Book of abstracts

9-TH SYMPOSIUM ON VACUUM BASED SCIENCE AND TECHNOLOGY

organized by
Faculty of Technology and Education
Koszalin University of Technology

November 17-19, 2015
Kołobrzeg, Poland

9-th Symposium
on Vacuum based Science and Technology

in conjunction with the

14-th Annual Meeting of the German Vacuum Society (DVG)

organized by

Faculty of Technology and Education
Faculty of Technology and Education
Koszalin University of Technology

Clausius Tower Society

in cooperation with

BalticNet PlasmaTec Association **Society of Vacuum Coaters**

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Preface

Dear Colleagues,

it is real pleasure to welcome you again in Kołobrzeg during the **9 th Symposium on Vacuum based Science and Technology**, organized by the Faculty of Technology and Education, the unit of the Koszalin University of Technology, under auspices of the Polish Vacuum Society (PTP) and the German Vacuum Society (DVG) and held in conjunction with the 14th Annual Meeting of the DVG.

The Symposium organization is supported by the Clausius Tower Association and the Baltic-Net Plasma-Tec Association.

Mission of the Symposium is to initiate and support cooperation within the European plasma science community and to provide a forum for discussion of recent research results as well as an exchange of expertise in the field of vacuum and plasma based science.

This year, the **Rudolf-Jaeckel Prize**, awarded by the DVG for outstanding achievements in the field of vacuum based sciences, will be presented to **Professor Franz J. Giessibl** from the Regensburg University.

The **Clausius Session**, already traditionally organized during the Symposium is addressed this year to young generation. We invited our young colleagues to attend educational lectures given by renowned scientists reporting on recent achievements in solar cells technology and offering an adventure with the fascinating world of plasma.

This year the Symposium is focused on the following topics:

- Plasma physics and techniques
- Vacuum science, techniques and trends
- Protective coatings and thin films
- Characterization of surfaces and thin films
- Thin films for solar cells and sensors
- Plasma based surface treatment technologies
- New trends and concepts of plasma based deposition processes

The Symposium is accompanied by the **Industry Exhibition** attended by representatives of leading companies offering vacuum equipment, complete solutions for plasma based technologies as well as advanced research equipment.

The tutorial course entitled: **Application of Reactive Sputtering** is offered to young scientists and industry engineers by the Society of Vacuum Coaters (SVC).

This Book of Abstracts will guide you through the Symposium topics and sessions. You are welcome to attend invited lectures, oral presentations and the poster session.

Authors of contributions accepted for oral presentation during the Symposium are encouraged to submit their manuscripts to **VACUUM**.

I wish you fruitful discussions and good time spent in Kołobrzeg where I hope to meet you again.

Witold Gulbiński
Symposium Chairman

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REACTIVE HIPIMS: CHALLENGES AND INDUSTRIAL SOLUTIONS

H. Gerdes, R. Bandorf, M. Vergöhl, G. Bräuer

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Reactive sputtering is a well-established technology for industrial sputtering. The process stabilization is carried out by varying the average power or the reactive gas flow. As input parameters the partial pressure of the reactive gas, the target voltage or the plasma emission is commonly used.
For reactive HIPIMS challenges regarding process control arise. For instance, the change of discharge voltage is strongly depending on the capacity of the used HIPIMS power supply. Even the plasma emission is lower in HIPIMS discharges (time-averaged) since the duty cycle is normally in the lower percentage range.
This talk will give an overview of different approaches for a reactive process control in combination with HIPIMS. The discussed feedback systems are based on plasma emission monitors either with optical filters for single emission lines or with a spectrometer. The controllers are regulating the oxygen flow by piezo-valves or mass flow controllers or by changing the off-time and therefore the average power.
The different approaches will be presented for Alumina, Zirconia, and Titania on different sputtering plants equipped with planar magnetrons or even rotatables. The results include the voltage and current characteristics as well as the deposition rates and selected film properties.

A PERSPECTIVE ON DEVELOPMENTS IN PLASMA ASSISTED PVD PROCESSES

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Plasma assisted (PA) Physical Vapour Deposition (PVD) processes first came into prominence in the mid 1960's. We may call this the "1st Wave" of PAPVD and at that time the main application was metallic coatings to provide protection against corrosion and wear and the process was in a simple DC diode configuration. In the 2nd Wave of development, the emphasis moved to ionisation enhancing additions to the system (eg using thermionic enhancement) and the main coatings were ceramic compounds. In the next (3rd Wave) development phase the attention focussed on sputter deposition processes, and in particular those which utilised methods in increase the level of ionisation in such systems (e.g. using unbalanced and closed-field magnetrons and pulse-power systems). The coating systems studied then began to emphasise complex multi-layered and nanocomposite coating systems. The deposition systems evolved further, to encompass innovations such as "rotatable" cylindrical magnetrons. Now we are in the 4th Wave of PAPVD developments, which has focussed on further refining the 2nd and 3rd Wave systems and introducing improved process control together with improvements in hardware designs and monitoring systems to enhance throughput and coating consistency. Hybrid systems are also being developed which combine different processes together (such as duplex nitriding plus coating) to further enhance coating performance for a widening range of application sectors.

TOWARDS HARD YET TOUGH CERAMIC COATINGS

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Over the past decades, hard and super hard ceramic coatings have been developed and widely used in various industrial applications. Meanwhile, an increasing number of studies have realized that the toughness is just as crucial, if not more, than hardness especially for ceramic coatings. However, hardness and toughness do not go naturally hand in hand. In other words, hard coatings usually are brittle and less durable while toughened coatings are of lower strength. For practical engineering applications, it is more desirable to have coatings with high hardness without sacrificing toughness too much. In this talk, a review is presented on continuous progress to realize hard-yet-tough ceramic coatings from an angle of hardening as well as toughening.

HOLLOW CATHODE BASED PLASMA SOURCES FOR REDUCED AND ATMOSPHETIC PRESSURES

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The paper gives a picture of the development of hollow cathode based plasma sources both for reduced and atmospheric pressures. The linear hollow cathodes in several arrangements for generation of plasma over large areas and suitable for further scale-up are presented. Examples of surface processing and coating by PVD, both by the Hollow Cathode Discharge and Hollow Cathode Arc, are given. The hybrid reactor combining hollow cathode and microwave plasmas are described. The hollow cathodes can be operated in both, PVD and PE CVD regimes, depending on process parameter. These regimes can even be combined within one process. A new type of planar magnetron in which the target is coupled with a magnetized hollow cathode is introduced. Detailed principles of such arrangements are explained. Concepts of Fused Hollow Cathode (FHC), Microwave Antenna (MWA) and Hybrid Hollow Electrode Activated Discharge (H-HEAD) cold atmospheric plasma sources are discussed. A non-equilibrium atmospheric plasma source utilizing the Fused Hollow Cathode can be used for gas conversion and for surface treatment at ambient atmosphere. The Hybrid Hollow Electrode Activated Discharge cold atmospheric plasma source, capable of generating plasma plumes more than 15 cm long, enables treatment of 3-d and complex geometry objects even at low gas flows. The plasma source with a coaxial geometry based on the Fused Hollow Cathode (FHC) geometry was used for generation of plasma inside water and the ethanol-water mixtures.

DEPOSITION, CHARACTERIZATION AND APPLICATIONS OF THIN FILMS ON GRANULAR SUBSTRATES

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In this work physical vapor deposition (PVD) techniques for the manipulation of the surfaces of granular materials are discussed. Several challenges have to be addressed for this purpose:
Extensive intermixing of relatively large amounts of powders, granulates or fibres under vacuum conditions is mandatory for uniform coating due to the line of sight process of PVD processes. This is not trivial due to agglomeration effects and different trickling behaviour of granular matter in vacuum as compared to ambient atmosphere. An intermixing device based on rotation and concussion of a specially shaped powder container is presented. Up to one litre of granulate can be coated with high uniformity using this set-up.
Also the determination of the thickness of coatings on granulates is an involved task. We present an optical method based on advanced image processing techniques to determine the thickness of transparent coatings on small transparent particles. This method is used to characterize the above mentioned process in respect to thickness uniformity and thickness distribution on statistically significant amounts of particles and to compare measured thicknesses to those predicted by a theoretical model.
Finally, possible applications of coated granulates are presented. The focus is laid on hollow glass microspheres coated by a catalytic material for the controlled storage and release of hydrogen. This concept may yield high storage efficiency for hydrogen combined with low total weight and safe handling. Also other applications like coating sintering powders by diffusion barriers or fundamental aspects of measuring the conductivity of metal coated insulating particles will be discussed.
From these examples future prospects of coated granulates in several technological fields will be highlighted and innovative concepts for new functional materials will be presented.

This work is supported by the Austrian Science Fund, Projects, P-22718 and TRP-281

POWER SUPPLY TECHNOLOGY FOR CUTTING-EDGE PLASMA APPLICATIONS

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One of the fundamental component of the sputtering system is a power supply. It is used to provide a sufficient energy to the system to ignite and sustain the plasma discharge. Variety of industrial plasma applications give a high demand on power supply flexibility and functionality. Advanced power supply features such as fast arc management, automatic duty cycle adjustment or voltage regulated operation in reactive processes are key factors which enable continuous process optimization. In this contribution the DC, High-Power Impulse Magnetron Sputtering (HiPIMS) and Bipolar power supply technology is reviewed and highlights of recent power supply improvements and their influence on coating processing are discussed. First, the motivations for DC pulse technology is illustrated with benefits in the industrial scale production of photovoltaic cells. The reduction of faulty wafers due to arcing in Indium-Tin Oxide layer deposition process and the influence of reverse voltage on the optical properties of TiO_2 coatings in Pulsed-DC technology are given as examples. Secondly, the operation principles and benefits of HiPIMS will be briefly reviewed. The CrN coatings prepared with HiPIMS and DC technology will be compared to demonstrate the advantages of highly ionized, droplet free HiPIMS deposition of dense and homogenous coatings. Finally, the dual magnetron sputtering processes where a rectangular voltage and current output wave is used will be characterised. Thus, the voltage and current waveforms in Bipolar power supplies will be described. The prospective of altering the energy and negative oxygen ions flux responsible for local fluctuations of electrical properties of transparent conductive oxide (TCO) coatings by choosing an appropriate Bipolar output waveform will be discussed in details.

