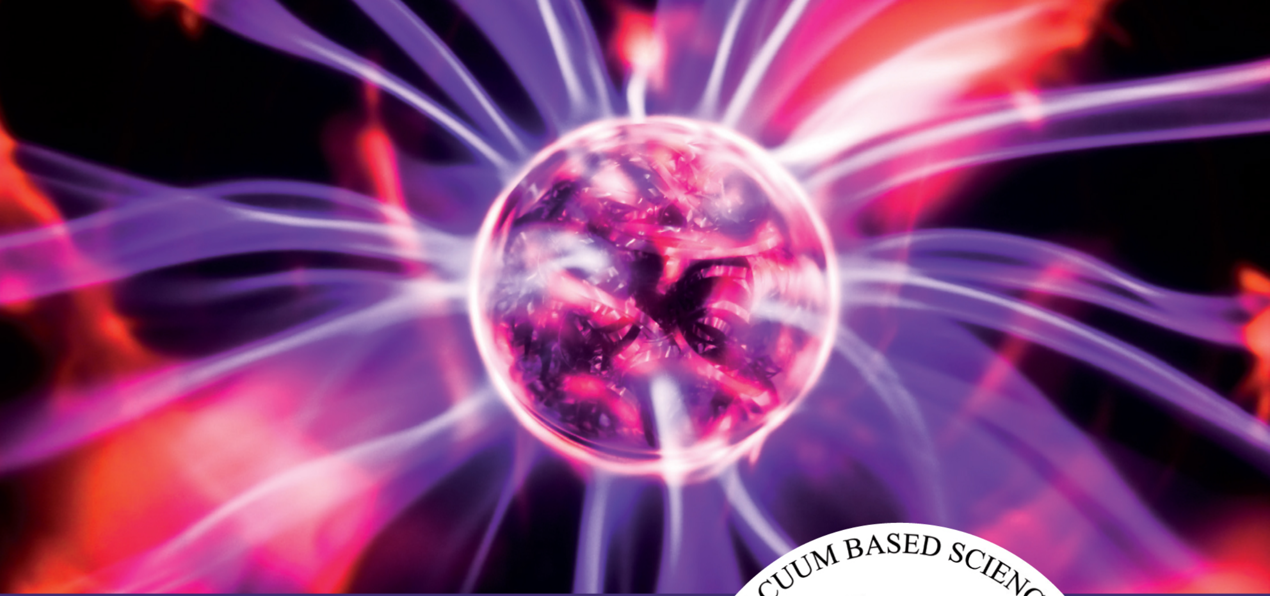

Book of abstracts

11th SYMPOSIUM ON VACUUM BASED SCIENCE AND TECHNOLOGY

organized by
Faculty of Mechanical Engineering
Koszalin University of Technology

November 19-21, 2019
Kolobrzeg, Poland

11-th Symposium on Vacuum based Science and Technology

organized by

Faculty of Mechanical Engineering
Koszalin University of Technology

under the auspices of

Polish Vacuum Society PTP

German Vacuum Society DVG

in cooperation with

BalticNet PlasmaTec Association

Society of Vacuum Coaters

November 19-21, 2019
Kołobrzeg, Poland

ISBN 978-83-7365-519-5

Chairman of Editorial Board
Zbigniew Danielewicz

Editor
Bogdan Warcholiński

Text setting
Roman Olik

Cover design
Justyna Horków

Wydawnictwo Uczelniane Politechniki Koszalińskiej
75-620 Koszalin, ul. Racławicka 15-17

Koszalin 2019, 1st Edition, No. of quires: 2.88, B-5, No. of copies: 100
Printed by: INTRO-DRUK, Koszalin

SCIENTIFIC AND PROGRAM COMMITTEE

- Dr Ralf Bandorf, Fraunhofer Institute for Surface Engineering and Thin Films IST, Braunschweig (DE)
- Prof. Jolanta Baranowska, Westpomeranian University of Technology, Szczecin (PL)
- Prof. Ryszard Czajka, Poznań University of Technology (PL)
- Prof. Marek Godlewski, Institute of Physics, Polish Academy of Sciences, Warsaw (PL)
- Prof. Grzegorz Greczyński, Linköping University (SE)
- Prof. Jerzy Morgiel, Institute of Metallurgy and Materials Science, Cracow (PL)
- Prof. Jan Walkowicz, Koszalin University of Technology (PL)
- Prof. Krzysztof Zdunek, Warsaw University of Technology (PL)

SYMPOSIUM CHAIR

Witold Gulbiński, Koszalin University of Technology
Faculty of Mechanical Engineering
e-mail: *witold.gulbinski@tu.koszalin.pl*
phone: + 48 94 34 78 626

LOCAL ORGANIZING COMMITTEE

Koszalin University of Technology
Faculty of Mechanical Engineering

- Bogdan Warcholiński
- Roman Olik
- Karina Grześkowiak
- Magda Małecka

e-mail: *svbst@tu.koszalin.pl*
phone: +48 94 34 78 626

Dear Colleagues,

It is my pleasure to welcome you at the 11 Symposium on Vacuum based Science and Technology.
This year the scope of our meeting is focussed on modern thin film deposition techniques. Invited speakers will share with us their expertise on modern hard coatings and magnetron sputtering, including process modelling and control. HiPIMS and high current pulsed vacuum arc deposition techniques will be discussed, also in the framework of training course offered by the Society of Vacuum Coaters. Oral and poster sessions will give us an overview of recent results and research challenges in our laboratories.
I wish to attract your attention to the Industry Exhibition where modern research and technological equipment will be presented.
In the framework of traditionally organized Clausius Session, our students will be invited to attend the popular scientific lecture on the plasma in the Universe.

Thank you for coming and have a good time in Kolobrzeg this Autumn!

Witold Gulbinski
Symposium Chairman

TEPRO SA *www.tepro.com.pl*	TEPRO
EDVAC Sp. z o.o. *www.edvac.pl*	EDVAC TECHNIKA PRÓŻNIOWA
Atlas Copco Polska Sp. z o.o. www.atlascopco.com	Atlas Copco
Kurt J. Lesker Company *www.lesker.com*	Kurt J. Lesker Company
MSA System *http://msasystem.pl/*	MSA SYSTEM
SHIM-POL *www.shim-pol.pl*	shim-pol
AMP Sp. z o.o. *www.amphtt.com*	AMP Technologie Obróbki Cieplnej

Invited Speakers

INV1 – **Prof. Diederik Depla,** Department of Solid State Physics
Ghent University (BE)

MODELING REACTIVE MAGNETRON SPUTTERING:
OPPORTUNITIES AND CHALLENGES

INV2 – **Prof. Jindrich Musil,** Department of Physics and NTIS – European Centre of Excellence, University of West Bohemia (CZ)

ADVANCED HARD NANOCOATINGS DEPOSITED BY
MAGNETRON SPUTTERING: PRESENT STATE AND TRENDS

INV3 – **Prof. Tomas Nyberg,** Solid State Electronics, Angstrom Laboratory, Uppsala University,(SE)

STUDIES OF NON-SATURATED REACTIVE SPUTTERING
PROCESSES

INV4 – **Dr. Matjaž Panjan,** Jožef Stefan Institute, Ljubljana, (SI)

SELF-ORGANIZATION OF PLASMA IN MAGNETRON
SPUTTERING DISCHARGES

INV5 **– Prof.Yuri Suchorski,** TU Wien, Institute of Materials Chemistry (AU)

UHV AND MODEL CATALYTIC STUDIES:
A SUCCESSFUL SYMBIOSIS?

INV6 – **Prof. Xiubo Tian,** Institute of Plasma Surface Engineering & Equipment School of Materials Science & Engineering, Harbin Institute of Technology, Harbin (CN)

HIGH-CURRENT PULSED ARC DISCHARGE FOR DEPOSITION
OF ta-C AND DLC FILMS

Oral presentations

Claussius Sesion Lectures

L1 – **Prof. Dr. Holger Kersten**
Institut for Experimental and Applied Physics, University Kiel, Germany,
"The Universe - a world of plasma"

Poster Presentations

P1 NANOSCALE MODEL CATALYTIC STUDIES IN UHV: BRIDGING THE HIGH VOLTAGE GAP
P. Winkler, J. Zeininger, M. Raab, G. Rupprechter and Y. Suchorski

P2 NANOSCALE MODEL CATALYTIC STUDIES IN UHV: CONTRIBUTION TO BRIDGING THE MATERIALS GAP
J. Zeininger, M. Raab, P. Winkler, G. Rupprechter and Y. Suchorski

P3 CYLINDRICAL MAGNETRON WITH MULTIPLE DYNAMIC AREAS OF TOROIDAL PLASMA AND CATHODE OR ANODE POLARIZATION
Marek Betiuk, Zbigniew Łataś, Piotr Domanowski, Wojciech Gajewski, Marcin Żelechowski

P4 MICROFRICTION OF ZrCN COATINGS CHARACTERIZED BY ATOMIC FORCE MICROSCOPY
T.A. Kuznetsova, V.A. Lapitskaya, S.A. Chizhik, B. Warcholinski, A. Gilewicz

P5 DETERMINATION OF FRACTURE TOUGHNESS OF COATINGS BY INDENTATION METHOD WITH THE AFM CRACKS DIAGNOSIS
V.A. Lapitskaya, T.A. Kuznetsova, S.A. Chizhik, B. Warcholinski, A. Gilewicz

P6 DLC FILM-POLYMERIC SUBSTRATE INTERACTION
Agata Niemczyk, Dariusz Moszyński, Jolanta Baranowska

P7 TRENDS IN BORIDING UNDER GLOW DISCHARGE CONDITIONS
Michał Kulka

MODELING REACTIVE MAGNETRON SPUTTERING: OPPORTUNITIES AND CHALLENGES

Diederik Depla

Department of Solid State Physics
Ghent University (BE)

Reactive magnetron sputter deposition is an often used technique to deposit compound thin films. It is a conceptually easy method and its description can be summarized in a few lines. Recent research clearly demonstrates however that behind the apparent simplicity this physical vapor deposition technique is driven by complex entangled physical and chemical processes. The addition of a reactive gas further increases this complexity. To get a better understanding, our team has developed the reactive sputter deposition (RSD) model. In the first part of the presentation, this model will be briefly discussed, and used as a guidance to illustrate the above statement by some recent experimental results. The well-known hysteresis behavior of most deposition parameters as a function of the reactive gas flow will be used as a first example to show that our knowledge on reactive magnetron sputtering still expands every day. A second example concerns a longstanding open question about the deposition rate during reactive magnetron sputtering. The latter is difficult to predict due to the simple fact that the oxide sputter yield during reactive magnetron sputtering differs from the typical values measured by ion beam experiments. As the measured sputter yield also depends on the discharge current, it is necessary to adjust our traditional view on target poisoning.

ADVANCED HARD NANOCOATINGS DEPOSITED BY MAGNETRON SPUTTERING: PRESENT STATE AND TRENDS

Jindrich Musil

Department of Physics and NTIS – European Centre of Excellence, University of West Bohemia, Univerzitni 22, CZ – 306 14 Plzen, Czech Republic
Institute of Physics v.v.i., Academy of Sciences of the Czech Republic, Na Slovance 2, Praha 8, CZ – 18221, Czech Republic

Corresponding author: musil@kfy.zcu.cz

The lecture reports on magnetron sputtering of advanced hard nanocoating and is divided in three parts. The first part briefly reports on the enhanced hardness of nanocomposite coatings, the formation of the X-ray amorphous coatings with thermal stability and oxidation resistance above 1000 deg C and flexible hard coatings. Reported results can be used in the development of the flexible ceramic coatings, the surface strengthening of brittle materials, the prevention of (i) cracking of the functional coatings and (ii) the cracks formation on the surface of bended materials. The second part is devoted to the energy vs. property relations in sputtered coatings. It is shown that a key role in formation of the flexible hard coatings with fully reproducible properties plays the energy delivered into growing coating by bombarding ions and fast neutrals. In part 3 two sputtering processes are described in detail (1) the low-temperature sputtering of coatings at low pressures p of sputtering gas and (2) the new sputtering sputtering technology based on three strongly non-equilibrium processes at atomic level: (1) extremely fast heating of the coating material to high temperatures up to its melting with low substrate heating, (2) high pressures ($\geq$1000 GPa) used in formation of the coating from its melted material and (3) extremely fast cooling rate ($\geq 10^{10}$ K/s) of the created coating material. The principle of this technology is explained. The utilization of this technology for creation of advanced coatings with new unique properties is demonstrated in sputtering of the following coatings: (1) the alloy coatings with high-temperature beta-phase sputtered at low substrate temperature T close to RT, (2) the overstoichiometric and dinitride coatings, and (3) the superhard flexible Ti coatings with high hardness (up to 20 GPa) several times higher than that of the bulk Ti metal. Special attention is devoted also to the formation heterostructural coatings composed of elements or compounds with different crystal structure.

