nearsurface regions in thin films. IBA is known for its high sensitivity and does not require reference materials for elemental analysis. At MTF STU, we have a state-of-the-art Time-of-Flight Elastic Recoil Detection Analysis (ToF ERDA) spectrometer, which provides comprehensive information on a sample's elemental composition in a single measurement. This includes depth profiles of light impurities and the elemental composition of the matrix, ranging from hydrogen to uranium. A notable feature of ToF ERDA is its capability to detect and quantify trace amounts of light elements, such as hydrogen and helium.

In this contribution, we present our results on the investigation of ion-induced radiation damage and its effects on the mechanical properties of various candidate structural materials using nanoindentation and ToF ERDA analysis.

#### Acknowledgment

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#### Mechanism of nanostructure formation on metal surface by an impact of slow highly charged ions under arbitrary angle of incidence

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We adapt our recently developed cohesive energy model (CEM) [1] to comprehensively examine how impact geometry affects nanostructure formation on metal surfaces by an impact of slow highly charged ions (HCI). The model comprises two steps.

In the CEM I step, we use the micro staircase model based on the quantum two-state vector model (TVM) [2] for studying the neutralization process above the surface, resulting in the neutralization energy deposition [2] and the ion-atom interaction potential model for studying the kinetic energy loss as a result of collisional cascade below the surface [3]. The outcome of the processes above and below the surface is the total energy deposition into the solid inducing the rearrangement of atoms and target destabilization which contribute to the materials` morphology alteration [3]. The synergy between the neutralization energy and deposited kinetic energy enables us to define the threshold in ionic velocity, known as the critical ionic velocity [3,4], which is associated with the specific nanostructure formation. When neutralization energy dominates, hillock-like structures are expected, whereas crater-like formations emerge in the case of kinetic energy loss dominance.

Furthermore, according to our model, the formation of specific nanostructures depends on both the ion velocity [3] and chosen collision geometry [5]. Thus, particular nanostructures can be intentionally created by tuning the ion velocity and impact geometry [5].

In the subsequent CEM II step, we incorporate the concept of cohesive energy, considering surface modification as a consequence of alterations in solid cohesive energy due to total energy deposition [1]. This approach allows us to derive a general expression for nanostructure diameters across various ion-surface systems. We point out that the CEM diameters of surface nanostructures depend on charge and velocity, aligning with recent experimental data in the case of normal incidence [6].

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#### **Oral – sponsor presentation**

## Enhancing Magnetic Sensing & Next-Gen AR Devices – Superior Performance with Ion Beam Processing

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This presentation highlights the capabilities of ion beam processing, especially ion beam etching (IBE), in producing ultra-precise surface structures. The tunnel-magneto-resistance effect (TMR) is used in modern high-precision sensors ranging from angular position sensors in the automotive industry to read-out sensors in the hard-disk-drive industry. TMR sensors leverage the tunnel magnetoresistance effect, which occurs in a magnetic tunnel junction (MTJ), consisting of two ferromagnetic layers separated by a thin insulating layer. The multilayer composition of the TMR sensor leads to issues regarding the necessary etching process for electrically contacting the sensor. Using argon ions, IBE can accurately remove magnetic layers without causing corrosion, a common issue with chemical etching methods. This results in high-quality sensors with better thermal stability and increased signal output. In addition, another area of application is highlighted: the advancing waveguide fabrication for Augmented reality (AR) devices. AR glasses are built to enrich real-world images with additional digital information. This will be useful for applications such as assembly instructions or medical surgery support, where detailed information must be shown at the correct spot while keeping the hands of the user free. Ion beam etching plays a crucial role in fabricating optical waveguides, particularly slanted surface relief gratings. These gratings are essential as in- and out-couplers of light into and out of the waveguide to produce lightweight near-eye AR displays. IBE allows the creation of complex structures with high accuracy, essential for the precise light guidance required in AR applications.



Figure 1. SRG for AR devices



Figure 2. scia Mill 200



Session 6

Invited

## Semi-continuous metal films as building blocks for optical coatings

Jordi Sancho-Parramon<sup>1</sup>, Elizabeth Hedl<sup>2</sup>, Nirmala Maria D'Souza<sup>3</sup>, Željko Samec<sup>1</sup>, Vesna Janicki<sup>1</sup>,

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Optical coatings have traditionally relied on stacking dielectric (non-absorbing) layers with thicknesses comparable to the wavelength and exploiting the interference effects among the multiple beams reflected at the interfaces between layers. In recent years, novel coating designs have been proposed based on the use of highly lossy materials that enable, for instance, obtaining perfect absorption using layers with thicknesses much smaller than the wavelength [1]. In this talk, we will explore the use of semi-continuous metal films for optical coating design. We will first review how these films, formed during the island growth mechanism of metals on dielectric surfaces, exhibit highly tunable optical properties linked to their morphology. Thus, the film optical response can encompass the localized surface plasmon resonance of isolated particles, the infrared-enhanced absorption of metal islands aggregates and the Drude-like response of a percolated metal network. Next, it will be shown how these films can be incorporated into multilayer systems to create optical coatings with multiple functionalities. The focus will be on asymmetric Fabry-Perot resonators, which exhibit diverse optical responses depending on the morphology of the top semi-continuous metal layer. Such multilayer structures can be used to optically detect temperature changes exploiting the morphological changes induced in the film upon thermal annealing [2]. Overall, it will be illustrated how the rich optical behaviour of semi-continuous metal films combined with their ease of production using well-established thin film technology, make them attractive building blocks for innovative optical coatings.

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## ITO nanostructures for label free electro-optical modulated LMR sensing.

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Physical vapor deposition and atomic layer deposition are vacuum based techniques for preparation of thin films and nanostructures, that found a great use in fabrication of variety of sensors and biosensor. This study investigates the effects of forming of a thin dielectric layer on the optical, electrochemical, and electro-optically modulated responses of indium tin oxide (ITO)-coated optical fiber sensors. ITO's properties, including optical transparency, high refractive index, and electrochemical activity, make it suitable material for lossy-mode resonance (LMR) effect in the optical domain while also functioning as an electrode in the electrochemical domain. The dielectric layer on the ITO surface was precisely created to the nanometer scale using atomic layer deposition (ALD). This serves as a benchmark for forming biological or chemical layers in label-free sensing applications with these dual-domain sensors. It was found that the sensor exhibits responses in both the optical and electrochemical domains upon dielectric coating formation on the ITO surface. Numerical and experimental analyses demonstrated that the dielectric layer significantly influences the effectiveness of electro-optical modulation. Changes in ITO's refractive index and extinction coefficient at its interface with the layer are induced by modulating the density of free charge carriers. The study showed that dielectric layer thickness changes, down to sub nanometer, can be precisely monitored with modulation. Such precision is challenging to achieve with standard optical measurements. These findings present new opportunities for employing electro-optical modulation in label-free sensing and biosensing, particularly when detecting small biological species such are pollutants in wastewaters or small concentrations.



## Thin ZnO films prepared by plasma-enhanced atomic layer deposition for enhanced photocatalytic applications

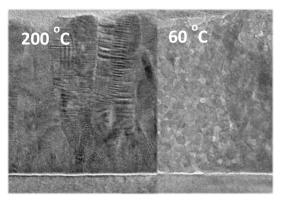
<u>Aleš Omerzu<sup>1</sup></u>, Daria Jardas Babić<sup>1</sup>, Robert Peter<sup>1</sup>, Krešimir Salamon<sup>2</sup>, Tina Radošević<sup>3</sup>, Damjan

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Atomic layer deposition (ALD) is an advanced deposition technique that allows us to synthesize high-quality thin ZnO films. By varying the parameters for thin film synthesis, different structural and physical properties of the films can be obtained [1-4]. The most important synthesis parameter is the deposition temperature. In thermal ALD, the temperature range for the deposition of high-quality films is between 120 °C and 180 °C. An upgrade of conventional ALD is



plasma-enhanced atomic layer deposition (PEALD), which enables deposition at lower temperatures without impairing the film quality and growth rate. The photocatalytic activity of the films deposited by the PEALD method shows a maximum value for films deposited at temperatures below 100 °C, and their efficiency is higher than that of the best thermal ALD films. In the present study, we compared the crystal structure, optical properties and photocatalytic activity of thin films deposited at 60°C, 80°C, 100°C and 200°C using the PEALD method. The TEM images show that the films synthesized at lower temperatures have smaller, roundish grains, while the films synthesized at higher temperatures consist of larger, elongated crystals that are perpendicular to the substrate. The smaller grain structure has a much higher concentration of localized surface states, resulting in better photocatalytic activity.

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Invited

## Nanomechanical characterization of solid-state Li-ion battery components

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Recently, the demand for safe, reliable portable power has risen, triggering significant research on solid-state batteries (SSB) as high-performance alternatives to conventional lithium-ion batteries. In fact, during electrochemical cycling extreme volume change, strain, mechanical degradation of the solid/solid interfaces of the SSB can occur which eventually results in battery cell failure. In this presentation, a comprehensive nanomechanical investigation of amorphous and crystalline modification of MoO3 cathode active material and Lithium phosphorous oxynitride (LiPON) solid state electrolyte is discussed. Understanding the mechanical behavior of the thin-film electrodes and solid electrolyte layers is crucial for the development of more durable and reliable SSBs. Nanomechanical properties of the electrode layers were analyzed by custom developed in situ nanoindenter [1] along with acoustic emission studies. We found that amorphous-type electrode materials are more durable against electro-chemo-mechanical-aging related battery performance loss in allsolid-state Li-ion batteries compared to their crystalline counterparts. Moreover, previous studies [2] noted unexpected ductility and strain recovery in amorphous LiPON, with sharpended tip indentations revealing pile-up formation and densification as the main deformation mechanisms. Our work presents nanoindentation experiments with a spherical tip, revealing a novel mechanical behavior—a sudden deformation event followed by slow strain recovery during unloading. This unique deformation is likely linked to the material's special structure, featuring isolated phosphate tetrahedra embedded in a Li matrix with occasional N bridge bonds between tetrahedra.

Keywords: Solid state batteries, Li-ion batteries, nanoindentation, plastic instability, strain recovery, MoO3, LiPON

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#### Session 7

#### Plenary

## Discovery of advanced materials using artificial intelligence methods and advanced characterization techniques

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Materials are the cornerstone of clean energy innovation, essential for advanced batteries, solar cells, low-energy semiconductors, thermal storage, and CO<sub>2</sub> capture and conversion. However, the lengthy and expensive process of developing new materials—often spanning 10 to 20 years—remains a bottleneck in the shift to a low-carbon future. Hence, significant challenges lie in discovering new materials within a huge chemical space and accomplishing an exhaustive characterization of materials at the sub-micrometre and atomic scales.

In this presentation, I will outline a combined experimental and Artificial Intelligence (AI) approach for discovering and optimizing advanced materials. I will highlight recent AI techniques used to predict novel energy materials with tailored properties, aligned with the Materials Genome Initiative [1]. Additionally, I will briefly describe a deep-learning method using a graph neural network (GNN) model to identify promising energy storage materials, trained with large datasets of structural and electronic properties from sources like the Materials Project, NOMAD, and another extensive database.

The AI-predicted candidates will be experimentally tested using advanced materials characterization techniques, including synchrotron radiation and neutron-based methods. I will present recent results from an innovative k-space nanoscope, Nano-ARPES (Nano Angle-Resolved Photoelectron Spectroscopy), which offers exceptional lateral resolution and robust spectroscopic and electronic characterization at the nano- and mesoscopic scales. This technique reveals the momentum- and spatial-resolved electronic band structure, highlighting the effects of heterogeneities and confinement on valence band electronic states at the Fermi level [2-6].