ADVANCES IN DEPOSITION EQUIPMENT AND PROCESS TECHNOLOGY FOR HIPIMS COATINGS FOR CUTTING TOOLS

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HiPIMS is characterised by short power pulses with an extremely short signal rise time. A completely new way of designing HiPIMS power supplies and for integrating them into a commercial coating machine will be presented. This novel approach avoids any cable between the HiPIMS pulse units and the respective cathodes. Other features are a full synchronisation between the HiPIMS sources and a dedicated table Bias and a HiPIMS frequency as high as 4.000Hz. This results in highest ionization, reduced resputtering and a so far unachieved deposition rate for HiPIMS. This paper presents how this new hardware design turns the advantages of the HiPIMS technology such as enhanced film adhesion, denser morphology and better coating uniformity all around 3D objects into user benefits for cutting tool applications.

Commercially used coatings for cutting tools are frequently of the Ti1-xAlxN type. Adding Si to the composition is a proven technique for machining materials above 55HRC. The study shows how a dedicated HiPIMS multilayer film design considerably improves the adhesion characterised by outstanding scratch loads and provides an appropriate toughness to support ultra-hard TiSiN layers. SEM cross sections show a dense morphology of HiPIMS coatings. Performed nanoindenter test data reveal how HiPIMS films combine high hardness and relatively low Young's modulus indicating a high coating toughness in a way most favourable for metal cutting.

Indexable inserts account for about 60% of the worldwide metal cutting market. HiPIMS accelerates the trend of using PVD sputter coatings for inserts by a hitherto unknown evenness of the coating distribution all around flank and rake face as well as a perfect film formation at the cutting edge. Case studies show how the effective bombardment of the growing film with highly ionized species further improves the wear resistance for e.g. cast iron machining.

SYNTHESIS AND CHARACTERISATION OF MeB_X (Me–Cr, Nb, Mo) THIN FILMS BY NON-REACTIVE DC MAGNETRON SPUTTERING

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Transition metal diborides (MeB_2) with the AlB_2-type structure have many unique properties such as high hardness, high conductivity and oxidation resistance One of the most studied diboride compounds is TiB_2 but also other transition metals like Cr, Nb and Mo can form the simple AlB_2 type structure.

Non-reactive magnetron sputtering is an excellent technique to deposit thin films of different MeB_2 phases. It is well-known that phases with rather simple crystal structures are preferably formed in magnetron sputtering where the quenching rates of the incoming atoms are high. Such metastable MeB_2 films may be chemically more reactive in a tribocontact and form a lubricating tribofilm of metal oxides and layered BO_x. Some metal oxides such as MoO_3 and boric acid (forming in humid atmosphere from BO_x) have been predicted to exhibit low friction coefficients. Consequently, it is possible that metastable MeB_2 films with the AlB_2-structure may exhibit excellent low friction properties.

In this study we have investigated the microstructure, mechanical and tribological properties of dc magnetron sputtered MeB_{2-x} films from Me/B targets (Me= Cr, Nb, Mo). The films were characterized with XRD, XPS, TEM, nanoindentation and tribological ball-on-disk method. All films exhibited the AlB_2-type structure with substoichiometric MeB_{2-x} grains surrounded by a tissue phase of amorphous B. All films were substoichiometric with respect to boron. The films exhibited much higher hardnesses compared to bulk samples, which could be attributed to a hardening effect of the tissue phase. Friction measurements confirmed the hypothesis that a significant tribofilm formation is present on the metastable MoB_{2-x} films. However, a reduced friction coefficient could not be observed. In contrast, the NbB_{2-x} films exhibit very low friction coefficients (0.16) because of boric acid ($B(OH)_3$) formation. General trends in transition metal diborides formation and properties will be explained in detail.

SURFACE PROPERTIES AND BIOLOGICAL EVALUATION OF SI-DLC FILMS FABRICATED BY A MULTI-TARGET DC-RF MAGNETRON SPUTTERING

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The Si-doped diamond-like carbon films manufactured using magnetron sputtering are not extensively described in the literature in terms of biological response. Hence, in this study the blood compatibility, cell proliferation and cytotoxicity of the Si-DLC coatings prepared by a multi-target DC-RF magnetron sputtering were investigated in reference to their surface properties. The Si-DLC films were deposited on a Ti6Al7Nb alloy. The XPS analysis revealed that the content of Si in the obtained coatings varied from ~4 at.% to ~14 at.%. The morphology examination using SEM showed that the surface of the Si-DLC coatings is uniform and homogenous without any local defects or delamination. Moreover, there is no evidence of Si conglomerates on the surface of the examined coatings. The biological evaluation proved that the fabricated DLC coatings improve the cell proliferation in comparison to a bare substrate. Furthermore, the live/dead test of endothelial cells indicated that the Si-DLC coatings are not cytotoxic and that with the growing Si concentration, the cell proliferation increases. According to the obtained results, the addition of Si caused the initial decrease in the platelet adhesion and activation level. However, further increase in the Si content resulted in a more thrombogenic character of the surface. The changes in the biological response towards the examined Si-DLC coatings were correlated with the changing surface properties. The small addition of silicon to the DLC films increased the hydrophilicity and polarity of the surface, while higher Si concentration resulted in turn in more hydrophobic surfaces. The presented results differ from those obtained by the authors for the Si-DLC coatings fabricated on the AISI 316 LVM steel using the same synthesis method. Therefore, it may be concluded that the substrate influences the biological behaviour of the Si-DLC coatings and thus, the deposition parameters should be adjusted to the basic biomaterial.

EUV INDUCED LOW TEMPERATURE PHOTOIONIZED PLASMA

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Ionization of gaseous media can be obtained by an electrical discharge or intense laser pulse irradiation. In both cases the electron collisional ionization is a dominating mechanism leading to plasma creation. In both cases electrons are accelerated by an electric field and some threshold must be exceeded to initialize the discharge or a laser spark. Quite different possibility offers irradiation with X-rays or extreme ultraviolet (EUV). In this case a single photon carries enough energy to ionize any atom or molecule. Thus ionization is possible even with low intensity radiation beams.

In this work investigations of photoionized plasmas were performed using laser-produced plasma extreme ultraviolet/soft X-ray (LPP EUV/SXR) sources with different parameters. The sources were based on three different laser systems with pulse energies ranging from 0.8 J to 500J and pulse duration 0.2 - 10 ns. Laser plasmas were produced by irradiation of double stream gas puff targets with Xe or KrXe mixture as the working gas. EUV or SXR radiation was focused using grazing incidence collectors of different types.

Different gases were injected into the interaction region, perpendicularly to an optical axis of the irradiation system, using an auxilary gas puff valve. Irradiation of the gases resulted in ionization and excitation of atoms and molecules forming photoionized plasmas. Spectra in SXR/EUV range were measured using a grazing incidence, flat-field spectrograph (McPherson Model 251), equipped with a 450 lines/mm toroidal grating. Spectra in UV/Vis range were measured using an Echelle Spectra Analyzer ESA 4000. Density distribution of photoionized plasmas was measured using laser interferometry. In all cases the most intense emission lines in EUV/SXR range were assigned to singly charged ions. Other spectral lines corresponding to doubly, triply and even quadruply charged ions were also recorded. In case of UV/Vis spectral measurements, however, atomic or molecular spectra were dominating.

Ti-C:H + Ti_xN + αTi(N) COPOSITE LAYER FOR APPLICATION OF TITANIUM Ti6Al4V ALLOY IN SPACE MECHANISMS

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Technologies related to space exploration are one of the most rapidly developing areas in which the newest technological achievements are used. Special attention in this study is paid to mechanical systems and to improving their tribological properties to ensure trouble-free operation and increase their energy efficiency. As it is impossible to use conventional lubricants in vacuum environments, or specialized synthetic vacuum greases due to the prevailing temperature range of 160-200C, there has been a development of solutions incorporating surface engineering technologies, which increase wear resistance and reduce the friction coefficients of the associated components.

Titanium and titanium alloys, due to their high relative strength as well as corrosion resistance and paramagnetic properties, are often used in devices operated in space conditions. However, due to their low wear resistance, their use in mechanisms operated in space is significantly reduced in favour of high-chromium steels.

The paper will show combination of novel surface treatments of Ti6Al4V alloy to increase its wear resistance and improve dry contact friction coefficient with Si_3N_4 and ZrO_2 ceramics to apply in light, hybrid bearings for use in space mechanisms. Obtained results exhibit that composite layers: nitrided layer + Ti-C:H coating type may prove effective in increasing the range of application of titanium alloys in mechanical systems and makes them competitive to high-chromium steels.

The technological aspects of producing such composite layers on Ti6Al4V alloy, microstructure, hardness and surface topography parameters of the produced hybrid layers, as well as the tribological “ball on disc” test results carried out in a laboratory and low vacuum environment at different temperature range, will be presented.

NANOCOMPOSITE TiN-Si_3N_4 COATINGS ON TEXTILES

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Magnetron sputtered amorphous Si_3N_4 films with included nanocrystals TiN are described in this paper. Target was prepared from powders mixture of 90 at.% Ti and10 at.% Al by hot pressing. Deposition was carried out with controlled gas flow of Ar and N_2. The most important parameter for deposition such composite structure is a substrate temperature not higher then 0,2 reduced temperature. Deposition parameters and structural properties investigated by X-ray and TEM techniques are presented. Heat reflection from a substrate with deposited TiN-Si_3N_4 film thickness about 100nm is noticeably higher.
Such coatings could be used for several applications which require high reflectance of IR. The adequate characteristics of reflectivity were measured. The temperature growth at the back side of substrate covered by TiN-Si_3N_4 coating was measured. The difference between covered and uncovered substrate are essential. It implies that the professional cloths for firemen and other services could be covered by such coatings.

Paper was financially supported by NCRD Project- Defense, Security No DOB-BIO6/04/104/2014.

MULTILAYER CARBON-PALLADIUM FOR HYDROGEN DETECTION

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In this paper we present the structural properties of C-Pd films applied for hydrogen sensing, obtained in physical vapor deposition (PVD). C-Pd types of film were deposited on quartz plates by PVD method. Fullerenes (C_{60}) and palladium acetate were evaporated during 10 minutes from separated thermal sources at dynamic vacuum (10^{-6} mbar). The temperature of palladium acetate source and the distance from substrate to the sources was the same for all films.

The cross-sectional TEM observations were performed for the samples prepared with the use of focused ion beam (FIB) method. All the specimens were studied with Titan Cubed microscope equipped with EDX spectrometer. In Fig.1 were presented images and results of EDX analysis for the three films. The modulation of Pd concentration with the film thickness is characteristic for all the samples, however some differences are noticed.