STUDIES OF NON-SATURATED REACTIVE SPUTTERING PROCESSES

S. Berg and T. Nyberg

*Solid State Electronics, Angstrom Laboratory, Uppsala University, Box 534,
751 21 Uppsala, Sweden*

Corresponding author: tomas.nyberg@angstrom.uu.se

Several models for reactive sputtering of oxides and nitrides have been published over the years. All these models show that for a large supply of the reactive gas, the composition of the deposited films will saturate at the stoichiometric compound for the oxide or nitride. Excess supply of the reactive gas will not cause the film compositions to significantly exceed the stoichiometric values. Since such film compositions saturate at stoichiometric oxide or nitride we choose to call such processes saturated reactive sputtering processes. The well-known "Berg model" is one of the existing models that quite well describes such saturated processes.

Using reactive gases like hydrocarbons, silanes or hydrogen sulphide during reactive sputtering may result in decomposition of the gases into solid carbon, silicon or sulphur respectively, that will be deposited onto the substrate even if they do not chemically form a compound. Such processes may result in film compositions ranging from (0 – 100%) of C, Si or S respectively. There exists no natural limitation of the composition of the deposited film. We therefore choose to call such processes non-saturated reactive sputtering processes.

The growth mechanisms for such processes are not very well understood and have not been extensively studied. Further, the original Berg-model cannot be used to correctly describe these processes. However, a new simple first order model that may serve to illustrate the behaviour of non-saturated processes is presented here. Experimental findings is presented and compared to modelling results.

SELF-ORGANIZATION OF PLASMA IN MAGNETRON SPUTTERING DISCHARGES

Matjaž Panjan

Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia

Corresponding author: matjaz.panjan@ijs.si

In magnetron sputtering, plasma plays an essential role in the sputtering process and growth of thin films. The plasma properties can be controlled by an electrical potential that is applied to the cathode. For this reason, magnetron sputtering is generally classified as DCMS (continuous cathode voltage), HiPIMS (pulsed voltage) and RFMS (oscillatory RF voltage). Magnetron cathodes come in several geometries, including planar (e.g., rectangular or circular) and cylindrical, while the magnetic field configuration can vary from balanced to unbalanced. Despite different types of cathode voltages, magnetron geometries and magnetic field configurations, the basic physical process responsible for gas ionization and sustainability of discharge are the same. Dense plasma is obtained with the crossed configuration of magnetic and electric fields, which forces electrons to travel on long trajectories within the torus-like area above the cathode. As it will be shown in the talk, the drifting motion of electrons in the $\mathbf{E}\times\mathbf{B}$ direction results in the formation of dense and often periodic regions called spokes. Such plasma structures have been observed in all types of magnetron regimes, cathode geometries and for a wide range of discharge conditions (i.e., pressures and discharge currents) [1-3]. The universal presence of spokes indicates that plasma self-organization is fundamental process in magnetron discharges. In the talk, I will present our latest investigations of plasma self-organization in different regimes of magnetron sputtering. I will discuss the formation of different spoke patterns and their dynamics with respect to the discharge conditions, their influence on the motion of charged particles and the importance of spokes on the ionization process. I will demonstrate that electric fields within spokes play an important role in providing energy to electrons and thus in sustaining the discharge.

[1] A. Anders *et al.*, *J. Appl. Phys.*, **111** (2012) 053304
[2] M. Panjan *et al.*, *Plasma Sources Sci. Technol.*, **24** (2015) 065010
[3] M. Panjan *J. Appl. Phys.*, **125** (2019) 203303

UHV AND MODEL CATALYTIC STUDIES: A SUCCESSFUL SYMBIOSIS?

Yuri Suchorski

TU Wien, Institute of Materials Chemistry, Getreidemarkt 9, 1060 Vienna, Austria

Corresponding author: yuri.suchorski@tuwien.ac.at

The importance of catalysis in our life is hardly possible to overestimate: the products range from our clothes, fuel for our cars, mobile phones, etc., to plastic garbage bags which we use to cast things away. Traditionally, catalysis has been developed by a *trial and error* approach, but to be able to predict and to create more efficient catalysts, it is necessary to understand the atomic mechanisms of catalytic processes.

Since heterogeneous catalysis takes place on surfaces, it is natural to use surface analysis techniques to study catalytic reactions. Most of such techniques need UHV, therefore, a typical approach has been to use model catalysts such as single crystal surfaces of precious metals and a gas phase in the UHV pressure range to mimic the real processes. A big amount of understanding has been collected in this way in the last decades, culminating in the Nobel Prize in 2007 for G. Ertl. Unfortunately, the data obtained under conditions typical for surface analysis can be not always extrapolated to real catalysis taking place under high pressure. In addition, single crystals can hardly model real catalytic nanoparticles.

The present contribution will focus on these differences between surface science and real catalysis, known as the *pressure and materials gap*, and on the ways how to bridge it. A row of sophisticated model systems, such as nanoparticles of precious metals on oxidic supports, nanoislands of oxides on catalytically active metal surfaces, apexes of nanotips as models of single catalytic particles, etc., will be discussed in course of societally relevant catalytic reactions such as CO and H2 oxidation. Examples of catalytic applications of UHV based analytic techniques, such as PEEM, MIEEM, FIM/FEM, FIAES and of the novel *kinetics by imaging* approach will be presented.

This work was supported by the Austrian Science Fund (FWF) [SFB F45 FOXSI].

HIGH-CURRENT PULSED ARC DISCHARGE FOR DEPOSITION OF ta-C AND DLC FILMS

Prof. Xiubo Tian

Institute of Plasma Surface Engineering & Equipment
School of Materials Science & Engineering
Harbin Institute of Technology, Harbin (CN)

FABRICATION OF SELF-ORGANIZED FISHNET STRUCTURES FOR CARBON NANOTUBE FOREST METAMATERIALS

Adam Pander[1,2,*], *Takatsugu Onishi*[1], *Akimitsu Hatta*[1,2], *and Hiroshi Furuta*[1,2,†],

[1] *Department of Electronic and Photonic Systems Engineering, Kochi University of Technology, Tosayamada, Kami, Kochi 782-8502, Japan*
[2] *Center for Nanotechnology, Research Institute, Kochi University of Technology, Tosayamada, Kami, Kochi, 782-8502, Japan*

Corresponding: * adam.michal.pander@kochi-tech.ac.jp, † furuta.hiroshi@kochi-tech.ac.jp

In recent years, a great deal of attention has been brought to metamaterials, artificial media composed of subwavelength structures grouped together and designed to manipulate electromagnetic (EM) waves in a way to exhibit novel properties not observed in nature, such as negative or zero index of refraction. Nowadays, diverse designs of metamaterials are being studied and developed based on metallic, all-dielectric and semiconductor properties, which also includes a variety of nonlinear materials, among which carbon nanotubes (CNTs) were also investigated, due to their excellent anisotropic electrical and optical properties.

Recently, our group presented CNT forest split-ring resonator (SRR) metamaterials and frost column-like CNT forest fishnet metamaterials, which showed very promising results for the applications in solar cells, antennas, energy storage, etc. However, the major drawback, related to the fabrication method limited the development of prototype devices. The large area production of metamaterials has low cost-effectiveness since the commonly used lithographic methods based on the electron beam, focused ion beam or laser interferometry are usually expensive and low-throughput processes.

In this work, we present an alternative bottom-up approach of the preparation of novel self-assembling CNT forest fishnet structures, using self-organizing polystyrene nanosphere (Ø800 nm) monolayers as a mask during the catalyst deposition before the CNT growth. The morphology of fishnet structures, as well as, the nonlinear optical properties of fabricated metamaterials were investigated in relation to the variation of the fishnet hole size and wall thickness.

Acknowledgments:
This work was supported by KAKENHI No. 17K06205.

ELECTRICAL AND OPTICAL PROPERTIES OF THE WO_3 THIN FILMS DEPOSITED BY THE GLAD MAGNETRON SPUTTERING TECHNIQUE

A. Rydosz[1], K. Kollbek[2], K. Marszałek[1], K. Dyndał[1], W. Andrysiewicz[3],*

[1] AGH University of Science and Technology, Department of Electronics, Al. Mickiewicza 30, 30-059 Krakow, Poland
[2] AGH University of Science and Technology, Academic Centre for Materials and Nanotechnology, Mickiewicza 30, 30-059 Krakow, Poland
[3] CBRTP SA, Zlota 59, 00-120 Warszawa, Poland

Corresponding author: artur.rydosz@agh.edu.pl

The deposition process and investigation of the physical properties of WO_3 thin films are presented in this paper. The tungsten trioxide (WO_3) thin films were deposited based on the previously confirmed results for gas-sensing applications. The deposition system and the schematic representation of GLAD technique are described. The system is based on the ECR (ECR-UHV-20532-001) manipulator from Kurt J. Lesker Company (Hastings, Easy Sussex, UK). A tungsten metal target (Kurt J. Lesker Company) of 50 mm diameter (99.995% purity) was employed for reactive sputtering (80% Ar/20% O2). The flows of argon and oxygen were controlled by MFC (mass flow controllers) 1179B (MKS Instruments (Andover, Massachusetts, USA). The substrate tilt angle various from 45°–90° and 0°, and the sample rotation at a speed of 20 rpm was stabilized by ECR manipulator. The *W* target was placed at a distance of 200 mm form centerline of the substrate. The base vacuum and deposition vacuum were $5 \cdot 10^{-6}$ mbar and $3 \cdot 10^{-2}$ mbar, respectively. The *W* target was firstly presputtered to remove any contamination in pure Ar for 10 min at 50 W, then the O_2 was introduced to chamber. After the presputtering processes, the sputtering time was adjusted to obtain films with different thicknesses, the power, temperature were fixed at: 50 W, 300°C The films were obtained with utilization of DC-MF sputtering mode by power supply from DORA Power System. After deposition the samples were annealed at 400°C/4h in air. Thin WO_3 films were deposited on glass, quartz and Si substrate. The crystal structure, morphology optical and electrical properties were determined by XRD, SEM, EDX, optical spectroscopy and spectroscopic ellipsometry as well as I-V measurements.

QUASI-AMORPHOUS, NANOSTRUCTURAL CoCrMoC/a-C:H COATINGS FOR CORROSION PROTECTION OF MEDICAL GRADE STEEL

E. Dobruchowska[1], *T. Suszko*[1], *G. Greczynski*[2], *D. Adamczewska*[1], *J. Morgiel*[3], *W. Gulbiński*[1]

[1]*Koszalin University of Technology, ul. Sniadeckich 2, 75-453 Koszalin, Poland*
[2]*Thin Film Physics Division, Department of Physics (IFM), 581 83 Linköping, Sweden*
[3]*Institute of Metallurgy and Materials Sciences, Polish Academy of Science, ul. Reymonta 25, Kraków 30-059, Poland*
Corresponding author: ewa.dobruchowska@tu.koszalin.pl

The requirements imposed on metallic biomaterials, applied in orthopaedic implantology or maxillofacial surgery, force to initiate studies for developing new materials, distinguished by a set of appropriately chosen parameters, as well as to gain a complete characterisation of the existing ones. Currently, Cr-Ni-Mo stainless steels and Co-Cr-Mo alloys are among the most common metallic biomaterials used for the production of temporary and long-term implants. These materials exhibit high biotolerance and good physico-chemical properties due to their ability to passivation i.e. formation a seal layer on the surface protecting the alloy interior against body fluids, when implanted. However, in dynamic conditions, the implants are exposed to fatigue processes, corrosion and friction wear. The corrosion rate and the release of metal ions depend on the repassivation time, which was found to be longer for Cr-Ni-Mo steel compared to Co-Cr-Mo. The observation became a motivation to design cobalt-based coatings increasing the corrosion resistance of stainless steel (AISI 316L, ISO 4404-316) and thus extending its lifetime in the environment of body fluids. CoCrMoC/a-C:H coatings were synthesised by reactive magnetron sputtering of medical Co-Cr-Mo ($Co_{64}Cr_{31}Mo_5$, ISO 5832-4) alloy in the C_2H_2/Ar atmosphere. The evolution of their structure, chemical and phase composition with increasing carbon content (from 0 up to 43 at.%) were investigated by X-ray diffraction, X-ray photoelectron spectroscopy and transmission electron microscopy. The results obtained have shown that microstructure of deposits evolves from biphasic, containing fcc and hcp metallic phases (for low carbon content), through nanocrystalline and amorphous (for 13 at.% C) to quasi-amorphous nanostructural (for 43 at.% C). In the latter case, segregation of amorphous carbon leads to the formation of a self-organised tubular nanostructure. Evaluation of corrosion resistance of CoCrMoC/a-C:H and 316L-coating systems was performed in Hanks' Balanced Salt Solution by means of electrochemical impedance spectroscopy and potentiodynamic polarisation tests. The studies performed have revealed high chemical inertness of all coatings under investigation. Passive layers formed on their surfaces exhibit a capacitive character, and their resistance is nearly two orders of magnitude higher compared to the medical Co-Cr-Mo alloy. Furthermore, the coatings containing more than 13 at.% C significantly increase the corrosion resistance of 316L stainless steel. The positive values of the corrosion potentials as well as total pitting corrosion resistance have been observed for these substrate-coating systems. The features observed are associated with structural properties of the CoCrMoC/a-C:H coatings.

