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C1-II-P-THU-P2-15 Enhanced photocatalytic activity of titanium dioxide photonic crystals modified with photodeposited platinum nanoparticles.

<u>M.Sc. Joanna Ginter¹</u>, M.Sc. Kaja Spilarewicz-Stanek¹, Dr Aneta Kisielewska¹, Prof. Ireneusz Piwoński¹ ¹University of Lodz, Faculty of Chemistry, Department of Materials Technology and Chemistry, Lodz, Poland

C1-II-P-THU-P2-16 Tunable wettability of thin polymer films on microstructured silicon surfaces

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C1-II-P-THU-P2-19 Dynamic wettability control through stretching of bilayer polymer films

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C1-II-P-THU-P2-20 Silver/hydroxyapatite hybrid coatings on Ti-6Al-4V surfaces by sol-gel method

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C1-I-P-TUE-P1-1

Role of polydopamine as a primer for catalyst coating on polyurethane foams

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Considering the adhesive properties of dopamine in mussel foot, its polymeric form (polydopamine) is widely studied as a surface modifier, to improve the adhesion of coatings. Recently, it has been used as a primer to coat a PDMA film by a catalytic Pd/alumina layer (Liu et al. Chem. Eng. J. 301, 2016, 35). This primary layer was a key step for getting a stable catalytic membrane.

In the present study, we report the use of polydopamine (PDA) as a primer to allow the coating of polyurethane foams (PUF) by a Pd/alumina catalytic layer. A previous successful work showed the immobilization of a TiO_2 photocatalyst on PUF (Pardieu et al. Chem. Commun. 52, 2016, 4691).

Polyurethane foams are in general not used in structured reactors, mainly because they can not easily be coated with a catalytic layer. They nevertheless would bring several advantages such as an easy availability, a low price, an easy handling and a better uniformity than metallic foams which generally suffered from successive process operations.

Cylindrical polyurethane foams were cut to fit a Mahoney-Robinson reactor. They were first coated with a PDA layer and then successfully coated with a Pd/alumina powder catalyst. The coated foam was used in successive batch hydrogenations of α -methylstyrene. 6 successive runs were possible, reaching a total turnover number of 36000 mol(cumene)/mol(Pd)). The activity of the catalyst was still high at the end of the 6th run (more than half the activity of the fresh catalyst). No palladium leaching was noticed.

The use of coated polyurethane foams in catalytic reactions is thus possible, whereas limited to low temperature reactions due to the thermal stability of polyurethane.



Coated foam deep characterization using X-ray tomography

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Gas-solid catalytic reactors where the catalyst is deposited on a solid foam structure are of interest to scientific and industrial communities as an alternative to conventional packed beds and monoliths, mainly due to improvement in pressure drop, thermal control and radial mixing.[1]

Washcoating the catalyst on their "random" macroporous structure without deterioration of its advantages is a key point. The coating methods, already described [2], depend on both the substrate and the foam nature. Scanning Electron Microscopy, widely used, requires a sample preparation (inclusion in resin, cutting, polishing, ...) but informations obtained are only adapted to small sample and this technique remains quite local.

In the lab, recent works take advantage of the use of X-ray tomography associated to the common characterization methods (adhesion test, SEM, ...) in order to verify the homogeneity and thickness of the catalytic layer on these porous solids (Figure 1 : Example of catalyst-coated foam (cylinder diameter: 20mm, length: 24mm) and X-ray tomography associated)

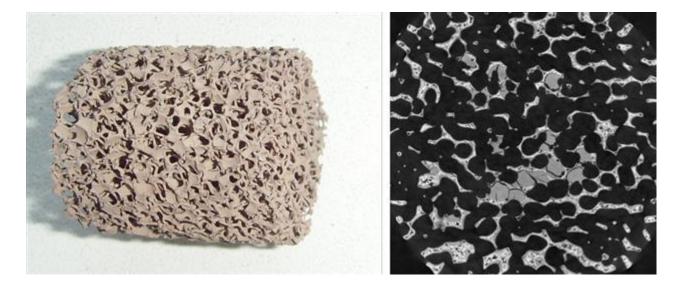
X-ray tomography is a non-destructive method and does not require special preparation. Associated to image analysis, it allows a volume visualization of the catalyst layer, an important attractive feature for this type of solids. However, this technique depends on X-ray absorption properties of the materials involved (solid and deposit). In this work, to extend the use of this X-ray tomography visualisation technique, contrast agents have been tested.

[1] J. Leveque, R. Philippe, M.-L. Zanota, V. Meille, F. Sarrazin, L. Baussaron and C. de Bellefon, Hydrodynamics and mass transfer in a tubular reactor containing foam packings for intensification of G-L-S catalytic reactions in co-current up-flow configuration, Chemical Engineering Research and Design doi: 10.1016/j.cherd.2016.03.017

[2] V. Meille, Review on methods to deposit catalysts on structured surfaces, Appl. Catal. A, vol. 315, pp. 1-17, 2006.

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New Optical Adhesive Reliability Evaluation for Silicon Photonics

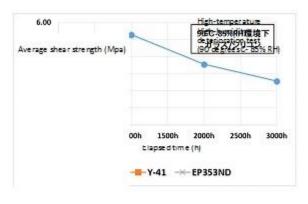
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For the progress of FTTH (fibre-to-the-home), the performance of optical adhesives is an important issue for increasing the reliability of optical networks. In addition, in recent years, the demand for optical adhesives for silicon and glass in silicon photonics has been increasing. In this paper, we will report the results of the durability of the newly developed Y-421 optical adhesives between silicon and glass.

The ratio of base resin and hardening accelerator in Y-421 is 100:40, that of Y-41 is 100:10, and that of Epotek-353ND is also 100:10. The adhesion surface between the float glass and silicon was 8 mm2 (2 × 4 [mm2]). The initial shear strength was measured, and the shear strength after the 90 degrees C- 85% RH high-temperature high-humidity accelerated aging test after 100, 200, 300, 500, 1000, 2000, and 3000 h were measured.

Figure 1 shows the shear strength after the high-temperature high-humidity (90 degrees C- 85% RH) test for Y-421, Y-41, and Epotek-353ND after the elapsed time in hours. From Fig. 1, it can be seen that the strength of Y-421 increased until 1000 h, and after 2000 h the strength declined slightly. On the other hand, Epotek-353ND and Y-41 showed a decline of strength after 500 h or 750 h. The fracture morphology was silicon fracture until 100 h, glass fractures were also observed. And after 2000 h and 3000 h, the shear strength was similar to the initial strength. From these results, we can deduce that the fracture morphology was silicon strengths. We can say that Y-421 is a more relaible than Epotek-353ND and Y-41. Y-421 showed a highly reliable bond durability for silicon photonics.





Multiscale characterization of the bio-tribological amorphous carbon coatings (a-C:H) implanted by metallic nano-particles

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The amorphous carbon coatings (a-C:H or DLC) due to exceptional properties (low fraction coefficient, high wear resistance as well as biocompatibility) are used on a wide scale in different industry e.g. medicine, aircrafts, electronics. However, the disadvantage is inherent thermal stability and high residual stress, which cause weak adhesion of the coating to the substrate materials.