The all films exhibited spontaneous formation of the palladium rich layers. The thickness of the layers can be controlled with the temperature of fullerene evaporating source. Appropriate choice of ratio of the rate flow of the reactants plays a key role in the segregation process. Simple model of the Pd segregation based on diffusion and nucleation explains some features of the layers` structure. Knowledge of the mechanism of segregation is very useful for the design of the layers used for the detection of hydrogen and its compounds.

This project is co-financed by the European Regional Development Fund within the Innovative Economy Operational Programme 2007-2013 No UDA-POIG.01.03.01-14-071/08-09

CRACK INITIATION AND PROPAGATION IN THE VERY HIGH CYCLE FATIGUE REGIME OF THE HIGH-STRENGTH STEEL 100Cr6:
VACUUM EXPERIMENTS AS A SIMULATION TOOL FOR VHCF FAILURE

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Often non-metallic inclusions are the cause of fatigue fracture in high-strength steel stressed by cyclic load. High stress amplitudes lead to a failure which is observed at low numbers of cycles. The fracture is caused by cracks starting from inclusions at the surface of the material. Instead, for low stress amplitudes allowing more than 10^6 cycles, crack initiation at subsurface inclusions is observed. In the vicinity of these inclusions a fine granular area (FGA) can be found, which seems to be essential for the failure which occurs at very high cycle fatigue (VHCF).
Since the VHCF failure usually takes place at inclusions somewhere in the bulk the initial stage is not easily detectable. Nevertheless, to investigate the mechanism of crack initiation and propagation at early stages before damage occurs we prepared artificial defects at the surface via laser or ion beams. To hinder the free access of Oxygen during mechanical testing the samples are kept under vacuum conditions at pressures below 10^{-6} mbar.
We present characteristic analytical results which we obtained by Electron Transmission Microscopy TEM and Atom Probe Tomography APT for both the VHCF failures starting from inclusions in the bulk material and those initiated by the simulated defects at the surface as well. It will be seen that obviously in both cases similar processes lead to the fatigue fracture. A mechanism for crack initiation and propagation along the FGA will be proposed.

MECHANICAL PROPERTIES AND CHEMICAL ANALYSIS OF Si-INCORPORATED CARBON COATINGS PREPARED BY MAGNETRON SPUTTERING ON Ti6Al7Nb SUBSTRATE, FOR POTENTIAL BIOMEDICAL APPLICATIONS

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One of the possible answers to the constantly increasing demand on the market of the medical equipment is not only introduction of new materials, but also surface modification of already available ones. In case of metal implants, the already used approach involves application of diamond like carbon coatings (DLC). Further tailoring of coatings properties can be obtained by introduction of dopant elements into the carbon matrix.

In this study, silicon doped diamond-like carbon (Si-DLC) coatings were synthesized on silicon wafers (111) and titanium alloy (Ti6Al7Nb) using a multitarget DC-RF magnetron sputtering. The silicon content between 0 and 13.88 at.% was obtained by increasing the RF sputtering power of Si target up to 60W. Characterisation of chemical structure of the synthetized thin films was performed with help of X-ray photoelectron spectroscopy (XPS), Raman spectroscopy and Fourier Transform Infrared Spectroscopy (FTIR). In all the cases the improvement of mechanical properties of the synthetized coatings in comparison to the substrate material was observed. The micro-hardness of obtained Si-DLC coatings was on the level of pure DLC and equal to about 9.2 GPa. by the addition of Si the improvement of adhesion is observed even on the level of 34%.

Amount of the dopant alters the hydrophobicity of the coating and the character of the connection of silicon and carbon matrix. High atomic concentration of Si results in broad absorption at 810cm^{-1} which corresponds to Si-C bonds in close proximity to methyl groups, while for low content of dopant the maxima at 1070 cm^{-1} is present, related to Si-O-Si bond system. Well visible is also the depletion of the intensity of the Id/Ig ratio due to doping. The decreasing trend in the position of G band can be related to lowering the stress level in the coating or increase in the sp3 fraction.

EFFECT OF RELATIVE HUMIDITY OF THE ENVIRONMENT ONTO TRIBOLOGICAL PROPERTIES OF NANOCOMPOSITE LOW FRICTION COATINGS

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Low-friction MoS_2-based coatings deposited by PVD methods draw attention of researchers from decades due to their excellent tribological properties. The purpose of the present paper was to characterize the structure and tribological properties of magnetron sputtered MoS_2-based coatings doped with Ti and/or W in environment of controlled humidity. Four different coatings were investigated (MoS_2, MoS_2+W, MoS_2+Ti and MoS_2+Ti+W). The coatings were deposited in a vacuum chamber equipped with four planar magnetrons with targets from sintered pure MoS_2 and MoS_2+Ti powders and metallic Ti or W. The magnetrons were powered from 4 medium frequency current sources. The coatings 1.4÷1.6 μm thick were deposited onto hardened Vanadis 23 HSS substrates and Si wafers. Tribological properties were examined with ball-on-disc technique using CSM tribotester at 5%, 25%, 50%, 75% and 95% RH against 6,35 mm diam. 100Cr6 steel and Al_2O_3 ceramic balls. Based on wear profiles the wear rates were calculated for all tests. The tribotester used in the experiments was equipped with dedicated environmental chamber with humidity controller. The surface morphology of the coatings was investigated with use of SEM equipment, chemical composition was examined with EDS attachment to the SEM and phase composition was identified by GIXRD technique. Adhesion of coatings to Vanadis 23 steel substrate was estimated with use of the scratch test method. The investigations allowed to determine the chemical and phase composition of the manufactured coatings and to elucidate the tribological characteristics of the coatings at different relative humidity levels in the environment against metallic and ceramic counterbodies. The values of friction coefficients ranged between μ=0,032 for MoS_2(Ti,W) coating at 5% RH and μ=0,28 for MoS_2 coating at 95% RH against 100Cr6 steel and between μ=0,036 for MoS_2(Ti,W) coating at 5% RH and μ=0,59 for MoS_2 coating at 95% RH in case of the Al_2O_3 one.

Fe-NANOCLUSTERS: GENERATION VIA MAGNETRON SPUTTERING AND CHARACTERIZATION BY SNMS AND TEM

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A magnetron sputtering and gas-aggregation technique was used to create metallic and sub-oxidic nanoclusters with cluster sizes from 3 nm up to some 10 nm. The direct characterization of the clusters emitted from the magnetron cluster source by time of flight measurements is extremely costly and difficult. Instead, analyzing the clusters deposited on various substrates delivers direct information on their sticking at the surface. Sputter depth profiling with Secondary Neutral Mass Spectrometry SNMS delivered the total amount of the deposited material i.e. the Fe mass fraction and the Fe coverage on the Cu substrates. Individual particle properties as cluster size and chemical and structural composition of the particles were analyzed by Transmission Electron Microscopy TEM. The deposited nanoparticles contain a crystalline Fe core surrounded by a Fe_3O_4 shell. Even so the direct bombardment mode of plasma based SNMS is not capable of lateral resolution the cluster size and the cluster composition may be determined also using the SNMS depth profile measurements. The evaluation of the characteristic particle properties is based on suitable assumptions and we show that SNMS and TEM both deliver comparable and reasonable values for the size and the elemental composition of the nanoparticles.

UNPRECEDENTED INCREASE OF 316L STEEL CORROSION RESISTANCE AFTER CATHODIC CAGE PLASMA NITRIDING AND CARBONITRIDING IN A SODIUM CHLORIDE SOLUTION

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There has been much research on the modification of processes conducted under glow-discharge conditions in recent years. Some of the most common glow-discharge treatment methods include: nitriding and carbonitriding at cathodic potential and at plasma potential (i.e. with an active screen). The advantages of these methods include complete control of the phase composition, thickness and topography of the produced layers. Another advantage is the methods' capability to process austenitic steel, in which nitrogen does not diffuse as easily compared to ferritic or martensitic steels. However, these methods also have their drawbacks. In the case of conventional processing at cathodic potential, researchers have to deal with the so-called edge effect, which makes the method unsuitable for processing complexly-shaped specimens. Austenitic steels cannot be processed using the method either, as it does not provide these metals with the required corrosion protection. The reason for this is that the method leads to a significant development of surface roughness and an increase of residual stress. However, the edge effect is not present when using the active screen plasma process. A uniform layer is formed which is characterised by a low level of roughness, decreased residual stress and reduced thickness compared to layers produced at cathodic potential. Despite their reduced thickness, the layers show better corrosion resistance in the presence of halide ions, which is of particular importance in the case of austenitic steels. These steels are widely used e.g. in bone and joint implants, and as such are exposed to physiological fluids, which are particularly aggressive for this steel grade.
Nitrided and carbonitrided layers produced on X2CrNiMo17-12-2 (AISI 316L) steel using active screen plasma nitriding and carbonitriding have been characterised. All the processes were carried out at 440 °C in pulsed glow-discharge conditions using a frequency of 100 Hz. The layers were tested for their microstructure, phase composition, surface morphology, roughness and corrosion resistance in an aqueous solution of sodium chloride. The investigations have demonstrated that the glow-discharge processes presented have a significant impact on the functional properties of AISI 316L steel, especially its corrosion resistance in a chloride environment.

PTFE THIN FILMS OBTAINED BY PULSED ELECTRON BEAM DEPOSITION AND PULSED LASER DEPOSITION METHODS

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The aim of the work is a comparison structure and properties of Polytetrafluoroethylene (PTFE) coatings deposited by different physical vapor methods. PTFE layers were manufactured using the Pulsed Electron Beam Deposition (PED) and Pulsed Laser Deposition (PLD) process. These techniques are promising alternative to traditional manufacturing of polymer layers because of its ability to produce very thin films with a well-controlled stoichiometry.
In our experiments PTFE coatings were obtained on <100> Si substrate. The deposition was carried out at constant parameters: RT substrate temperature, pulse frequency of 5 Hz. Nitrogen was used as the background gas. The gas pressure was varied between 0.40 and 1.46 Pa. It has been found that the nitrogen pressure in the chamber has a major impact on the coating thickness. The presence of a PTFE structure was confirmed by means of Fourier Transform Infrared Spectrometry (FTIR) and X-ray diffractometry (XRD). The surface morphology and roughness were characterized by atomic force microscopy (AFM).