SURFACE SINTERING OF W-BASED POWDER TARGETS PERFORMED VIA ELECTROMAGNETIC DISCHARGE CONDITIONS – NOVEL APPROACH OF FILMS SYNTHESIS IN MAGNETRON SPUTTERING

B. Wicher[1], *R. Chodun*[1], *M. Kubiś*[2], *K. Król*[3], *K. Nowakowska-Langier*[4], *K. Zdunek*[1]

[1]*Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska St. 141, 02-507 Warsaw, Poland,*
[2]*Institute of Heat Engineering, Faculty of Power and Aeronautical Engineering, Warsaw University of Technology, Nowowiejska St. 21/25, 00-665 Warsaw,*
[3]*Institute of Microelectronics and Optoelectronics, Koszykowa St. 75, 00-662 Warsaw,*
[4]*National Centre for Nuclear Research (NCBJ), A. Soltana St. 7, 05-400 Otwock, Poland*

Corresponding author: Bartosz.Wicher.dokt@pw.edu.pl

Reproducibility of bulk magnetron targets constitutes a crucial factor, which consistently determines method of them manufacture up-to date, therefore a 3-dimensional (3D) sintering of green body is being developed, so far. Unfortunately, consolidation of bulk targets includes some important limitations, mainly concerns straightforward form of them (reduced size and constrained geometry), narrow chemical composition availability and more importantly, being not remain on a cost-effective basis. Accordingly, we put in place of that, a novel approach of only surface sintering (2D) of W-based powder mixture, by way of electromagnetic discharge conditions, with no additional mechanical press. By taking this assumption into consideration, the energy exchange between electrically excited plasma-particles (ions and electrons) and the free surface of loosely-packed powder will be investigated through the density measurements (AccuPyc II 1340) of mass transport effect, occurred within. Further, detailed approximation of the plasma flux energy dissipation at a gradually densified tungsten target interface, would be studied by usage of well-founded techniques, including; X-ray diffraction (XRD), thermal diffusivity analysis (LFA) and the micro computed tomography (μCT), as well. Subsequently, surface-consolidated W and W-B-C targets account for a novel vapor source concept, will be adjusted to the films synthesis due Gas Injection Magnetron Sputtering (GIMS), respectively. It will appear to be a relevant, whether kind of flaws occurred within targets, affects utilitarian properties of synthetized W and WxBCy layers. For this purpose, HRTEM images were taken from the identified metastable β-W phase, followed by a promising low-resistivity values, in terms of advanced spintronic devices application. Apart from that, an unusual combination of high hardness and moderate ductility was achieved for the nanocrystalline WxBCy film, which otherwise has been predicted by ab-initio calculations, previously. These suggest, the surface-sintering becomes an interesting in its simplicity approach, of any material target preparation, possibly influencing the performance of coated tools during real-life applications.

ELECTRICAL AND OPTICAL PROPERTIES OF THE THIN ZnO LAYER WITH Al AND Mg DOPPED GRADIENT COMPARED TO THE MULTILAYER STRUCTURE BASED ON THE SAME MATERIALS

K. Marszalek [1,2], *G. Putynkowski*[1], *K. Leszczynska*[1],

[1]*CBRTP SA, Zlota 59, 00-120 Warszawa, Poland*
[2]*AGH University of Science and Technology, Mickiewicza 30, 30-059 Krakow, Poland*

Corresponding author: karolina.leszczynska@cbrtp.pl

The deposition process and investigation of the physical properties of ZnO: Al and ZnO: Mg thin films are presented in this paper. The Al doped ZnO and Mg doped ZnO thin films were deposited by ALD method in Beneq reactor. Atomic Layer Deposition permit to manufacture layer by layer very precise nanometer structure with unique properties of the coatings, with the high repeatability.
The great advantage of such technique of manufacture of doped thin ZnO films is law temperature in comparison with diffusion processes. Thin ZnO: Al and ZnO: Mg films were deposited layer by layer of ZnO then Al_2O_3 then ZnO several times and so on. Finally a thin film structure with Al gradient is obtained in a remarkable lower temperature then a system obtain by diffusion of Al. Thin film system were deposited on glass and Si substrate. The crystal structure, optical (refractive index and extinction coefficient) and electrical properties were determined by XRD, SEM, and spectroscopic ellipsometry and sheet resistance measurement.

STRUCTURE AND PROPERTIES OF TiO_2/nanoTiO_2 BIMODAL COATINGS OBTAINED BY A HYBRID TECHNIQUE COMBINING MS-PVD AND ALD METHODS

M. Staszuk, D. Pakuła, Ł. Reimann, A. Kloc-Ptaszna

Silesian University of Technology, Faculty of Mechanical Engineering,
Konarskiego Street 18A, 44-100 Gliwice, Poland

Corresponding author: marcin.staszuk@polsl.pl

The magnetron sputtering method has been known for many years, and is still an attractive and modern direction in the development of material engineering. Atomic layer deposition (ALD) is currently a very promising direction of investigation, and one of the leading features of coatings obtained by this method is a very high degree of coverage reaching the order of 80-100%, which is due to the fact that the reactants only contact each other on the coated substrate by what gas phase reactions are eliminated. The synergy achieved by combining these two technologies will allow to obtain layers with properties impossible to obtain using these methods separately. The purpose of this work is to investigate the impact of TiO_2/nanoTiO_2 coating production conditions obtained in a hybrid process combining magnetron sputtering (MS-PVD) and atomic layer deposition (ALD) methods on the structure and selected properties of these layers.

Research methodology included among others imaging using electron microscopy and scanning probe microscopy as well as spectroscopic analysis in micro-areas. Structure and morphology studies of the manufactured layers were carried out using the SEM scanning electron microscope and AFM atomic force microscope. The tribological properties of the investigated materials will be determined by the pin-on-disc method. In order to determine the corrosion resistance of the investigated materials, anode polarization curves were recorded using the electrochemical potentiodynamic method. Additional information about the electrochemical properties of the investigated samples, among others: the quality of tested coatings, their tightness and resistivity were obtained thanks to electrochemical impedance spectroscopy (EIS).

Studies have shown that the production of hybrid layers allows coatings with electrochemical properties to outweigh the properties of layers produced by a single method. In addition, the main mechanisms of corrosion and tribological wear were determined on the basis of SEM observations after corrosion research's and after tribological investigations.

THE INFLUENCE OF LASER TEXTURING ON THE PROPERTIES OF PVD COATINGS

D. Pakuła[1], M. Staszuk[1], M. Pancielejko[2]

[1] *Silesian University of Technology, Institute of Engineering Materials and Biomaterials, Konarskiego Street 18A, 44-100 Gliwice, Poland*
[2] *Technical University of Koszalin, Faculty of Technology and Education, Śniadeckich Street 2, 75-453 Koszalin, Poland*

Corresponding author: daniel.pakula@polsl.pl

The main goals of the work is to determine the effect of synergy of hybrid surface treatment of tool materials using physical deposition methods and laser texturing on the structure and properties of newly developed surface layers on cemented carbides and sialon. In the assumed research model two variants of hybrid surface layer manufacturing process are provided: laser texturing of the substrate before coating deposition and laser coating texturing after its deposition. Textured substrates of sintered carbide and sialon ceramic were coated with the following PVD coatings: (Ti,Al)N, Ti(C,N), (Cr,Ti)N, (Al,Ti)N and (Al,Cr)N. As part of the work, researches of the structure using scanning electron microscopy (SEM), analysis of chemical composition using EDS spectrometers, morphology investigations using atomic force microscopy (AFM) and confocal microscope were made. In addition, mechanical properties including microhardness, "scratch test" adhesion, "pin on disc" wear resistance and roughness were also tested. As a result of researches, it was found that the creation of a hybrid layer - laser texturing/ PVD coating - on tool material contributes to the improvement of exploitative properties of the tested material. The honeycomb texture (in a microscale) and LIPSS nanoripples (in a nanoscale), created as a result of selective laser processing on the tool material, increase the adhesion of the tested PVD coatings to the substrate. It was also found that the hybrid layers, thus produced, reduce friction coefficient as well as the width and depth of wear, as was observed during the "pin on disc" tests.

MAGNETIC FIELD SENSORS BASED ON AN ULTRA-THIN ACTIVE CHANNELS

W. Koczorowski[1,2], *S. El-Ahmar*[1], *M. Przychodnia*[1], *T. Grzela*[1]

[1] *Institute of Physics, Faculty of Technical Physics, Poznan University of Technology, ul. Piotrowo 3, 60-965 Poznań, Poland*
[2] *Center for Advanced Technology, Adam Mickiewicz University, ul. Uniwersytetu Poznańskiego 10, 61-614 Poznań, Poland*

Corresponding author: wojciech.koczorowski@put.poznan.pl

In the last few years the applications of the 2D-materials in the wide range of the technology are observed. It is related to interesting physical features which are offered by: graphene (G) and thin layers of topological insulators (TI) or transition metal dichalcogenides (TMD's) materials. These thin layers allow to construct various types of 2D-materials based devices with extremely miniaturised active channels. This is promising approach for semiconductor devices miniaturisation, where the single atomic layer is a natural limit of the channel thickness.

This work involves: description of the fabrication procedures for micrometric scale magnetic field sensors in planar architectures and its characterisation. The main technique used for the devices fabrication is mask-less optical lithography. DC magnetron metal deposition under High Vacuum condition method is used for electrodes formation and vacuum Ar^+ ion sputtering process is applied for channel formation in case of G layer. The examples of operational magnetic field sensors with different designs, containing G and Bi_2Se_3 flakes as the active channels will be discussed in details. Used architectures of the sensors reported within this work include: standard Hall's cross, planar extraordinary magnetoresistance (EMR) and modified raster pattern geometry magnetoresistor - strip pattern geometry (SPG).

Acknowledgements:

This work has been partially supported by the Polish Ministry of Science and Higher Education under the project No. 06/62/SBAD/1922.

EXPERIMENTAL REALIZATION OF DOUBLE HALL SENSOR STRUCTURE USING VACUUM BASED TECHNIQUES

S. El-Ahmar[1], *W. Koczorowski*[1,2]

[1] *Institute of Physics, Faculty of Technical Physics, Poznan University of Technology, ul. Piotrowo 3, 60-965 Poznań, Poland*
[2] *Center for Advanced Technology, Adam Mickiewicz University, ul. Uniwersytetu Poznańskiego 10, 61-614 Poznań, Poland*

Corresponding author: semir.el-ahmar@put.poznan.pl

Hall effect devices are a group of magnetic field sensors commonly used in many areas of measurement, control and automatics. Unavoidable and harmful feature of all Hall sensors is the generation of the offset voltage at the output electrodes. That offset overlaps with the Hall voltage and by that decreases the sensor accuracy, particularly at low magnetic fields. Therefore, in order to obtain a Hall sensor with high magnetic sensitivity and accuracy, the offset must be eliminated or at least reduced.