In the presented research, in order to improve properties of the pure a-C:H coatings, the group of metallic nano- particles (Zr, Cu, Nb, Ta, AgPt, and Ag) has been implanted into their structure. Bio-tribological coatings were deposited on advanced Carbon–Fiber-Composites and conventional tool steel (316L) substrates by magnetron sputtering technique. Nano-composite coatings were subjected to complex biological and tribological characterization. Biological analysis included the analysis of blood- material interaction and eukaryotic cells adhesion to coatings surface. Tribological analysis were connected with micro- hardness, scratch and wear tests. The applied biological and mechanical analysis allowed the optimal bio-tribological parameters to be indicated for the potential application as protective coatings for metallic medical tools. The optimal parameters were found for the a-C:H coatings reinforced by Zr nano-particles.

Microstructural characterization of as-deposited coatings and after bio-mechanical tests was studied by means of Transmission Electron Microscopy and X-ray techniques. Thin foils for TEM observations were prepared using Focused Ion Beam method equipped with an Omniprobe lift-out system. The technique allows cutting out thin foils directly from the places of interest. Microstructural description of a-C:H coatings reinforced by Zr nano-particles showed wear mechanism cracking process in micro and nanoscale as well as very good coating adhesion to the metallic substrate. The X-ray technique indicated the positive reaction metallic nano-particles on the residual stress reduction.

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Bio- compatibile, wear resistant, decorative coatings for biological, corrosive fluids interaction

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Requirements for tribological protective coatings for medical tools, which would increase their wear and corrosion resistance, are very high. They should withstand corrosive body fluids environment, high – temperature as well as guaranty the adequate wear resistant properties. The main goal of the work was the bio- corrosion and bio- tribological

(wear in body fluids) properties increase together with aesthetic impression improvement

of metallic surfaces by application of advanced Zr/ZrN and Zr/ZrN+a-C:H multilayer coatings as well as their multiscale characterization. Coatings contained different phases ratio (metallic to ceramic). They were deposited by the application of the magnetron sputtering technique on metallic substrates (tool steel 316L). The bio- corrosion test was performed

in Ringer solution at 37 degree Celsius in order to simulate human fluids. The biological analysis was based on the cytotoxicity investigations. Tribological experiments included indentation, scratch and wear tests. All investigated coatings were characterized by high hardness, wear resistance, as well as, from the biocompatibility point of view, they were nontoxic. Anyway, the bio- tribological investigations indicated the optimal bio- tribological properties for coatings with higher amount of ceramic phase in the structure and with amorphous carbon layer (a-C:H) at a top (as an outer layer). The microstructure

of as deposited coatings as well as after bio- tribological experimants was analyzed

by the transmission electron microscopy techniques (TEM). Thin foils for TEM observations were prepared by the Focused Ion Beam technique (FIB), which allowed to get information about the microstructure changes directly from places of interest (namely from deformed zones caused by the bio- tribological interaction on coatings surface). Two types of wear mechanisms were noticed. The first one was based on the layer by layer coating remove and the tribo- film formation. The second one was connected with the coating cracking.



The microstructure of weld overlay Ni - base alloy deposited on carbon steel by laser QS- Nd:YAG

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Ni- base alloys are used as a one of the most important coating material and can be applied in a different environments and elements of devices having various applications. To protect the surface of the material from aggressive gases at high temperature, there are use different kinds of the Ni- base alloys. One of such alloy is a Ni-Cr-Mo-W-called commercial Inconel 686. This alloy based on NiCr22Mo9Nb (Inconel 625) , in which content of molybdenum increased and niobium was replaced with the addition of tungsten, in order to counteract the strong segregation in the microstructure, which results in homogeneity of the chemical composition of the structure. Inconel 686 is also characterized by greater resistance to high temperature corrosion and heat resistance than Inconel 625. The Inconel 686 alloy was deposited on 13CrMo4-5 steel by laser QS - Nd:YAG. In the poster the microstructure (SEM, TEM) and chemical composition (EDS) of obtained weld ovelrays were investigated.



Characteristic of LaCoO3 thin layers made by PLD for application to NOx gas sensor

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Sensors for detecting NOx gases, CH4, C3H8 belong to the group of chemical sensors, in which chemical information coming from the environment are converted into analytically useful signal. The chemical sensor based on the solid electrolyte represents a group of potentiometric sensors. The potentiometric sensors consist sensing, counter electrodes and electrolyte. In this group of solid electrolyte sensor is zirconium oxide ZrO2 doped with Y2O3. LaCoO3 belong to a group perovskite we used as sensing electrodes, as counter electrode we proposed platinum electrode. In the first step of our research we analysis the morphology of the perovskite thin films on ZrO2 doped Y2O3. The thin films LaCoO3 was deposited on ZrO2 doped with Y2O3 (3%, 9,5% mol) by PLD method. A laser ablation system was equipped with the Nd-YAG laser Continuum Powerlite DLS (Digital Laser Source) (P=266 mm) and a chamber Neocera. The deposition conditions were: laser pulse frequency 10 Hz, energy density on the target 7,8 J/cm2 , laser pulse duration 4 ns, the substrate was heated at T=750, 850, 1000 °C. To determine morphology and structure we used TEM, XRD, SEM and also AFM to determine effect of different process temperature on the thin films roughness. Thin films have a nanocrystalline structure without cracks and with small droplets on the surface. Increase of the process temperature results in the increasing of thin films roughness.

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Boronizing of Ti-Al alloys using the paste method with an optimized slurry of amorphous boron nanoparticles

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The main goal of this study was to achieve a boride layer formation at lower temperature and/or shorter time in the furnace in comparison with the traditional method of pack boronizing. For this purpose, the research was organized in three stages. First, pack boronizing experiments were performed, in order to have a reference point for the comparison. Its kinetic parameters were determined using metallographic analysis. Then, the production of amorphous boron nanoparticles slurry was carried out using ultrasonic bath and high speed stirring. The optimization of the slurry took place by differentiating parameters such as solids level and dispersant agent concentration. The stability of the slurry was estimated via Z-potential and particle size distribution measurements. Finally, boronizing experiments were performed at a temperature range and various time durations using the optimized slurry. Subsequently, the kinetic parameters of the diffusion process were subjected to comparison with the ones of pack boronizing method. Some collateral measurements with XRD and SEM were carried, too. In this presentation some representative results are exhibited, from which a conclusion about the effectiveness of this method could be extracted. References (indicatively):

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Gaseous nitriding of iron whiskers

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Nitriding is an important thermochemical surface engineering process to introduce nitrogen into the surface of ferrous alloys. Thereby, typically a nitride compound layer is formed at the surface and a diffusion layer follows as larger depths, leading to improvement of fatigue properties and to a higher surface hardness. Nucleation of the iron nitride phases at the surface of the alloy is often crucial for the compound layer's microstructure. To obtain an improved understanding of the nucleation process and of the early stages of compound layer development we strive at investigating the formation of iron nitrides at reduced dimensions.

One approach for this is use of Fe substrates of reduced dimensions. Here we present results obtained on α iron whiskers, which have been grown by reduction of ferrous chloride in hydrogen gas at 823 K by Brenner's method [1]. The whiskers were treated in a NH₃/H₂-containing gas atmosphere at 773 K - 873 K to transform them into γ' -Fe₄N phase. The initial iron whiskers as well as the nitrided iron whiskers were characterized by scanning electron microscopy including electron backscatter diffraction. The microstructures observed on the nitrided iron whiskers are compared with the microstructures observed from similarly treated polycrystalline α -Fe plates at early stages of γ' -Fe₄N phase formation.

Reference

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Assessment of the possibility to improve working life of the shaping tools in the Conform extrusion process

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The paper presents results of examinations related to possibilities of improving working life of the shaping tools in the Conform extrusion process.

