Surface Engineering - Applied Research and Industrial Applications
Room Grand Hall - Session GP-ThP

Surface Engineering - Applied Research and Industrial Applications (Symposium G) Poster Session

GP-ThP-1 Effect of Plasma Nitriding and Modulation Structure on the Adhesion and Corrosion Resistance of CrN/Cr_2O_3 Coating, *C Huang, F Yang, Y Tsai,* ***Chi-Lung Chang,*** Ming Chi University of Technology, Taiwan

The increasing demand for high performance coatings has led to the production of coatings which are becoming more sophisticated in terms of their engineered microstructure and properties. An extensive interest of high power impulse magnetron sputtering (HiPIMS) as a novel PVD technology in academia and industry owing to their dense and smooth coatings properties when compared to traditional PVD technologies. However, the adhesion strength still not insufficient, therefore a plasma nitriding process is introduced before the HiPIMS process. On the other hand, the carbon steel has a very poor corrosion resistance in an atmospheric environment, which one is needed to solve by various surface treatment technologies in environmentally friendly.

In this study, a continuous process combines both vacuum plasma nitriding and HiPIMS CrN/Cr_2O_3 multilayered coating technology is used. Before CrN/Cr_2O_3 multilayer coating, the vacuum plasma nitriding is carried out at different nitrogen flow rate for 1~5 h with a bias output of HiPIMS power between 0.5 and 2.5 kW, which is at a fixed temperature of 400℃. After that CrN/Cr_2O_3 multilayer films with different bi-layer thickness were deposited onto carbon steel and silicon p-type (100) wafer substrates at 400℃ using Cr targets with a nitrogen (or oxygen)/argon flow ratio of 0.2, by high power impulse magnetron sputtering technique. The bi-layer thickness was varied by time-controlled with a switch in between the nitrogen and oxygen to obtain different nanoscale multilayered period thickness. The modulation structure characteristics of the CrN/Cr_2O_3 multilayer films between 100 nm to 5 nm were systematically investigated. The results have demonstrated that both the nitriding effect and the modulation period (Λ) was strongly to affect adhesion strength between carbon steel and the coating. In addition, the corrosion resistance of carbon steel is strongly improved by the CrN/Cr_2O_3 multilayer film.

GP-ThP-2 Study on SiN and SiCN Film Production using PE-ALD Process with High-density Multi-ICP Source at Low Temperature, ***Hohyun Song,*** *H Chang,* Korea Advanced Institute of Science and Technology, Republic of Korea

SiN and SiCN film production using plasma-enhanced atomic layer deposition (PE-ALD) is investigated in this study. A developed high-power and high-density multiple inductively coupled plasma (multi-ICP) source is used for a low temperature PE-ALD process. High plasma density and good uniformity are obtained by high power N2 plasma discharge. Silicon nitride films are deposited on a 300-mm wafer using the PE-ALD method at low temperature. To analyze the quality of the SiN and SiCN films, the wet etch rate, refractive index, and growth rate of the thin films are measured. Experiments are performed by changing the applied power and the process temperature (300–500 °C).

GP-ThP-3 PEO Coatings for Adhesive Bonded Aluminium Structures, ***Dominic Shore,*** *A Rogov, A Matthews, A Yerokhin,* The University of Manchester, UK

Plasma Electrolytic Oxidation (PEO) has received much attention in research for the production of oxide coatings with excellent tribological performance and corrosion resistance. This study looks into a further application of PEO as an alternative to conventional anodizing techniques for the preparation of aluminium for adhesive bonding. Conventional anodizing processes have been applied to adhesive bonded aluminium components to promote increased bond durability tracing back to the first half of the 20th century. However, Conventional anodizing procedures generally use strongly acidic electrolytes which have a substantial environmental impact. Anodizing procedures are generally multi-stage processes which can be resource intensive and time consuming. PEO offers an alternative route for the production of well adhered oxide coatings where weak alkaline electrolytes can be utilised in place of the highly acidic electrolytes associated with conventional anodizing processes. PEO has the further potential to reduce the number of additional treatments prior to and after the anodizing stage offering scope for resource and time saving. To provide bond durability comparable to that offered by the conventional anodizing techniques currently used, PEO coatings must promote sufficient mechanical interlocking and physical/chemical affinity between the surface and adhesive in addition to good corrosion resistance.