Double Hall sensor structure (DHSS) can be used for effective voltage offset reduction. DHSS is composed of two standard Hall sensors that are activated with two currents from electrically independent current sources. The advantage of DHSS is that it can be manufactured with the standard thin film technology enabling efficient miniaturization of the system. DHSS can effectively be used as well for the measurements of Hall effect in ultra-thin layers containing the two dimensional electron gas, such as the epitaxial graphene. A record offset reduction by four orders of magnitude to the DC microvolt level is achieved. The strongly reduced offset is thermally stable, provided that the single Hall sensors of the DHSS have equal temperature coefficients of resistance and the heating/cooling procedure is performed under isothermal conditions.

Acknowledgements:

This work has been partially supported by the Polish Ministry of Science and Higher Education under the project No. 06/62/SBAD/1922.

THERMAL EVOLUTION OF THE PLATINUM BUFFER LAYER ON GRAPHENE/Ge(110)/Si(110) SAMPLES

T. Grzela[1], *M. Przychodnia*[1], *S. El-Ahmar*[1], *T. Ciuk*[2], *K. Kwiecień*[1], *R. Czajka*[1], *W. Koczorowski*[1,3]

[1]*Institute of Physics, Poznan University of Technology, ul. Piotrowo 3, 60-965 Poznań, Poland*
[2]*The Łukasiewicz Research Network - ITME, ul. Wólczyńska 133, 01-919 Warsaw, Poland*
[3]*Center for Advanced Technology, Adam Mickiewicz University, ul. Uniwersytetu Poznańskiego 10, 61-614 Poznań, Poland*

Corresponding author: tomasz.grzela@put.poznan.pl

Recently graphene was considered as prospective material platform, which can significantly increase the development of microelectronics. However, despite promising physical properties of the graphene, several technical issues need to be solved, towards the full use of its potential in microelectronics. One of the major problem is the creation of effective, stable and low resistance electrical contacts to the surface of graphene. The high contact resistance that exists between metal electrodes and the graphene must be decreased. One of the potential solution involves the formation of an additional buffer layer of particular metal having a large work function difference with graphene e.g. titanium, platinum.

This work will present our results of the thermal evolution of thin Pt layer (10 nm) deposited on the graphene/Ge/Si system. Graphene layer was synthetized by CVD technique on commercially available Ge(110)/Si(110) substrates. DC magnetron metal deposition under high vacuum condition method was used for Pt layer formation. An analysis of the changes in the metallic layer surface morphology, its thermal stability, as well as the influence of the graphene on interactions between Pt layer with both graphene and semiconducting substrate (Ge/Si), was mainly based on the Scanning Tunneling Microscopy (STM) technique. STM measurements and sequential annealing of the samples were realized under ultra-high vacuum conditions.

Given the fact that in the typical CMOS technology the production of electronic devices is a multistage process involving several annealing steps, we believe that our findings will allow to optimization of the formation process of electronic devices containing graphene as an active channel.

Acknowledgements:

This work has been partly supported by the Polish Ministry of Science and Higher Education projects no. 06/62/SBAD/6208 and 06/62/SBAD/1922, as well as from the statutory funds of The Łukasiewicz Research Network - ITME.

OPTIMISATION OF ZrC GRADIENT COATINGS

Szparaga Łukasz[1]*, Mydłowska Katarzyna*[1]*, Gilewicz Adam*[1]*,*
Bartosik Przemysław[1]*, Ratajski Jerzy*[1]

[1]*Faculty of Mechanical Engineering, Koszalin University of Technology,*
Śniadeckich 2, 75-453 Koszalin, Poland

Corresponding author: lukasz.szparaga@tu.koszalin.pl

Gradient coatings belonging to the group of FGM (Functionally Graded Materials), deposited by PVD (Physical Vapor Deposition) techniques, enable effective possibilities for shaping of changes in mechanical properties and the state of residual stress in the substrate/coating systems. This directly results in the possibility of optimising of these coatings due to their anti-wear properties, by appropriate selection of changes in material properties of the gradient layer, such as Young's modulus, yield strength, Poisson's ratio and thermal expansion coefficient. The aim of the research was to develop an optimisation procedure using FEM (Finite Element Modelling) models supporting designing of a gradient coating with high fracture toughness, low wear and high adhesion to the substrate. In the mathematical model, the gradient coating was represented by so-called transition functions (describing the spatial change in mechanical properties) and the adopted decision criteria were functions of stress states and deformations in the substrate/coating system generated as a result of tangential and normal mechanical loads. Optimisation of the gradient coating structure was carried out on the example of ZrC coatings deposited by the Magnetron Sputtering PVD (MS PVD) technique, which mechanical properties can be changed by controlling the carbon concentration in the coating. Using the developed optimisation procedure, a number of prototypes of gradient coatings with improved anti-wear properties compared to bi-layer coatings were selected. i.e. with higher critical loads in scratch test and lower wear ratios in the ball-on-disc tests.

Acknowledgements
This work was supported by the National Science Center, Poland
[grant: Opus 11, No. 2016/21/B/ST8/01738]

EVOLUTION OF PHASE COMPOSITION AND ANTIBACTERIAL ACTIVITY OF ZrC THIN FILMS DEPOSITED BY MAGNETRON SPUTTERING

Czerwińska Ewa[1], *Mydłowska Katarzyna*[1], *Gilewicz Adam*[1],
Dobruchowska Ewa[1], *Szparaga Łukasz*[1], *Jakubczyk Ewa*[2],
Ceynowa Przemysław[1], *Ratajski Jerzy*[1]

[1] *Faculty of Mechanical Engineering, Koszalin University of Technology,*
Śniadeckich 2, 75-453 Koszalin, Poland
[2] *Centre for Engineering Materials, Mechanical Engineering Sciences,*
University of Surrey, Surrey, Guildford, Surrey, GU2 7XH, UK

Corresponding author: katarzyna.mydlowska@tu.koszalin.pl

In the present study, thin coatings of ZrC were deposited by reactive, pulsed magnetron sputtering of zirconium target in Ar–C2H2 atmosphere on 304L steel substrates. As a part of the work their phase composition, microstructure, coating chemistry and mechanical and antibacterial properties were studied. The phase composition and microstructure were studied using standard techniques such as X-ray diffraction (XRD) and with Raman spectroscopy. The surface chemistry of the coatings was analyzed by using a surface sensitive technique such as X-ray photoelectron spectroscopy (XPS). A correlation between the phase composition, microstructure and mechanical and antibacterial properties of ZrC coatings was developed. Based on the obtained results, it was concluded that the wide possibility of shaping the mechanical properties of ZrC coatings in combination with relatively good antibacterial properties after exceeding 50 at. % of carbon concentration in coatings and high protective potential of these coatings makes them a good candidate for medical applications.

Acknowledgements
This work was supported by the National Science Center, Poland
[grant: Opus 11, No. 2016/21/B/ST8/01738]

ULTRA-FAST GROWTH OF CUPRIC OXIDE THIN FILMS FOR PHOTOVOLTAIC APPLICATIONS

M. Ozga [1], *B.S. Witkowski* [1], *J. Kaszewski* [1], *R. Pietruszka* [1], *P. Sybilski* [1],
M. Godlewski [1]

[1] *Institute of Physics Polish Academy of Sciences, al. Lotnikow 32/46,
PL-02-668 Warsaw, Poland*

Corresponding author: ozga@ifpan.edu.pl

Cupric oxide is a p-type semiconductor with direct and narrow energy gap between 1.2 eV – 2.1 eV. Due to many beneficial properties CuO is classified as a good material for photovoltaic applications. It represents cheaper and safer alternative to thin film solar cells based on CIGS and CdTe.

In this work, we introduce an extremely simple, fast and safe technology for deposition of cupric oxide thin films from aqueous solution in an open system. Developed method is based on a dynamic reaction in the hydrothermal process. It is characterized by high growth rate (process lasts only several minutes), low growth temperature (below 100°C) and simple control of the films thickness. The process does not require high purity of used materials or sophisticated equipment, so the presented approach is really low-cost technology. Characterization of the obtained thin films and their potential applications in solar cells also will be presented.

This work was partially supported by the National Centre for Research and Development TECHMATSTRATEG1/347431/14/NCBR/2018.

S-PHASE ANTIMICROBIAL COATINGS DEPOSITED ON AUSTENITIC STAINLESS STEEL BY REACTIVE MAGNETRON SPUTTERING

Słowik J.[1]*, Pendrak K.*[2]*, Fryska S.*[1]*, Baranowska J.*[1]

[1]*West Pomeranian University of Technology, Szczecin, Faculty of Mechanical Engineering and Mechatronicts, Chair of Materials Technology; al. Piastów 19; 70-310 Szczecin, Poland*
[2]*West Pomeranian University of Technology, Szczecin, Faculty of Biotechnology and Animal Husbandry, Department of Immunology, Microbiology and Physiological Chemistry (Department of Microbiology and Biotechnology), Piastów 45, 70-311 Szczecin*

Corresponding author: justyna.slowik@zut.edu.pl

Along with the increasing problem of hospital infections, there is a need to introduce solutions that limit the development of microorganisms on sinks, elements of ventilation systems and touch surfaces such as door handles or handrails. These are mostly made of austenitic stainless steel, which are corrosion resistant and has aesthetic metallic surface. The main disadvantages of this steel are a lack of antibacterial properties, and low resistance to scratches, which results in the creation of additional sites favouring the development of biofilm. In order to address these issues hard S-phase coatings with copper were deposited on austenitic steel components.

Coatings with different copper content in the S phase structure were obtained by the reactive magnetron sputtering method by simultaneous sputtering of austenitic steel and copper targets in a nitrogen atmosphere. The differences in copper content were obtained by changing the intensity of copper target sputtering through the variation of the current generated on the target. The coatings were tested for chemical and phase composition as well as for mechanical properties and antimicrobial activity. It was confirmed that the copper content in the coating increases as the current applied to the copper target increases. The presence of S-phase was evaluated by X-ray diffraction. The coatings with the addition of copper were characterized by good antimicrobial properties, especially against *Staphylococcus aureus*.

SURFACE AND PHASE TRANSFORMATIONS OF Mg-La SUPPORTED COBALT CATALYSTS FOR AMMONIA SYNTHESIS

Aleksander Albrecht[1]*, Adam Sarnecki*[1]*, Paweł Adamski*[1]*,*
Dariusz Moszyński[1]

[1]*West Pomeranian University of Technology, Szczecin, Faculty of Chemical Technology and Engineering, Institute of Inorganic Chemical Technology and Environment Engineering, Pułaskiego 10, 70-322 Szczecin, Poland*

Corresponding author: adam.sarnecki@zut.edu.pl

More and more restrictive environmental requirements impose the need for the new catalysts for ammonia synthesis to be durable, inexpensive and eco-friendly. Catalysts containing cobalt are a promising group of candidates to replace conventional iron-based ones. Most of the latest proposals of the cobalt-based ammonia synthesis catalysts include supported catalysts. Cobalt-based catalysts supported on a mixed magnesium-lanthanum oxide proved to have promising activity in the ammonia synthesis. This kind of structure guarantees a large surface specific area and strong electron-donor character, important in ammonia synthesis catalysis.

Although the chemical composition of catalyst precursor is well known, the question of the chemical composition of active catalysts during a chemical reaction is still open. Metallic cobalt is supposed to be an active phase of these systems. Therefore the reduction of cobalt oxides in catalyst precursor is required.

In the present work, the activation process of cobalt catalysts was examined. The reduction of precursors was performed in a High Pressure Cell (HPC), being a part of an ultra-high vacuum (UHV) system, at 550°C under hydrogen atmosphere. After the reduction HPC was evacuated down to the pressure of 10^{-6} mbar and the sample was transferred under UHV to the analysis chamber of an electron spectrometer. X-ray photoelectron spectroscopy (XPS) was used to investigate the evolution of the surface composition. Qualitative and quantitative surface compositions of precursors and activated catalysts were determined. In-situ X-ray Diffraction (XRD) experiments were done under similar conditions in order to determine phase transformations during the activation process.

XRD experiment has not confirmed the presence of cobalt in the samples. The phase transformations occur only for La-containing compounds. XPS experiment showed that the reduction of cobalt occurs only on the surface and it depends on the sample preparation method.

Aleksander Albrecht thanks the Polish Ministry of Science and Higher Education for support through the project "Diamentowy Grant" no. DI2018003348 funded in years 2019-2022.