The coatings deposited on the substrate samples composed of AlCrN film and covered by low-friction layer as DLC, TiC, CrCN, MoS₂ were the subject of the studies. The AlCrN layer was deposited by PVD lateral rotating ARC-cathodes (LARC). The DLC and TiC, CrCN, MoS₂ were deposited by PACVD and magnetron sputtering technology on the X6CrNiMoTi 17-12-2 respectively.

Tribological tests (coefficient of friction, wear) were performed in sliding friction conditions using the pin(ball)-on-disk method, where a ceramic Al_2O_3 ball was used as the counter-sample. The studies were performed at ambient temperature with loading force of 10 N and linear velocity of 0.2 m/s. In sliding dry friction conditions, after the break-in time, the friction coefficient for the investigated elements is set in the range between 0.14-0.63 depending on the coating type. The investigated AlCrN/DLCII and AlCrN/TiC coatings reveals high wear resistance. These coatings could be used to ensure improvement of working life of the shaping tools the Conform extrusion process.



Wear resistant and electrically conductive composite coatings on nonferrous metal substrates

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The paper presents results of examinations related to the structure, as well as mechanical and physical properties of composite coatings deposited on non-ferrous metal substrates. The emphasis was given to the preparation of special composite electrodes by spark plasma sintering (desired composition of them is a mixture of Ni, W, Cu, Al and TiB₂, TiC etc.) and deposition of them on Cu, Al, Ti substrates (and their alloys) by electro spark deposition in order to increase wear resistance of the substrate material and achieve good electrical conductivity for welding electrodes and sonotrode cutting tools applications. Final step of the research was to evaluate their friction properties (coefficient of friction, wear). The produced coatings were subjected to tribological tests with application of high temperature tribometer – THT by CSM Instruments. Tests were carried out by pin-on-disc method.



Mechanical properties and thermal behavior of Zr(–Hf)–Cu thin-film metallic glasses

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Metallic alloys are commonly fabricated as crystalline materials by a relatively slow-cooling casting of a melt. Magnetron sputter deposition as a non-equilibrium process with high cooling rates (higher than 10⁶ K/s) allows us to prepare metallic alloys also as thin-film materials in an amorphous glassy state.

Recently, we have showed that Zr–Cu thin-film alloys can be prepared as metallic glasses in a very wide composition range (30–65 at.% Cu) by non-reactive magnetron co-sputtering. In the present study, we focus on characterization of their mechanical and thermal behavior in more detail. In addition, we investigate the effect of an incorporation of Hf into the Zr–Cu thin-film metallic glasses on a potential improvement of their behavior. The films were deposited using three unbalanced magnetrons equipped with Zr, Hf and Cu targets in pure argon. The magnetrons with the Zr and Hf targets were operated in a dc regime while the Cu magnetron in a high-power impulse regime. The films were deposited without an external heating onto rotating substrates.

Mechanical properties of the Zr–Cu thin-film metallic glasses are strongly dependent on the elemental composition. A gradual growth of hardness with increasing Cu content up to 70 at.% correlates well with an evolution of the glass transition temperature and the crystallization temperature. This behavior can be explained by an increasing concentration of icosahedral clusters having the highest atomic packing density. The Zr–Cu films prepared with the Cu content higher than 50 at.% or at a moderate substrate bias exhibit a tendency to be more resistant to the formation of shear bands during indentation. A continuous substitution of the Zr by the Hf at the same concentration of the Cu leads to a gradual increase in the hardness and the thermal stability of the glassy state.



Selective electron-beam alloying of aluminium with vanadium

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It is known that surface alloying of aluminium with different transition metals by high intensity energy fluxes, such as electron-beam treatment is able to form AI3X (X=Ti, Nb, V, Hf, etc.) – hard intermetallic compounds with high melting point. In this study on commercially pure aluminium substrate bilayer structures of Ti/V were deposited by direct current magnetron sputtering (DCMS), followed by selective electron-beam surface alloying using circular sweep mode. The crystallographic structure of alloyed layers was studied by X-ray diffraction (XRD) with Cu K α characteristic radiation. The microstructure and the chemical composition were studied by scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX) respectively. Metallographic investigations were conducted in order to evaluate the mechanical properties of each sample. The obtained results are compared with respect to the applied technological parameters of the selective electron-beam alloying.



Thermal stability and mechanical properties of TiAIN/VN nano-multilayer films

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Transition-metal nitride hard films are widely used as coatings for cutting tools due to their high hardness. Nano-multilayer films deposited alternately by two kinds of materials at nanometer-scale thickness could exhibit an anomalous increase of hardness and wear resistance compared to the corresponding monoliths. Therefore, it supplied an alternative way to obtain the film with excellent mechanical properties.

TiAlN films are attracting more attentions owing to their superior properties as compared TiN especially in high temperature applications. The metastable cubic-phase TiAlN undergoes a two-step decomposition process during annealing: first to c-TiN and metastable c-AlN domains and, subsequently, to c-TiN and stable h-AlN. A coherency to the parent matrix of c-AlN domains results in the age hardening and good mechanical properties and machining performance of TiAlN. Recent reports showed that the age hardening could be enhanced by straining of the TiAlN sublayers in the nano-multilayers.

In this work, TiAlN/VN nano-multilayer films were synthesized by reactive magnetron sputtering method in order to improve the hardness and age hardening of TiAlN film. The microstructure, mechanical properties and thermal stability have been characterized by XRD, SEM, TEM, nano-indentation and CETR-UMT-3 tribometer. The results show that TiAlN grows epitaxially over VN layers under the critical layer thickness in the TiAlN/VN nano-multilayers. Correspondingly, the hardness and modulus of TiAlN/VN nano-multilayer films remarkably increase to 38.3 GPa and 683.1 GPa respectively, as compared with 27.5 GPa and 484.9 GPa of the TiAlN single film. The friction coefficient of TiAlN/VN nano-multilayer films, 0.4, is much lower than that of TiAlN single film (0.9). In addition, TiAlN/VN nano-multilayer film exhibit higher hardness than TiAlN single

film at 1000°C, indicating that the age hardening of TiAlN is enhanced by insertion of VN sublayers. In brief, the TiAlN/VN nano-multilayer films have great potential as protective coatings on cutting tools.



Formation and structure of TiN/ZrN multilayer coatings deposited on tool steel

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The formation of coatings with high hardness, toughness, wear- and corrosion-resistance, suitable for applications in the area of medicine (anti-corrosion and wear-resistance coatings of different medical instruments, artificial joints and implants) and machine engineering (protective coatings on cutting, forming and petal processing instruments and bearings) are problems of great importance nowadays. TiN/ZrN multilayer coating system is a hard, wear resistant material which is widely used for cutting and forming tools and other components operating in an abrasive wear environment.

In this study TiN/ZrN multilayer coatings was deposited on tool steel by direct current magnetron sputtering. The structure of the coatings was observed by XRD (X-ray diffraction) with CuKα characteristic radiation (1.54 Å). The measurements were conducted in Bragg-Brentano (B-B) symmetrical mode, from 20° to 80° at 2θ scale. The step has been chosen 0.1° with counting time 10 sec. per step. The microstructure of the obtained multilayer coatings was investigated by Scanning Electron Microscopy (SEM), as backscattered electrons have been used. The accelerated voltage was 20 kV. The chemical composition was studied by Energy-Dispersive X-ray Spectroscopy (EDX). The surface of the coatings was observed by Atomic Forced Microscopy (ATM). Nanoindenter tester (Brucker, USA) was used to measure the nanohardness and Young's modulus.