ELECTRON BEAM WELDING – TECHNIQUES AND TRENDS - REVIEW

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Electron beam welding, despite long history and widespread arc and laser technology is still widely used in industry. The main application this high efficiency welding process is: automotive, electronics, electrical engineering, aerospace and mechanical engineering industry. The technology ensures high-quality welded joints from the all structural metals in a wide range of thickness from 0.025 mm to 300 mm. It is also used for the production of films and coatings by deposition and surface modification. In the paper approximated examples of the use of the electron beam given by the welding, rapid prototyping, texturisation surface, cladding with wire and powder as well as alloying. Provides information about the possible techniques that can be used during these processes. The trends in the electron beam welding are presented.

PHOTOELECTRON SPECTROSCOPY UNDER REACTIVE CONDITIONS

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A challenge in the study of heterogeneous reactions is the analysis of the surface composition during the reaction process. Analysis methods suitable for in situ studies may not have sufficient surface sensitivity, while surface science techniques usually require ultra-high vacuum (UHV) for operation. However, the surface structure in UHV is not necessarily the same as under ambient pressure conditions. For example, many adsorbed species and reaction intermediates only exist on surfaces in equilibrium with gases. Thus, the study of surfaces under more realistic conditions is necessary to gain a better under-standing of heterogeneous reactions. To close this "pressure gap", X-ray photoelectron spectroscopy (XPS) has been adapted to operate under near ambient pressure (NAP) conditions. XPS provides quantitative elemental and chemical information about the near-surface region of solids including the makeup of adsorbed species; and the method is suitable for the investigation of the surface composition at increased pressure using a synchrotron source and spectrometers designed to operate at pressures in the mbar region. Recently, a growing number of papers report the adaptation of these spectrometers for operation with a laboratory X-ray source. The in situ study of heterogeneous catalytic reactions is one application of these lab-based instruments. Probing water-solid interfaces is another challenge of surface science where such a method can be employed. Technical and instrumental aspects of la-based NAP-XPS with a conventional X-ray source will be discussed and different applications detailed presented.

NANOCOMPOSITE GRAPHENE - HEXAGONAL BORON NITRIDE

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Graphene is a modern day marvel, composed of few-layer carbon sheets. It has novel properties with versatile applications, including sensors, nano-electronics and lithium ion batteries. The high conductivity of graphene is not suitable for certain electronic devices in which electrical conductivity is needed to be controlled. Being thermally insulating, graphene cannot be used for applications involving high heat generation. Hexagonal boron nitride (h-BN) is an electrically insulating material with high thermal conductivity and isoelectronic with graphene. We propose a hybrid nanostructure composed of graphene and h-BN nanosheets. In our laboratories, methods for synthesizing both materials have been developed. Graphene has been synthesized by carbon dioxide conversion while h-BN nanosheets were generated by autoclave pyrolysis techniques. The dry ice method of burning magnesium in solid carbon dioxide is an efficient and cost effective process to prepare few-layer graphene materials. In this methodology, non-toxic chemicals are involved, carbon dioxide and magnesium strips are used as starting materials. On the contrary, pyrolysis of boron and nitrogen containing precursors in a stainless steel autoclave is a commercially scalable technique suitable for industrial applications. We have successfully combined both of these methodologies to synthesize h-BN and graphene hybrid nanocomposites. The products are being characterized via Fourier-Transform Infrared (FT-IR) Spectroscopy, Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Energy Dispersive Spectroscopy (EDS) and X-Ray Diffraction (XRD) analysis.

CHEMICAL AND STRUCTURAL SURFACE PROPERTIES OF CARBON NANOMATERIALS DECORATED WITH Pd – ELECTRON SPECTROSCOPY AND QUASES ANALYSIS

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Carbon nanomaterials, like carbon multiwall nanotubes (MWCNTs), graphene oxide (GO), reduced graphene oxide (RGO) and their Pd decorated composites were studied using electron spectroscopy methods, *i.e.* X-ray photoelectron spectroscopy (XPS) and reflected electron energy loss spectroscopy (REELS), revealing their chemical (XPS) and structural surface properties (REELS).
The atomic content, content of C sp^2/sp^3 hybridisations and oxygen groups (hydroxyl C-OH, epoxy C-O-C, carbonyl C=O, carboxyl C-OOH) in carbon nanotubes and graphene materials were studied by the XPS from the photoelectron survey spectra and photoelectron spectra recorded from carbon (C 1s) and oxygen (O1s) atoms.
The average number of layers in the stacking nanostructures of graphene materials was studied by the REELS spectra of primary electron energy loss on electrons forming π and $\pi+\sigma$ bonds, considering the values of: (i) the energy loss on π and $\pi+\sigma$ electrons and (ii) intensity of surface and bulk components of energy loss features on $\pi+\sigma$ electrons.
Surface morphology and structural properties for evaluating the Pd nanoparticles surface coverage, Pd nanoparticles diameters, Pd nanoparticles density, thickness of the PdO_x overlayer on Pd nanoparticles, were studied using XPS aided with the QUASES Tougaard software. In this analysis the photoelectron spectra of Pd 3d, O 1s and O KLL Auger transitions, including the inelastic background in the vicinity of the analysed peaks, were considered. The spectra recorded from the investigated samples are fitted to the respective spectra recorded from Pd and PdO_x standards. The results obtained from the XPS-QUASES analysis were confirmed by the results from the XRD and STEM. The work demonstrates possibility of applying electron spectroscopies as a complementary tool for determining the structural properties, especially useful for analysis of the surface within the information depth of analysis.

GRAPHENE NANO PLATELET FILMS AT BIO IMPLANT MATERIALS BY MODIFICATION OF PECVD PROCESS AND IT'S ELECTRODE BEHAVIOUR

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Graphene Nano Platelets (GNP) films can contribute to enhance surfaces of bioactive materials and can be considered as non-cytotoxic and hemocompatible. For example it is reported they do not reduce cell viability and do not cause a ß-thromboglobulin release above physiological level.
In this work we consider the temperature and bias tension dependence a GNP film deposition process in a RF-PECVD chamber. The films were deposited at relevant test substrates like Ti, TiAlV, 316L and glassy Carbon as well as Si [100] for reference. The plasma process was performed using a high temperature and low pressure CO/H plasma. A negative self-bias tension was applied to the substrate.
The films have a mean thickness 100 nm. Their morphology investigated by SEM, STEM and TEM shows different forms like upright standing, lateral isotropic oriented flat graphene platelets of about 10 nm thickness, the same but waved platelets with longer extension and in the beginning of growth small rounded structures, forming to platelets later. This way a scalable void volume and density between the GNP can be managed by the plasma process. The range scales from 10 nm to about 100 nm. They all show graphitic electron diffraction pattern (SAED) measured by TEM.
The electric properties of GNP coated electrodes have been investigated by cyclic voltammetry. They show promising properties for applications as electrodes with nano scaled biocompatible surface.

SPECIFIC COATING/SUBSTRATE INTERFACE FORMED DURING COATING DEPOSITION WITH THE USE OF MODIFIED IPD METHOD

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This work concerns the characterization of the effects of the application of impulse changeable gas concentration, carried out by gas injection by impulse valve system, providing gas in individual doses, to the discharge region of impulse plasma coaxial accelerator in the IPD method (Impulse Plasma Deposition). Reported studies focuses on physical - chemical state of the coating/substrate interface forming in the deposition process of "valve" modified IPD method (in previous standard of IPD method, working gas is distributed continuously and dynamic value of the working pressure does not vary. The starting point for the formulation of research tasks were extremely good results of the durability tests of cutting tools coated with TiN, deposited during the modified IPD process, which was reported in one of our previous work[1]. We have expected the explanation of this stunning properties in the state of interfacial region of coating and substrate. The studies of phase boundary region showed, that the interface has a complex chemical structure. Straight under the nitride coating, the presence of a thin zone of the substrate material, highly enriched by products of erosion of accelerator electrodes was found. On this basis, it has been hypothesized for shallow implantation (subplantation) of plasma ions in the substrate with the presence of the competing phenomena of partial sputtering and subsequent re-condensation. Additionally, advanced HRTEM studies revealed the local formation of epitaxial zones at the interface which play a role of coating "anchors" to the substrate which play a crucial part in good adhesion of the whole system. This examples of local epitaxial growth have been noticed in the HiPIMS method.

IMPROVING THE PERFORMANCE PROPERTIES OF THE PVD PRODUCED NITRIDE LAYERS ON LIGHT ALLOYS USING A HYDROTHERMAL TREATMENT

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Covering light alloys, such as aluminum and magnesium alloys, with nitride surface layers is one of the prospective ways of improving the intrinsically poor surface properties of these metals, in particular their tribological and corrosion resistance. The layers are usually produced by the PVD methods using magnetron sputtering or arc evaporation. Even though the layers thus produced significantly increase the wear resistance of the alloys, their effect on the corrosion resistance is however unsatisfactory because of the poor tightness characteristic of PVD-produced products. The tightness acquires the crucial significance when the substrate is a highly-active magnesium alloy, and hence came our idea to tighten the layers by subjecting them to a post-deposition chemical treatment. The present paper presents the results of our experiments with two new hybrid surface engineering methods using a final hydrothermal treatment tightening of the composite titanium nitride layers PVD produced on the AZ91D magnesium alloy. The proposed methods permit increasing the performance properties such as resistance of the layers to wear and corrosion to values comparable with those of steel (the corrosion resistance achieved using the proposed treatment exceeds that of stainless 316L steel, and the tribological resistance is close to that of 100C bearing steel). The developed methods are also suitable for treating aluminum alloys, which gives them a more general applicative character.

OPTIMIZATION OF THE DEPOSITION PARAMETERS OF Cr/DLC COATINGS WITH THE MODIFIED CATHODIC VACUUM ARC METHOD FOR SINTERED CARBIDES TOOLS

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The purpose of the present study was to determine the optimal values of selected deposition parameters of Cr/DLC coatings with the modified cathodic vacuum arc method which ensure obtaining of their most advantageous properties from the perspective of their application for the coating on sintered carbides tool substrates for woodworking. An analysis was conducted of the investigations into the influence of the selected deposition parameters of Cr/DLC coatings on the accepted optimization criteria with the use of the Taguchi module. Adhesion, hardness and friction wear resistance were accepted as the optimization criteria of Cr/DLC coatings for sintered carbides substrates. It was established on the basis of the statistical analysis of the research results that in order to ensure a high adhesion of Cr/DLC coatings to sintered carbides substrates, a thick Cr sublayer (0.03 μm) and a DLC coating (0.9 μm) is to be used, which is deposited at a low argon pressure (0.01 Pa); no substrate bias (the floating potential) is to be used. In order to obtain high hardness and friction wear resistance, higher values of substrate bias voltages (-80 V) and a low pressure of argon (0.01 Pa) are to be used. Depending of the deposition parameters applied, it is possible to obtain DLC coatings in a wide range of hardness (20-60 GPa). The properties of Cr/DLC coatings that are deposited with optimized parameters may indicate the possibility of their application for woodworking or tools for wood-like materials in order to increase their durability.