Work has been carried out on the optimisation of PEO coatings on aluminium alloys with regards to the application of adhesion bonding. Through the use of varying processing parameters and different electrolyte compositions, coatings with properties designed to promote enhanced bond strength are being developed. Topological features of the developed PEO coatings, including surface porosity which is important for the promotion of mechanical interlocking in the adhesive bond have been analysed. Chemical analysis of the surface composition of the oxide and the adsorption groups formed at the oxide/adhesive interface has been carried out to deduce the chemical nature of the adhesive bonding occurring on PEO coatings formed under different conditions. As a result, a scientific description as to how the surface features of PEO coatings contribute to adhesive bond durability is being summated, which will lead to further optimisation of PEO coatings for this application.

GP-ThP-5 Hydrogen Barrier Coatings Deposited by Magnetron Sputtering: A Study of Different Oxide Materials and Their Microstructure on the Hydrogen Permeability Properties, ***Sofia Gimeno,*** Fersa Bearings, Spain; *J Garcia,* Universidad Publica de Navarra, Spain; *I Quintana, L Mendizabal, C Zubizarreta,* Physic of Surfaces and Materials Unit, IK4 – TEKNIKER, Spain

White Etching Cracks (WEC) is the main cause of premature failure on bearings used in wind turbine sector. This premature failure mode is still under debate due to the lack of a clear theory about how it is produced and which are the factors involved in the WEC damage. One of the most important theories is based on Hydrogen embrittlement, due to hydrogen diffusion in steel under rolling contact fatigue (RCF) [1].

The development of novel surfaces which -are able to avoid or delay the diffusion of Hydrogen would be considered as a key point to solve the WEC problem. In this study, films based on WO_3, SiO_2 & Al_2O_3 oxides grown by pulsed dc magnetron sputtering have been designed to act as barrier to Hydrogen permeation, delaying or avoiding the diffusion of this in 100Cr6 steel. The objective of this study is to analyze the behaviour of the different oxide materials, as well as the influence of the morphology of coatings on the permeation of Hydrogen.

WO_3, SiO_2 & Al2O3 thin films were deposited on 100Cr6, Niquel and silicon substrates. The adhesion, microstructure and crystal structure of oxide films were investigated and correlated with barrier properties against hydrogen permeation.

Hydrogen permeability of different films was studied using differential pressure method described in ISO 15105-1:2007. Nickel was used as substrate due to the known and high permeability of this material to hydrogen.

GP-ThP-6 Process for Obtaining TiO_2/SiO_2 Systems using Magnetron Sputtering RF from Ceramic Targets: Studies on their Anti-Reflective Response, ***Dario Zambrano,*** *R Villarroel, R Espinoza,* Universidad de Chile, Chile

Nanostructured systems of SiO_2 and TiO_2 produced by magnetron sputtering technique have allowed the development of optical filters that maximize the conversion of light in different devices and applications [1–4]. The anti-reflective coatings (ARCs) of SiO_2 / TiO_2 systems allow obtaining a greater contrast and better image quality in applications such as TV screens, cell phones, projectors, among others. In addition, they improve the quantum efficiency in photovoltaic panels, increasing the power generation capacity [5].

In this research nanostructured films composed by multilayer stacking of SiO_2 and TiO_2 were studied, considering the working pressure and the partial pressure of O_2 as variables for the modification of the microstructure of each monolayer and the influence on the anti-reflective response of the multilayer SiO_2/TiO_2 system.

The nanostructured layers of TiO_2 and SiO_2 were synthesized by magnetron sputtering RF, using ceramic targets of TiO_2 and SiO_2. The modulation of the optical performance of the ARCs were measured using spectrophotometry and ellipsometry. On the other hand, the microstructure of the ARC's was obtained using FE-SEM, AFM, XRD, and FTIR.

REFERENCES

[1] M. Mazur, D. Wojcieszak, D. Kaczmarek, J. Domaradzki, S. Song, D. Gibson, F. Placido, P. Mazur, M. Kalisz, A. Poniedzialek, Functional photocatalytically active and scratch resistant antireflective coating based

on TiO2 and SiO2, Appl. Surf. Sci. 380 (2016) 165–171. doi:10.1016/j.apsusc.2016.01.226.

[2] M.A. Green, S.P. Bremner, Energy conversion approaches and materials for high-efficiency photovoltaics, Nat. Mater. 16 (2016) 23–34. doi:10.1038/nmat4676.

[3] S. Chu, Y. Cui, N. Liu, The path towards sustainable energy, Nat Mater. 16 (2017) 16–22. doi:10.1038/nmat4834.