The obtained results demonstrate the possibility of formation of hard and wear resistant multilayer coating of TiN/ZrN on the tool steels. It was shown the opportunity of formation of surface coatings with good stoichiometry and great mechanical properties with the chosen technological parameters, applied during the deposition.



Atomic layer deposition of tin oxide thin films using tetraethyltin to produce high-capacity Li-ion batteries

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Thin-film power sources are the most advanced and sometimes the only feasible components for use in smart cards, microchips with integrated power supplies, various portable devices, and medical implants. Currently, most batteries of this type have lithium based anodes but this material has safety issues. Tin oxide thin film anodes are safer and have high capacity and cycling performance.

In this research, we deposited thin films of tin oxide on substrates of silicon and stainless steel by using atomic layer deposition (ALD) with tetraethyltin precursor. Various coreactants such as water, oxygen, remote oxygen plasma, hydrogen peroxide, and ozone were used. We compared the growth features of the thin-film tin oxides and characterized their compositions, morphologies, phases in detail by using spectral ellipsometry, scanning electron microscopy, atomic force microscopy, X-ray diffraction, X-ray reflectivity, X-ray photoelectron spectroscopy.

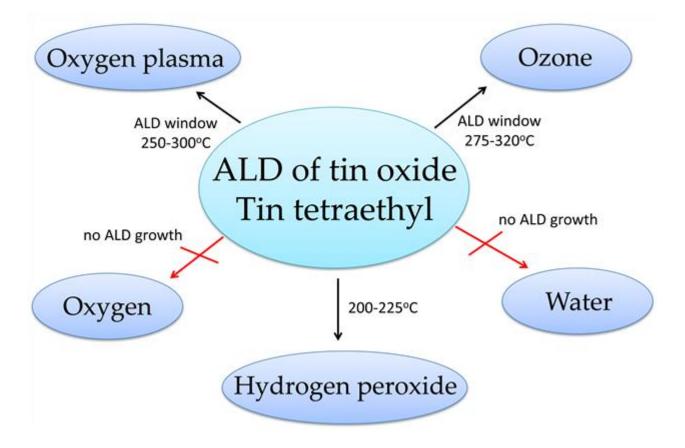
We optimized the process parameters of ALD, determining the optimal deposition temperature, pulse time of TET, and coreactants. Using oxygen plasma used as the oxidizer allowed to deposit coatings over a wide range of temperatures, but the temperature greatly affected the film morphology. The films deposited using H_2O_2 and O_3 oxidizers showed good conformity, density near that of bulk SnO_2 , and low roughness. The films deposited below $300^{\circ}C$ were amorphous. All deposited films contained tin in the +4 oxidation state, but also contained excess oxygen and carbon contamination.

All samples exhibited very stable cycling electrochemical performance. The "crystalline" thin films showed a specific capacity above 900 mAh/g with a Coulombic efficiency over 99%. The samples demonstrated excellent cycling performance through over 500 cycles.

This research was conducted using the equipment of the resource centers of the Research Park of the St. Petersburg State University «Innovative Technologies of Composite Nanomaterials», «Centre for Physical Methods of Surface Investigation», «Centre for X-ray Diffraction Studies», «Nanotechnology Interdisciplinary Centre».

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Structure and properties of TiO2/TiN coated EBM modified Ti alloy for biomedical application

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It is well known that the Ti alloys are widely used to manufacture implants but they have poor shear strength and wear resistance that makes them less desirable for articulating surfaces. Additionally, although Ti shows inert behavior, the bone attachment is difficult and the body encapsulates the material. The application of hard PVD coatings on properly prepared Ti alloy surface could basically improve both the mechanical properties and osseointegration of the implant surface via tight bonding both to substrate and bone.

The substrate material used in the present study was Ti-5Al-4V alloy in different conditions – as received, solution treated and overaged. In order to enhance the bone fixation strength, the surface roughness and microscale organization of both coarse lamellar and bimodal microstructure was improved by electron-beam modification (EBM). After that the nanoscale modification that increases the surface area and biocompatibility was obtained by deposition of bilayered TiO2/TiN coatings. For these two different PVD techniques: cathodic arc deposition and magnetron sputtering were compared. This work concentrates on the nature and strength of the applied surface treatments. Light optical microscopy, AFM, SEM and XRD technique were carried out to examine the effect of the EBM and coating deposition on the microstructure, surface texture and phase composition. The effect of the applied surface modification on the hardness values in depth of the substrate is discussed. The experiment then moves onto investigating the surface hardness and tribological performance of the system by employing nanoindentation and scratch analysis, respectively. Yet the effect of the combined treatment (EBM and PVD coating) on the mechanical strength and tribological properties of the implant material is unexplored.



A TUBE (InTerconnection under UHV of ChamBers for Elaboration, and Characterization for Novel Materials) FOR MULTI-MATERIAL GROWTH AND MULTI-TECHNIC CHARACTERIZATION UNDER ULTRA HIGH VACUUM

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The TUBE is a unique tool that combines a wide range of specific thin film deposition/growth analysis and post-deposition treatment techniques, all connected under Ultra High Vacuum (UHV).

In a context where fundamental physics, as well as the functionality of the devices, are no longer driven by the material bulk properties but by surface and interface properties, materials have to be grown, controlled and characterized in a UHV environment in order to avoid fatal contamination.

The TUBE is a linear UHV pipeline where samples are transported with 3 motorized and automated monorail trains. By means of transfer sticks, two inches sample holder can be exchanged between the train and the chambers.

In this way 10 growth chambers (MBE, PVD, PLD...) are connected to 8 analysis chambers where structural as well as functional characterization can be carried out (XPS, Ellipsometry, AFM...). As-deposited samples can be transported from the growth chamber to either one of the analysis chambers in an environment with a base pressure of 1×10-10 Torr, which protects them from oxidation or other structural or chemical changes related to atmosphere exposure. Different materials which need different deposition process can be done in order to create multi-material sample with an atomic scale control.

The TUBE is made of 2 UHV connections, a 40 meters one in the scientific hall and a 30 meters one in the industrial partners hall allowing fundamental and applied projects in interdisciplinary fields, coupling a multi-material and multi-analysis approach.

In conclusion this tool is not only going to give access to a platform composed of state of the art deposition, analysis and patterning chambers, but it will also be providing and developing the skills and competences in thin film growth and characterization.



ANALISIS OF PLASMA NITRIDING PARAMETERS APPLIED IN HIGH SPEED STEEL CUTTING TOOLS

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Nowadays, industry has made great efforts to reduce production costs and at the same time increase the quantity and quality of their manufactured products. The machinery processes intend to provide shapes and finishes in metallic components, by using tools to turning, drilling, milling and others processes. During the machining, a factor that determines the lifecycle of the tools is the wear. As an alternative to improve the wear resistance of those tools, the machining industries used techniques as heat treatment and coating, in order to reduce the effects caused by the fatigue in the material. In this paper, we will discuss the plasma nitriding, samples of High Speed Steel tools submitted to a 30 min pre-sputtering treatment with 30 sccm H2 at 300°C to the proper cleaning, than they were nitrided in different temperatures 400°C, 450°C and 500°C in atmospheres with 20% N2 and 80% H2 during 4 hours. In doing that, we increase the surface hardness of the samples and is expected to improve the lifecycle of the tools in the industry. The microhardness test show that the average surface hardness of 450°C sample was 1339 HV while the non-nitrided was 820 HV achieving an improvement of 163% on the hardness.