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Invited

## Microstructural Design of Cathodic Arc Deposited fcc-TiAlN Coatings: A Path Toward Sustainable Hard Coatings

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The microstructural design of hard coatings is the key to enhancing wear protection in cutting tools. Through optimized deposition techniques and parameter adjustments, it is possible to boost the performance while maintaining simple chemistries, which contributes to coating sustainability. This presentation explores how factors such as substrate properties and deposition parameters influence the microstructure of coatings synthesized by cathodic arc deposition. In particular, the role of bias voltage as a tool to control the microstructure will be examined, with attention to how cemented carbide substrate characteristics—like phase composition and WC grain size—affect the results. The study demonstrates how substrate-template effects and bias voltage can be used to control texture, crystallite size, and residual stress in fcc-TiAIN coatings. Additionally, Ta alloying along with bias voltage grading during deposition offers further control over microstructural features. While Ta is used as a model element here, future work may focus on replacing it with more sustainable alternatives. This research highlights the potential of microstructural design as a sustainable strategy for tailoring coating performance, providing flexibility to either leverage or mitigate substrate effects based on application needs.



## Novel plasma-supercavitation technology for water cleaning

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Water scarcity is a critical issue affecting billions globally. Climate change and population growth exacerbate the situation, leading to severe water shortages and impacting health, development, and economies. An increasing number of waterborne pollutants affect the safety of the water environment. One type of these pollutants is also waterborne viruses, such as human, animal or plant viruses. Of human viruses, norovirus, rotavirus and hepatitis are the most problematic and they pose significant health risks. These viruses can lead to diarrhea and gastroenteritis but can also cause other, more serious illnesses. Preventing the spread of waterborne viruses with inactivation is essential for protecting public health.

One of the new technologies that can be used to tackle the problem of micropollutants in water is non-equilibrium plasma. However, so far, the only type of plasma that could be used to treat water was atmospheric pressure plasma [1], where in most studies, only the surface of liquid or small volumes was treated.

To overcome these limitations, we designed a device where plasma can be ignited at subatmospheric pressure inside a stable supercavitation bubble that is formed inside a flowing liquid. Our innovative method (US11807555B2) is the first plasma system to synergistically integrate two advanced oxidation technologies: plasma and supercavitation. In our initial research utilizing this method, we used bacteriophage MS2, which is a human enteric virus surrogate. It is the first study where sub-atmospheric plasma was used to inactivate viruses in water.

With our patented method and device [2], we successfully inactivated more than 5 log10 PFU/mL of bacteriophage MS2 in a 0.43 L of recirculating water with under 4 minutes of treatment [3]. Our device is constantly being improved, optimized, and used to inactivate other waterborne micropollutants.

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## Combination of advanced oxidation technologies for removal of micropollutants

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The presence of micropollutants such as pharmaceuticals and endocrine-disrupting chemicals (EDCs) in water bodies, even in concentrations ranging from nanograms per liter to micrograms per liter, poses a significant worldwide problem. These substances can have profound and far-reaching impacts on both environmental and human health. Their presence in drinking water sources raises serious public health concerns, as traditional water treatment processes often fail to effectively remove these contaminants. Therefore, enhanced water treatment methods, including advanced oxidation processes (AOPs), are crucial to address this pressing issue. We have combined two potent AOPs, cavitation, and gaseous plasma, to tackle the removal of several micropollutants (valsartan, sulfamethoxazole, naproxen, diclofenac, tramadol, propyphenazone, carbamazepine, 17β-estradiol, and bisphenol A) simultaneously in a water matrix, and evaluated their removal rates in terms of varying process parameters, such as plasma power, water temperature and treatment time. The removal rates, yield, and characteristic half-lives  $(t_{1/2})$  achieved with the combination of two AOPs are benchmarked toward scientific literature that used atmospheric-pressure plasma jets, dielectric barrier discharges, and pulsed corona and spark discharges. The highest removals of all compounds were achieved at a water temperature of 25 °C, treatment time of 30 min, and plasma power of 53 W. Micropollutants 17β-estradiol, bisphenol A, and diclofenac were removed to the greatest extent, namely  $\geq$  89%, while the removal of other compounds ranged from 33–65%.



#### Session 8

#### Invited

Unraveling plasma self-organization of magnetron sputtering discharges

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The magnetron sputtering is a widely used plasma-based technique for deposition of highquality thin films. It is classified by the type of voltage that is applied to the cathode: DCMS (continuous voltage), HiPIMS (high-power impulse voltage) and RFMS (oscillatory voltage). The basic physics of the sputtering process is similar in all regimes. The crossed magnetic and electric field of a magnetron device confines electrons close to the cathode. The electrons gyrate around the magnetic field lines, bounce from the cathode sheath and drift in the **E**×**B** direction. Such field configuration greatly extends the trajectories of electrons and increases the probability of gas ionization. However, different motions of electrons (**E**×**B** direction) and ions (**E**-directed motion) cause spatial separation of charge, which consequently leads to the formation of azimuthally non-uniform plasma structures.

Dense azimuthal plasma structures called spokes have been reported for all types of magnetron sputtering regimes; DCMS [1], RFMS [2] and HiPIMS [3,4]. Spokes typically exhibit a triangular shape, form periodic or semi-periodic patterns and rotate in  $-E \times B$  direction (DCMS) or  $E \times B$  direction (HiPIMS). They were observed for various magnetron geometries (planar and cylindrical) and for a wide range of discharge conditions typically used in the deposition of thin films. The universal presence of spokes demonstrates that plasma self-organization is a fundamental process in magnetron sputtering physics. In the talk, I will review the latest findings about the self-organization of plasma in different magnetron regimes and discuss the properties of spokes for a range of discharge currents and working gas pressures.

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## Upscaling the plasma reactor for the treatment of seeds and seed-borne toxins in agriculture use

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In the scope of an industrial project, "Plasma Grain" (TRL 6-9), we have upscaled and constructed the plasma reactor to the industrial scale. A mobile plasma reactor was built, which fit in the back of the truck and treated large quantities of seeds (up to a kilogram per second). The innovative idea was to treat seeds on the spot and sow them in the soil right after plasma treatment, thus also reducing the CO<sub>2</sub> footprint due to significantly shortened transport routes.

The system was built with two differently coupled plasma reactors: an inductively coupled (IC) and capacitively coupled (CC). Seeds were treated with plasma to eliminate the seed-borne toxins (mycotoxins) for safe use in the food industry and as a feed for a stock. Other seeds were used for planting and were treated to inactivate the fungi and moulds from the surface; thus, a higher germination rate was expected.

Maize (a total of seven hybrids) was treated and sown in the Interkorn fields for two consecutive harvest years. Each year, plant numbers (no. of plants per m<sup>2</sup>) at emergence and at the tasselling time were recorded, as well as the final yield (kg/ha). Maize grown from untreated seeds served as the control treatment. In two of the four hybrids in the first harvest year, the statistically significant highest yield arose from plants from plasma-treated seeds. In the second harvest year, the highest yield was from plants from plasma-treated seeds in three of the four hybrids [1]. Preliminary results also showed great potential for mycotoxin decontamination from maize using IC plasma. HPLC showed up to a 90% decontamination rate of mycotoxins after plasma treatment.



Figure 1. Mobile plasma reactor for seed treatment.

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## Hydrogen interaction and oxide permeation barrier formation in B2-ordered Fe-Al intermetallic alloy

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The interaction of hydrogen with metals is a topic of growing importance due to the challenges in realizing the Hydrogen Economy. Ongoing advances in understanding the properties of metals and alloys have led to the engineered design of materials for targeted applications, including hydrogen storage and transport. [1,2] However, since the interaction of hydrogen with alloys can differ significantly from that with their pure metal constituents, predicting mass transport phenomena is troublesome, and systematic experimental investigations are required. Binary Fe-rich iron aluminide intermetallic alloys are well recognized as structural materials for engineering components operating in harsh environments at elevated temperatures. Upon exposure to air, a protective superficial alumina scale is formed on the surface of Fe-Al, which protects the underlying metal against external agents. [3] This work explores two critical aspects of the B2-ordered Fe3Al alloy: its interaction with hydrogen and the in-situ formation of a self forming hydrogen permeation barrier. The gas permeation technique was employed to determine the diffusivity and permeability from 200 to 500 °C, a range rarely explored in the literature. Our findings confirmed that the interaction of hydrogen with the Fe3Al alloy differs substantially from that of the pure metals involved. The in-situ formed aluminium oxide resulted in a highly impermeable layer with a permeation reduction factor greater than 1000. This straightforward method provided a very efficient hydrogen barrier with permeabilities that have not previously been reported [4], except for pure Al, which lacks the mechanical properties of the alloy.

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## 3D Printing of Semiconductors using Electron Tweezers

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Our research introduces a novel approach for fabricating germanium microstructures within an ultra-high-vacuum scanning electron microscope (UHV-SEM). The technique combines Electron Tweezers with the Vapour-Liquid-Solid process for growing of nanowires. Electron Tweezers enable precise manipulation of two-component liquid droplets using only the electron beam of the SEM. This phenomenon is not widely explored in current literature. Our research opens new possibilities for in situ manipulation and microfabrication in SEM.

The principle of **Electron Tweezers** manipulation is based on thermomigration. The electron beam exposure creates a local temperature gradient in a germanium substrate and induces a directional thermomigration of nearby AuGe or AlGe droplet. We propose that the temperature gradient in substrate causes a concentration gradient in droplets, leading to in dissolution-diffusion-deposition flow [1] of Ge atoms through the droplet (See Figure 1a).

A horizontal germanium **structure is 3D printed**, when during a deposition of Ge from an effusion cell, a droplet is very slowly moved by the electron beam using a Python script. Behind the slowly moving droplet, a horizontal wire grows through the VLS method. An example outcome of this 3D printing process is in Figure 1b.

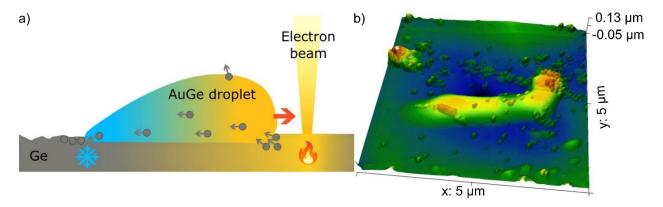


Figure 1. a) AuGe droplet on Ge substrate with temperature gradient induced by the electron beam exposure. The droplet is moving toward the warmer area, i.e., the droplet is following the electron beam. b) AFM image of a germanium structure 3D printed using an electron tweezers.

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#### Session 9

Plenary

## Practical Functional Oxide Thin Films for Electronic Devices

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Opportunities of functional oxides for applications in electronics are huge. However, oxides in electronics are quite scarce. The challenges stem from both intrinsic and extrinsic materials problem, e.g. composition, defect and interface control. Also, current thin film deposition routes cannot always deliver the performance of bulk materials. This talk looks at some of the reasons for the aforementioned challenges and shows ways to overcome them. Recent examples are given focusing on oxides for neuromorphic computing using resistive switching.



Invited

Nanoscale Ferroelectricity and Electrochemical Dynamics in Hafnia-Based Films

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The discovery of ferroelectricity in nanoscale films of binary oxides, particularly hafnia, has reignited interest in understanding the emergence of ferroelectric properties at the nanoscale. The unusual size scaling of polarization in these films is a focus of intense investigation. Given the sensitivity of polarization in hafnia-based films to the oxygen content, it has been hypothesized that polarization switching is intertwined with electrochemical reactions. Though these processes have been shown to co-occur, their relationship remains unclear. We conducted direct observations of the electrochemical state within regions exhibiting preset polarization directions in hafnia-based ferroelectric films, employing hard x-ray photoelectron spectroscopy (HAXPES). Our findings reveal more pronounced electrochemical changes in films with lower polarization, suggesting a lack of direct linkage between polarization and electrochemical changes. These results mark progress in unravelling the origins of the ferroelectric response in hafnia-based films.



## Fabrication of NbTiN superconducting nanowire single-photon detector

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Advances in optical quantum technologies have been achieved thanks to improvements in the performance of single-photon detectors. There are several types of detectors, but superconducting nanowire single-photon detectors (SNSPDs) have proven to be highly promising due to their impressive features, such as almost 100% quantum efficiency, timing jitter of only a few picoseconds, and count rates reaching 10<sup>9</sup> counts/s with minimal number of dark counts [1,2]. Thanks to these properties, SNSPD can be used in many areas, such as photonic quantum computers, quantum cryptography, quantum networks, and space research.