Keywords: Modified Cathodic Vacuum Arc; Cr/DLC coatings; Sintered carbides tools; Taguchi optimization method; Mechanical properties; Tribological properties.

VACUUM MEASUREMENT - PIRANI WITHOUT ZERO PRESSURE

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Currently for heating a filament on target temperature a minimum power rating is necessary even in the high vacuum range. The reason is the loss of heat over the filament suspension.
This interference-prone power offset is usually many times higher than the dissipated heat by heat conduction due to the gas, i. e. the measuring signal.
Practically this limits the measuring range at the low pressure side ("zero pressure").
With a new solution (patent pending) the heat loss over the filament suspension is compensated by the supply of heating power with separate connections.
So the measuring signal is free of an offset (firm capacity, „Zero Pressure" capacity).
It can be measured without interference and amplified. Practically this means a wider measuring range in the direction of high vacuum.

BalticNet PlasmaTec Session

BALTICNET-PLASMATEC A CLUSTER SUPPORTING INTERNATIONAL COOPERATION

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Registered as a non-profit association, BalticNet-PlasmaTec (BNPT) is an international cluster – located in Greifswald, Germany – which stands for a technology and market-oriented cooperation of science, research and economics in the field of plasma technology. As one of the most important plasma technology clusters in Northern Europe, BNPT's aim is to raise the perception of the plasma technology in society. BalticNet-PlasmaTec is a contact partner for interested parties who intend to expand their own technical and economic potential in using plasma technology. BalticNet-PlasmaTec has currently 70 members in 15 countries, most of them are from the industry.

BalticNet-PlasmaTec based on five working groups. The working group Plasma & Environment focuses on plasma application for environment protection. All activities in the area of water cleaning are bundled in this working group.

The main interest of the working group Plasma & Bio is the combination of physical plasmas with life sciences and medicine. The Plasma & Surface working group is engaged with low pressure plasma applications like coating technologies. The working group Education organises placements, initial and further trainings, events for employees and students like summer schools and workshops. All activities which increase the visibility of plasma and the growing of the cluster are bundled in the working group Marketing.

The main activities of the cluster include:

- Connecting partners from R&D and industry
- Defining, participating, organising and managing international projects
- Cooperation in R&D and scientific marketing
- Acquiring funds
- Organising exchange of teachers, students and employees
- Placement of graduates, doctoral candidates and skilled personnel
- Presenting partner products, services and R&D results
- Presenting the cluster and the partner at fairs, conferences, workshop etc.
- Organising workshops, congresses and informative meetings

You will find more information about the international cluster BalticNet-PlasmaTec at: http://www.balticnet-plasmatec.org

MID-PRESSURE RF DISCHARGE FOR RADICAL PRODUCTION

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The radicals (O and N) produced in Ar or He based discharges are often used in the treatment of a variety of material surfaces. We have developed a radical source based on a capacitive RF discharge working in the pressure range of 2-20 Torr with N_2 or O_2 diluted in Ar carrier gas. The use of Ar as the carrier gas allows to ignite the discharge at low applied voltages similarly to He based discharges but radical losses by diffusion are significantly decreased. In addition, excited species of Ar may actively participate in the production of O and N radicals.

The properties of RF discharge as a function of gas composition and discharge current were characterized by various electrical and optical methods. The electrical characterization allowed to determine the plasma power, reduced electric field and electron densities inside the plasma column. The optical emission spectroscopy allowed to determine the gas temperature, the concentration of Ar atoms in 1s states and the main radical production mechanisms. The analysis of the production mechanisms suggests that the production of O radicals depends mainly on the O_2 fraction in the Ar while the production of N radicals depends on the discharge current. The concentration of N radicals in the afterglow of discharge was additionally estimated by NO titration.

The radical source was tested for the treatment of several materials. The treated samples were placed downstream from the discharge to prevent the direct treatment by plasma. Treatment with nitrogen radicals were used to passivate GaAs surface by producing a thin GaN layer. Treatment with oxygen radicals were used for the activation of heat sensitive graphene surface and for cleaning of TiO_2.

ADDED VALUE TROUGH SURFACE TECHNOLOGY

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Surface Technology is one of the core elements within Schaeffler´s strategic concept "Mobility for Tomorrow". The focus areas of future mobility are environmental drives, urban and interurban mobility and the corresponding energy chain. Environmental drives are one of the major factors that determine energy efficiency and environmental compatibility of mobility. Therefore, development of energy-efficient tribological systems continues to take top priority. The surface properties of engine components must be adjusted to more stringent environmental requirements while friction losses can be minimized by surface technology.

Numerous products benefit from tailored coatings to improve or just enable the function provided. For innovative functional parts, it is extremely important to consider coatings as design elements and integrate them in the product development process at a very early stage. In the future, the role of thin film coatings as a design element will strongly increase in further technical applications. Close collaboration between research and production, industry and research institutes is required to achieve this challenging goal. The Schaeffler Group delivers more than 100 million high-quality PVD- and (PA)CVD-coated components (Triondur®) every year and, along with its comprehensive coating tool box, enables outstanding applications, preserves resources and meets increasing customer requirements. As a result, customers benefit from all over the world and receive innovative, customized solutions of highest quality.

PLASMA DEPOSITION OF FUNCTIONAL NANOCOMPOSITES

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Nanocomposite films consisting of metal nanoparticles in a dielectric organic or ceramic matrix have unique functional properties with hosts of applications. The present talk demonstrates how plasma and other vapor phase deposition techniques can be employed for tailoring the nanostructure and the resulting properties. Vapor phase deposition, inter alia, allows excellent control of the metallic filling factor and its depth profile as well as the incorporation of alloy nanoparticles with well-defined composition. The metallic nanoparticles typically form via self-organization during co-deposition of the metallic and matrix components due to the high cohesive energy of the metals and the low metal-matrix interaction energy. Various methods such as sputtering, plasma polymerization, and evaporation have been applied for the deposition of the matrix component, while the metallic component has mostly been sputter-deposited or evaporated. Moreover, gas aggregation cluster sources were utilized to obtain independent control of filling factor and size of the embedded nanoparticles. In most applications, a high filling factor close to the percolation threshold is essential because the functional properties often require short range interaction between nanoparticles. Examples include optical composites with tuned particle surface plasmon resonances for plasmonic applications, magnetic high frequency materials with cut-off frequencies well above 1 GHz, sensors that are based on the huge change in the electronic properties near the percolation threshold, and biocompatible antibacterial coatings with tailored release rate.

ON THE ROLE OF ENERGY INFLUX DURING PLASMA-SURFACE PROCESSES

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Since the thermal conditions at substrate surfaces affect essentially the interaction of elementary processes during plasma treatment of solid surfaces (deposition, etching, modification), the experimental determination of the energy influx from plasma to substrate is of great importance. The total energy influx can be measured by special calorimetric sensors, e.g. by passive or active thermal probes, respectively.
The method of passive thermal probes (PTP) is based on the determination of the temporal slope of the substrate surface temperature in the course of the plasma process. The heating curve as well as the cooling curve (after switching-off) are fitted by suitable functions and the time derivatives at same environmental temperature are calculated. 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 the sensors bias voltage the energetic contribution of charge carriers can be determined.
As alternative method of a continuously operating active thermal probe (ATP) has been developed which does not need to be calibrated and which compensates the environmental effects as well as the heat conduction by the probe holder. By means of controlled electrical heating the probe is set to a given working temperature and then the energy supply supporting the fixed operating temperature is measured. The energy influx by the plasma is compensated by decreasing the heating power and is directly displayed in J/cm^2s. Even, if the probe is designed as double probe the directionality of the energy influx can be determined.
By using thermal probes of different materials it is even possible to verify the effect of surface recombination, secondary electron emission and sputtering in respect to the energy balance of a substrate in plasma processing. By comparison of the experiments with model assumptions on the involved plasma-surface mechanisms the different energetic contributions to the total energy influx can be separated.
The methods will be demonstrated for various process plasmas, e.g. magnetron sputtering (HiPIMS), asymmetric rf-discharge, ion beam source operation, and ECR afterglow.

INFLUENCE OF Ti LAYER AND Si SUBSTRATE ON THE PROPERTIES OF THE C-Ni/Ti/Si MULTILAYER

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C-Ni film deposited on different substrate are interesting materials by means of their application as field emitters or sensors. The problem in obtaining of such films on flat Si substrate is adhesion. To avoid the film lamination we applied titanium under layer that should improve its adhesion to the Si substrate.
Structural studies results of carbonaceous films containing Ni nanocrystllities (C-Ni) deposited on silicon and titanium/silicon substrates are presented. Silicon n-type <100> substrates with resistivity 1-10 Ωcm were used. Some samples were covered with thin titanium films by means of magnetron sputtering. Then C-Ni films were prepared by Physical Vapour Deposition (PVD) method in which C60 and nickel acetate were evaporated from two separate sources at dynamic vacuum of 10^{-6} mbar and with different technological parameters such as deposition time, current intensity through sources. Scanning Electron Microscope (SEM) was applied to study morphology and topography of films. SEM images in low angel backscattered electrons (LABE) and secondary electrons (SE) mode were analyze to obtain information on morphology of observed objects.
High electric field used in field emission experiment was used for testing adhesion of C-Ni film deposited on Ti/Si substrate. The electric field of the order 10^3 V/μm was applied to test such adhesion. SEM results show the topography of the films after adhesion experiment.