[4] H.K. Raut, V.A. Ganesh, A.S. Nair, S. Ramakrishna, Anti-reflective coatings: A critical, in-depth review, Energy Environ. Sci. 4 (2011) 3779–3804. doi:10.1039/c1ee01297e.

[5] W.-J. Ho, J.-C. Lin, J.-J. Liu, W.-B. Bai, H.-P. Shiao, Electrical and Optical Characterization of Sputtered Silicon Dioxide, Indium Tin Oxide, and Silicon Dioxide/Indium Tin Oxide Antireflection Coating on Single-Junction GaAs Solar Cells, Materials (Basel). 10 (2017) 700-709. doi:10.3390/ma10070700.

GP-ThP-7 Microstructure Evolution of Overlay Welded Duplex Stainless Steel Joints, ***Paola Andressa Luchtenberg***, *R Torres, P Soares, P Campos,* Pontificia Universidade Católica do Paraná, Brazil

Duplex stainless steels have a good mechanical properties, wear and corrosion resistance and fatigue strength. This material have a higher price compared to austenitic steel, in some cases to lower the cost, companies use low carbon steel or low alloy coated with duplex stainless steel, than t his material can be employed through overlay welded coatings on mild steel components and equipment. In this work, the aim was to evaluate the overlay properties obtained through deposition of ER 2209 duplex stainless steel alloy on a mild steel ASTM A 516 Gr 60. The deposition was performed through GMAW welding process. The coatings were deposited using four heat input levels, which showed influence in phase balance in weld metal austenite/ferrite morphology. Microstructural characterization by optical microscopy and quantification of ferrite by ferritscope, showed that heat input, cooling rate and grain size influences on the formation of secondary phases, because this type of phase precipitate from ferrite or primary austenite. The reheating change the microstructure influencing positively or negatively in corrosion behavior of DSSs . The secondary austenite that precipitates due to the welding process in DSSs are grain boundary austenite (GBA), Widmanstätten austenite (WA), partially transformed austenite (PTA) and intragranular austenite (IGA) and each of them has their characteristic precipitation, like temperature, grain size, cooling rate. However, the corrosion resistance can be avoided by means of appropriate welding procedures and the phase control such as ferrite/austenite balance.

GP-ThP-11 Ion Beam Assisted Deposition of DLC for Sheet Metal Forming Tools, ***Lars Pleth Nielsen***, *K Almtoft, C Jeppesen, C Mathiasen, P Pedersen,* Danish Technological Institute, Denmark

A deposition process combining high-energy ion implantation of chromium ions into a condensing Fomblin-oil has been developed and charaterized. The composition of the IBAD-DLC film was measured by RBS and contain a mixture of elements (C, H, O, F and Si) from the condensing oil (Penta Phenyl Trimethyl Trisiloxane) besides the impinging high-energy Cr ions. The friction properties have been evaluated by pin-on-disk to be below 0.05. The hardness of the IBAD-DLC was measured to 10.7 GPa. Hardness, scratch tests and friction properties will be compared with PVD-based DLCs. Examples of different industrial applications of the IBAD-DLC coating in connection with sheet metal forming in the food sector will be presented.

GP-ThP-13 Effect of Interaction between Microbial Fluid and Electrode on Performance, ***Yu-Chen Liu***, *Y Yang,* National Taipei University of Technology, Taiwan

For the electrode reaction controlled by electron transfer, the electrode material has a great influence on the reaction rate. In terms of the material's conductivity, the reaction rate of the MFC will be improved by the high electron conductivity of the anode and the cathode. About the electrode area, as the electrode reaction is carried out at the electrode-solution interface, the reaction rate will be proportional to the electrode area, so a high surface area of the electrode development is extremely important. In addition, the metal surface modification, in order to develop a corrosion-resistant and highly collectible metal electrode modification is also an important research topic.

Electrodes of the Microbial fuel cell whether used in sewage treatment plants or marine rivers or lakes, the purpose of the production of electricity will be associated with the behavior of the fluid. Fluid behavior for the system efficiency and electrode usefulness would have a great impact. In this study, we focused on the interaction of microfluidic fluids with multi-morphological metal electrodes in the system. Through the observation of fluid mechanics and numerical simulation, we hope to understand the interaction between fluid and electrode, understand the interaction between bacteria and electrodes.