In this work, we describe fabrication technology, which we use to produce single-photon detectors based on a thin superconducting NbTiN film located on a suspended Si<sub>3</sub>N<sub>4</sub> membrane. The SNSPD consists of a nanowire (70-150 nm in width) patterned into a meander shape from an NbTiN layer prepared by magnetron co-sputtering. All technological steps must be precisely coordinated to achieve high detection efficiency. This includes designing the shape of the nanowire, i.e. the ratio of its width to the gap (filling factor). The proper composition of the NbTiN layer, as well as the thickness of the bottom reflective layer and the thickness of the membrane, plays a crucial role. Using an electron-beam lithography and an optimized dry etching process, we can repeatedly prepare detectors with a precise meandered shape and a critical temperature of around 11K. The meander was characterized using an AFM and SEM microscope and electrical measurements at low temperatures. The simulations show that thickness-dependent optical conductivity shifts the absorption peak of the nanowire, affecting its optical absorption.

#### Acknowledgments

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 J. Chang, J. W. N. Los, J. O. Tenorio-Pearl, N. Noordzij et al. APL Photonics, 6(3): 036114, 2021

This work has been supported by the European Union Digital Europe Programme (101091548), the Recovery and Resilience Plan of SR (1789/2023), by SRDA (No. APVV-20-0425) and Scientific Grant Agency of the SR (VEGA No. 2/0168/22). We thank Dušan Lorenc from International Laser Centre of SCSTI for coupling the detector to optical fiber.



Structure and growth of cobalt oxide epitaxial layers

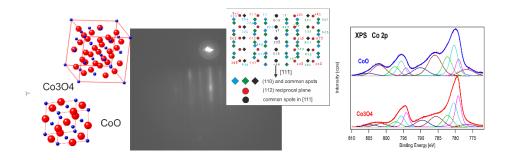
Karel Mašek, Oleksandr Leiko, Josef Mysliveček, Viktor Johánek

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Previous experiments performed on polycrystalline layers have shown that cobalt oxide layers exhibit interesting catalytic properties in methanol to hydrogen conversion [1]. However, basic research consisting in determination of fundamental physico-chemical properties is performed on model well-defined systems for which the relation between structural and chemical properties can be recognized. Thus, the preparation of the well-defined system and its complete structural description is the first of the studies.

In this work we investigate the growth and structure of well-ordered CoO and Co3O4 ultrathin films prepared on Cu(111) and Ir(100) single crystal surfaces by repeated cycles of metallic cobalt deposition, RF plasma oxidation, and temperature annealing. The films were investigated by Reflection High-Energy Electron Diffraction (RHEED), X-ray Photoelectron Spectroscopy (XPS) and other surface sensitive techniques to described their surface chemical composition and structure. The diffraction patterns revealed CoO cubic and Co3O4 spinel structures with (111) epitaxial plane parallel to the substrate surface. The homogeneous chemical state of the layers and different deposit-substrate interaction for copper and iridium were confirmed by XPS. The formation of cobalt oxide – substrate interface was deduced from RHEED patterns as well as from the XPS measurement. While we were able to prepare both types of cobalt oxide on iridium, only CoO layer has grown on the copper substrate. The results are compared to the layers prepared by Physical Vapor Deposition (PVD) [2].



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JVC-19 19<sup>th</sup> Joint Vacuum Conference

Session 10

#### Invited

Microbiologically induced corrosion of power plant steel components

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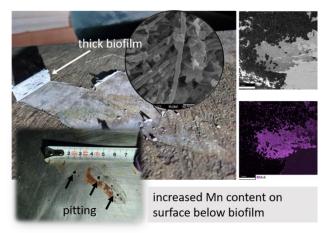
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Rivers are important sources for the production of green energy that can help reduce greenhouse gas emissions, both directly as a source of power for hydroelectric plants and as a cooling medium for nuclear power plants. In Slovenia 25 % of electric energy is produced by hydroelectric power plants, a large part on the Sava river. In addition, 40 % of electric energy is produced by the nuclear power plant Krško, that uses the Sava river for cooling purposes.

Corrosion is a significant threat to material lifespan in an aqueous environment, it can cause major damage to hydroelectric and nuclear power plants if not controlled. Most components are constructed from corrosion-resistant stainless steels, but even these can corrode under certain conditions through Microbiologically Induced Corrosion (MIC).

The data obtained in our research group indicate that MIC can cause a specific turbine blade degradation at hydroelectric power plants and heat exchanger in the nuclear power plant in the lower Sava river. The phenomenon has only occurred in the last few couple of years, and is related to changes in the environment, namely low water flows during the extended dry periods and rising water temperatures. For the time being the phenomenon appears to be locally induced as only one hydroelectric was severely affected. However, with increasing climate change it is expected that prolonged dry periods will be more frequent and when combined with increased river nutrient concentrations and temperatures, more power plants located along the lower Sava river will be affected. A similar phenomenon was observed in the proximally located nuclear power plant Krško cooling system where increased MIC problems were reported over the last couple of regular refits in the power plant.

This contribution will present our latest findings on MIC mechanisms in the power plants, discuss contributing environmental factors, and propose mitigation strategies to enhance the longevity and safety of these critical energy sources.





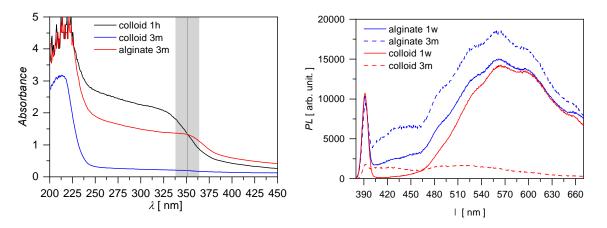
## Alginate-stabilized colloidal ZnO NPs for biomedical applications

## Kinga Kutasi<sup>1</sup>, Zsolt Tóth<sup>2</sup>

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The reactive oxygen and nitrogen species and NPs enriched hydrosols/hydrogels are expected to be good candidates for the RONS and metal ions' delivery in the case of different therapies, such as wound healing/disinfection or cancer therapy. It is also expected that hydrosol can retard the dissolution of colloidal NPs. Here we present the indirect plasma assisted deposition of RONS and ZnO NPs into alginate hydrosols. Accordingly, the alginate solutions are prepared from RONS and ZnO NPs enriched water. RONS are deposited into 32 ml of DIW with a surface-wave microwave discharge generated with the help of a surfatron wave launcher [1]. The liquid is positioned at 12 mm, from the edge of the discharge tube and treated with an argon discharge for 8 min. Under these conditions nitrite, nitrate and hydrogen peroxide of significant concentrations can be deposited into the treated water [1]. The acidification induced by the plasma treatment is neutralized by using high reduction Zn metal powder during plasma treatment, thus assuring also the stability of the deposited RONS [2]. The ZnO NPs are deposited into the filtrated RONS enriched water by laser ablation of Zn foil [3]. The 1064 nm Nd:YAG laser is focused using a 250 mm lens on the target positioned from the lens at 125 mm distance in 20 ml solution. The energy delivered to the target surface is 320 mJ, the duration of the ablation is 60 s with 6 ns pulses at 20 Hz repetition rate. It has also been shown recently, that in the alkaline RONS enriched liquid the dissolution of ZnO NPs is retarded. Herein it is demonstrated, that the ZnO NPs embedded into alginate hydrosol are stable for months, as shown by the UV-VIS absorption spectra and photoluminescence emission of DIW and alginate based colloids, respectively, presented in the Figure below.



Acknowledgement: This work was supported by Hungarian NKFIH Science Founds K-132158.

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Oral

Atmospheric plasmas for depositing sulfonated plasma-polymerized films for use in

## liquid contacts

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The various forms of oxidized sulfur, such as sulfate  $(SO_4^{2-})$ , or sulfonic acid  $(SO_3H)$  group, exhibit surface functionalities, which are of interest to biomaterials, sensors, fuel cells, and ion exchange membranes for liquid separations<sup>1,2</sup>. Sulfonated thin films are often deposited on porous membranes or polymer substrates for use in liquid-coating contact applications, rather than on smooth surfaces such as glasses, Si wafers, or metals. In the case of the application of the coatings in contact with liquids, the stability of the sulfonic groups  $(SO_3^{-})$  in the plasma-polymerized coatings on Si wafers has never been realized<sup>2,3</sup>.

This study investigates the stability of sulfonated coatings when exposed to liquids. Using dielectric barrier discharge (DBD) plasmas at atmospheric pressure, sulfonated coatings have been synthesized on Si wafers through a plasma polymerization process. As a precursor for the deposition of sulfonated plasma-polymerized coatings on Si wafers, a combination of "Acrylic Acid (AA)+ H<sub>2</sub>SO<sub>4</sub>+ Sulfur" is used. The stability of deposited coatings has been evaluated when in contact with an acidic (pH=5.5) buffer solution, they have found to have good adhesion to substrates, hydrophilicity and physical stability. X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR-IRRAS) have been used to evaluate the chemical stability, while profilometry was used to determine the film thickness (40-120 nm). As a result of both FTIR and XPS analysis, it is confirmed that the grafted functional groups, SO<sub>x</sub>(H) and -COOH, could sustain in the coating after the flushing of buffer solution for 200 hours, though the atomic percentage of sulfur has been stabilized at 1.15 % compared to 3.25% prior to the flushing. Finally, compared to previously reported methods<sup>2,4</sup>, this method is simple to set up, inexpensive, and capable of producing highly stable sulfonated coatings.

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Plasma-generated vacuum-UV radiation for inactivation of viruses in water

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With an increasingly intense anthropogenic activity, it is becoming more difficult to ensure a safe supply of purified water. Standard water disinfection methods such as chlorination, filtration or UV radiation all have certain shortcomings related to their efficiency towards different contaminants, cost, and production of harmful by-products. Advanced oxidation processes rely on in-situ production of chemically reactive species, such as hydroxyl radicals (OH<sup>•</sup>), and have been gaining significant attention as a novel technique for water purification, since they react rapidly and indiscriminately against all organic contaminants. There are several methods for producing OH<sup>•</sup> radicals in water. One of which is by utilizing photons in the vacuum ultraviolet (V-UV) wavelength region (100–200 nm), which are energetic enough to break down water molecules into hydrogen and OH<sup>•</sup> radicals. However, their application to liquid water presents a technical limitation due to their interaction with various substances. Our study utilized a special, hermetically sealed chamber, with an MgF<sub>2</sub> window facing the low-pressure plasma radiation to treat water containing MS2 bacteriophages. By varying the applied power, gas inside the chamber, and the amount of V-UV exposure and OH<sup>•</sup> scavengers, we demonstrated that 1) V-UV intensity influences the efficiency of virus inactivation, 2) ozone is produced when air is present in the treatment chamber and, with the aid of UV radiation, decomposes to OH<sup>•</sup> radicals in water to contribute to the inactivation of viruses. With optimal treatment parameters, the technique could inactivate 9 log<sub>10</sub> viruses in under a minute. The study provided essential information for further understanding of such treatment approaches.

Acknowledgments:

The authors acknowledge the financial support from the Slovenian Research Agency (research core funding No. P2-0082) and research project No. L2-2617



Session 11

#### Invited

## Revealing nanoscale mechanism in the jumping crystals

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Thermosalient materials (colloquially known as "jumping crystals") are a class of solidstate materials that exhibit mechanical motion, such as bending, twisting, or even jumping, when heated or cooled. These materials undergo solid-to-solid phase transitions when exposed to changes in temperature that involve a reorganization of the crystal lattice without melting, leading to a rapid release or absorption of mechanical energy. Jumping crystals are typically anisotropic which leads to differential expansion or contraction when the material undergoes a phase transition and customarily exhibit uni- or bi-axial negative thermal expansion. Due to their ability to produce rapid and significant mechanical movements they can be used as nanoscale actuators in microelectrochemical systems, temperature sensors, energy harvesting devices, smart materials and devices or for pharmaceutical and drug storage and delivery.