Deposition and investigation of resistant Al/Ni coatings deposited by pack cementation

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Steel is a commonly used material in construction applications. However its good mechanical properties, steel oxidizes quickly and easily, especially in elevated temperature environments

which has economic and environmental consequences. Developing a coating layer of proper materials with improved properties can improve high temperature, wear and oxidation durability. Ideal materials for such applications appear to be Nickel and Aluminum, for which have claimed to possess high-temperature mechanical strength and oxidation resistance, especially their compounds with iron. In this work such coatings were prepared by the combination of two simple, economic processes comparing with the already used techniques. An initial Ni layer was electrochemically deposited on steel substrates followed by Al deposition via pack cementation thermochemical process, performed at low temperature in order not to affect seriously the mechanical properties of the substrate. Furthermore an attempt was made to insert inert oxide nanoparticles in these coatings in order to enhance further their durability. The characterization of structure and phase identification was performed by X-ray diffraction and X-ray photoelectron spectroscopy, and the morphology and elemental analysis was examined by Scanning electron microscopy. The final coatings were found to contain Ni-Al phases, mainly Al3Ni2 and AlNi3, also Ni-Fe phases appeared at the substrate/coating interface, due to the diffusion of Ni layer in the substrate during thermal treatment at the aluminization step. Thermogravimetric and electrochemical corrosion measurements reported a significant increase of the samples resistance, comparing to bare steel, while coatings with nanoparticles were revealed to withstand better the applied aggressive environments.



Research on surface wettability of copper and copper-based alloys for antimicrobial surfaces.

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Among various applications copper and its alloys are used as a material for antimicrobial surfaces. One of the most important properties of the surface of any material is its wettability, which is especially important in case of materials intended for antimicrobial surfaces. The paper present the results of research on modification techniques of the copper-based alloys towards obtaining more hydrophobic surfaces. The applied techniques include various combinations of thermo-chemical treatments. The treated copper-based materials were in the next step subjected to evaluation of their surface morphology and water droplet contact angle.

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Zn-ZnO core-shell nanoparticles deposited onto tantalum nanostructures

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Development of multifunctional material for medical devices, such as dental implants, are of great interest due to their multifunctional requirements. Biocompatibility, osseointegration, bioactivity and high chemical resistance are some of the indispensable characteristics of these types of devices. However, during the last few decades, antibacterial features have been being incorporated, since the probability of infections can be largely reduced, and, hence decrease the implants rejection rates. In this sense, to reduce the appearance of bacterial infections, this study proposes the incorporation of antibacterial zinc oxide nanoparticles on tantalum nanostructured surfaces.

Zn/ZnO core-shell nanoparticles were deposited by DC-pulsed magnetron sputtering in an Ar atmosphere using a high purity Zn (99.99%) onto nanostructured anodized tantalum. The deposition conditions, such as discharge pressure, deposition time and the substrate bias voltage were varied in order to obtain different particle size and distributions that control the functional properties.

The structure, morphology and composition of the NPs were determined using both a conventional JEOL 2100F TEM and an aberration-corrected FEI Titan. The results revealed that only altering the size of the nanostrutures (dimples) on the surface, the ZnO nanoparticle growth can be modified, changing the particle size and changing their distribution. On the other hand, zinc ions release and surface antibacterial activity was determined by inductively coupled plasma and halo test (zone of inhibition - ZOI), respectively.



MAG-CW WELDING PROCESS WITH ADDITION OF POWDER OBTAINED FROM COATED ELECTRODE OK 8358

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The wear and tear of equipment and components in the industrial and agricultural branches as well as numerous branches of activity, represents a great factor of depreciation of capital and source of expenses with maintenance and replacement of mechanical components. Often it entails stopping the production line in order to restore and repair, limiting production, directly influencing the output of an industrial segment. As wear is a surface phenomenon involving mechanical removal, the solutions found through the coating solder have been shown to be highly effective both to prevent and to minimize or recover the different forms of wear of the metals. In this way, MAG-CW welding procedures have been applied in order to minimize these additional costs for an industrial branch. However, the implementation of procedures such as these refers to a cost of operation, which in turn, meets the purpose of the use, the improvement of the operating procedures can further optimize the application of the process in industrial productions.

Thus, this work is done in the modification and optimization of the MAG-CW welding process in the wear coating procedures. Thus, a MAG-CW welding process with powder addition was used, this being obtained after the removal and grinding of the electrode coating OK 8358, making use of a machined process and as a base material the ASTM A36 steel due to be enough used in industry. The process begins by deposition of the powder in the test body, afterwards the welding is carried out with the pre-established parameters of tension, current, wire speed and speed of the welding equipment. Satisfactory results were obtained in the welding process, since by surface analysis a geometric regularity of the strands (width, height) along its length is observed, with little slag and low index of spatter.



Study on the Electrochemical Characterization of Dimensionally Stable Anode for Electroplating Applications

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The electrodeposited copper foil process was reduction copper from electrolyte using DSA and titanium plate cathode. High current is applied in this process, copper is deposited at a high rate on the cathode, and oxygen evolution reaction increased in the anode. Increasing oxygen reaction was facilitated degradation on the surface of anode, corresponding increase applied voltage and power consumption. Therefore, the long-term stability of DSA is the most important property for electrodeposition of copper thin film. DSAs were fabricated two or three components metal oxide such as iridium, ruthenium, tantalum and platinum because of their electrochemical performance and stability. In order to enhance the long-term stability of DSA for copper electroplating process, in the present study, noble metal oxides with excellent electrochemical properties was used and optimum condition was determined the ratio of noble metal oxides and surface pre-treatment of titanium substrate and heat treatment. The composition and surface morphologies on the surface of DSA were characterized by field emission scanning electron microscopy (FE-SEM), energy dispersive X-ray spectrometer (EDS or EDX). The effect of the surface pretreatment of titanium substrate and ratio of noble metal oxides were estimated by accelerated test at the highly current density conditions. Nano-dimple of titanium treatment led to surface of nano structure, which is ascribed to an increase a thirteen-fold increase in the lifetime of DSA. The improvement of accelerated life time of DSA seems to be closely related to adhesion between metal oxide coating layer and substrate.



Surface patterning to improve joint strength of SiC and SiC/SiC

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The effect of a new controlled atmosphere plasma etching on the surface morphology of silicon carbide and SiC/SiC composites was investigated with the aim of improving adhesion with an epoxy adhesive. Scanning electron microscopy and atomic force microscopy revealed that the plasma treatment induced the formation of a nanostructured layer on the SiC and SiC/SiC surface.

The joints have been performed with a commercial epoxy adhesive (Hysol[®] EA9321). A study has been carried out to understand the effectiveness of the plasma etching on the adhesive/substrate interface, and the mechanical strength of the joint. The apparent lap shear strength of the joined SiC and SiC/SiC samples has been measured before and after etching. The plasma etching surface modification was also compared to the Selective Thermal Removal, an innovative process developed by the authors and used to obtain a brush-like SiC/SiC surface in view of joining. The full exploitation by the aerospace industry of the attractive and unique properties of SiC based composites or monolithic SiC calls for effective joining technologies that may help to assemble them in complex shapes or to combine them with metallic parts forming hybrid structures.



Removable Parylene Based Bilayer for Barrier Corrosion Protection of Metallic Archaeological Artefacts

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A combination of acrylic resins and microcrystalline waxes are commonly used for preservation and conservation of metallic archaeological artefacts. Nowadays, novel, more efficient polymers may be considered for alternative conservation methods thanks to the development of advanced materials and technologies during recent years. The usage of barrier films for this purpose appears promising. Barrier films act as a gas protection of materials against external influences and they are used in wide range of industrial fields such as microelectronics, food packaging, anticorrosion layers etc.