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INFLUENCE OF THE SUBSTRATE TOPOGRAPHY ON THE CONSTITUTION OF Li-RICH Li-Ni-Mn-Co-O THIN FILM BATTERIES

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$Li(Ni_{1/3}Mn_{1/3}Co_{1/3})O_2$ as a cathode material shows compared to the well-established $LiCoO_2$ a better thermal stability, higher reversible capacity (290 $mAhg^{-1}$), good rate capability and is environmentally friendlier. One way to enhance the electrochemical properties like Li-diffusion rate or rate capability is to increase the surface between cathode and electrolyte. By sputter etching the surface topography of stainless steel substrates have been varied. Onto this modified stainless steel substrates Li-rich Li-Ni-Mn-Co-O thin film cathodes have been deposited by non-reactive r.f. magnetron sputtering from a ceramic $Li_{1.11}(Ni_{0.37}Mn_{0.19}Co_{0.33})O_{1.77}$. Coating thickness is about 100 nm. The samples were heat treated for one hour at 600 °C in a $Ar{:}O_2$ atmosphere. Composition and microstructure were investigated comprehensively. The elemental composition was determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) in combination with carrier gas hot extraction (CGHE). During sputter etching the roughness of the stainless steel substrates could be increased from R_a = 4 nm up to R_a = 64 nm. The microstructure of the films was characterized by X-ray diffraction (XRD), unpolarized micro-Raman spectroscopy and Atomic-force microscopy (AFM) at room temperature. Electrochemical characterizations of as deposited and annealed films were carried out by impedance spectroscopy (IS). Correlations between process parameters, microstructure and electrochemical behaviour are discussed in detail. These films are promising candidates for manufacturing of all solid state thin film batteries.

ENHANCING THE Pt-COATING PROCESS ON ABS BY PLASMA TREATMENT

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Nowadays many people use contact lenses. For hygienic and safety reasons a proper cleaning of the lenses is crucial before everyday use. One popular cleaning system on the market uses a hydrogen peroxide solution to clean the lenses. In that cleaning step also a platinum-coated star shaped piece of ABS (Acrylnitril-Butadien-Styrol) is added to the cleaning solution to degrade the peroxide. So the lens is ready for usage after 6h without any further needs to remove the remaining hydrogen peroxide.

In the production of those ABS-stars chromic acid is used to oxidize and roughen the surface before the platinum coating is applied. A high degree of oxidation leads to better Pt-bonding. But chromic acid is a very toxic and environmentally harmful agent. In the following work we will present some results of our experiments to replace the treatment with chromic acid by a plasma treatment. The degree of oxidation and the surface morphology of the untreated, the chromic acid treated and the plasma treated ABS-samples were compared to make meaningful assumptions on the Pt-bonding capabilities.

ENERGY STORAGE PLASMA ELECTRONİC DEVİCES WİTH POROUS ZEOLİTE CATHODES

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In this work, we have studied the transport processes in a plasma electronic devices (PED) with a wide pressure range up to atmospheric pressure (AP) (4-760 Torr) at electrode gap *d* (50 μm), and diameter *D* (22 mm) of the active areas for porous zeolite (ZC) and GaAs semiconductor cathode (SC) in an air and hydrogen dc microdischarges. The PED with SC cannot effectively operate at AP in air media compared with SC in H_2 media and ZC. Operating efficiently of the system will be based on the use of ZC, which is a good absorber of gas molecules in their nanopores. Thus, the discharge initiates from the surface and channels of the ZC, unlike conventional planar PED with GaAs SC. The plasma emission intensity of PED with ZC is higher than plasma emission for chamber with GaAs photocathode. This device might find an application in for generating and sustaining a stable, uniform and homogeneous non-thermal AP plasma. In summary, these results promise of H_2 gas can be absorbed into a pores and nanoscaffold of zeolites and properties of cheap and natural zeolite provides efficiently work for plasma light source applications. Therefore, we confirm that the porous zeolites is a suitable as solid state H_2 storage electrode materials for energy storage and conversion in PED and can serve as a source of UV-radiation if pressure and electric field are sufficiently high.

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INCREASED IONIZATION DURING MAGNETRON SPUTTERING

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In this work the influence of the process gas on the degree of ionization during magnetron sputtering is investigated. For this purpose, transient calorimetric measurements were performed in a typical substrate position and in the toroidal plasma region. The transient method allows for an estimation of plasma parameters such as the electron temperature T_e as well as the electron and ion densities, n_e and n_i, respectively. It also gives access to the total energy influx and the different contributions originating from charged and neutral particles as well as surface processes like recombination or film formation. For a comparison the experiments were performed using three different process gases, Ar, Ne and Kr.
Since it is well known that the degree of ionization of the process gas and the ion impact at the substrate play an important role for the optimization of thin film properties, the knowledge of how to influence these characteristics is of great value.
With T_e having a greater influence on the degree of ionization than n_e, this work has been focused on tailoring the electron temperature. Due to the higher ionization potential of Ne compared to Ar and Kr, a higher degree of ionization was achieved by using Neon. In addition, QCM measurements were performed to calculate the deposition rate, quantifying the sputter yield of the three different gases.
Due to the calorimetric data obtained simultaneously with the electrical currents, it is possible to calculate the total energy flux and its components from different species of the plasma.

ELECTRICAL MEASUREMENTS FOR THE CONTROL OF NANOPARTICLE GROWTH IN AN ACETYLENE PLASMA

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Synthesis of nanoparticles is an important part of research in nanotechnology. Nanoparticles are not only of fundamental interest in complex plasmas but also of practical importance in material processing. Understanding the processes during their formation in plasmas is essential for the preparation of nanoparticles of well defined size and composition. Especially the early stages of the particle growth are not well investigated since they are experimentally inaccessible by standard methods like Mie-scattering.
In order to get a better insight into the early stages of particle growth, a novel collection method based on neutral drag was tested. A capacitively coupled discharge driven at 13.56 MHz, where multiple growth cycles can be obtained, was used for the experiments. Size distributions of the nanoparticles at different stages of the growth cycle were determined ex-situ by electron microscopy. The obtained size distributions have been correlated with in-situ measurements of the bias voltage and the phase angle between discharge current and voltage. The observed correlations, which can be used for prediction of the particle growth, are qualitatively explained. This work was supported by the DFG SFB-TR24.

C/Ni/Pd NANOCOMPOSITE FILMS FOR CO_2 AND NH_3 SENSING

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Innovative three step method was applied to obtain nanocomposite C/Ni/Pd films sensitive to CO_2 and NH_3. This method was developed in Tele-& RadioResearch Institute. Presented here C/Ni/Pd films were deposited on conductometric transducers with alumina ceramic substrates and the interdigitized Pt electrodes. Prepared films have a high surface per volume ratio, their thickness is 300-500nm. Dynamic performance of developed sensors towards CO_2 and NH_3 was examined at room temperature. It was found that the film structure and sensing properties depend on the content of Ni and Pd in the film. The layers containing both nano-Ni and nano-Pd grains could be applied for different gas sensor design. Changing content of Ni or Pd allows for sensing different gases. Ni/Pd films obtained on different substrates were described in papers. These preparation methods are complicated, material and time consuming. The new method developed in our group is cheap and simple and easily could be implemented in industry.

DURABILITY CHANGES OF COBALT-TUNGSTEN CARBIDE TOOLS AFTER ION IMPLANTATION

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Deep hole drilling tools (where the hole depth is ten-fold greater than its diameter) are very desirable in the modern engineering. Among various types of these tools, the solid drill heads are used. This type of tools is a response to changing needs of the market, ie. among others, large variety of production, short production series, a quest for more efficient processing methods. In these heads, the position of an external insert is fixed using cobalt-tungsten carbide guide pads.
In our research, we used ion implantation technique to increase the tool life. For example, nitrogen ion implantation of carbide guide pads increases its lifetime by a factor of two. The guide pads were tested in industrial conditions. Additionally, SEM observations, EDS analyses and selected tribological tests were conducted for the initial and the modified tools.

ELECTRON MICROSCOPY STUDIES OF C-Pd FILMS

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C-Pd nanomaterials react with gas containing hydrogen. Composite C-Pd films were investigated and described in our previous papers. These films were obtained by PVD process based on thermal evaporation of two starting materials, fullerene (C_{60}) and palladium acetate, placed in separate containers and heated resistively to temperatures corresponding to their melting points.
The result of electron microscopy SEM and TEM studies of C-Pd films are presented in this paper. The films were obtained by PVD method. The technology parameters were different for various processes. The films with palladium content between few wt. % and few tens wt.% were prepared in this way.
Quantitative and qualitative analysis of the topography and morphology of C-Pd films obtained under different technological conditions processes were carried out using scanning electron microscopy (SEM) and the structure of these films were studied with transmission electron microscopy (TEM).
The results of studies of electrical changes due to hydrogen interaction with the film study show that the reactivity of these films toward hydrogen and its compounds depends on the palladium content, palladium nanograins size and distribution in the film volume.

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QUALITY EVALUATION OF HS6-5-2 TOOL STEEL / CrN/CrCN AREOLOGICAL SYSTEMS WITH VARIOUS MODULATION PARAMETER λ USING RECATEST METHOD

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The literature publications suggest that the industry, particularly the woodworking sector, is very interested in multi-layer coatings based on chromium nitrides and chromium carbonitrides. As it was proven during previous research works conducted at The Koszalin University of Technology, 3 μm thick multi-layer CrN/CrCN coatings composed of 7 CrN/CrCN modules significantly reduce the wear of planer knives, particularly during working of beech wood.

The presented examinations were performed on 5 μm multi-layer CrN/CrCN coatings characterized by various values of modulation parameter λ. The coatings composed of 4, 8, 12, 16, 24, and 32 CrN+CrCN modules were synthesized in plasma stream from low-pressure arc source using the PVD-Arc technique in a TINA 900M device. The HS6-5-2 tool steel toughened to the hardness of 62 HRC was used as the substrate. The microstructure of multi-layer coatings was revealed during metallographic examinations of spherical microsections. Examinations of adhesion and determination of mechanical wear symptoms were performed using the scratch test method. The nature of areological system microstructure and geometry changes in the scratch area was evaluated using the Recatest method

MODIFICATION OF SURFACE PROPERTIES OF ta-C COATINGS BY THE SUBSTRATE BIAS VOLTAGE ADJUSTMENT

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The surface of material plays a basic role in determining various processes such as dyeing penetration, chemical absorption, biocompatibility and others. Properties of material surface - composition, roughness, topography, wettability can influence phenomena at material interface and are very important for various advanced applications. Modern techniques are widely used for surface parameters modification. The aim of the study was the comparative analysis of the surface properties of ta-C coatings depending on the substrate bias voltage, in the range of − 25 to − 200 V. Experiments were performed on a modernized industrial vacuum arc device C55CT. Two graphite cathodes, with a DC-arc current of 50 A, high-current arc pulses of 1400 A, were used for carbon (ta-C) deposition.