This fascinating phenomenon was comprehensively studied in the anticholinergic agent oxitropium bromide. The thermosalient effect, manifested in forceful jumps of up to several centimetres, was investigated by a combination of structural, microscopic, spectroscopic, and thermoanalytical techniques, providing data on which to base a proposed nanoscale mechanism for the phenomenon. Direct observation of the effect in a single crystal and structure determination of both phases revealed that the jumping of the crystals is a macroscopic manifestation of a highly anisotropic change in the cell volume characterized by the negative uniaxial thermal expansion. The cell distortion is accompanied by a conformational change of the oxitropium cation, which triggers increased separation between the ion pairs in the lattice at nearly identical separation between the cation and the anion within each ion pair. At the molecular level, the cation acts as a molecular shuttle composed of two rigid parts (epoxy-aza-tricyclic-nonyl portion and phenyl ring) that are bridged by a flexible ester linkage. The structure of the rigid, inert aza-tricyclic portion remains practically unaffected by the temperature, suggesting a mechanism in which the large, thermally accumulated strain is transferred over the ester bridge to the phenyl ring, which rotates to trigger the phase transition.

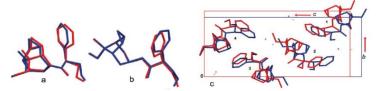


Fig 1. Structures of phase A (OXTBA, blue) and phase B (OXTB-B, red) of oxitropium bromide

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<sup>2.</sup> Skoko, Ž. et al. J. Cryst. Growth 2017, 17, 4445



#### Oral

Radical-assisted covalent binding of biomolecules to amine plasma polymers

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Even though synthetic polymers are promising materials for bioapplications, lacking biocompatibility hinders their applicability. Coating such material with a thin film of plasma polymer (PP) containing polar groups increases the surface free energy, making the polymer hydrophilic. This facilitates immobilization of proteins or enzymes, as well as cell attachment and growth. Our previous publications showed that coating polycaprolactone nanofibers with amine PPs or carboxyl PPs with bound platelet-rich plasma highly improves cell viability [1, 2]. Immobilization of biomolecules is often mediated by chemical coupling through a linker to avoid nonspecific binding. However, recent studies [3] have shown that the deposition of amine PPs using enhanced ion bombardment produces a high number of free radicals, which are capable of covalently binding biomolecules to the PP surface without losing functionality. In this work, we proved abundant, long-lived radicals even for amine PPs prepared by a typical low-pressure plasma polymerization (with no additional ion bombardment). Although the radical density was the highest directly after the deposition, PPs preserved 60% of the original radical amount after 3 months of storage. The decrease was attributed to radical recombination in bulk or their diffusion to the PP surface, followed by reactions with adsorbed molecules. Interestingly, the radical density does not scale with the average discharge power but with the on-time power, and the deposition rate is similar during the off-time and ontime. It means that the radicals in the material grown during the off-time are created in the on-time because of the ion penetration effect. Finally, as proof of concept, we demonstrated a radical-assisted direct immobilization of a biomolecule, bovine serum albumin, to amine PPcoated quartz crystal microbalances.

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## Exploring the effects and stability of organic molecule modifications on CVD-synthesized monolayer MoS<sub>2</sub>

Antun Lovro Brkić<sup>1</sup>, Antonio Supina<sup>1</sup>, Davor Čapeta<sup>1</sup>, Lucija Dončević<sup>2</sup>, Lucija Ptiček<sup>3</sup>, Šimun Mandić<sup>1</sup>,

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Even though two-dimensional (2D) materials are promising candidates for the next generation of devices due to their unique properties, there are several issues that need to be addressed before these materials can be implemented in commercial devices. Firstly, precise and controlled synthesis and methods of manipulation are necessary for their successful integration, as well as the ability to fine tune the properties of interest like doping, band gap, or optical response. Several ways of tuning the properties of 2D materials are extensively researched, main ones being mechanical modulation and functionalization (e.g. with molecular patterning). In our research, we are interested in the modification of the 2D materials' properties by utilizing a layer of organic molecules.

Starting point of our research was the synthesis using chemical vapor deposition (CVD), transfer and characterization of monolayer MoS<sub>2</sub> samples. Next step was studying effects of prolonged exposure to ambient conditions and different solvents, in order to develop optimal procedures and to distinguishing the effects of the solvent from those of the organic molecules. Under the course of our research, we developed a simple method for modification of 2D materials by deposition of the organic molecule solution on the 2D material [1]. We further investigated the stability of our modified samples, as well as the reversibility of this modification. By analyzing the optical signatures of the samples using photoluminescence spectroscopy (PL), Raman spectroscopy, and surface quality using atomic force microscopy (AFM), we demonstrate that the modification of 2D MoS<sub>2</sub> with organic molecules leads to a stable surface modification that retains its optical signature, while heating of the modified samples restores their original optical signatures, indicating the re-establishment of the optical properties of the pristine MoS<sub>2</sub>.

### [1] A. L. Brkić et al., Nanomaterials 13, 2115 (2023)



### Poster session 1

## ALD HfZrOx and InGaOx Thin Films for Flash Memory Application

### <u>Changhwan Choi<sup>1</sup></u>, Boncheol Ku<sup>1</sup>, Kyung Soo Park<sup>1</sup> and Hyun-Yong Yu<sup>2</sup>

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Flash memory device continuously needs high bit density with small footprint, and costeffective process as technology node advances. Memory performance has been improved using adopting various materials and innovative device structure. However, there are still several challenges such as high operating voltage, low programming speed, and difficult cell size scaling, which requires alternative materials, device structures and advanced circuit design. Among alternative materials being investigated to replace the current charge trapping layer (Si<sub>3</sub>N<sub>4</sub>) and channel material (poly-Si), ferroelectric and oxide semiconductor materials have garnered a great interest. Ferroelectric thin film can be adopted due to scalability and operating power reduction compared to current SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub>/SiO<sub>2</sub> (ONO) layer. In addition, oxide semiconductor thin film can be used as a channel materials thanks to high mobility and amorphous properties. Nonetheless, further research should be attempted before they are adopted in 3D flash memory device structure.

We demonstrated 3D vertical Gate-All-Around (GAA) vertical-channel FeFET using ferroelectric (FE) Zr-doped HfO<sub>2</sub> (HfZrOx) and indium-gallium oxide (InGaOx) channel. Subthreshold swing of 90 mV/dec, large memory window (MW) of 2.5V, 2 bits/cell, and reliable 10-years retention are reported. Our result indicate that ferroelectric and oxide semiconductor materials can be promising candidates to overcome the current ONO and poly-Si materials in 3D NAND flash memory device.

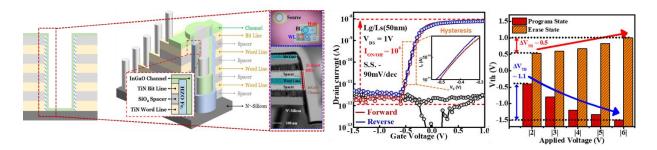


Figure: final 3D flash memory device structure, and their corresponding I-V, and memory characteristics [Ref] B. Ku et al, International Memory Workshop (IMW), 2024

JVC-19 & 30<sup>th</sup> ISMVST 29.9-4.10.2024 Podstrana, Croatia

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P1



## Photocatalytic activity of TiO2 thin films embedded with Cu nanoparticles prepared by atomic layer deposition technique

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Among the various metal oxide semiconductors, titanium dioxide (TiO2) has emerged as the most promising material for photocatalytic applications due to its excellent properties such as high chemical stability, non-toxicity, and suitable energy positions of the conduction and valence band edges. However, the biggest problem in the photocatalytic usage of TiO2 is its wide band gap of about 3.2 eV, which limits the optical absorption in the material only to UV light. One of the methods to increase the photocatalytic efficiency of TiO2 is to use metal nanoparticles coated on the surface, or embedded in the matrix of the semiconductor material. In this way, the absorption of visible light in the material is increased due to the effect of localized surface plasmon resonance in the metal nanoparticles. In this work, we have employed atomic layer deposition (ALD) method to synthesize a composite material consisting of Cu nanoparticles embedded in TiO2 thin films. The concentration of Cu nanoparticles was controlled by the number of pulses of the copper precursor during the ALD growth of the material. The chemical composition of the Cu-TiO2 thin films was determined by x-ray photoelectron spectroscopy (XPS), while the surface morphology of the samples was investigated in detail by scanning electron microscopy (SEM). In addition, the crystal structure of TiO2 films embedded with Cu was determined by x-ray diffraction (XRD) measurements. Lastly, the photocatalytic efficiency of the ALD-grown samples was evaluated by monitoring the decomposition of methylene blue in aqueous solution under simulated solar irradiation. TiO2 samples containing copper show an almost twofold increase in the photocatalytic activity, compared to pure TiO2 films, demonstrating the highly beneficial effect of Cu nanoparticles on the applications of TiO2 for organic dye degradation.



## High-Temperature Tribology of Multilayered CrN/(Cr,V)N/VN Protective Coatings

## Domen Korbar<sup>1,2</sup>, <u>Aljaž Drnovšek<sup>1</sup></u>, Sandra Drev<sup>3</sup>, Miha Čekada<sup>1</sup>

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With the advancement of modern high-speed machining and molding technologies, there is a growing demand for protective hard coatings that exhibit excellent tribological performance across various temperatures. CrN coatings pose high oxidation resistance, anti-corrosive and anti-adhesive properties. However, CrN coatings still face challenges due to relatively low hardness and poor tribological performance. Combining CrN with other nitrides via solid solution or multi-layering has proven to be an effective strategy to improve these properties. Recently, VN has attracted increasing interest because it quickly oxidizes to form Magnéliphase vanadium oxides, which have easy slipping shear planes and low melting point, thus becoming lubricious under stress and high temperature.

To study tribological properties at elevated temperatures, we deposited multilayered CrN/(Cr,V)N/VN coatings. All the coatings in our study were deposited using an industrial DC-magnetron sputtering system. We used two triangle-like segmental Cr/V targets, one chromium, and one vanadium target. This unique setup allowed us to produce coatings with different Cr and V concentrations in a single deposition process by placing samples on different heights of the vertical axis in the chamber. Ball-on-disk tests were performed on a high-temperature tribometer at room temperature, 300, 600, and 700 °C. Results show a decrease in friction coefficient at maximum temperature. These coatings' mechanical and structural properties before and after the tribological tests were studied using nanoindentation, SEM, and TEM.

Ρ3



## Pressure dependent nonlinear spectral broadening of ultrashort laser pulses in hollow-core fiber (HCF)

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Hollow-core fibers (HCFs) are a remarkable type of optical fiber that have significantly transformed light transmission and control in various photonic applications. Unlike traditional optical fibers that guide light through a solid glass core, HCFs utilize a hollow core, surrounded by a microstructured cladding. This distinctive structure greatly reduces the interaction between light and the fiber material, allowing for exceptionally low-loss light transmission, particularly at wavelengths where conventional fibers typically face challenges due to absorption and scattering. The use of hollow-core fibers with laser pulses has opened new frontiers in fields such as high-power laser delivery, nonlinear optics, and ultrafast pulse propagation.

Manipulation of ultrashort pulses can be achieved by pulse propagation through the fiber core in vacuum conditions or through a noble gas-filled core. High pulse energies, noble gas characteristics, and the geometry of the fiber core can lead to a series of nonlinear effects such as self-phase modulation and four-wave mixing that change the spectrum of the pulse. We aim to utilize modified laser pulses in ultrafast spectroscopy techniques such as femtosecond transient absorption (fs-TA) and two-dimensional electronic spectroscopy (2DES), where the spectrum of the pulse, and consequently the pulse duration, play a crucial role. Here, we investigate the spectral and temporal characteristics of pulses propagated through HCF using the Frequency-Resolved Optical Gating (FROG) technique, depending on noble gas pressure and incident pulse energies. Using an optical compressor, we managed to compress pulses propagated through the fiber to a pulse duration of less than 15 fs.

**P4** 



## 

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Investigating strain in two-dimensional (2D) materials is particularly compelling due to its potential to modify their lattice constants, lattice vibrations, thermal conductivity, electronic and optical properties, as well as their chemical reactivity and overall device performance. However, the field of "straintronics" faces significant challenges. Most research relies on spatially averaging techniques that fail to provide insights into local mechanical and electronic properties. Additionally, creating crystallographically precise, tunable strain and forming narrow strain-induced junctions for nanoscale device applications remain formidable tasks.