The polymer coating for such a special application must fulfil a number of specific requirements. The protective coating should last for a long time and it should be easily removable to allow future tests. The treatment should not change the optical appearance of artefacts and the anticorrosive coating should act as a diffusion barrier against oxygen, moisture and atmospheric pollutants.

Based on our previous research, parylene (poly-p-xylylene) polymer was chosen as a suitable candidate for the preparation of barrier films thanks to its desirable properties such as excellent barrier properties, transparency and formation of conformal coating without defects. The soluble interlayer made from the special silicon acrylic lacquer Laksil was applied between metal and parylene film to ensure removability of coating.

The presented contribution deals with the deposition of Laksil/parylene bilayer on iron, copper, brass and bronze model samples and on the authentic iron artefact. The physical and chemical characteristics of bilayer were determined using the profilometry, SEM, colouristic measurements and standard corrosion tests. The removal efficiency of protective bilayer was evaluated by EDX and TGA analysis and almost full removal was confirmed. The barrier properties of our new bilayer coating were much better in comparison with conventional conservation coating composed of Paraloid B72 acrylic resin and Revax microcrystalline wax.



ZnO as antireflective coating for thermochromic VO2 films

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Thermochromic materials are used as coatings on smart windows in order to regulate the indoor temperature. VO2 is the most common thermochromic material, since its critical transition temperature (TC = 68 oC) is close to Room Temperature. Below TC it is highly transparent in IR, while above TC it becomes opaque in IR, and as a result it is the appropriate candidate to control the internal temperature of rooms for energy efficient buildings. However, the poor visible transmittance is a critical parameter that has to be improved. This can be done either by doping or by depositing a suitable overlayer which can act as antireflection (AR) coating on VO2 films.

In the present work, ZnO was examined as AR coatings on VO2 films. The ZnO layers were deposited by dc magnetron sputtering at Room Temperature on VO2 thermochromic films which had been grown by rf sputtering technique on commercial Pilkington K-Glass substrates at a low temperature of 300 oC. The thickness of ZnO AR coatings was varied between 22 nm and 100 nm. It was found that a 30 nm ZnO AR coating enhanced the luminous transmittance of VO2 thermochromic films by about 8% from 38% without AR coating to 46% with AR coating. Moreover, solar modulation was slightly increased by about 1%, while critical transition temperature and width of transmittance hysteresis loop remained unchanged of AR coating. Thus, ZnO can be the appropriate AR coating to enhance luminous transmittance of VO2 thermochromic films.



The effect of Iridium on the properties of zinc oxide films

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The backbone of the current microelectronics industry are components based on silicon semiconductors. However, the perspectives for further developments are limited due to material constraints like non availability for flexible devices, optical opacity and need for high temperature processing. The emerging class of oxide semiconductors is able to overcome many of those restrictions, especially because some of them can be prepared as thin (transparent) films under comparatively moderate conditions.

ZnO is a wide band gap semiconductor (Eg ~ 3.3eV) with high exciton binding energy (~60 meV), has good transparency and its conductivity approaches that of the widely used n-type ITO. However, achieving p-type ZnO in a controllable, reproducible and reliable manner remains difficult. A different class of p-type zinc oxides with iridium has attracted attention recently, due the some remarkable features of their electronic structure. Despite the very limited available data on Zn-O-Ir system, theoretical calculations have shown that while light Ir doping inhibits p-type conduction and transparency, high Ir concentrations hole trapping does not play an important role in p-type conductivity in these materials. Similar findings concerning p-type conduction have been reported for the case of nickel aluminate spinel. In this investigation we report on the properties of rf-sputtered ZnON:Ir. The ZnON films were deposited from a zinc nitride target and the dopant was introduced by placing on the target surface Iridium pellets. The films were prepared in different amounts of Oxygen in Ar plasma and the morphological, structural, optical and electronic properties of the films were examined just after deposition as well as after annealing. The ZnON films containing 5 at.% of Ir assume high transparency which is improving upon annealing, while resistivity seems to depend more on Ir content than oxygen content in plasma.

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Influence of mode of electrodeposition, grain size on mechanical propertice of electrodeposited nanocrystaline nickel coatings.

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This paper presents results of conducted measurements of mechanical and tribological properties of nanocrystaline nickel coatings. Nickel coatings were electroplated on electropolished copper. There were two sets of samples with different grain size, one set of 10 samples electroplated using saccharine as a grain refinement agent, second set of 10 samples deposited with pulsed current with varied properties of current to obtain coatings with different grain size. Prepared samples were subjected to tensile tests, micro- and nanoindentation, grain size measurement under XRD. Moreover, there were measured friction coefficient and wear resistance at the macroscale with the custom tribometer and at the microscale using LFM and small loads. The Hall-Petch effect was observed in both mechanical and tribological testing. The hardness as well as friction coefficient and wear resistance increase with the decrease of grain size.



Structure and properties of the soft magnetic Fe-Zr-N nanofilms with enhanced mechanical characteristics

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The Fe-Zr-N films with broad composition range (total formula is Fe99-43Zr0-35N0-12O2-15) were deposited in Ar+N2 gas mixtures (0, 0.05 or 0.15 % of N2) by magnetron sputtering of Fe target covered with Zr plates (0, 2.4, 5.3, and 13.4 wt.% Zr). The films were annealed in vacuum at temperatures 300÷600°C to control the amount of amorphous phase. The structure and phase composition were investigated by TEM, SEM, XRD, and GDOES methods. Mechanical and tribological properties were estimated by nanoindentation and pin-on-disk test. Magnetic properties of as-deposited and annealed films were studied by VSM.

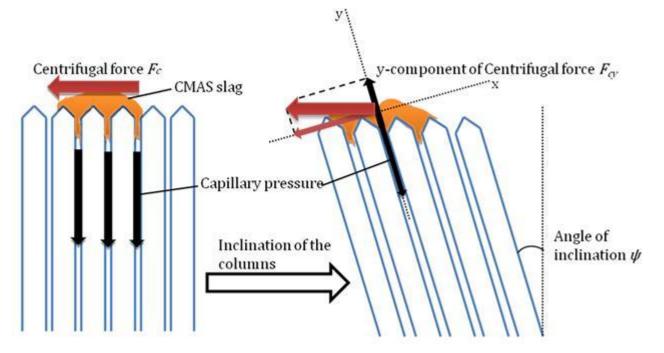
Results showed that depending on the chemical composition the single-phase (bcc α -Fe(Zr,N) solid solution), two-phase (α -Fe(Zr,N) + Fe4N or α -Fe + ZrO2–x), or three-phase (Fe4N + Fe3N + α -Fe(Zr,N)/FeO) films with a nanocrystalline, amorphous, or nanocomposite (amorphous + nanocrystalline) structure are formed. The formation of the solid solution is confirmed by the fact that the bcc-phase lattice parameter (2.868 Å – 2.918 Å) is higher than that of pure α -Fe (2.866 Å). The average grain sizes of the bcc phase have been found to be in the range of 2–28 nm, depending on total concentration (Zr+N) in the films. It is found that the as-deposited nanocrystalline films had a columnar structure with columns elongated in a film growth direction. The columnar structure was more pronounced after annealing at temperatures above 400° C. Fe-Zr-N coatings exhibited the hardness in range of 14-19 GPa, elastic modulus 156-174 GPa, elastic recovery 55-72%. It was shown that the as-sputtered and annealed at 300-600°C films are strong ferromagnets with high saturation induction (up to 2.1 T) and low coercive field (as low as 0.5 Oe). The correlations between the Bs and Hc magnitudes and the chemical composition of the films, their phase and structural states and magnetic structure are discussed.