GP-ThP-15 Design of Low-Pressure Chemical Vapor Deposition Reactors Using Vertical Cavity Surface Emitting Lasers, ***Seungho Park***, *Y Noh, Y Kim,* Hongik University, Seoul, Republic of Korea; *B Kim, H Kim,* Viatron Technologies, Republic of Korea

VCSEL modules were investigated to design a LPCVD reactor for promising industrial applications due to advantages of excellent irradiation uniformity, rapid power controllability, and especially extended spatial scalability. Each VCSEL cell radiates perpendicularly from the wafer surface, differently from the conventional EEL.

A laser beam emitted from the VCSEL diffuses slightly as its power load increases, which is critical to irradiation uniformity and spatial scalability. The divergence angles best-fitting the radiative fluxes that were measured experimentally increased monotonically with a very small slope.

Through the experimental investigation on the temperature distribution on the silicon wafer irradiated by the high-power VCSEL beams, the optimal structure for the VCSEL heating system that ensured the uniform irradiation was obtained for wafers of 300 mm in diameter.

Based on the single-step chemical reaction mechanism for the deposition of polycrystalline silicon with silane gas species, the factors for the Arrhenius equation depicting the deposition process were obtained by the comparisons of numerical simulations and the available experimental results.

A simple reactor structure was used to investigate the variation in deposition rates on the wafer during the VCSEL LPCVD process. On the wafer surface the boundary condition of the energy conservation equation for the numerical simulation was given by the heat flux distribution calculated from the radiative irradiation from the VCSEL arrays, rather than the temperature distribution that has been widely used in simulations.

Comparisons of the deposition thicknesses calculated from the simulations under static and practical rotative conditions indicated that the wafer exclusion region increased considerably in the rotative condition due to the decrease in deposition rates in the edge region of the wafer. In order to minimize the wafer exclusion region, the VCSEL emitters which exerted influence on the wafer edge region were controlled to increase the emissive power slightly. As a result, the wafer exclusion zone was reduced considerably, without the help of additional structures to stabilize the gas flow and to reduce the energy loss in the wafer edge region commonly applied in practice.

GP-ThP-16 Optical, Mechanical and Anti-corrosive Property Investigation of Tantalum Oxynitride Thin Films for Hard Coating Applications, ***Jignesh Hirpara***, *R Chandra,* Indian Institute of Technology Roorkee, India

Mechanically hard coating with anti-corrosive behaviour is high in demand for the coating of metal surface in the medicine and industries. Tantalum oxynitride film deposited through reactive magnetron sputtering was investigated for its mechanical and anti-corrosive behaviour. XRD study revealed monoclinic phase with average crystalline size 270Å. Surface topographic was studied through AFM. Micrographs showed high uniformity with low roughness (<100 nm). Highly magnified FE-SEM imaging cleared the formation of nanoscale granules. Optical spectroscopic results demonstrated good transparency of the film (around 85%). Potentiodynamic polarization test and electrochemical impedance spectroscopy were performed with 0.1 M NaCl electrolyte, which demonstrated the high anticorrosive behaviour of the film. The measurement of mechanical hardness of the film was done using the Nanoindentation technique, which has presented the high value of reduced modulus (E_r) 132.98 GPa and hardness (H) 7.30 GPa. Hence, this material with high mechanical strength, transparency and anti-corrosive behaviour is quite suitable for the protective coating of the metal surface.

GP-ThP-17 Synthesis and Properties of Two-dimensional Zirconium Phosphate/Polyimide Nanocomposites as Anticorrosion Coatings, *G Lai,* National Chin-Yi University of Technology, Taiwan; *I Tseng,* Feng Chia University, Taiwan; *T Huang, P Tsai,* ***Mei-Hui Tsai***, National Chin-Yi University of Technology, Taiwan

In this study, novel zirconium phosphate/polyimide (ZrP/PI) nanocomposite was developed as an anti-corrosion coating material. The two-dimensional ZrP was prepared by the thermal reflux method and modified by the surfactant Jeffamine M1000. Various contents (0.5 ~ 5 wt.%) of ZrP were then homogeneously blended within PI matrix to obtain ZrP/PI coatings

with enhanced anticorrosion effect on cold-rolled steel electrodes. The characteristics of two-dimensional ZrP and nanocomposites were confirmed by Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscope (SEM) analyses. A series of electrochemical measurements such as corrosion potential (E_{corr}), polarization resistance (R_p), corrosion current (I_{corr}) and electrochemical impedance spectroscopy (EIS) were performed to evaluate the performance of the anticorrosion coatings.