Advancing contact angles were measured by Wilhelm's method (Kruss K12) at temperature 20^{o}C. The surface free energy SFE and its polar and dispersion parts estimations were made by Owens-Wendt-Rabel-Kaeble', Van Oss and Fowkes methods. The SFE energy values were in the range 40 - 45 mN/m, polar part was grown from 4.79 mN/m to 8.49 mN/m with increasing bias potential.

The results demonstrate the possibility to modify surface parameters of ta-C coatings by changing technological parameters and the substrate bias voltage adjustment. The changing of surface roughness, surface free energy and polar parts is very challenging for various industrial applications of ta-C coatings.

INVESTIGATIONS OF CARBON CATHODE SURFACE BEFORE AND AFTER THE PASSAGE OF COMBINED DC VACUUM-ARC WITH SUPERIMPOSED HIGH-CURRENT ARC PULSES

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The paper presents the results of studies of carbon cathode surface before and after the passage of the combined DC vacuum-arc with superimposed high-current arc pulses. The surface morphology of the cathode was investigated by the scanning electron microscope. The phase structure of the cathode surface was investigated using Raman spectroscopy and X-ray Photoelectron Spectrometry (XPS). Investigations of surface morphology of carbon cathodes using Scanning Electron Microscopy (SEM) showed, that secondary nuclei of high-density are formed on the cathode surface after passing of the combined DC vacuum-arc, which results in the formation of a globular structures. Results of the phase structure analysis by Raman spectroscopy showed that even at a minimum operation time (5 sec) of the combined DC vacuum-arc broadening of the peaks 1355 and 1583 cm^{-1} occurs, which means that the carbon cathode surface undergo phase transformation. Results obtained by XPS spectrometry demonstrate that the globular structures formed on the cathode surface are composed of sp^3 bonded carbon atoms and carbon oxides. The research results described in this paper show that in the process of passing of the combined DC vacuum-arc with superimposed high-current arc pulses through the cathode surface the globular structures with high content of sp^3 bonds are formed. The amount of these structures increase with the process duration, which may influence the conditions of the ta-C coatings deposition.

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THERMOMECHANICAL ANALYSIS OF THE INFLUENCE OF SUBSTRATE NITRIDING ON THERMAL STABILITY OF SUBSTRATE/CrCN COATING SYSTEMS

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Operational properties of tools coated with antiwear coatings deposited by PVD methods, depend significantly on thermochemical conditions of interactions between coating and its substrate. During the exploitation of tools, their temperature may rise to 1000 ^{o}C or more, due to this fact, its mechanical properties strongly depends on thermally induced phenomena such as: migration of lattice defects, phase transformation, mutual diffusion or coatings decohesion. The objects of research were systems composed of substrates made of 42CrMo4 steel (non-nitrided and nitrided) and CrCN coatings. The thicknesses of the analyzed coatings were 4 μm and 8 μm. Thermal stability of the systems was investigated using thermo-mechanical analysis (TMA) with temperature modulation. In particularly, measurements of the reversible thermal expansion coefficient α_{AC} (measured at 200°C), and changes in the length of the systems ΔLs (referenced to a length of a system after the deposition process) indicate a significant differences in thermal stability of the investigated systems. Also for the purposes of system's viscosity assessment, the time delay τ between the temperature signal and the dilatometric response of the systems were measured. Summary results indicate that nitriding of substrates made of 42CrMo4 steel, caused significant increment of thermal stability of systems. Furthermore, basing on the obtained results it is possible to identify the influence of the gas atmosphere (argon - air) during annealing of the system and the thickness of coating (4-8 μm) on the dilatometric response. The changes in residual stresses in substrate/coating systems may also be indirectly evaluated using ΔLs

THE INFLUENCE OF THE COMPOSTION OF THE TiAlN ANTI-WEAR COATING ON THE COURSE OF THERMOMECHANICAL INTERACTION TO THE SUBSTRATE IN THE FUNCTION OF TEMPEARATURE

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This publication presents an innovative use of the TMA (Thermomechanical Analysis) method with the temperature modulation to describe changes in the conditions of thermomechanical interactions between the substrate and the PVD coating with varying composition. The substrates are made of WCCo10 sinter, while the coatings Ti_{1-x} Al_xN differ with respect to AlN content with x = 0; 0.3; 0.55; 0.63; 0.67; 0.72; 0.79 and 1.
The principle of the identification of changes to the conditions of thermomechanical interactions between the substrate and the PVD coating relies upon the measurement of changes in the reversible thermal expansion coefficient α_{AC} of the substrate, which result from the variable composition of the adhesive TiAlN coating. In this method, the substrate functions as a "sensor", with the aid of which changes can be registered to the thermomechanical loads of the substrate by the coating. Furthermore, this method permits detection of the diversified courses of the evolution of stresses in the coatings that result from the variations of the coating composition.
In the publication, the results are presented of the measurements of changes to time delays τ between cyclical changes to the temperature of the samples of the WCCo10 - TiAlN systems examined and dilatometric responses that followed from the variable compositions of the TiAlN coating. In this way, it was possible to define changes to the rheological properties of the physical models of the WCCo10-TiAlN systems examined that are of significance from the perspective of the prediction of their service life.

ROLE OF METAL IMPURITIES IN GENERATION OF DEFECTS IN ANODIC COATINGS Nb_2O_5

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Key role in formation of the properties of the anode layers of niobium pentoxide is related to defects of their own or additive nature, while the main defects are oxygen vacancies . The aim of this work – analysis of additive influence in niobium on formation of defects in Nb2O5.The object of the study were layers of Nb2O5, formed by anodic oxidation of the sintered niobium pellets of two types (denoted as A and B), differ by the presence on the surface of A type samples of Mg additive, controlled by X-ray photoelectron spectroscopy (XFES). It shall be noted that XFES - initial analysis of the niobium powder applied at generation of samples of both types, has shown no differences in the composition of additives; presence of Mg has only been detected on the surface of the pellets formed by high-temperature vacuum powder sintering. This fact affirms that additives located in the niobium powder as a result of sintering are concentrated on the pellet's surface probably according to the bulk diffusion mechanism. Anodic oxidation of niobium was similarly carried out in two stages: in a galvanostatic mode, and subsequent aging at a constant voltage. Concentration of charged defects is determined from the current-voltage characteristics (C - V) according to Mott -Schottky equation. The results can be interpreted as follows: at the first (galvanostatic) stage of anodic oxidation of samples part of A atoms displaced from the metal oxide pellets are represented by additive metal atoms (Mg), which behavior in electric field is determined by mechanisms specific for anionic defects. As a result, after galvanostatic stage less quantity of defects as compared to B sample case will be carried out to the oxide / electrolyte border. Further, at the transition to the potentiostatic oxidation mode, Mg^{2+} cations along with the basic (anionic) defects are moved to the outer boundary of the oxide layer, which leads to an increase in concentration of the charged defects in the surface layer Nb_2O_5.

ADHESIVE POROUS DLC-COATINGS BY MODIFIED PECVD PROCESS

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For special applications, e.g. in medicines techniques highly porous surface coatings are required, e.g. as reservoir of functional materials or membrane films. For these applications adhesive and mechanical and chemical stable thin films are required with excellent adhesion and defined porosity. Diamond like carbon (DLC) is very promising because of its chemical inertness, excellent mechanical properties and complete biocompatibility, but for DLC coatings with controlled porosity little is referred.

In this work porous DLC coatings were prepared on relevant metal substrates by a modified PECVD technique: plasma enhanced growing and plasma etchings have been combined in an alternating process with nanometre scaled multilayer growing. The plasma was excited using a parallel-plate capacitive coupled RF system. For growing, Ar and C_2H_2 have been used as working and precursor gas, respectively, and for etching Ar and O_2. The number of alternating steps and the RF bias was varied. The morphology, Hardness and elasticity, the wear resistance and the adhesion have been investigated by SEM and TEM, Nanoindentation tests (MST Nano Indenter XP), grinding calottes with defined geometry and by bending tests, respectively, and the porosity was quantitatively obtained by digital imaging processing. The sp^3/sp^2 ratio in the carbon film was proved by XPS.

This investigation revealed that DLC coatings with controlled and homogeneously spread porosity can be achieved. The average pore diameter was depending on substrate bias during growing between 3 and 20 µm. The pores volume was estimated up to 15 %. The DLC films exhibited excellent adhesion and wear resistance and the obtained hardness was between 5 and 10 GPa. Further investigation is now aimed at increased pore volume at remained mechanical properties.

VACUUM PUMPS AND THEIR EFFECTIVE APPLICATIONS

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Vacuum technology is largely used in many branches of industry and has undergone rapid development during last six decades. The number of technical procedures in vacuum processes is high, and it has a large spectrum of the application. The pumping operation is mainly divided into wet and dry vacuum procedures. Distinctions between the two categories are described briefly and the basic pumping procedures are given in the following paragraphs. The choice of the required working pressure and required pump is considered in this article. Therefore, the most suitable pumps for use in the particular pressure regions are described. If the working region varies during a process, different pumping systems must be fitted to the vessel. As experiments have shown the pumping speed of all rotary pumps falls off rapidly when it closes to the attainable ultimate pressure. Therefore, the lowest limit for the normal working pressure region of these pumps should be that at which the pumping speed still amounts to about 50 % of the nominal pumping speed.

The issue of discussion is also the optimum pumping properties, setting the pressure range in which a single-stage rotary pump is sufficient, having a high pumping speed, and the most economical pumps to purchase. Moreover, the combination of two-stage rotary pumps with roots pumps work economically. The comparative description of these pumps and the vapor ejector pump has shown that the last one operates more effectively, and might be used for a longer time without much maintenance.

The comparative description done between the application of the special diffusion pumps which combine the properties of vapor ejector pump (high critical backing pressure and high throughput in the medium vacuum region) with the outstanding properties of diffusion pumps (high pumping speed in the high vacuum region and comparatively low ultimate pressure).