Employment of the operational centrifugal forces of the turbine to resist the Calcium-Magnesium-Alumninosilicates infiltration in a EB-PVD thermal barrier coating: A numerical simulation.

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As future gas turbine engines require increased operating temperatures to improve efficiency, the resistance to CMAS (Calcia-Magnesia-Alumino-Silicate) induced degradation has been rendered critical for the development of forthcoming thermal barrier coating (TBC) systems. Although mitigation strategies have appeared, most of them approach the problem strictly from a chemical reaction point of view. This work aims to study the dynamic aspect of CMAS infiltration in the TBC and evaluate the efficacy of a proposed TBC structure with inclined columns. With an inclination of the columns in the right direction, the TBC can benefit from the centrifugal forces acting on the infiltrating slug and limit its penetration in the coating. A model based on the one dimensional momentum balance for a laminar flow in a non-circular conduit is used and the effect of TBC column inclination angle and hydraulic radius of inter-columnar gaps is explored. It is shown that with appropriate TBC structure design the infiltration of CMAS can be permanently arrested prior complete infiltration of the TBC occurs, by the act of the centrifugal forces generated by the operating turbine engine.



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Silicide coating on Ti-46Al-8Ta (at.%) – assessment of growth mechanism through diffusion-couple experiments

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Advanced alloys based on intermetallic phases γ -TiAl and α 2-Ti3Al are intensively studied as lightweight structural materials for aircraft and automotive industry. However, their applications are limited because of insufficient corrosion resistance at high temperature. Among many possible surface modifications, silicide coatings seem to be particularly promising since they produce a SiO2-TiO2 scale, which constitutes an effective barrier against the penetration of aggressive species. In this work a silicon-rich coating with good adhesion was deposited on a Ti-46Al-8Ta (at%) alloy in a two-step process, comprising physical and chemical vapour deposition (magnetron sputtering and pack cementation, respectively). As the development of multilayer and multiphase coating was not fully understood, diffusion-couple experiments were undertaken to elucidate the growth mechanism.

Diffusion couples, consisting of Si wafers and Ti-46Al-8Ta (at%) alloy plates were assembled in molybdenum holders and annealed at the temperature of 950 oC or 1050 oC under an argon atmosphere for 8, 24 or 48 hours. Cross-sections of the diffusion couples were analyzed by SEM/EDS and phase composition was determined by XRD.

The diffusion-couple experiments revealed complexity of coating growth, involving two-way transport of reactants - inward diffusion of Si and outward diffusion of Ti/Ta) - accompanied by segregation of aluminium from the Ti/Ta silicides. The investigations will be further supplemented by thermochemical calculations and diffusion simulations.

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The influence of the phosphorous content and heat treatment on the nanomicro-structure, thickness and micro-hardness of electroless Ni-P coatings on steel

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Electroless Ni-P coatings were obtained on steel substrates using different bath compositions, which lead to different phosphorous contents of the coatings. The effect of the P-content was experimentally studied on the thickness of the coating, on its nano-micro-structure and on its micro-hardness. The as-received samples were nano-crystalline and their micro-hardness was found to decrease with increasing the P-content. Upon annealing at 400 oC a new Ni3P phase was formed and the nano-cystalline Ni-rich grains coarsened to micrograins. In this annealed state the micro-hardness was found to increase with increasing the P-content. A complex model was built to explain the experimental results. It was supposed that the as-received Ni-P coating contains almost pure Ni nano-grains surrounded by segregated P atoms. When the grain is fully covered by the P atoms, further grain growth is inhibited and the coating can grow further only due to nucleation of a new grain. Thus, the size of the grains was found inversely proportional to the P-content of the Ni-P alloy. The need for a larger number of nucleation events with decreasing grain size explains why the coating has a smaller thickness for smaller grain size, i.e. higher P-content. The inverse Hall-Petch rule was found for the grain size dependence of micro-hardness of the as deposited samples due to the grain boundary sliding of relatively hard Ni-rich nano-crystals along the soft P atoms (higher P-content lead to lower microhardness through smaller grain size). After annealing the micro-hardness was found to increase with the volumetric phase fraction of the harder Ni3P phase within a relatively soft Ni matrix, i.e. it was found to increase with the P-content of the Ni-P coating. The extrapolated value for the micro-hardness of the Ni3P phase is found about 757 ± 20 HV0.01.



Characterization of Yb3+ Doped Y2O3 Thin Films Prepared by Electron Beam Evaporation Method

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In this study, Yb doped Y2O3 powders were prepared from nitrate precursor solutions with different Yb3+ dopant ratios by sol-gel method. The powders were pressed to form pellets. The pellets were evaporated by using electron beam evaporation method and Yb3+Y2O3 thin films were deposited on both glass and silicon wafer substrates. The films were investigated by X-ray diffraction analysis (XRD) and spectrophotometer. Phase composition of the thin films was investigated by X-ray diffractometer with thin film attachment. Morphology of the thin films on substrates was characterized by using scanning electron microscopy (SEM). The effect of dopant ratio on optical properties was investigated by using spectrophotometer. Also, The cell parameters of thin films were calculated by using Cohen-Wagner method.



Enhanced photocatalytic activity of titanium dioxide photonic crystals modified with photodeposited platinum nanoparticles.

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Titanium dioxide (TiO₂) photonic crystals (PCs) with a spatial periodic distribution of refractive index in homogenous dielectric matrices have attracted remarkable attentions in the last two decades due to the appearance of photonic band gap (PBG). Because the propagation of certain wavelengths of incident light can be inhibited in the PCs structures, they have been explored for universal applications such as optical communications, novel biological and chemical sensors and in photocatalysis. This kind of structure is characterized by an open, interconnected macroporous ordered structure and nanosized wall thickness. Upon illumination, photons near PBG edges propagate with strongly reduced group velocity leading to the occurrence of slow photon effect (SPE). If this phenomenon of SPE overlaps the edge of semiconductor absorption band, the photocatalytic properties of TiO₂-PC can be strongly enhanced. In addition to the abovementioned structural method, another effective way of photocatalytic enhancement is modification of TiO₂-PCs by platinum nanoparticles (PtNPs). They have a broad absorption band of the electromagnetic spectrum and therefore reveal unique properties also used in photocatalysis. Besides, the electron-hole recombination rate can be reduced by the participation of PtNPs in mechanisms of electron transfer and trapping. Photocatalytic redox processes under UV illumination can be therefore improved [1,2].

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PtNPs and the structural properties of TiO_2 -PC on the photocatalytic properties of obtained PtNPs/TiO_2-PCs.

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Tunable wettability of thin polymer films on microstructured silicon surfaces

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Hybrid nanomaterials and nanostructured surfaces are very promising for novel applications and the development of smart materials. In particular, micro/nanostructured silicon fabricated by laser processing, known as black silicon, has attracted great interest due to the wide range of applications in optoelectronic devices, microelectronics, microfluidics, and biomedical devices. Wettability is an important property of solid surfaces that is governed by the chemical composition and roughness of the surface. Several applications require control of the surface wettability of solid materials and reversible control of the wettability can be achieved by stimuli-responsive surfaces. Poly(N-isopropylacrylamide) (PNIPAM) is a thermo-responsive polymer, which switches between hydrophilicity and hydrophobicity at its lower critical solution temperature (LCST). In this work, microstructured silicon surfaces were fabricated by a pulsed Nd:YAG laser system with pulse duration 5 ns, average energy 70 mJ per pulse, and 532 nm wavelength at a repetition rate of 10 Hz. The silicon wafers were placed in a vacuum chamber with 600 mbar of SF₆ environment and irradiated by approximately 1000 pulses. The microstructured surfaces of silicon have large specific surface area with dualscale roughness in the micro- and nanoscale and present a superhydrophilic behavior. To tune this behavior, coatings of different polymers have been applied on the microstructured silicon surfaces. The surfaces were coated with thin polymer films of Polystyrene (PS), polystyrene-b-poly(isopropylacrylamide) (PS-PINIPAM) and a solution of PS/PS-PNIPAM by drop casting. The film properties were characterized by SEM, profilometry, and contact angle measurements. Contact angle measurements show a great difference on the wetting behavior of the microstructured silicon surface before and after film coating.