To address these challenges and systematically explore the effects of strain on specific 2D materials and their heterostructures, we have developed a novel approach. We grow monolayer transition metal dichalcogenides on pre-patterned, stepped substrates, where the 2D material bends over step edges. This method enables precise control over the crystallographic direction of applied strain and facilitates the formation of sharp lateral junctions with finely tuned band gaps and work function variations. As a case study, we present the synthesis of monolayer MoS<sub>2</sub> on a graphene-covered Ir(332) substrate using molecular beam epitaxy (MBE) under ultra-high vacuum (UHV) conditions. Scanning tunneling microscopy (STM) and Atomic force microscopy (AFM) topography images confirm the successful growth of  $MoS_2$  islands on the gr/Ir(332) substrate, with individual islands seamlessly traversing multiple step edges and bending accordingly. Further characterization using Kelvin probe force microscopy (KPFM), nano Fourier transform infrared spectroscopy (nano-FTIR), and Raman spectroscopy reveals the presence of strain within the system and significant changes in work function induced by MoS<sub>2</sub>. This work highlights a successful bottom-up approach for structurally modifying and straining 2D materials, with promising implications for future applications in nanoscale devices.

Keywords: 2D materials, MoS<sub>2</sub>, MBE growth, intercalation, AFM imaging

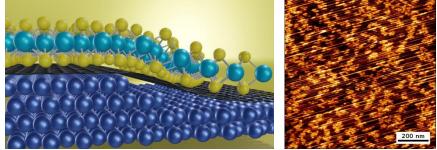


Figure 1. Left, a model of  $MoS_2$  island covering atomic steps present on gr/Ir(332) substrate, right, an AFM topography of a  $MoS_2/gr/Ir(332)$  sample.

Ρ5



## Characterization of Alumina Scales on Fe-Al Alloys: Enhancing High-Temperature Hydrogen Permeability Resistance

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Binary Fe-Al alloys are pivotal high-temperature structural materials known for their outstanding corrosion and wear resistance. Their reduced weight compared to structural steels makes them attractive for various applications. A key feature of these alloys is the formation of a protective alumina ( $Al_2O_3$ ) scale on the surface when exposed to oxygen-rich environments, which significantly enhances their performance. This study focuses on the characterization of alumina scales formed on a Fe32.5Al alloy, particularly in the context of their impact on hydrogen permeability.

The investigation began with a Fe32.5Al cast sample, which was oxidized in a controlled oxidative environment and heated to 500°C. Initially, a thin oxide layer (~1.5 nm) was formed during thermal treatment in high vacuum, as estimated from XPS analysis. This initial layer showed a slight reduction in hydrogen permeation. Subsequent oxidation in pure oxygen at 500°C resulted in a denser, more impermeable oxide layer (~5 nm), achieving a Permeation Reduction Factor (PRF) >1000, marking the lowest effective permeability ever recorded on a metal substrate at 400°C.

Characterization techniques such as XPS and SEM were employed to analyze the composition and morphology of the alumina scales. The results revealed that the initial oxide layer was amorphous, while the oxide formed at 500°C was highly crystalline, indicating a phase transformation to  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. This transformation is significant for the material's permeability properties, as the dense crystalline alumina layer provides an effective barrier against hydrogen permeation.

The findings underscore the importance of controlled oxidation processes in developing alumina scales with optimal protective properties. By achieving a dense and crystalline alumina layer, Fe-Al alloys can be enhanced for high-temperature applications, combining lightweight characteristics with superior resistance to hydrogen permeation. This study provides critical insights into the interplay between material characterization and functional performance, paving the way for advanced applications of Fe-Al alloys.

Acknowledgement: Slovenian Research and Innovation Agency (ARIS), research core funding P1-0417, Research project no. J2-440.

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P6



## High resolution, compact Raman spectrometer based on CMOS camera

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We present a Raman spectrometer that reaches spatial resolution of <1  $\mu$ m (dependent on the focusing objective); is cost-effective and provides high spectral resolution of 2.6 cm-1. Good performance has been the achieved by use of high grade optical components for Raman beam coupling to the entrance slit of the spectrometer, and later, and the choice of an industrial-grade uncooled CMOS camera as detector. The camera has good quantum efficiency (~70%) and low readout noise. The drawback of using uncooled camera is that the integration time is limited to less than 10s due to higher dark current. Moreover, by characterization with the 2D MoS2 samples, we have demonstrated the spectrometer has throughput and resolution comparable to research-grade confocal Raman microscopy systems. The reduction of cost and size compared to research-grade commercial devices will enable an even wider spread of Raman spectroscopy for real time monitoring of chemical reactions or deposition processes.

P7



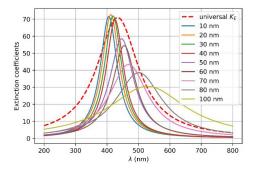
## Determination of dielectric functions of colloidal silver nanoparticles in LSPR relevant wavelength range

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Importance of dielectric functions in context of interaction of light with nanoscale matter is huge since they govern all optical properties [1]: dispersion, absorption, scattering, reflection, transmitance, polarization etc. In this work, the method for extraction of dielectric functions from UV-Vis spectra of colloidal silver nanoparticles is presented as a bypass to standard experimental techniques like ellipsometry [2] in constrained wavelength range [400..600] nm. The approach is based on developed Mie scattering fitting function with additional conditions on dielectric functions behaviour at localized surface plasmon resonance wavelength (LSPR) [3]. It allows extraction of universal dielectric functions which are valid for *a priori* unkown sizes of silver nanoparticles in diameter range [20..100] nm as well as dielectric functions of fixed size silver nanoparticles. Once known, these dielectric functions can be used for direct fit of UV-Vis spectra to determine size and consequently concentration of colloidal silver nanoparticles. The obtained results allow qualitative insights into electronic structure and dynamics of electronic motion in silver nanoparticles under influence of external EM field [4].



**Figure 1.** Mie extinction coefficients  $K_E(\lambda)$  for monodisperse colloidal silver nanoparticles in size range [10..100] nm. Dashed curve shows universal  $K_E(\lambda)$  which substitutes all other curves in fitting function for UV-Vis spectra.

- [1] D. J. Fabian, *Solid-state spectroscopy*, vol. 285, no. 5761. 1980.
- [2] H. U. Yang, J. D'Archangel, M. L. Sundheimer, E. Tucker, G. D. Boreman, and M. B. Raschke, "Optical dielectric function of silver," *Phys. Rev. B Condens. Matter Mater. Phys.*, vol. 91, no. 23, 2015, doi: 10.1103/PhysRevB.91.235137.
- [3] J. Car and N. Krstulović, "Fitting Procedure to Reconstruct the Size Distribution and the Concentration of Silver Colloidal Nanoparticles from UV-Vis Spectra," Nanomaterials 2022, 12, 3302. https://doi.org/10.3390/ nano12193302
- [4] Y. He and T. Zeng, "First-principles study and model of dielectric functions of silver nanoparticles," J. Phys. Chem. C, vol. 114, no. 42, pp. 18023–18030, 2010, doi: 10.1021/jp101598j.



## Effect of Sputtering Power on Low Concentration Impurities in Binary Oxides for Semiconductor Applications : A ToF-ERDA Characterization Study

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The performance of semiconductors is heavily influenced by impurity concentrations. Power density is one of sputtering parameters that significantly affects these impurities. Higher power density reduces the amount of contaminants in the thin film. This study looks at how power density in magnetron sputtering impacts hydrogen, carbon, and other impurities in silicon, titanium, copper, silver, and palladium binary oxides, which are common in the semiconductor industry.

We focused on a narrow power density range of 200-300W using DC reactive magnetron sputtering on a 3-inch target. Six samples of each material were prepared under fixed conditions of 5\*10^-3 mbar pressure in an Argon and Oxygen atmosphere with a flow rate of 40/10 sccm. Data analysis was done on films of the same thickness, ignoring the surfaces of both the substrate and the film.

Using Time-of-Flight Elastic Recoil Detection Analysis (ToF-ERDA), we could detect very low but important changes in impurity concentrations and understand how they relate to the sputtering power density. This detailed investigation into low impurity levels has not been done to this depth before. The results offer new insights into optimizing deposition parameters to improve film purity and performance.

The Slovak Research and Development Agency, grant No. APVV-20-0220.

P9



## Low-energy ion bombardment induced reduction of TiO<sub>2</sub> grown by Atomic Layer Deposition

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Titanium dioxide (TiO<sub>2</sub>) is one of the most studied photocatalytic materials, known for its numerous advantages over other semiconductor materials, such as chemical stability, non-toxicity, biocompatibility, and availability. Recent research has demonstrated that structural defects in TiO<sub>2</sub> significantly affect its physical and chemical properties. These include electrical, optical, and mechanical characteristics, its reactivity, surface chemistry, and the ability to participate in chemical reactions. Oxygen vacancies are the most significant defects in TiO<sub>2</sub>, making the controlled creation of these point defects a common technique for modifying its properties. Low-energy ion bombardment is a frequently employed approach for introducing structural defects in metal oxides and creating new structural and chemical phases and nanostructured surfaces [1, 2].

In this study, the anatase TiO<sub>2</sub> thin films synthesized by Atomic Layer Deposition (ALD) were modified by the low-energy  $H_2^+$  and  $Ar^+$  ion bombardment at room temperature. The bombardment was carried out inside an ultra-high vacuum chamber of an X-ray Photoelectron Spectroscopy (XPS) instrument. The impact of ion bombardment on the chemical composition of  $TiO_2$  surfaces as a function of bombardment time was assessed by measuring photoemission spectra around the Ti 2p and O 1s core-levels, and the valance band. The relative fractions of chemical states of Ti and O atoms as a function of bombardment time were determined from the numerical deconvolutions of the corresponding photoemission lines. Secondary Ion Mass Spectrometry (SIMS) was employed for in-depth elemental analysis and film thickness measurements. In addition, the crystalline structure of the surface region of the irradiated films was determined by Grazing Incidence Xray Diffraction (GIXRD), while the impact of ion bombardment on the surface morphology and film thickness was studied with Scanning Electron Microscopy (SEM). The observed differences in oxide reduction kinetics under  $Ar^+$  and  $H_2^+$  ion bombardment are explained, on one hand, by the high preferential sputtering under Ar bombardment and, on the other, by the high chemical reactivity of hydrogen.

<sup>[1]</sup> M.I. Nandasiri et al., Instability of Hydrogenated TiO2, J. Phys. Chem. Lett., 6 (2015) 4627-4632.

<sup>[2]</sup> B.M. Pabón *et al.*, Formation of titanium monoxide (001) single-crystalline thin film induced by ion bombardment of titanium dioxide (110), Nat. Commun., 6 (2015) 6147.



P11

# Visualizing intercalation of 2D material using AFM based techniques: MoS2 on graphene/Ir(111) case study

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Two-dimensional (2D) materials, especially transition metal dichalcogenides (TMDs), exhibit remarkable physical properties such as 2D superconductivity, magnetism and bandgaps tunable via functionalization, strain, and substrate interactions [1]. Intercalation, particularly self intercalation, influences TMD-substrate interactions, altering their electronic structure [2]. While scanning tunneling microscopy (STM) offers atomically resolved insights, it is limited in scan area and requires ultra-high vacuum conditions. We demonstrate that atomic force microscopy (AFM) with its advanced optical and electrical modes can be used as a method to visualize local intercalation on a larger scale. This capability is showcased using molybdenum disulfide (MoS2) grown on top of graphene (Gr) on Ir(111) substrate. MoS2 islands exhibit distinct coalesced morphology and lower coverage near graphene wrinkles due to sulfur intercalation which weakens the MoS2-Gr interaction. AFM phase imaging reveals noticeable contrast around wrinkles, quantitatively confirmed by a local drop in Young's modulus. Kelvin probe force microscopy (KPFM) maps out intercalated areas based on intercalation induced local changes in electronic structure of both Gr and MoS2 [2], likely causing observed variations in the work function in the vicinity of wrinkles. Photo-induced force microscopy (PiFM) further delineates intercalated regions in this 2D heterostructure. Our results show the versatility of AFM based techniques in visualizing atomic-scale phenomena and emphasize the importance of micrometer scale imaging techniques for studying materials grown by molecular-beam epitaxy (MBE).