CORRELATION BETWEEN MODULATION FREQUENCY AND ENTHALPY OF ATOMIZATION DURING PULSED MAGNETRON SPUTTERING OF SELECTED TRANSITION METALS

G W. Strzelecki[1]*, K. Nowakowska-Langier*[1]*, S. Okrasa*[1]*, M. Jacniak* [1]*, M. Kuk*[1]*,*
K. Zdunek[2]

[1] *National Centre for Nuclear Research, Andrzeja Sołtana 7, 05-400 Otwock, Poland*
[2] *Faculty of Materials Science and Engineering, Warsaw University of Technology, Wołoska 141, 02-507 Warsaw, Poland*

Corresponding author: grzegorz.strzelecki@ncbj.gov.pl

Plasma generation parameter of modulation frequency (f_{mod}) in modulated Pulsed Magnetron Sputtering (mPMS) is studied as an influence on the synthesis of thin metallic films by using a unique power supply unit with phase modulation of the plasma generating waveform in the range of 100kHz. In this study, materials were selected from the d-bloc and ranked according to their enthalpy of atomization ($\Delta H_{at}^{\ominus}$). External characteristics of the power supply are measured with an oscilloscope (U(t), I(t)) and corresponding parameters are calculated: power per package and fill factor. Obtained results for synthesised films and synthesis conditions are correlated with enthalpy of atomization and plasma parameters. The effects of f_{mod} on sputtering effectiveness are discussed.

INVESTIGATION OF THERMOMECHANICAL EFFECTS IN TiAlN COATINGS DEPOSITED BY HIPIMS TECHNIQUE ON WC-Co SUBSTRATES

Myśliński Piotr[1], *Greczyński Grzegorz*[2], *Szparaga Łukasz*[1], *Mydłowska Katarzyna*[1], *Ratajski Jerzy*[1]

[1] *Faculty of Mechanical Engineering, Koszalin University of Technology, Śniadeckich 2, 75-453 Koszalin, Poland*
[2] *Thin Film Physics Division, Department of Physics (IFM), Linköping University, SE-581 83 Linköping, Sweden*

Corresponding author: lukasz.szparaga@tu.koszalin.pl

The conducted research developed issues regarding the use of the modulated temperature thermomechanical analysis (MT TMA) for investigating thermomechanical effects in substrate/coating systems. The object of the research were $Ti_{1-x}Al_xN$ coatings deposited on a cemented carbide WC-Co substrates, using the HIPIMS (High Power Impulse Magnetron Sputtering) technique. The scope of the research included the analysis of the influence of aluminum content in the coating on the thermal and mechanical properties of the substrate/coating system, assessed on the basis of the dilatometric response of substrates to applied thermal loads. In particular, was determined the dependence between the temperature evolution of the value of coefficient of thermal expansion (CTE) of the systems substrate and the content of aluminum in the coating. In addition, the values of differences between linear elongations of substrates/coating systems after deposition and at the end of imposed thermal loads cycles were determined, which in combination with information about CTE change, enabled qualitative assessment of changes in residual stresses in the coatings of the analyzed systems. The scope of experimental research also included the assessment of changes in rheological properties of systems, based on the analysis of phase shifts between applied thermal load and the dilatometric response of the system's substrate. Using the obtained experimental results of changes in thermal and mechanical properties of the systems, it was also possible to determine the genuine substrate's temperature during the coating deposition process.

EFFECT OF HIPIMS PULSE MODE AND PULSE PARAMETERS ON REACTIVE SiO_x DEPOSITION PROCESS

Anna W. Oniszczuk[1], *Piotr Różański*[1], *Paweł Lesiuk*[1]
Arutiun P. Ehiasarian[2], *Daniel Loch*[2]

[1]*TRUMPF Huettinger Sp. z o.o., Marecka 47, 05-220 Zielonka, Poland*
[2]*National HIPIMS Technology Centre, Materials and Engineering Research Institute, Sheffield Hallam University, Howard Street, Sheffield S1 1WB, UK*

Corresponding author: Anna.Oniszczuk@TRUPMF.com

Silicon oxides are widely used thin films. Due to their optical transparency and dielectric properties they are commonly used in optical applications. A high hardness and good scratch resistance are desired to maintain optical performance over a long period of use. The High Power Impulse Magnetron Sputtering technology (HIPIMS) is well-known for depositing a hard and dense coating, providing the opportunity to improve the properties of SiOx thin films. Effect of HIPIMS discharge on the Si deposition process stability is unknown. In this work we discuss HIPIMS operating on Si targets, both on 4” laboratory size and on an industrial 1321 x 127 mm target. Process stability in a reactive O_2 atmosphere is investigated in uni-polar (monopulse) and bi-polar (bipulse) pulsing modes. Effect of the O_2 partial pressure on the plasma ionization is investigated using optical emission spectroscopy and a Langmuir flat probe. A positive influence of a reverse voltage on arcing rate is discussed. Moreover, it was observed that arcing rate can be lowered by changing the pulse shape, especially with the reduction of the current rising rate. Finally, the effect of the pulse duration and frequency on the cathode current, cathode voltage and ion flux is investigated in detail.

NON-CONVENTIONAL DIAGNOSTICS BY CALORIMETRIC AND FORCE PROBES FOR PLASMA-BASED THIN FILM DEPOSITION

H. Kersten, T. Trottenberg, A. Spethmann, M. Klette, L. Hansen

Institute for Experimental and Applied Physics, Kiel University, Kiel, Germany

For an optimization of plasma-based processes as thin film deposition or surface modification, respectively, suitable diagnostics are required. In addition to well-established plasma diagnostic methods (e.g. optical emission spectroscopy, mass spectrometry, Langmuir probes, etc.) we perform examples of "non-conventional" diagnostics, which are applicable in technological plasma processes. Examples are the determination of energy fluxes by calorimetric probes and measurement of momentum transfer due to sputtered particles by force probes. In particular, energy and momentum transfer transport through the plasma sheath combined with the possibility to measure the effect of charge carriers as well as energetic neutrals are of interest and become possible by these diagnostics.

Total energy fluxes from plasma to substrate have been measured by special calorimetric sensors. A typical method is the passive thermal probe (PTP) based on the determination of the temporal slope of the substrate surface temperature (heating, cooling) in the course of the plasma process. By knowing the calibrated heat capacity of the sensor, the difference of the time derivatives yields the integral energy influx to the surface. Simultaneously, the electrical current to the substrate can be obtained and by variation of bias voltage the energetic contributions of charge carriers can be determined. By comparison with model assumptions on the involved plasma-surface mechanisms the different energetic contributions to the total energy influx can be separated.

Furthermore, for thin film deposition by sputtering it is essential to determine the sputtering yield as well as the angular distribution of sputtered atoms. In addition to simulations (TRIM, TRIDYN etc.) an experimental determination of the related quantities is highly demanded. For this purpose, we developed a suitable interferometric force probe. The sensitive probe bends a few µm due to momentum transfer by the bombarding and released particles, i.e. sputtered target atoms and recoiled ions. By knowing the material properties of the cantilever and by measuring its deflection, the transferred momentum (e.g. the force in µN range) can be determined experimentally. In the present study, measurements are compared with TRIM simulations for different experimental discharge conditions.

STABILITY OF Cu_3N OBTAINED BY PULSE MAGNETRON SPUTTERING METHOD AFTER ANNEALING

S. Okrasa[1], K. Nowakowska-Langier[1], R. Chodun[2], K. Król [3],G. Strzelecki[1], R. Minikayev[4], K. Namyślak[1], M. Wilczopolska[1], K. Zdunek[2]

[1] National Centre for Nuclear Research (NCBJ), Material Physics Department, Andrzeja Soltana 7, 05-400 Otwock-Swierk, Poland
[2] Faculty of Materials Science and Engineering, Warsaw University of Technology, Woloska 141, 02-507 Warsaw, Poland
[3] Institute of Microelectronics and Optoelectronics, 75 Koszykowa Str., 00-662 Warsaw, Poland
[4] Polish Academy of Sciences, Institute of Physics, 32/46 Aleja Lotnikow Str., 02-668 Warsaw, Poland

Corresponding author: sebastianokrasa@gmail.com.

Synthesis of Cu_3N layers by surface engineering methods encounter difficulties in receiving a stoichiometric material due to accompanying to those methods thermal effects. As is known, Cu_3N is a metastable semiconductor that, due to its properties is a very promising material but also characterized by a narrow range of thermodynamic stability of the Cu3N phase, which decomposes during heating (250-400°C).

In this work, synthesis of copper nitride layers was conducted employing modulated Pulsed Magnetron Sputtering (mPMS). As our earlier research shows [1], it is a method with which we are able to obtain layers of copper nitride with different lattice parameters (due to changing stoichiometry). In order to determine the stability of obtained material and the effect of changes on its' structure, the samples were subjected to annealing at different temperatures. The annealing processes were carried out in vacuum during 90 minutes at temperatures ranging from 130°C to 400°C. Afterwards the material was characterized in terms of phase composition by X-Ray diffraction and Raman spectroscopy measurements. Furthermore, its' functional properties were measured. Structural analysis and comparisons to the control sample were carried out. Changes in the material were identified and stages of Cu_3N decomposition were described with correspondent temperatures at which they occure.

PHOTOVOLTAIC STRUCTURES BASED ON ZnO/Si HETEROJUNCTION

R. Pietruszka[1], *B.S. Witkowski*[1], *M. Ozga*[1], *M. Godlewski*[1]

[1] *Institute of Physics, Polish Academy of Sciences, Aleja Lotnikow 32/46,*
PL-02668 Warsaw, Poland

ZnO, is an intensively studied semiconductor with a 3.3 eV band gap at room temperature, what makes it a promising candidate for photovoltaic (PV) applications. Most of the studies concentrate on the use of ZnO as a transparent electrode (transparent conductive oxide (TCO)). Other possible applications include the use of ZnO as a buffer layer and/or a n type emitter in Si- based solar cells, as we demonstrated recently. In our work we demonstrate a n-type ZnO structure with a 3D ZnO-nanorods as an alternative low cost materials for construction of efficient ZnO/Si solar cells. Zinc Oxide and ZnO:Al (AZO) films were deposited by atomic layer deposition method (ALD). The 3D zinc oxide nanorods were grown by a hydrothermal method.

Various versions of PV cells of the same architecture were investigated, with a thickness of silicon wafers varied between 50 and 200 µm. Electrical parameters of the wafers are 2 Ωcm and a hole concentration is 10^15 cm^-3.

For 200 µm thick solar cells, we received light conversion efficiency up to 14%. For thinner silicon films the Jsc value decreases from 38 mA/cm2 to 30 mA/cm2, whereas the Voc remains similar, of about 500 mV. The solar cells efficiency varies between ~10% for the cells constructed on the thinnest Si wafers.

This work was partially supported by the National Centre for Research and Development TECHMATSTRATEG1/347431/14/NCBR/2018.

MODIFICATION OF THE AMORPHOUS FeSiB ALLOY SURFACE LAYER BY USING INTERFERENCE PULSED LASER HEATING

Agnieszka Radziszewska[1], *Jan Kusinski*[1], *Olaf Czyz*[1], *Roman Ostrowski*[2], *Krzysztof Morawiec*[3], *Piotr Dłużewski*[3], *Małgorzata Kac*[4]

[1]*AGH University Of Science And Technology, Al. Mickiewicza 30, 30-059 Krakow, Poland*
[2] *Military University of Technology, Institute of Optoelectronics, Warsaw, 2 Gen. S. Kaliskiego, 00-908 Warsaw, Poland*
[3]*Institute of Physics Polish Academy of Sciences, Al. Lotnikow 32/46, 02-668 Warsaw, Poland*
[4]*Institute of Nuclear Physics Polish Academy of Sciences, ul. Radzikowskiego 152, 31-342 Krakow, Poland*

Corresponding author: kusinski@agh.edu.pl

The production of the controlled microstructure from amorphous state has become of special interest for technological applications. In some cases, a suitable heat treatment, which leads to partially nanocrystalline microstructure, may provide an improvement in specific properties. In this paper we describe the generation of nanocrystalline seed islands created in the $Fe_{80}Si_{11}B_9$ amorphous alloy, by pulsed laser interference crystallization (PLIC) and show that seeded growth can be achieved. The use of laser beam, of relatively low energy (120 mJ), but with a subsequent overlapping of heated areas (from 50 to 500 pulsed laser shots), resulted in development nanostructure in the heated areas of the amorphous ribbon. The structural properties of the microcrystalline islands in $Fe_{80}Si_{11}B_9$ amorphous matrix are analyzed by light, scanning electron (SEM), transmission electron (TEM, also using Fresnel and holographic magnetic imaging), and atomic force microscopy (AFM), as well as by HRTEM observations. They indicate that pulsed laser interference heating involves structural transformations in the subsurface layer up to ~300 nm. By means of Fresnel magnetic imaging in TEM, it was possible to show that nanocrystalization of the $Fe_{80}Si_{11}B_9$ amorphous alloy caused significant refinement of the magnetic domain structure. Images of holography fringes carrying information about phase shift of electron wave interacting with a sample. The phase shift consists of contributions introduced by electric and magnetic fields. Certainly, in the laser crystallized FeSiB amorphous ribbon, the detailed distribution of lines of magnetic flux at a nanometer scale are visualized. In the absence of external magnetic field phase shift contours were parallel to the edge in the amorphous matrix, while Fe(α) crystallites involves visible shift of magnetic lines. Interestingly, phase shift contours, after switching on external magnetic field, drastically altered their configuration. It is clearly seen that external magnetic field, perpendicular to the sample surface, rotates the phase shift contours by almost 90 degrees in the surface plane of the sample. These and other results obtained for FeSiB alloy samples will be presented and discussed.