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The role of Ostwald ripening and coalescence in photocatalytic growth of silver nanoparticles on titanium dioxide coatings

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The growth of silver nanoparticles (AgNPs) on titanium dioxide (TiO_2) surface can be conducted by a photocatalytic reduction of Ag+ ions, using electrons trapped on the UV-excited TiO_2 surface as reducing agents, leading to the formation of AgNPs/TiO₂ nanocomposites. However, the comprehension of AgNPs growth mechanisms providing to various sizes and shapes of nanoparticles is challenging scientific task.

 TiO_2 coatings were prepared on silicon wafers by the sol-gel dip-coating method according to the procedure reported in our previuous studies [1]. Photocatalytic growth of AgNPs on the TiO_2 coatings was performed in solutions containing Ag+ ions having different concentrations under various UV illumination times.

The goal of this work was monitoring the nucleation and growth of AgNPs on TiO_2 in short and long illumination intervals supposing that the the growth process is multidirectional. The important factors governing the initial nucleation of AgNPs are accessibility of Ag+ ions for reduction and their local concentration near the TiO_2 surface [2]. Besides, in further stages of AgNPs formation, the growth mechanism is determined by the size of clusters and the distance beetween them.

It was found, that Ostwald ripening mechanism is typical for low concentrations of Ag+ ions where newly generated unstable clusters dissolve, which can be observed as a lowering of a number of small-size AgNPs during illumination. Released in this process Ag+ ions are subsequently used to enlarge bigger AgNPs. On the other hand, if nanoparticles grow near together and concentration of Ag+ is sufficiently high, they subject to coalescence providing larger nanostructures having elongated shapes.

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Effect of the preferred orientation on the electrochromic properties of tungsten oxide coatings grown by a LPCVD system

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In recent years, chromic coatings have attracted the scientific interest due to the significant technological applications involved, such as smart windows, self-dimming rear mirror, electrochromic display, sensor etc. Especially, electrochromic (EC) coatings, such as those based on tungsten trioxide (WO₃), have the ability to change their transmissive properties in the presence of a small electric voltage, a property essential for the construction of energy efficient windows. Many techniques have been employed for their development such as sputtering, pulsed laser deposition, sol-gel, spray pyrolysis and chemical vapor deposition (CVD).

In this work, a low pressure chemical vapour deposition (LPCVD) system has been used for the development of WO₃ layers on FTO substrates, using tungsten hexacarbonyl (W(CO)₆) as precursor. X-ray Diffraction (XRD), Raman Spectroscopy, Field-Emission Scanning Electron Microscopy (FE-SEM) and cyclic voltammetry were employed for the characterization of these samples, so that the effect of the deposition parameters on the basics characteristics and the electrochromic behaviour can be studied. As found out, the change of the preferred orientation of monoclinic WO₃ can significantly affect the Li+ ions intercalation–deintercalation process and the stability of the layers.

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Dynamic wettability control through stretching of bilayer polymer films

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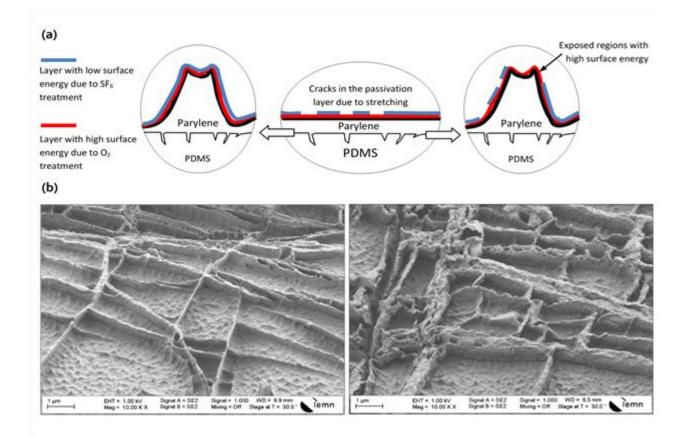
We produce PDMS films which are CVD coated with a thin parylene layer (~ 3 μ m). We show how these surfaces can be engineered by combining stretching induced deformation of the top layer followed by a plasma treatment to control their wettability and render them superhydrophobic. Indeed, due to the difference in the material properties, stretching will induce bilayer instabilities manifested by wrinkling of the parylene layer.[1] The chosen treatment combines oxygen (O2) plasma pretreatment with a sulfur hexafluoride (SF6) plasma treatment to achieve superhydrophobicity without altering the PDMS bulk properties (e.g. mechanical properties). This treatment was proposed by Bi [2] and creates nanostructures on top of the micron scale wrinkles produced by the stretching step.

Using dynamic contact angle measurements, we show that the thus obtained surfaces exhibit super hydrophobicity with an Advancing Contact Angle of 167°, a Receding Contact Angle of 138° and low hysteresis. We study the effect of a second mechanical stretching applied to the surfaces. Interestingly, this step significantly increase adhesion with RCA under 10°. Through a combined AFM, SEM and XPS study we attribute this switching behavior to microstructure alterations of the surface leading to the exposure of oxygen rich defects (C=O) and (O2C=O) produced by the O2 plasma step.

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Silver/hydroxyapatite hybrid coatings on Ti-6Al-4V surfaces by sol-gel method

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It is well known that Ti–6Al–4V alloys are widely used in the human body [1]. Release ions from implanted materials after the implantation may provoke not only to mechanical failure of implant but also to local pain and swelling in the body. Thus, the implants need specific surface modification in order to minimize the adverse effects. The sol–gel is a low-cost process to coating of implants and it is relatively easy to control the deposition parameters [2, 3]. In this study, silver-doped hydroxyapatite (HA) hybrid films were successfully coated on the Ti–6Al–4V surfaces. Calcium nitratetetrahydrate (Ca(NO3)2·4H2O) and di-ammonium hydrogen phosphate ((NH4)2HPO4) and silver nitrate (AgNO3) were used as precursors to obtain Ag doped HA structure. The corrosion susceptibilities of samples have been analyzed in Ringer's solution under in-vitro conditions by potentiodynamic polarization scanning (PDS) technique.

The surface of the coating was homogeneous and dense in appearance, and some micro cracks were observed as shown in Fig. 1a. The EDS result of the coating layer justifies the presence of Ca, P and Ag phases in the coating layer. Also, main elements of the substrate were identified in the analysis due to the nano scale coating layer (Fig. 1a). The corrosion tests showed that coated sample has nobler corrosion potential and lower passivation current density than uncoated sample. In other words, the corrosion resistance of Ti–6Al– 4V after coating with Ag doped HA increases. The study has been financially supported by TUBITAK (The Scientific and Technological Research Council of Turkey) under project number 114M437.

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FIGURE 1. (a) SEM image and the EDS result of region A on the image and (b) PDS curves of coated and uncoated samples

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