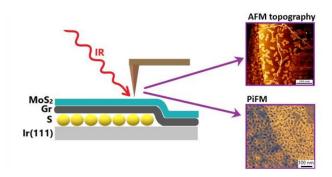


Figure 1. A schematic illustration of MoS2/Gr/Ir(111) heterostructure partially intercalated with Sulphur, alongside the representative AFM topography and PiFM image.

[1] Monga, D., et al. Materials Today Chemistry 2021, 19, 100399.

[2] Pielić, B., et al. Tailoring the intrinsic interactions in a two-dimensional multivalley semiconductor by selfintercalation. npj 2D Materials and Applications Journal Specifications (accepted for publication).



UV and solar-driven photocatalysis of organic dyes using ZnO-Ag heterojunction nanoparticles synthesized by one-step laser synthesis in water

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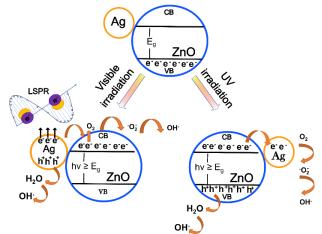
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In this research, ZnO-Ag heterojunction nanoparticles (NPs) were obtained with a novel onestep pulsed laser ablation (PLA) synthesis technique [1]. ZnO-Ag NPs were obtained by simultaneously ablating ZnO and Ag targets in water using a pulsed Nd:YAG laser, achieving facile and clean synthesis. The PL spectra show that ZnO-Ag NPs have a lower recombination rate of the e<sup>-</sup> - h<sup>+</sup> pairs, implying a higher photocatalytic activity. The photocatalytic efficiency of ZnO-Ag NPs was carried out under UV and solar irradiation against Methylene Blue, Rhodamine B, Crystal Violet, and Methyl Orange dyes. It was confirmed that the presence of Ag increases the photocatalytic efficiency against all studied pollutants due to the prolonged charge separation and the existence of localized plasmon surface resonance which enables solar-driven photocatalysis. Ultrafast transient absorption spectroscopy was employed to understand the charge carrier's photoexcitation process involved in the ZnO-Ag NPs photocatalytic route under the influence of visible irradiation. This research demonstrates a simple one-step synthesis of Ag-ZnO heterojunctions via the PLAL method for efficient solardriven photocatalysis against various organic pollutants.



**Figure 1**. Schematic illustration of ZnO-Ag photodegradation mechanism under UV and visible irradiation. [1] R. Radičić et al. Appl. Surf. Sci. 669 (2024) 160498

P12



Application of electrochemical impedance spectroscopy in studying the mechanism of metal oxide nanoparticles used in ion-selective electrodes

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In recent years, metal and metal oxide nanoparticles (MONPs) have gained huge popularity among scientists who are still developing new methods of obtaining them and looking for new areas of application. One of applications is using the nanoparticles in the construction of electrochemical sensors. The research of ion-selective electrodes with solid contact (SCISEs) as a layer of MONPs (copper, iron and zinc oxides) were conducted, while electrochemical impedance spectroscopy (EIS) method was used to examine their electrical parameters.

The EIS is becoming increasingly popular and is mainly used as a tool for studying the electrical and electrochemical properties of materials and systems. It consists in applying an electrical impulse (of known intensity or voltage) to the tested electrodes and observing the obtained response. EIS enables the separation and characterization of kinetic processes occurring in different electrochemical systems. In relation to ISEs, it is a fast and convenient method that allows for the study of charge transfer kinetics at the interface between the ISM and the internal electrode. It allows for the optimization, characterization and monitoring work of ISM, and also determination of many parameters, such as double layer capacitance or bulk membrane and charge transfer resistance. Moreover, thanks to using this method, it is possible to determine whether the tested sensors are still working properly or need additional calibration.

The obtained results allowed to prove that the presence of the MONPs intermediate layer in the SCISEs construction caused a decrease in the bulk resistance of the membrane and an increase in the capacitance of the double layer. The charge transport across the interface was also faster and easier, what resulted in better potential stability and increased durability of the electrodes over time [1].

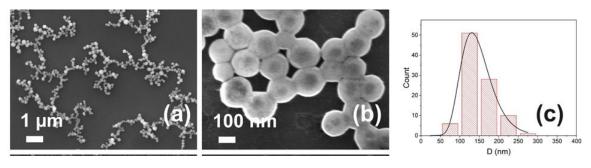


Fig. SEM images of ZnONPs (a,b) with corresponding size-distribution (c).

[1] K. Pietrzak, N. Krstulović, D. Blažeka, J. Car, S. Malinowski, C. Wardak, Metal oxide nanoparticles as solid contact in ion-selective electrodes sensitive to potassium ions, Talanta 243 (2022) 123335



## High entropy nitrides: High temperature study of Cr–Hf–Mo–Ta–W–N thin films

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In general, high entropy alloys (HEAs) are multicomponent materials containing at least five principal elements. The research on HEAs has also been extended to cover multicomponent ceramics, such as oxides, nitrides, carbides, and borides, due to their promising properties. High entropy ceramics can exhibit very good mechanical properties at low as well as high temperatures, outstanding structural stability, and high oxidation resistance. This makes them promising candidates for next-generation replacements of traditional materials in many areas of the industry.

In present study, we focused on the structure and properties of high entropy nitrides (HENs) from the Cr–Hf–Mo–Ta–W system. Cr–Hf–Mo–Ta–W–N films were prepared by magnetron sputtering at ambient temperature and at elevated temperature of 700°C. During deposition, the nitrogen flow was fixed to 20 sccm, while the target composition was changed in order to prepare films with a wide range of the elemental compositions. The nitrogen content in the films was in the range of 35 – 52 at.%, and the content of metals in the films was changing in a wide range. After deposition the elemental composition, structure and mechanical properties were investigated. Subsequently, the films were annealed in a vacuum to temperatures up to 1200°C and after cooling down to room temperature the structure and mechanical properties were examined.

It was found that appropriate deposition conditions result in the preparation of HEN films with a simple FCC structure given by the employed elements typical for high entropy nitrides. High hardness of 20 GPa of these films in the as-deposited state was retained even after annealing to 1200°C in a vacuum. Changes in the structure after annealing will also be discussed.



## Structural investigation of the heat-treated Ti/Al/TiN/Au contact layer on n-GaN - role of TiN

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In this work, we present the structure of the conventionally used Ti/Al/TiN/Au/n-GaN contact layer as a result of heat treatment for different TiN thicknesses. The Ti/Al/TiN/Au contact layer is the standard ohmic contact to n-type GaN. The ohmic character of the Ti/Al/TiN/Au/n-GaN junction can be achieved after heat treatment at 700°C in nitrogen. The complex layer system formed as a result of heat treatment has been investigated in numerous studies. However, commonly while analyzing XRD patterns of the junction, the possibility that the TiN layer would not act as a diffusion barrier in case of heat treatment above 500°C is not taken into account. Hence the XRD peaks have been usually explained by the formation of Al-Ti alloy layers. In the publications in which TEM investigations were also performed, heat-treated layers without an Au layer were generally examined, so the role of Au could not be explored. Our TEM investigations showed that in fact TiN does not act as a diffusion barrier layer in a full ohmic contact stack and that both Au and Al diffuse through it, as shown in Figure 1, which illustrates the diffusion processes. Additionally, we show that N leaves GaN enabling the formation of AIN and TiN layers on the surface of GaN. In addition, an Au<sub>2</sub>Al alloy layer is also formed and a spinel-type Al<sub>2.67</sub>O<sub>4</sub> phase can be observed on the surface of the sample, which can cause roughening of the surface.

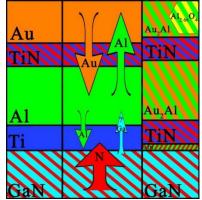


Figure 1. Diffusion processes during heat treatment. [1]

[1] Zs Fogarassy, A Wójcicka, I Cora, A S Rácz, S Grzanka, E Dodony, P Perlin, M A Borysiewicz, Materials Science in Semiconductor ProcessingOpen AccessVolume 1751, 2024, 108250



On the challenges in reactive HiPIMS co-sputtering of Y and Ti for oxide layers

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Reactive HiPIMS (High-power impulse magnetron sputtering) is a sophisticated technique that requires precise tuning of several sputtering parameters (e.g. sputtering power, pulse length and frequency, reactive gas partial pressure, pumping speed, etc.) to achieve stable layer growth. In the case of co-sputtering of two targets (yttrium and titanium) – which exhibit completely different behaviour in reactive environments (secondary electron emission increases for  $YO_y$ , while decreases for  $TiO_y$ ) – this seems a great challenge. The first trial was to set the most critical parameter (the O<sub>2</sub> gas pressure). In order to do this, the hysteresis behaviour of reactive sputtering was investigated and the oxygen flow rate was optimized separately for Y and Ti, to achieve a stable plasma and reasonable sputter rate for both cases. During the co-sputtering, the reactive gas inlet was pulsed with a chopping time of about ten seconds.

Another challenge is to control the Y - Ti composition of the deposited layer. For non-reactive sputtering this can be done simply by tuning the power on each target, but in the reactive case it would upset the delicate balance of the system. By varying the number of pulses in the burst applied on the two targets, the desired change in layer composition was achieved. By using the above mentioned techniques and the micro-combinatorial one [1] we could produce a combinatorial Y-Ti-O sample in which the Y –Ti ratio varied laterally over a wide range. Figure 1a shows the photograph of the HIPIMS chamber during the sputtering process, while Figure 1b shows the resulting combinatorial Y-Ti-O layer on a Si wafer, which exhibits a lateral composition gradient.





Figure 1: a, Reactive burst HiPIMS sputtering of Y (left) and Ti (right) b, resulting combinatorial Y<sub>x</sub>Ti<sub>1-x</sub>O<sub>y</sub> sample

[1] Sáfrán G. et al. Materials 2023, 16, 3005. https://doi.org/10.3390/ma16083005

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P17

# Advanced structural characterization of Gallium Oxide by transmission electron microscopy

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Intrinsic properties of  $Ga_2O_3$  makes it a promising candidate for high power electronics, suitable for fabrication of solar-blind detectors for UV-C radiation.

Beside the stable monoclinic  $\beta$ , other polymorphs ( $\alpha$ ,  $\gamma$ ,  $\kappa(\varepsilon)$ ,  $\delta$ ) are relevant candidates for applications. The most crucial step in developing devices is the structural characterization of the polymorphs: understand their detailed crystal structure, growth mechanism, thermal stability and phase transformations up to 900°C, and the structural relationship between polymorphs at the atomic scale. Atomic scale study of these can be achieved by conventional and scanning transmission electron microscopy combined with elemental mapping and image simulations. In situ TEM annealing is an adequate method to study transformations alive.

In our work we would like to present five polymorph transformations were studied by us applying *ex-situ* and *in situ* TEM heating measurements. The  $\kappa \rightarrow \beta$  transformation was crystallographically described *in situ* in the microscope under vacuum, and using differential scanning calorimetry. The  $\kappa \rightarrow \gamma$  transformation was also studied with *ex-situ* heating measurement on a  $\kappa$ -Ga-oxide thin film, where the role of O<sub>2</sub> fugacity of heating measurements on transformation of  $\kappa$  structure was discovered and the interfaces between  $\kappa /\beta$  and  $\kappa /\gamma$  were modelled. Our second group of TEM experiments were carried out on radiation induced phase transformation of  $\beta$ -Ga<sub>2</sub>O<sub>3</sub>. First, the  $\beta \rightarrow \gamma$  phase transformation was crystallographically described. Later the structural and textural development of the  $\gamma \rightarrow \beta$  transformation was monitored by ex situ heating (series of samples at different temperatures from RT up to 1100°C) and in situ TEM annealing experiments. The experimental data were excluded by simulations using JEMS software applying multislice approach for TEM image simulations.