RHENIUM-BASED COATINGS BY CVD AND PVD TECHNIQUES

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Accessible Re resources in the Earth crust are extremely limited ($<7\cdot10^{-8}$ wt.%). On the other hand Re has been recognized as a very important engineering material irreplaceable in numerous applications in chemical, nuclear, aviation, aerospace, electric, automobile or defense industries due to a unique combination of physical and chemical properties. For example, it has the second highest melting point, the third Young's modulus and the fourth highest density of all metals. It has one of the highest strength hardening exponent of all elements, a high hardness, good tribological properties, superior tensile and creep rupture strength up to ~2000 ^{0}C (the last one is twice that of tungsten) and due to its BCC elementary cell it doesn't undergo ductile-to-brittle transition. Moreover, it is the only refractory metal which doesn't form carbides. Due to this unique combination of properties it is used in a number of high-tech applications, as, e.g., diffusion barrier coatings for graphite, superhard ReB_2 coatings for high speed machining, for enhancement of tensile strength and ductility of refractory metals (as Mo or W), as Pt-Re bimetallic catalysts for converting low-octane naphtas into high-octane hydrocarbon products or for hydrogenation of fine chemicals, for replacement of hard chrome internal coating of large and medium gun barrels due to environmental limitations against noxious hexavalent electrolytic chromium deposit, for wear- and arc-resistant electrical contacts, for high temperature protection of radiation heaters and high temperature thermocouples, for thermo-photovoltaic or thermo-electric power generators based on $ReSi_2$, for marine bio-antifouling or decorative coatings (for replacement of precious metals).
Six new CVD and PVD deposition techniques of Re-based decorative and protective coatings are being elaborated in the Lodz University of Technology (the PVD ones are based on the newly developed GPMS technique).

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PROPERTIES OF THE DETONATION DIAMONDS NANOPOWDER (DND) MODIFIED WITH MW PECVD+R SYSTEM

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Plasma processing has become an established method for surface modification, etching or deposition of plasma detonation nanodiamonds powder DND. Current practise is to set the external plasma parameters to empirically obtained optimum values and maintain these values through the whole process. Currently, there is a great interest in methods of modifying carbonaceous materials by which they gain new properties. There are known methods of mechanical, chemical or plasma functionalization. This paper is devoted to the construction of the innovative MW PACVD + R system for plasma-chemical modification of diamond nanopowders, thanks to which it gains new – biological properties. The carbonaceous material obtained in the process was subjected to a series of tests that are to verify and determine its new features.

THE INFLUENCE OF MAGNETRON DISCHARGE POWER ON THE SINGLE PROBE LANGMUIR MEASUREMENTS AND RESULTED PLASMA PARAMETERS OF TITANIUM SPUTTERING PROCESSES

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The pulse density modulation is often used to regulate the average power level of magnetron sputtering discharges driven by the resonant type of medium frequency power supplies (such as DORA Power Systems). While the discharge power level is being changed than the number of pulses in group changes, but the discharge current pulses are the same from pulse to pulse – their parameters (duration time, amplitude) do not change with the discharge power. Such pulsed powering of magnetron source introduces discharge 'ON' and 'OFF' phases resulting in plasma presence (groups of pulses) and absence (between groups of pulses) phases, respectively. One can conclude that this fact has a great impact on the measured Langmuir probe current if its time-averaged value (i.e. its mean value, direct current) is recorded.

The goal of conducted research was to present the influence of medium frequency discharge power level on the direct current I-V characteristics of a single Langmuir probe and resulted plasma parameters caused by the pulse density modulation.

The sputtering processes of titanium were diagnosed at different discharge power levels. The measured Langmuir probe I-V characteristics and calculated plasma parameters showed strong dependence on the discharge power. The electron density (plasma density) was calculated to be of about 0.5; 1.3; 1.9×10^{17} m^{-3} at discharge power of 1; 3; 5 kW, respectively. As the discharge powering pulses stay the same with the discharge power level change such influence was unlikely to take place. Using time-resolved analysis of Langmuir probe current waveforms the origin of this influence was indicated. The influence of discharge power level on the single probe Langmuir I-V characteristics and resulted plasma parameters was eliminated using a simple method of scaling the results. Finally, the reliable plasma density was calculated to be of about 2.4×10^{17} m^{-3}.

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THE EFFECT OF DEPOSITION METHOD ONTO PROPERTIES OF NANOCOMPOSITE, LOW FRICTION nc-TiC/a-C(:H) COATINGS

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Low friction nc-TiC/a-C and nc-TiC/a-C:H coatings were deposited using two different methods: conventional magnetron spattering (MS) and a newly developed method - gas pulse magnetron sputtering one (GPMS). In the latter plasma was controlled by short gas pulses, during which pressure in vacuum chamber increased to several dozen Pascals. Frequency of gas impulses was 1 Hz. During deposition of hydrogenated nc-TiC/a-C:H coatings, hydrogen or methane was let into the vacuum chamber together with Ar. The nanocomposite coatings were deposited on hardened Vanadis23 steel plates (Ø 26 mm × 6 mm, 64 HRC) and pure Si wafers (10 mm × 10 mm × 565μm). The coatings were investigated with HRTEM, EDS and tribological ball-on-plate test. The GPMS method in comparison with the conventional sputtering allows to obtain:

- nanocomposite nc-TiC/a-C coatings with greater wear resistance during dry sliding friction.
- lower coefficient of friction of hydrogenated nc-TiC/a-C:H coatings when additional of hydrogen or methane gas was let into the chamber at a flow rate 5 sccm.
- significantly smaller dimensions of TiC nanocrystallites in the nc-TiC/a-C coatings structure.

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THE NITROGEN STABILIZED AUSTENITE COATINGS DEPOSITED BY REACTIVE MAGNETRON SPUTTERING WITH THE DIFFUSIVE SUB-LAYER

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Austenitic stainless steel is widely used in many industries; however, its low hardness and poor wear properties seriously limit these applications. One of the possible ways to enhance mechanical properties of austenitic stainless steel is an application of protective hard coatings. Austenitic stainless steel is designed for use in aggressive environments, therefore such coatings should ensure the maintenance of the corrosion resistance also in conditions of mechanical damage and be characterized by very good adhesion. The coatings made from nitrogen-stabilized austenite deposited by reactive magnetron deposition are particularly promising from this point of view. They are characterized by high hardness and corrosion resistance similar to that of austenitic stainless steel. Furthermore, it is possible to produce a diffusive connection between the coating and substrate, which is crucial for good adhesion.
The paper presents results of the investigation on the microstructure and phase composition of the nitrogen-stabilized austenite coatings, with a particular emphasis put on the possibility of producing a diffusion connection with the substrate. During the deposition process the temperature and nitrogen pressure were varied. The phase composition was revealed by means of X-ray diffraction. The elemental composition was evaluated using X-ray microanalysis (WDS, EDS) and glow discharge optical emission spectroscopy (GDOES). Scanning electron microscopy was used to investigate microstructure of the coatings. It was demonstrated that it is possible to obtain the coatings with diffusive sublayer in the austenitic substrate.

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TUNABLE SENSITIVITY UNDER ULTRA-VIOLET LIGHT IRRADIATION FOR ROOM TEMPERATURE NO_2 SENSING

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Atmospheric pollution is a major problem brought on by industrialization and urbanization, and many forms of air pollution disturb the delicate balance of life on earth. The eventual damage is not just on the environment but also on the human health, which is heavily at risk in polluted areas. The air pollutant species of most interest from the point of view of human health is nitrogen dioxide (NO_2). Nitrogen dioxide is an important atmospheric trace gas, above 10 ppm, not only because of its health effects but also because it absorbs visible solar radiation and contributes to impaired atmospheric visibility and as an absorber of visible radiation it could have a potential direct role in global climate change NO_2 gas. The acceptable ambient levels of NO_2 are in the range of 5-20 ppm. Therefore, it is required to detect NO_2 at these low levels.

In this work, HfO_2 samples were prepared by atomic layer deposition method and were investigated the effects of the UV light irradiation on the sensing characteristics. UV light irradiation can improve the sensing properties and result in enhanced gas sensing properties to NO_2. Our results showed that the sensor exhibited tunable sensitivity when UV irradiation time was controlled during the fabrication process. HfO_2 sensing layer exhibits good response to NO_2 gas, as well as good operational stability in the short term under UV light which has 200 nm wavelengths for 1 hour irradiation. The sensor exhibits high performances including high sensitivity and selectivity at room temperature.

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THE RELATION BETWEEN PHYSICAL AND MORPHOLOGICAL PROPERTIES OF ZnO/WASTE POLYSTYRENE NANO FLUID

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This research studies the relation between viscosity, density, surface tension, optical and microstructure properties of ZnO/waste polystyrene nanofluid. The nanofluid prepared by dissolving the waste polystyrene in toluene solvent then mixed with0.1, 0.3, 0.6, 0.9 and 1.2 wt% of ZnO nanoparticles (NPs) using sonication process. XRD and AFM are used to characterize ZnO NPs. Cone-plate viscometer and tensiometer is used to examine the viscosity and surface tension. UV- visible and optical microscopy is used to examine the microstructure and optical properties of the nanofluid. The results show that the viscosity and density decreases at lower ratio then increased at high ratio of ZnO NPs, while the surface tension increases at lower ration then increases at higher ratio of ZnO NPs. The UV visible shows that the blue shift peak of Plasmon surface resonance at 0.1 of ZnO NPs then red shift increased progressively up to 1.2 of ZnO NPs. The image of optical microscopy indicates lower agglomeration at 0.1 of ZnO NPs as compared with the other ratio. There is strong relation obtained between physical and morphological properties of the waste polystyrene nanofluid. In general from physical properties can predicate the morphological properties and vice versa.

EFFECT OF FIBRES REINFORCED ON CRYSTALLINITY AND FATIGUE BEHAVIOUR OF (POLY ETHER ETHER KETONE) COMPOSITE IN AEROSPACE APPLICATIONS

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In present paper the effect of fibres types (glass &carbon fibres) on crystallinity and fatigue behaviour, for high performance composite materials of poly ether ether ketone (PEEK)/glass &carbon fibre are studied ,which are used in the aircraft Industry. Three matrix materials (PEEK natural) , (PEEK+30% glass fibre) and (PEEK+30% carbon fibre) were used in this work. Deferent microstructure testing techniques (infrared (FTIR, SEM and DSC) are used, to identify the types of (PEEK), and evaluate of crystallinity intensity of the (PEEK) with fibre types, Also deferent mechanical testes (tensile test, impact strength and the fatigue test) are conducted to evaluate the effect of these additions on the mechanical properties for the prepared composite materials. The results of (FTIR)show the standard type of PEEK polymer with compared with the other types of matrix materials which are used in this study and show the addition of GFRP to PEEK decreases the crystalline of (PEEK) material, and vise verse for CFRP. The results show that the tensile, impact and fatigue properties of (PEEK) decreases with GFRP and increase with carbon fibre addition. SEM results show the different in topography of fracture surfaces of the samples that are subjected to fatigue test after failure at maximum load.

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