DC AND AC PEO TREATMENTS OF TITANIUM SUBSTRATE IN ACID ELECTROLYTES

Krzysztof Rokosz[1], Tadeusz Hryniewicz[1], Łukasz Dudek[1], Kornel Pietrzak[1], Raaen Steinar[2], Sofia Gaiaschi[3], Patrick Chapon[3], Winfried Malorny[4]

[1]*Division of BioEngineering and Surface Electrochemistry, Department of Engineering and Informatics Systems, Faculty of Mechanical Engineering, Koszalin University of Technology, Racławicka 15-17, PL 75-620 Koszalin, Polad*
[2]*Department of Physics, Norwegian University of Science and Technology (NTNU), Realfagbygget E3-124 Høgskoleringen 5, 7491 NO Trondheim, Norway*
[3]*HORIBA Jobin Yvon S.A.S., 16-18, rue du Canal - 91165 Longjumeau cedex -France*
[4]*Hochschule Wismar-University of Applied Sciences Technology, Business and Design, Faculty of Engineering, DE 23966 Wismar, Germany*

Corresponding author: rokosz@tu.koszalin.pl

The Plasma Electrolytic Oxidation (PEO), known also as Micro Arc Oxidation (MAO), is widely used in many industries and finishing of metal biomaterials, as well as ongoing development works on them all over the world due to new electrolytes and processing voltage shapes. Depending on the DC or AC mode of PEO used, different chemical compositions of the fabricated coatings, including their thickness and porosity, can be obtained. The obtained results clearly indicate that it is possible to obtain porous phosphate coatings enriched with biocompatible calcium and magnesium as well as with bactericidal zinc and copper. To characterize the PEO coatings, the SEM, EDS, XPS, GDOES, and corrosion polarisation methods, were used. It was also found out that the increase of PEO voltage results in increasing of coatings' thicknesses and their maximum pore diameters. In addition, it was observed that the largest amount of elements derived from electrolyte may be incorporated into the coating at short processing times and high POE voltages. It has also been noted that DC-PEO coatings are thicker than those ones fabricated in the AC-PEO process.

This work was subsidized by the Grant OPUS 11 of the National Science Centre, Poland, with the registration number 2016/21/B/ST8/01952, entitled "Development of models of new porous coatings obtained on titanium by Plasma Electrolytic Oxidation in electrolytes containing phosphoric acid with addition of calcium, magnesium, copper and zinc nitrates".

THE PHYSICOCHEMICAL PROPERTIES OF THE SURFACE MODIFIED PURE TITANIUM BY PVD METHOD

Anna Woźniak[1], *Marcin Adamiak*[1]

[1]*Chair of Engineering Materials and Biomaterials, Faculty of Mechanical Engineerin, Silesian University of Technology, Konarskiego 18A. 44-100 Gliwice, Poland,*

Corresponding author: marcin.adamiak@polsl.pl

The substrate material used in the research was pure titanium Grade II powder, the tested samples were manufactured by SLM method. The energy density delivered to the powder material during SLM process was 75 J/mm^3. All samples were subjected to the mechanical grinding and polishing and in the next stage the surface of the samples was modified by PVD CrN and TiN coatings. Observation of topography and surface morphology of tested coatings were performed in SEM and AFM microscopes. Contact angle measurements and roughness were also performed. In order to determine the corrosion resistance the potentiodynamic test and impedance test were performed. Based on the obtained results it can be concluded that the surface modification by PVD method, using CrN and TiN coatings lead to improvement of SLM produced elements properties. The SEM analysis for the samples after surface modification by PVD method for both group of the samples was properly carried out. The surface modification, regardless of the type of the coating lead to increase value of wetting angle and reduce the values of surface roughness and wear volume. For samples group after modification the lowest value of surface roughness were obtained for Grade_CrN. For samples group with TiN layer the mean value of contact angle was $\theta = 101.5 \pm 7^\circ$ and for Grade_CrN samples group the mean value was $\theta = 98.2 \pm 8^\circ$, which indicate the hydrophobic character of the surface. The highest value of surface roughness was obtained for the samples in initial state $R_a = 0.9 \pm 0.2$ µm and for this samples group the hydrophilic character of the surface was observed $\theta = 79.0 \pm 10^\circ$. The obtained results indicated the different pitting corrosion resistance for all tested samples. The higher value of resistance of the charge transition at phases boarded R_{ct} was recorded for the Grade_TiN samples group. Additionally, for samples with TiN coating the highest value of pitting corrosion potential E_{corr} = -91 mv and transpassivation potential E_{tr} = 2572 mV were recorded. The samples with TiN layer Grade_TiN are characterized by optimum properties.

SPECTRAL INVESTIGATION OF EUV INDUCED, LOW TEMPERATURE PLASMAS CREATED IN GASES AND AEROSOLS

A. Bartnik, W. Skrzeczanowski, P. Wachulak, T. Fok, Ł. Wegrzynski, J. Czwartos, H. Fiedorowicz, J. Kostecki

Institute of Optoelectronics, Military University of Technology, Warsaw, Poland

Corresponding author: andrzej.bartnik@wat.edu.pl

In this work investigations of low temperature plasmas, induced by irradiation of gases or aerosols with intense pulses of extreme ultraviolet (EUV), were performed. The EUV radiation was delivered from laser produced plasma (LPP) sources, developed in Institute of Optoelectronics. To obtain high intensity of the ionizing radiation, it was focused, using a specially prepared grazing incidence collector. The EUV pulse irradiated a small portion of gas or aerosol injected into a vacuum chamber, perpendicularly to an optical axis of the irradiation system, using an auxilary valve. The other option was to irradiate the medium injected into a vicinity of the LPP. Low temperature plasmas created this way, emitted radiation in a wide wavelength range. Spectral measurements of this radiation were performed in the EUV and VUV ranges using grazing incidence, flat-field spectrographs (McPherson, H+P Spectroscopy respectively). Spectra in UV/Vis range were measured using an Echelle Spectra Analyzer ESA 4000. The EUV/VUV spectrographs enabled to obtain only time integrated data while the UV/Vis spectrograph allowed for time resolved measurements.

Temporal measurements in the EUV range were performed for spectrally integrated signals, recorded using a specially prepared detection system, based on an EUV collector and an AXUV detector. The spectral measurements indicated that the most intense emission lines in EUV/VUV range originated from singly charged ions, however, lines corresponding to multiply charged ions were also recorded. In the UV/Vis range atomic or molecular spectra were also detected. For detailed spectral analysis various codes like LIFBASE, Specair, PGOPHER or PrismSpect were employed.

PLASMA-ENHANCED SYNTHESIS OF NANOSTRUCTURED ELETROCATALYSTS FOR FUEL CELL AND ELECTROLYSES APPLICATIONS

Gustav Sievers, Volker Brüser

Leibniz Institut for Plasma Science and Technology, Felix-Hausdorff-Str. 2,
17489 Greifswald, Germany

Corresponding author: brueser@inp-Greifswald.de

Hydrogen is considered to be the main future energy carrier. Its carbon-free production and conversion is one of the main challenges for the breakthrough in sustainable energy conversion technologies. Electrolysis and fuel cells are two important components of green technology.

In materials synthesis, plasma-enhanced surface modification and layer deposition methods are based on the presence of non-equilibrium states of reactive species in a plasma environment. They are therefore able to overcome limitations of traditional catalyst synthesis methods, giving rise to new reaction pathways and resulting in unique properties of nanomaterials.

Polymer electrolyte membrane fuel cells and electrolyzers have been recognised as a potential future power source for zero emission devices. Several plasma methods have already been developed for fuel cell catalyst synthesis. Aspects as corrosion resistance of catalyst support, content of platinum or alternative abundant catalysts respectively are of primary interest.

Nanostructuring of electrocatalysts is an important aspect of catalyst design as catalytic performance depends not only on the specific activity (reaction rate per surface area), but also on the dispersion of the catalyst. We present an industrially compatible, but effective preparation method for support-free nanostructured catalyst layers.

Claussius Sesion Lectures

THE UNIVERSE - A WORLD OF PLASMA

Prof. Dr. Holger Kersten, Institut for Experimental and Applied Physics,University Kiel, Germany,

Plasma as the „fourth state of matter" is the most dominant existence of matter in the Universe. Plasma exists in all of its modifications – from dense and hot thermal fusion plasmas in the interior of the Sun and stars to strongly non-isothermal rarefied plasma of the interstellar space. Whereas plasma is somewhat exotic on earth, it is the common state in the Universe – and the known states of matter on Earth are only the condensed forms of the cosmic plasmas.

A fascinating journey through the World of Plasma will be made during the presentation:

- plasma – the fourth state of matter
- gas discharges in laboratory and their applications
- natural plasmas on Earth and in the solar system
- Sun and stars – compact plasma spheres in the space
- interstellar and intergalactic matter
- summary

The popular scietific lecture will be completed by some amazing discharge experiments.

NANOSCALE MODEL CATALYTIC STUDIES IN UHV: BRIDGING THE HIGH VOLTAGE GAP

P. Winkler, J. Zeininger, M. Raab, G. Rupprechter and Y. Suchorski

TU Wien, Institute of Materials Chemistry, Getreidemarkt 9, 1060 Vienna, Austria

Corresponding author: yuri.suchorski@tuwien.ac.at

Field emission (FEM) and field ion (FIM) microscopy allow imaging metal surfaces on the nm-scale (FEM) or even with atomic resolution (FIM). Usually sharp tips with nm-sized apexes, resembling catalytic particles used in industrial catalysts, are applied as specimens. Using platinum group metal tips as model samples, catalytic reactions in a pressure range from 10-4 to 10-10 mbar can be visualized *in situ* using the above microscopies.

Techniques based on field emission of electrons (FEM) or ions (FIM) require a high electrostatic field in the order of volts per angström, which necessitates direct application of high voltages (up to 20 kV) to the specimen. Various surface science experiments performed using FEM/FIM, such as thermal desorption spectroscopy or catalytic ignition, furthermore require exact measurement and fast-acting control of the sample temperature. This is difficult under high voltage conditions: since it is hardly possible to apply voltages in the kV-range solely to the specimen, typically the entire specimen holder is exposed to the full applied voltage. This creates a “high voltage gap” between the temperature measuring and sample heating facilities (in UHV) being under high potential and the control devices operating (at atmospheric pressure) on ground potential.

To solve this longstanding problem, a novel sample temperature control system based on wireless data transmission was designed and built. The novel system simplifies bridging the “high voltage gap” and significantly improves experimental procedures. The system was tested by *in situ* studies of catalytic CO and H2 oxidation on a nm-sized Rh specimen. Both isothermal and temperature-programmed catalytic experiments were conducted, demonstrating the capabilities and performance of the new system. Generally, the system is applicable not only to FEM, FIM and AP techniques, but for all experiments requiring high voltages to be applied to a specimen.

Acknowledgement: This work was supported by the Austrian Science Fund (FWF) [SFB F45 FOXSI].