### Poster session 2

#### Ρ1

## PET surfaces treated with atmospheric pressure plasma jet and enhanced with ZnO nanoparticles: characterization and antibacterial activity

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The development of new technologies to prevent bacterial growth on surfaces without promoting bacterial resistance is crucial in the fight against microbial contamination. Polyethylene terephthalate (PET), one of the most common thermoplastic polymers, is widely used in the food and beverage packaging industry, making it an excellent candidate for safe-by-design surface modification that promotes antibacterial activity. The use of modified bioactive PET surfaces could extend product shelf life while reducing waste production.

In the present study, PET surfaces were subjected to atmospheric pressure plasma jet treatment and enhanced with ZnO nanoparticles, in different concentrations, synthesized by pulsed laser ablation in water. The effects of each surface modification were studied in detail using scanning electron microscopy, atomic force microscopy, Fourier transform infrared spectroscopy, energy dispersive X-ray spectroscopy, and contact angle measurements, while the antibacterial effect was tested. The results show a significant difference in surface properties between pure PET and modified PET surfaces, while the antibacterial tests demonstrate excellent activity against *E. coli* and *S. aureus*.



### Collective states driven far away from the equilibrium

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We show how an intense laser pulse drives collective state far from the equilibrium and into a metastable nonthermal state or melts the order. While the optical pump-probe (P-p) studies show a suppression of the coherent (collective mode) response [1], studies done by three pulse techniques have revealed various phenomena, from a rapid order parameter recovery [2, 3] to transitions into new states [4,5].

Here, a three-pulse technique [2] was used to study two systems: (NbSe<sub>4</sub>)<sub>3</sub>I, undergoing pseudo Jahn Teller distortion, and K<sub>0.3</sub>MoO<sub>3</sub>, a well-studied prototype CDW system. Both systems show no coherent phonon response in the high symmetry state above the phase transition. We used the sequence of three pulses: a strong D-pulse followed by the weak P-p sequence [2] to record the real-time collective response as a function of time after the strong D-pulse (t<sub>12</sub>). For excitation densities (D) comparable to complete quenching of the low-temperature phase, both systems show periodic modulations of strengths (amplitudes) and phases of collective modes as a function of t<sub>12</sub>, see e.g. Fig. 1. What is particularly interesting is that the collective mode amplitudes can be substantially enhanced in the driven state compared to the near-equilibrium case. Moreover, their phases reach values that are not observed in the conventional P-p studies. While some of the observations can be accounted for in terms of existing theoretical models [3,5,6], some, e.g. the nonanalytical dependence of phase on t<sub>12</sub>, seek for a new theoretical approach.

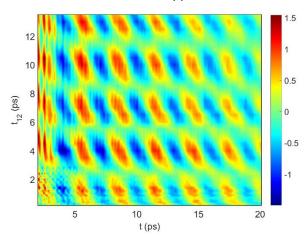


Fig. 1. The oscillatory part of the transients recorded at 5K on (NbSe<sub>4</sub>)<sub>3</sub>I, shown in false color coding as a function of time delay after the strong D-pulse,  $t_{12}$ . The time between the pump (P) and probe (p) is denoted by t. The fluence of D pulse was set to 2 mJ/cm<sup>2</sup> and the laser wavelength was 800 nm.

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[2] R. Yusupov et. al, Nat. Phys., 6 (2010) 681-684
[3] P. Kusar et. al, Phys. Rev. B, 83 (2011) 035104
[4] L. Stojchevska et al., Science 344 (2014) 6180
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[6] H. Schäfer et al., Phys. Rev. Lett., 105 (2010) 066402



## Characterization of low-pressure gaseous plasmas by optical emission spectroscopy

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Non-equilibrium plasma sustained in molecular gases in the pressure range of 1 - 100 Pa is useful for numerous applications, especially for tailoring the surface properties of heatsensitive objects. Such plasmas are typically sustained by electrodeless discharges coupled to a radio-frequency generator. Optical emission spectroscopy is among the most frequently used techniques for plasma characterization because of its simplicity. Appropriate calibration with standard radiation sources is necessary for the scientifically spotless interpretation of the spectra. While the method is regarded as qualitative, the comparison with available codes for plasma simulation and/or the addition of a small quantity of actinometer provides sufficient insight into the plasma composition. The most extensive radiation of plasma particles (particularly atoms) appears in the vacuum ultraviolet range, so a combination of spectrometers with the detection range between about 100 and 1000 nm provides adequate results. Several examples will be presented, and the spectra will be interpreted in a semiquantitative manner.

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Ρ3



## Synthesis and properties of Ge-based core-shell quantum structures

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Quantum dots and nanoparticels with core/shell structure enables manipulation the materials optical and electrical properties in a broad range. Depending on the type, size and shape of both core and shell, we observe different consequences of quantum confinement effect in them.

Here we compare effects in materials comprising quantum dots consisting of Ge core and various types of shell (Si, Si<sub>3</sub>N<sub>4</sub>, Al and Mn) which are embedded in dielectric matrices. The materials are prepared by magnetron sputtering deposition in self-assembling growth mode. This mode enables formation of 3D regular lattices of the quantum dots. Such materials have exceptional, very tunable properties like enhanced absorption, quantum confinement, multiple exciton generation, very high temperature coefficient of resistance, etc. These properties are very usable in various photo-sensitive devices and sensors.

Ρ4



### Femtosecond engraving computer-generated holograms

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Femtosecond laser micromachining is a technique used for the modification of various materials both on the surface and under the surface. In our laboratory, we have utilized this technique to modify different materials such as metals, ceramics, teeth, and polymers. Binary computer-generated holograms (CGH) can be written in transparent materials. While CGH computation is a well-researched field, challenges arise in their production and application due to high production costs and the need for specialized machinery. Holograms are mostly manufactured in the graphic industry, where they serve as security features in printed documents, among other uses.

We have successfully created holograms using a direct laser writing method with 120 fs pulses at 800 nm and a repetition rate of 1 kHz at the Institute of Physics in Zagreb, utilizing the Spectra-Physics Spitfire amplifier to generate femtosecond pulses from the Spectra-Physics Tsunami laser. We selected polycarbonate as our material of choice. The two-axis stage system from Micos was computer-controlled simultaneously with the laser. By tightly focusing the laser beam, an optical breakdown can occur within the bulk of the transparent material through multi-photon absorption. This absorption takes place in a confined space, resulting in permanent material modifications.

For a comparison I will also show reconstruction of the same CGH using other techniques: hologram generated with liquid crystal mask, Computer-to-Plate Process, Computer-to-Film Process and Monochrome Offset Printing.



## Fabricating PET/ZnO composite assisted by RF plasma processing

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Nanoparticles (NPs) can be incorporated into polymer matrices to significantly enhance their functional properties, including antimicrobial activity, barrier properties, biodegradability, and UV resistance [1, 2]. However, a major limitation of most low-cost polymers is their inherently poor surface characteristics. Cold plasma treatment has emerged as an effective technique to address this issue, improving surface energy, biocompatibility, morphology, wettability, and roughness, among other properties [3]. Radio frequency (RF) plasma generally produces a more uniform and stable plasma across the entire treatment area, making it particularly suitable for surface modifications.

In this study, ZnO NPs were incorporated into PET pretreated with oxygen RF plasma. The ZnO NP colloidal solution was synthesized using pulsed laser ablation in water at five different concentrations. The resulting PET/ZnO composite was characterized through various analytical techniques, including Fourier-transform infrared spectroscopy (FTIR), atomic force microscopy (AFM), contact angle goniometry, and X-ray photoelectron spectroscopy (XPS). A leaching test was also performed to evaluate the stability of nanoparticle incorporation within the polymer matrix. Furthermore, antimicrobial activity was assessed against *Escherichia coli*, and the gas transport properties of the composite material were investigated.

Acknowledgments:

This research was supported by the Croatian Science Foundation through the Outgoing Mobility of Assistants program (MOBDOK-2023).

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[3] H. Turkoglu Sasmazel et al., Molecules. 26 (2021) 1665.



## Silver nanoparticles in SiC matrix

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Silicon carbide (SiC) is a crystalline substance with a structure similar to diamond, with very high hardness, semiconducting properties, and high thermal and chemical resistance. SiC belongs to the group of wide bandgap semiconductors. Due to its properties, it has great potential for use in high-power, and high-radiation conditions under which conventional semiconductors cannot adequately perform. The properties of the material are significantly influenced by the metal atoms, or nanostructures, "inserted" into the basic matrix. In order to change the properties, silver nanoparticles were incorporated into the SiC matrix by simultaneous magnetron deposition. Depending on the deposition conditions, different structural, electrical and optical properties of the obtained samples were observed. The positive influence of silver nanoparticles on the observed properties was demonstrated.



Technological applicability of VO<sub>2</sub> layers prepared with different approaches

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The present work aims to summarise our work on  $VO_2$  layers, material preparation and applicability in microelectronics and micromechanics.  $VO_2$  films were prepared with four methods: classical, dense thin films were deposited with vacuum evaporation, sputtering and ALD, and nanostructures with hydrothermal growth. All of these methods have advantages and disadvantages, and there are some technological difficulties that tend to always come up in the use of  $VO_2$  materials which are hard but crucial to tackle. This paper is a summary of these with a special focus on practical issues.

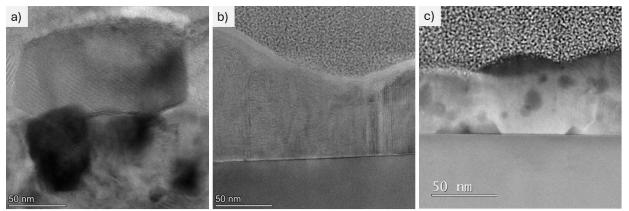


Fig.4. TEM images of different VO<sub>2</sub> layers: evaporated and post-annealed (a), sputtered (b) and ALD (c)

#### Acknowledgment

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## Grazing incidence high-energy heavy ion irradiation for surface nanopatterning

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High-energy heavy ion irradiation is a time- and cost-effective technique for tailoring material properties such as production of nanomembranes or surface nanopatterning. In particular, irradiation at grazing incidence angle is known to produce long surface ion tracks along irradiation direction [1]. Depending on the applied ion fluence, coverage of surface with ion tracks can be tuned to desired level. For high enough fluences, complete coverage of surface can occur and uniform nanoscale patterns can emerge. In this contribution we present first results of our investigations where special emphasis has been placed on the ion energy and choice of the material.

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## Pt nanoparticles on inert h-BN support: investigation of the CO<sub>2</sub> hydrogenation mechanism on the surface level

### <u>Tímea Hegedűs</u><sup>1</sup>, Imre Szenti<sup>1,2</sup>, Anastasiia Efremova<sup>1</sup>, Ákos Szamosvölgyi<sup>1</sup>, János Kiss<sup>1,2</sup>, Zoltán

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One of the goals of catalysis research is to design catalysts that can eliminate unwanted byproducts and achieve 100% selectivity for the target product without loss of activity. A detailed understanding of the role of the components that build up the catalyst is essential for its proper design. This is easy for oxides, but for pure metals, especially precious metals, determining their intrinsic activity under ambient conditions is much more complex as the catalyst supports used so far always play a role in the catalytic reaction directly or indirectly. Even  $SiO_2$ , regarded as inert shows some activity owing to the hydroxyl groups on its surface. In this work, we propose chemically inert and defect-free h-BN fibers with wide band gap and robust covalent bonds as an uncommon reference catalyst support in the CO<sub>2</sub> hydrogenation<sup>1</sup>. They were synthesized via a co-precipitation method and applied as support for size-controlled (4,7±0.6 nm) Pt nanoparticles. The catalytic activity of pure h-BN and Pt/h-BN was tested in the  $CO_2$  hydrogenation reaction. The fibers alone show no catalytic activity; however, Pt/h-BN exhibited low but notable activity (377 nmol/gs at 400°C) with almost 100% CO selectivity. XPS, TEM, and DRIFTS measurements indicate that h-BN has no effect on the metal particles or the reaction. The observed catalytic activity is attributed to Pt nanoparticles alone. CO vibration spectroscopy studies suggest that due to the lack of substrate-metal interaction Pt nanoparticles adopt an ideal spherical structure, resulting in several low coordination sites capable of CO<sub>2</sub> conversion<sup>2</sup>. Thus, h-BN fibers can serve as reference material in catalytic investigations with the proposed metal and reaction or under different conditions.

<sup>&</sup>lt;sup>1</sup> A. Efremova et al., Applied Surface Science, 571, January 2022 151326.

<sup>&</sup>lt;sup>2</sup> C. Lentz et al., Journal of Molecular Catalysis A: Chemical, 426, January 2017, 1-9.



## In situ TEM examination of the monoclinic VO<sub>2</sub> thin layer phase transformation to tetragonal VO<sub>2</sub>

### I Cora<sup>1</sup>, Zs Baji<sup>1</sup>, Zs E Horváth<sup>1</sup>, Z Szabó<sup>1</sup>, J Volk<sup>1</sup>, Zs Fogarassy<sup>1</sup>

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Vanadium oxide is of wide interest, thanks to the fact that it can be used in sensors, semiconductors, critical temperature sensors among others. Examining the phase diagram of vanadium oxide, we can see quite a lot of different vanadium oxide structures. In special cases phase transformations of the semiconducting V<sub>2</sub>O<sub>3</sub>, V<sub>3</sub>O<sub>5</sub> or VO<sub>2</sub> phases occur into a metallically conductive polymorph of vanadium oxide phase. The monoclinic, semiconducting VO<sub>2</sub> phase is reversibly transformed into the tetragonal, metallically conducting VO<sub>2</sub> phase at a relatively low temperature of around 68°C [1].

In this work, the *in situ* phase transformation of the monoclinic ( $P2_1/c$ ) VO<sub>2</sub> to tetragonal (rutile-type,  $P4_2/mnm$ ) VO<sub>2</sub> was investigated by transmission electron microscopy (TEM). The primary objective of the project was the production of monoclinic VO<sub>2</sub> layers, which can be intend use in application-oriented sensors in the future [2]. Even the initial layer deposition experiments were successful, since based on the electrical measurements, the deposited vanadium oxide layers became conductive at around 68°C, from which it could be concluded that a monoclinic VO<sub>2</sub> layer was successfully formed, which can reversibly transform into a tetragonal VO<sub>2</sub> phase above 68°C.

During the TEM examination, from the electron beam, the sample can locally easily exceed the temperature required for the phase transformation, so the monoclinic  $\rightarrow$  tetragonal VO<sub>2</sub> phase transformation can be observed even without extra heating during the TEM examination. However, the investigation is complicated since most of the peaks of the monoclinic (P2<sub>1</sub>/c) and tetragonal (P4<sub>2</sub>/mnm) structures of VO<sub>2</sub> are overlapping, only a few of them are indicator. Therefore, the layers were deposited to amorphous substrate by atomic layer deposition (ALD) in order to have random orientation of the ca. 50-100 nm large grains, so we probably have the proper orientation in plan view thinned sample.

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## Python Modeling of Beamline Vacuum System

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This paper presents a Python-based model for analyzing and optimizing vacuum systems in linear beamlines. The model processes input data from CSV files containing beamline geometry, material properties, pumping parameters, and outgassing rates. Using Python's math and numpy libraries, the model calculates pressure distribution along the beamline and identifies areas of high gas load. It provides insights into the impact of different materials, pumping speeds, and pump locations on the system's performance. While the model offers valuable estimates, actual results should be verified experimentally.



## The effect of impurities on the structure and mechanical properties of CoCrCuFeNi alloy films

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High-entropy alloy (HEA) films are often used as protective coatings; therefore their mechanical and anticorrosion properties are of main interest. Mechanical properties of films are fundamentally determined by their structure and morphology. Impurities, especially oxygen can inhibit structure evolution in vapour deposited films. Consequently, mapping their effect on film growth and structure is of outmost importance.

CoCrCuFeNi films were deposited by DC magnetron sputtering on thermally oxidized Si wafers. We used different types of ultra-high vacuum and high vacuum deposition conditions by introducing oxygen before sputtering. The initial background pressure was  $5 \times 10^{-8}$  mbar and we increased it in steps of one order of magnitude up to  $5 \times 10^{-5}$  mbar.

All films exhibit the same structure and morphology. The films have V-shaped grains which are the characteristic of competitive growth. Grains with <111> orientation grew faster at the disadvantage of grains with other orientation. Electron diffraction patterns showed that all films have fcc metallic HEA phase and there is no sign of an oxide phase. We presume that oxygen atoms were not able to segregate on the growing crystal faces and could not form covering layers, inhibiting grain growth. In accordance with structural investigation, nanoindentation measurements showed that all films have the same elastic modulus (~200 GPa) and hardness (~7.5 GPa).

We will show how the structure varies on a combinatorial sample where the partial pressure of oxygen is further increased during deposition. We aim to find the oxygen partial pressure where an impurity phase forms that hinders grain growth and disrupts the columnar structure.

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## Direct growth and application of 2D single crystals of Mxenes

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This work represents a comprehensive exploration of Mo<sub>2</sub>C MXene synthesis. We employed an advanced setup, specifically Ultra-High Vacuum Chemical Vapor Deposition (UHV-CVD) and Ultra-High Vacuum Scanning Electron Microscopy (UHV-SEM), for the direct growth of two-dimensional (2D) single crystal MXenes. Our primary objective was to gain insight into the surface properties of Mo<sub>2</sub>C. We utilized analytical techniques such as Scanning Electron Microscopy (SEM), and Energy-Dispersive X-ray Spectroscopy (EDS), Nano-SAM (scanning Auger Microscopy). Our observations revealed that, at elevated temperatures, copper (Cu) droplets coexisted with needle-like molybdenum (Mo) structures. However, a detailed understanding of such dynamics is still missing.

Therefore, the integration of Molecular Beam Epitaxy (MBE) apparatus with a Mo electron beam cell, direct epitaxial growth of Mo<sub>2</sub>C MXenes can be studied. Incorporating the vertical UHV e-beam evaporator enhances the method performance and provides better control over MXenes synthesis, offering new possibilities and insights for future applications.

Keywords: Chemical vapor deposition, Molecular beam epitaxy, Mo<sub>2</sub>C, SEM-EDS, Nano-SAM (scanning Auger Microscopy).



## Enhanced Photocatalytic Activity of ZnO Films with Fe2O3 Intermediate Layers for Efficient Water Purification

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Zinc oxide (ZnO) is a wide bandgap semiconductor with diverse applications, including photocatalysis. In the last two decades, atomic layer deposition (ALD) emerged as an excellent technique for synthesizing ZnO thin films, offering precise control of film thickness, high conformality and reproducible growth of defect-free films. ZnO films grown on flat solid substrates or nanoparticles have shown potential for water purification systems, due to their excellent photocatalytic properties. Powders or colloidal suspensions of nanoparticles are particularly efficient due to their high surface-to-volume ratio, which increases the number of available surface states serving as reaction sites for photocatalytic processes. However, separating the nanoparticles from the solution is challenging and expensive. Therefore, optimizing the design of highly efficient photocatalytic materials for water purification to achieve practical benefits is essential, as both a large surface area and high optical efficiency are crucial for the high photocatalytic activity of metal oxide films. This study investigated the growth of thin ZnO ALD films on glass substrates coated with an intermediate layer of nanoor microparticles of various sizes and shapes. Up to five different samples of Fe2O3 particles were used as an intermediate layer between ZnO film and glass substrate, producing a multifunctional material with a high surface area. To understand the photodegradation mechanism, detailed information on the morphology of the nanostructured ZnO surface and surface chemistry was obtained using scanning electron microscopy, secondary ion mass spectrometry, and X-ray photoelectron spectroscopy. Photocatalytic activity of samples was evaluated by monitoring the degradation of methylene blue in of aqueous solution under UV and solar light irradiation. The best Fe2O3-ZnO sample exhibited a photodegradation reaction rate approximately 60% higher than that of the reference ZnO film on glass substrate. These results demonstrate that ZnO with an intermediate layer made of Fe2O3 microparticles can be effectively applied for environmental applications.



Influence of chitosan-coated urinary catheters on adhesion of urethral cells

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Urinary catheters are often a source of hospital-related infections of patients. One possible solution to prevent such infections is the application of antibacterial coatings such as chitosan, which has promising antibacterial properties. However, there is a problem with the adhesion of chitosan to the surface of a catheter. Catheters are made of a long, flexible polymer tube, which is usually made of polyvinyl chloride (PVC). PVC is a hydrophobic polymer. Therefore, chitosan solution cannot completely wet the surface and adhere to it. One method that can be used to improve the wettability of polymer is plasma treatment. In this contribution, we present the influence of plasma treatment on chitosan adhesion and cell proliferation. Four different plasma treatment methods were used: solely  $O_2$  or  $CO_2$  plasma treatment, or a combination of  $H_2$  plasma pretreatment followed by  $O_2$  and  $CO_2$  plasma treatment. In all four treatment methods, the wettability was increased by the incorporation of oxygen functional groups. However, when using  $O_2$  or  $CO_2$  plasma treatment with  $H_2$ pretreatment, a superhydrophilic surface was obtained. In addition, H<sub>2</sub> pretreatment reduced the chlorine concentration in the PVC surface layer. Nevertheless, despite the different wettabilities of plasma-treated surfaces, all treatments enabled the adhesion of chitosan. More specifically, there were no differences when comparing  $O_2$  and  $CO_2$  treatment, but additional H<sub>2</sub> pretreatment seems to cause a slightly higher amount of adhered chitosan on the surface as shown by measuring the nitrogen concentration from chitosan. Also, no differences were found when comparing different incubation times of chitosan, i.e., 30 min, 1 h, and 3 h. The chitosan-coated PVC surfaces were then incubated with urethral cells to investigate the cytotoxicity of the chitosan. Cell adhesion and proliferation correlated well with the adhered chitosan, i.e., better cell proliferation was observed when H<sub>2</sub> pretreatment was used.



New stable ionization vacuum gauge as the transfer standard

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Dynamically generated pressure systems are often used as primary ultra high vacuum standards. To demonstrate the equivalence of the primary standards, it is necessary to compare them. The Slovenian IMT laboratory and the Czech CMI laboratory are preparing a bilateral comparison registered as Euramet project 1416 in the range from  $5 \cdot 10^{-7}$  Pa to  $9 \cdot 10^{-4}$  Pa.

A suitable transfer standard with the best possible stability is necessary for the comparison. The paper describes the design of the prepared transfer standard, which includes, in addition to an ionization extractor type vacuum gauge and a spinning rotor gauge, a new stable ionization vacuum gauge with previously proven excellent sensitivity stability.

This new gauge described in detail in ISO/TS 6737:2023 will be powered and evaluated by a special new IRC081 unit manufactured by Inficon. The measurement method and comparison evaluation principle are also described.



GaON thin films prepared by reactive sputtering of a liquid Ga target

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Wide bandgap Ga-based semiconductors, such as GaN and  $Ga_2O_3$  are receiving increasing interest in the field of power electronics and optoelectronics. However, there is quite large difference between their bandgaps. The bandgap of GaON is expected to be between them, thus it is worthwhile to explore optical properties of this material.

In our work, we investigated the possibilities of depositing GaON thin films by reactive sputtering. For the deposition, a liquid Ga target was used and the flow rate of the reactive gases (O<sub>2</sub> and N<sub>2</sub>) was varied. A combinatorial sample preparation approach was used to deposit a range of compositions in one sample. The pressure of the sputtering gas was 2 Pa and the sputtering power was fixed during all the deposition experiments. The elemental composition of the films was determined by energy dispersive spectroscopy. Optical properties (e.g. refractive index) were studied by spectroscopic ellipsometry and the optical bandgap of the samples deposited on quartz substrate was determined from transmission intensity measurements. The microstructure of the films was studied by transmission electron microscopy. Dependence of the refractive index and layer thickness on the applied reactive gas flow was explored. Furthermore, it was found that the bandgap energy can be altered by controlling the oxygen concentration of the film (through controlling the oxygen flow rate during deposition).