NANOSCALE MODEL CATALYTIC STUDIES IN UHV: CONTRIBUTION TO BRIDGING THE MATERIALS GAP

J. Zeininger, M. Raab, P. Winkler, G. Rupprechter and Y. Suchorski
TU Wien, Institute of Materials Chemistry, Getreidemarkt 9, 1060 Vienna, Austria

Corresponding author: yuri.suchorski@tuwien.ac.at

As a component of supported catalysts, noble metal nanoparticles are the main working horse of industrial catalysis. Due to the nanometric dimensions it is, however, difficult to study catalytic reactions on individual nanoparticles. Therefore, simplified systems are used for model studies, but the results often differ from those obtained with real catalysts. The resulting *materials gap* represents a longstanding but still current problem of catalytic research. A promising way to bridge the *materials gap* consists of using sharp nanotips (radius of curvature < 10 nm) made of catalytically active metal, *e.g.* Pt, Pd, or Rh, as model systems. Like nanoparticles, such nanotips consist of differently oriented nanofacets, but in contrary to individual nanoparticles, they can be prepared and characterized on a nm-scale or even with atomic resolution using UHV surface analysis techniques. Methods such as field electron microscopy (FEM) and field ion microscopy (FIM) allow visualization of ongoing catalytic reactions on such samples in real time. In turn, atom probe (AP) and field ion appearance energy spectroscopy (FIAES) provide atomic scale chemical information.

In the present contribution, we report nanoscale model catalytic studies of the H2 oxidation reaction and of the CO oxidation reaction on Rh nanotips. An UHV chamber containing an FEM/FIM system was used as a catalytic flow reactor and the ongoing reaction was imaged in real time with nm-resolution. Kinetic transitions between the catalytically inactive and active states caused by variation of CO and H2 pressure in the 10-6 mbar range were observed *in situ* and video-monitored. Transition points obtained by digital analysis of recorded video-files for a wide range of parameters (pressure, temperature) are summarized in kinetic phase diagrams allowing the direct comparison with other model systems across the *materials gap* and with industrially used catalysts.

Acknowledgements: This work was supported by the Austrian Science Fund (FWF) [SFB F45 FOXSI].

CYLINDRICAL MAGNETRON WITH MULTIPLE DYNAMIC AREAS OF TOROIDAL PLASMA AND CATHODE OR ANODE POLARIZATION

Marek Betiuk, Zbigniew Łataś, Piotr Domanowski, Wojciech Gajewski
Marcin Żelechowski

Institute of Precision Mechanics (IMP), Department of Advanced Technologies,
Laboratory for PVD Technology and Implantation, Duchnicka 3
01-796 Warszawa

Corresponding author: marek.betiuk@imp.edu.pl

The new cylindrical magnetron with a dynamic oscillatory magnetic field, as developed at the Institute of Precision Mechanics, The application of a magnetron and plasma environment for the synthesis of coating materials results in obtaining advanced metallic coating materials, nitric/carbide/oxide metals included in the groups IVB to VIIB of the periodic table (e.g. CrN, NbN, ZrN and ReN, etc.). Anode polarized magnetron can be used in the process of low-pressure glow nitriding. The design of the magnetron cooling system results in reliable operation in vacuum chambers within the range 20 °C to 650 °C. The magnetron is suitable for operation with current power supplies manufactured by national and European manufacturers.

MICROFRICTION OF ZrCN COATINGS CHARACTERIZED BY ATOMIC FORCE MICROSCOPY

T.A. Kuznetsova[1], *V.A. Lapitskaya*[1], *S.A. Chizhik*[1], *B. Warcholinski*[2],
A. Gilewicz[2]

[1] *A.V.Luikov Heat and Mass Transfer Institute of NAS Belarus, P.Brovki 15,*
220072, Minsk, Belarus
[2] *Koszalin University of Technology, Śniadeckich 2, 75-453, Koszalin, Poland*

Corresponding author: bogdan.warcholinski@tu.koszalin.pl

ZrCN coatings were formed on the polished steel substrates by magnetron sputtering using the mixture of acetylene and nitrogen and pure zirconum target at the various rates of acetylene flow in the reaction chamber (0-3 cm^3/min). The thickness of the ZrCN coatings was approximately 3 μm. Using atomic force microscopy (AFM) with a diamond tip in the Contact Mode under ambient temperature and humidity controlled conditions, we studied the surface morphology, roughness (Ra), friction coefficient (C_{fr}), friction force (F_{fr}), specific volumetric wear (ω) of ZrCN coatings with the construction of C_{fr} dependencies on the number of scan cycles. It was found that the coating obtained at a acetylene flow rate of 3 cm^3/min has the lowest values of C_{fr} (0,062±0,004) and of F_{fr} (196,3±14,6 nN) among the ZrCN coatings. The highest C_{fr} (0,103±0,013) and F_{fr} (324,5±42,1 nN) has ZrCN coating obtained at a acetylene flow rate of 1.5 cm^3/min. The dependence of the C_{fr} and F_{fr} on the consumption of acetylene for ZrCN coatings is nonmonotonic. It was determined that Ra on the area 1x1 μm changes with increasing of acetylene concentration in the gas mixture nonmonotonously too: from 5.27 nm for 1 cm^3/min, to 5.96 nm for 1.5 cm^3/min and to 2.68 nm for 3 cm^3/min. C_{fr} correlates with Ra and don't completely correlates with ω of ZrCN coatings. The depth of wear obtained with similar conditions is 2.2 nm for 1 cm^3/min, 6.6 nm for 1.5 cm^3/min and 7.7 nm for 3 cm^3/min.

Acknowledgments

This research was supported by the grant of Belarusian Republican Foundation for Fundamental Research BRFFR – SCST – Poland No. T18PLDG-002. Publication partly financed by the National Centre for Research and Development, Poland, BIOSTRATEG3/344303/14/NCBR/2018. The participation in the Conference was supported by the EU Program MOST (Mobility Scheme for Targeted People-to-People-Contacts).

DETERMINATION OF FRACTURE TOUGHNESS OF COATINGS BY INDENTATION METHOD WITH THE AFM CRACKS DIAGNOSIS

V.A. Lapitskaya [1], *T.A. Kuznetsova* [1], *S.A. Chizhik* [1], *B. Warcholinski* [2], *A. Gilewicz* [2]

[1] *A.V.Luikov Heat and Mass Transfer Institute of NAS Belarus, P.Brovki 15, 220072, Minsk, Belarus*
[2] *Koszalin University of Technology, Śniadeckich 2, 75-453, Koszalin, Poland*

Corresponding author: bogdan.warcholinski@tu.koszalin.pl

One of the main physical and mechanical properties of thin hard coatings is the fracture toughness. The fracture toughness is determined by various methods (bending, chipping, indentation), but not everything can be applied to the coatings. A common method for the fracture toughness determination the of the coating is the indentation method by introducing the Vickers pyramid into the coating surface, followed by measuring the parameters of the prints and the length of the radial cracks in the prints. Quantitatively, the fracture toughness is characterized by a critical stress intensity factor K_{1C}. Traditionally, optical microscopy is used to quantify the crack lengths, and in rare cases, scanning electron microscopy (SEM). The use of atomic force microscopy (AFM) is promising for the accurate detection of microhardness print parameters and determination the type of cracks on the surface. The AFM method allows one to reveal the features of deformation and fracture of the material from the topography of the surface, which are not determined by either optics or SEM. At low loads, the error in determining the length of cracks and indentation parameters by the optical method compared to the AFM reaches 30% (of which 20% is the determination of the crack length and 10% is the determination of the indent diagonal length).

Acknowledgments

This research was supported by the grant of Belarusian Republican Foundation for Fundamental Research BRFFR – SCST – Poland No. T18PLDG-002. The participation in the Conference was supported by the EU Program MOST (Mobility Scheme for Targeted People-to-People-Contacts).

DLC FILM-POLYMERIC SUBSTRATE INTERACTION

Agata Niemczyk[1], *Dariusz Moszyński*[2], *Jolanta Baranowska*[1]

[1] *West Pomeranian University of Technology, Szczecin, Faculty of Mechanical Engineering and Mechatronics, Chair of Materials Technology; al. Piastów 19; 70-310 Szczecin, Poland,*
[2] *West Pomeranian University of Technology, Szczecin, Faculty of Chemical Technology and Engineering, Department of Chemical and Environment Engineering; al. Piastów 42; 71-065 Szczecin, Poland,*

Corresponding author: Jolanta.Baranowska@zut.edu.pl

Heretofore, most of the research on DLC films have been focused on the characteristics of the variety of the properties and applications of the films deposited on metallic and ceramics substrates. Nonetheless, more recently an increase of scientific interest on the DLC films on the polymeric substrates is noted, broadening the DLC films exploitation areas and taking a part in the discussion on theories about the diamond-like structure creation. Therefore, the aim of the work was to obtain DLC films deposited on different polymeric substrates and study the interaction between the DLC film and the substrates. The graphite target was ablated with the KrF laser in vacuum at the room temperature. The structure of DLC films and the substrates were studied using Raman, Infrared and X-ray photoelectron Spectroscopies. The quality of the films, in terms of microcracks and delamination was evaluated by scanning electron microscopy (SEM). The DLC film characteristic was discussed in relation to the substrate materials and their surface properties.

TRENDS IN BORIDING UNDER GLOW DISCHARGE CONDITIONS

Michał Kulka

Poznan University of Technology, Institute of Materials Science and Engineering, pl. M.Sklodowskiej-Curie 5, 60-965 Poznan

Corresponding author: michal.kulka@put.poznan.pl

All the specified techniques of boriding, carried out under glow discharge conditions, were characterized and compared based on the available literature data. The technological aspects of boriding processes were analyzed, taking into consideration the advantages and disadvantages of each method. The effects of the boriding techniques on the microstructure of borided materials were shown. The mechanism of formation of active boron atoms or ions were described. The five main techniques of boriding under glow discharge conditions were specified: plasma gas boriding, plasma paste boriding, plasma electrolytic boriding, spark plasma sintering boriding, and double glow plasma surface alloying with boron.

LOW TEMPERATURE NITRIDING OF THE NANOBAINITIC X37CrMoV5-1 STELL

Michał Tacikowski[1], *Jerzy Ratajski*[2], *Kinga Grabowska*[1], *Szymon Marciniak*[1],
Jacek Rudnicki[1], *Paweł Marchlewski*[3], *Marcin Pisarek*[4]

[1] *Warsaw University of Technology, Faculty of Materials Science and Engineering, ul. Wołoska 141, 02-507 Warszawa*
[2] *Koszalin University of Technology, Faculty of Mechanical Engineering, Racławicka 15-17, 75-620 Koszalin, Poland*
[3] *Institute of Precision Mechanics, ul. Duchnicka 3, 01-796 Warszawa*
[4] *Institute of Physical Chemistry of the Polish Academy of Sciences, Kasprzaka 44/52, 01-224 Warsaw, Poland*

Corresponding author: michal.tacikowski@pw.edu.pl

Producing nanocrystalline high strength steels using heat treatment consist one of the prospective challenges for the modern materials engineering. Nanocrystalline structure may be obtained in certain steels grades by the austempering which result in the nanobainite formation of the ausferitte type which give steels an extremely high strength, associated with significantly improved ductility and toughness. However the wear resistance of the nanobainitic steels is often not satisfying for some kind of applications. In industrial practice when a high strength and toughness in combination with increased wear resistance is needed the structure steels are thermally hardened by quenching and tempering and then usually subjected to the gas nitriding process, which is usually executed in the temperatures between 500 and 600 ºC. However due to the low thermal stability of the austempered steels nanostructure the conventional nitriding procedure should be excluded. This impose the search for the adequate surface engineering solution, able to improve surface properties of the nanocrystalline steels without the harmful thermal effect on their bulk nanostructure and resulting mechanical properties. As it was found the nanostructurised X37CrMoV5-1 steel exhibits a sufficiently high thermal stability of the nanobainitic structure to conduct the nitriding processes in the temperatures range close to 400 °C. Therefore in the present work the effect of the low temperature gas nitriding on the structure and properties of the nanostructurised X37CrMoV5-1 steel was investigated in order to check its effectiveness, comparing to the conventional nitriding procedure and in particular with respect to alternative glow-discharge nitriding. The preliminary results shows that gas nitriding performed in temperatures close to 400°C is able to produce about 70 μm thick and up to 1460 HV0,05 hard nitrided layers which significantly increases the wear resistance of the nanostructurised X37CrMoV5-1 steel without destabilizing its nanostructure, what gives better performance than other nitriding options.

THE PROPERTIES OF AlCrBN COATINGS DEPOSITED USING CATHODIC ARC EVAPORATION

A. Gilewicz, P. Myśliński, E. Dobruchowska, D. Murzyński, Z. Galocz, D. Jakrzewski, K. Szafirowicz, B. Warcholiński

Koszalin University of Technology, Faculty of Mechanical Engineering, Raclawicka 15-17, 75-620 Koszalin, Poland

Corresponding author: adam.gilewicz@tu.koszalin.pl

AlCrN are one of the most widely studied hard coatings, especially if it is about their thermal resistance and mechanical properties. An improvement of mechanical properties of the coatings can be achieved by doping with a metallic or non-metallic element. The paper presents the results of deposition and testing the AlCrBN coating, especially the effect of nitrogen pressure during coating deposition. The coatings were synthesized by means of cathodic arc evaporation equipped with AlCrB targets with chemical composition (50:30:20).The coatings were deposited in nitrogen pressure p_{N2} ranged from 2 Pa to 5 Pa.

The morphology, phase and chemical composition of the coatings were studied by scanning electron microscopy, X-ray diffraction and energy dispersive spectroscopy, respectively. The mechanical and tribological properties were evaluated by nanoindenation and ball-on-disc test, respectively. Adhesion of the coatings was investigated using scratch test and additionally Daimler-Benz test. The corrosion properties of coating – steel substrate systems were investigated using potentiodynamic polarisation tests in 3.5 wt % NaCl solution. Corrosion potential, corrosion current density and polarization resistance were determined.

In AlCrBN coatings deposited at nitrogen pressure of 2 Pa the diffraction lines characteristic for the hexagonal AlN phase were observed. The increase in nitrogen pressure results in a decrease in the intensity of the observed AlN lines. In coatings formed under nitrogen pressure of 5 Pa there are diffraction lines from the cubic CrN phase. The hardness of the coatings is about 22-23 GPa and is independent of nitrogen pressure during coating deposition. The hardness of the coatings depends on the substrate bias voltage U_B and increases from 18.7GPa (U_B = -50 V) to23.1 GPa (U_B = -150 V). Similar effect is observed for arc current, 20.4 GPa (Ic = 80 A) and 24.0 GPa (Ic = 120). Critical load for coatings is relatively high, the lowest is about 67 N (p_{N2} = 2 Pa) and the highest - about 100N (p_{N2} = 2 Pa). The wear rate decreases with nitrogen pressure from 3.5×10^{-6} mm^3/Nm (p_{N2} = 2 Pa) to 3.9×10^{-7} mm^3/Nm (p_{N2} = 4 Pa).

The AlCrBN coating significantly increases the corrosion resistance of the HS6-5-2 steel substrate in the environment of 3.5 wt% aqueous NaCl solution. The corrosion potential of the coating is about 0.400 V higher in relation to the substrate. This is accompanied by a decrease in the corrosion current density by about two orders of magnitude and an increase in polarization resistance by about two orders of magnitude, which indicates a decrease in the speed of corrosion processes compared to an uncoated steel substrate.

Investigations partly financed by the National Centre for Research and Development, Poland, BIOSTRATEG3/344303/14/NCBR/2018.

MECHANICAL PROPERTIES OF AlCrN COATINGS DEPOSITED USING CATHODIC ARC EVAPORATION

A. Gilewicz[1], P. Myslinski[1], R. Jedrzejewski[2,3], B. Warcholinski[1]

[1] Koszalin University of Technology, Faculty of Mechanical Engineering, Raclawicka 15-17, 75-620 Koszalin, Poland
[2] West Pomeranian University of Technology Szczecin, Faculty of Mechanical Engineering and Mechatronics, 19 Piastow Ave., 70-313 Szczecin, Poland
[3] Lukasiewicz Research Network – PORT Polish Center for Technology Development, Stablowicka 147, 54-066 Wroclaw, Poland

Corresponding author: adam.gilewicz@tu.koszalin.pl

The Al-Cr-N coatings were formed from AlCr (50:50) cathode at various nitrogen pressure, substrate bias voltage and substrate temperature using cathodic arc evaporation. The relationship between technological parameters and properties of the coatings was investigated. The phase and chemical composition of the coatings, roughness, hardness, adhesion and thermal stability were analyzed by xrd, sem, edx, micro-indenter, rockwell and scratch tester and modulated-temperature dilatometer.

For coatings deposited at 350°C and 450°C the deposition rate increases to 3 Pa of nitrogen pressure and then decreases. With substrate bias voltage increase the deposition rate decreases. The deposition rate for coatings formed at 450°C is less from 4% to 25% than for coatings obtained at lower temperatures.

XRD analysis indicate that coatings crystallize in cubic CrN structure and show preferential orientation in (200) plane. With increase nitrogen pressure the preferential orientation changes to (111).

EDX analysis shows that with nitrogen pressure increase, and the Al/(Al+Cr) rate decreases. The microscopic observations indicate that number of macroparticles reduces with nitrogen pressure increase. The results of the studies show that with the increase of the nitrogen pressure and the substrate bias voltage, the surface roughness parameter Ra coatings decreases. The hardness of the coatings depends on the deposition parameters: with the increase of the nitrogen pressure in the chamber and the substrate bias voltage the hardness increase. With an increase in deposition temperature the hardness decrease. AlCrN coatings show very good adhesion to the substrate. The critical force for coatings formed at nitrogen pressure from 1 Pa to 5 Pa and temperature 350°C is nearly constant and is approximately 90 N. The coatings deposited at 450°C characterizes by about 20% lower critical force.

Investigations partly financed by the National Centre for Research and Development, Poland, BIOSTRATEG3/344303/14/NCBR/2018.

OPTIMIZATION OF THE ARCHITECTURE AND PROPERTIES OF MULTILAYER COATING TiAlN/Cr/W-DLC FOR APPLICATION ON TAPS FOR AUSTENITIC STEELS

J. Walkowicz[1], A. Czyżniewski[1], Z. Galocz[1], A. Urbanowicz[2], D. Ptaszkiewicz[2]

*[1]Koszalin University of Technology, Faculty of Mechanical Engineering,
Racławicka 15-17, 75-620 Koszalin, Poland*
*[2]Tool Factory Fanar Joint Stock Company, Płocka 11, 06-400 Ciechanów,
Poland*

Corresponding author: jan.walkowicz@tu.koszalin.pl

Multilayer hard/lubricant coatings, made of an inner hard layer and an outer soft lubricating layer, are used to coat tools for dry cutting. The hard layers used include e.g. TiN, TiAlN, AlTiN, and as lubricating layers carbon layers modified with carbide-forming metals are frequently applied. The cutting properties of coated tools are especially influenced by the properties of the lubricating layer and the properties of the transition layer between the hard and lubricating layers. Properties of carbon layers modified with carbide forming metals depend on the mutual contribution of the carbide phase (TiC, WC, CrC) and the amorphous carbon matrix a-C:H or a-C. When the content of the amorphous carbon phase exceeds 80%, the layers are characterized by the ability to graphitize during friction and, as a result, a low coefficient of friction under dry cutting conditions.

The aim of the presented research was to optimize the architecture and properties of the TiAlN/Cr/W-DLC coating designed for taps used for machining austenitic steels. Coatings were deposited by reactive magnetron sputtering in an industrial CC800/9ML device from CemeCon AG. In order to achieve high adhesion between hard (TiAlN) and lubricating (W-DLC) layers, a transition layer consisting of Cr layer, Cr/WC modulated layer, WC layer and WC/W-DLC gradient layer was used. Based on preliminary studies, it was assumed that the structure of the WC/W-DLC gradient layer, the thickness of the W-DLC lubrication layer, the thickness of the Cr layer, and the total thickness of the Cr/WC modulated layer and the WC layer significantly affect the properties of the entire coating. The architecture and properties of the coating were optimized using the Taguchi method. In the first stage the experimental plan based on the L9(3^4) orthogonal array was used, while in the second stage the plan based on the array L4(2^3) was applied.

THERMOMECHANICAL DIAGNOSTICS OF WEAR RESISTANT AlCrN COATING - HS6-5-2 STEEL SUBSTRATE SYSTEMS FOR THE OPTIMIZATION OF THEIR PVD TECHNOLOGY

P. Myśliński, A. Gilewicz, K. Mydłowska, B. Warcholiński

Koszalin University of Technology, Faculty of Mechanical Engineering,
Racławicka 15-17, 75-620 Koszalin, Poland

Corresponding author: piotr.myslinski@tu.koszalin.pl

There is a significant temperature rise in contact between the machine tool for woodworking and the workpiece. This activates and strengthens, often synergistic, most mechanisms leading to changes in the operational properties of coatings. Currently, the most commonly used method to determine the effects of modification of cutting tool working surfaces by deposition wear resistant PVD coatings is the test operation of these tools. The results of tests on the thermal stability of coatings at the stage of developing new technologies or their optimization are not widely used. The research concerned the identification of thermomechanical sensitivity of PVD coating systems - HS6-5-2 steel substrate to heat induced stresses in them. The tests were carried out with the use of samples constituting physical models of PVD coating - substrate systems. It was assumed that in the tested samples there is a variable thermomechanical load of the surface by the deposited adhesive coating. Therefore, in metrological terms, they can be objects with variable rheological properties as a function of coating deposition technology, including temperature and time. The conditions for testing such systems are provided by Temperature Modulated Thermomechanical Analysis MT TMA. AlCrN coatings were deposited using cathodic arc evaporation with various technological parameters. Variable parameters of the technology were: composition of cathode material (AlCr, AlCrSi and AlCrB), the substrate bias voltage during coating deposition (-100 or -150 V) and the arc current (50, 60, 80 or 100 A). The substrates of the systems are cylinders with a diameter of 3 mm and a length of 30 mm. They were subjected to heating twice at 450°C for 4 hours. in an argon atmosphere. The thermal stability of the conditions of thermomechanical interactions between the coating and the substrate in the sample was identified on the basis of relative changes in the effective thermal expansion coefficient αAC of the substrate at 200°C and 450°C determined under temperature modulation conditions and absolute increases in the linear deformations of the substrate $\Delta(\Delta L_s)$ recorded at 20°C after another diagnostic heating of the coating - substrate system at 450°C. The analysis of the above results makes it possible to indicate the coating technology whose application promises the greatest service life of these wear resistant coatings.

Investigations partly financed by the National Centre for Research and Development, Poland, BIOSTRATEG3/344303/14/NCBR/2018.

CORROSION PROPERTIES OF Al-Si COATING OBTAINED BY MAGNETRON SPUTTERING ON STEEL X5CrNi18-10

D. Murzyński[1], *E. Dobruchowska*[1], *Ż. Zienkiewicz*[1]

Koszalin University of Technology, Sniadeckich 2, 75-453 Koszalin, Poland

Corresponding author: dawid.murzynski@tu.koszalin.pl

Research were carried out to determine the effect of aluminum content on the physico-chemical properties of the Al-Si coatings. The coatings were deposited on X5CrNi18-10 steel substrates by reactive magnetron sputtering technique. Surface morphology of the samples obtained were characterized by scanning electron microscopy (SEM), and their composition by energy dispersive X-ray spectroscopy. An attempt was also made to determine the thickness of the coatings by the Kalotest method. Evaluation of corrosion resistance, including pitting corrosion, of the substrate-coating systems in 3 wt.% NaCl solution was performed by means of potentiodynamic polarization tests. Based on the polarization curves obtained, the parameters such as corrosion potentials, breakdown potentials and corrosion current densities were determined, and polarization resistance was calculated using the Stern-Gaery equation. The studies performed have revealed that a decrease in the aluminum content significantly increase the corrosion resistance of the Al-Si coatings. Further, it was shown that all coatings were characterized by a good adhesion to the substrates. The corrosion processes, as demonstrated by SEM images, were initiated only in the immediate vicinity of coating defects.

Koszalin University of Technology

www.tu.koszalin.pl

Faculty of Mechanical Engineering

www.wm.koszalin.pl

Polish Vacuum Society PTP

www.ptp.pwr.wroc.pl

German Vacuum Society DVG

www.physik.uni-kl.de/dvg

Society of Vacuum Coaters

www.svc.org

BalticNet PlasmaTec

www.balticnet-plasmatec.org

Clausius Tower Society of Koszalin

www.clausius-tower-society.koszalin.pl

ISBN 978-83-7365-519-5

WYDAWNICTWO POLITECHNIKI KOSZALIŃSKIEJ