GP-ThP-18 Improvement of the Corrosion Resistance in the ASTM F75 Alloy by Ball Burnishing, ***Eric Noe Hernandez-Rodrigues***, *D SilviaAlvarez, A Marquez Herrera, A Saldana Rovles, J Moreno Palmerin,* University of Guanajuato, Mexico

The ball burnishing is a process in which the surface of a material is compressed causing plastic deformation, and therefore changing the mechanical and chemical surficial properties. ASTM F75 alloy is used in the field of health for fabrication of orthopedic implants. In this application, the alloy is exposed to physiological fluids which are corrosive media. Therefore, improving the corrosion resistance is desirable in order to extend the lifetime of the implants. In this work, we propose the ball burnishing for improving the corrosion resistance of the ASTM F75 alloy. We implemented a design of experiments (DoE) methodology to minimize the corrosion. Two factors were analyzed in the DoE: burnishing force(F_B) and number of tool passes (N_P). The response variable was the corrosion current (I_{corr}). Tests were conducted in cylindrical ASTM F75 alloy samples (ø 25.4 mm x L 12 mm). The ball burnishing process was carried out in a conventional lathe and was only applied to one flat face of the samples. Burnishing force was varied from 150 N to 450 N, while tool passes was varied from 2 to 6. After burnishing, corrosion tests were performed by means of electrochemical polarization. Hank´s balanced salt solution was employed as the corrosive media. Tafel plots were used for determining I_{corr}. Under studied experimental conditions, $F_B$wasthe more significative factor in determining $I_{corr.}$ It was found that as N_P is set constant (at 2, 4 or 6 passes) and F_B is varied from 150 to 450 N, I_{corr} decreases (and therefore corrosion) when ball burnishing is performed with lower F_B values. On the other hand, when F_B is set constant (at 150, 300 and 450 N) little influence was found when N_P changes. A minimum I_{corr} value was found for 150 N and 4 tool passes. Under this condition I_{corr} was 4.9 nA in contrast to I_{corr} in unburnished samples which was 65.2 nA. These values showed a reduction of I_{corr} up to 92.5% in burnished samples, and therefore, a great improving in corrosion resistance.

GP-ThP-19 Surface Modification of Sputter Deposited γ-WO3 Thin Film for Scaled Electrochromic Behaviour, *R Chandra,* ***Gaurav Malik***, *S Mourya, J Jaiswal,* IIC, IIT Roorkee, India*; J Hirpara,* Indian Institute of Technology Roorkee, India

Here, we have reported the electrochromic properties of the highly ordered γ-WO3 nanoporous thin film grown directly on the indium tin oxide glass (ITO) coated glass substrate using DC magnetron sputtering in a reactive environment (Ar:O_2) at room temperature. To achieve the nanoporous-nanocrystalline behaviour of the active material, a thermal treatment (250°) was given to the active material, which modified the compact film surface into nanospheres. This surface modification is responsible to alter the physical, optical and electrochromic properties of the active material. The physical properties of the active material were characterized in detail using X-ray diffraction, scanning electron microscopy, atomic force microscopy, and energy-dispersive X-ray analysis. The optical and electrochromic behaviour of the active electrode material was analyzed using UV-Vis spectroscopy and cyclic voltammetry. The proposed device revealed large optical modulation, high reversible redox behaviour and good cyclic stability at least up to 1000 cycles. This electrochemically active architecture allows one to fabricate the device for energy harvesting applications at an elevated temperature. Our work indorses human comfort with financial benefits and plays a crucial role in "green nanotechnology".

GP-ThP-21 Nanotexturization and Passivation of Single Crystalline Silicon Surface for Passivated Emitter and Rear Contact Solar Cells, *C Hsu,* Xiamen University of Technology, China*; S Liu,* Da-Yeh University, Taiwan , Taiwan*;* ***Wan-Yu Wu***, Da-Yeh University, Taiwan*; S Lien,* Xiamen University of Technology, China

Passivated emitter and rear contact (PERC) solar cells are currently the most promising product in solar cell market. One way to improve the cell efficiency is to reduce the reflectance at incident surface while maintaining high passivation quality. In this study, nanostructured black silicon has been prepared by using metal catalysed chemical etching with a solution mixture of silver nitrate ($AgNO_3$) and hydrogen fluoride. The $AgNO_3$ concentration is varied from 0.015 to 0.075 M. An aluminum oxide and silicon nitride stack is deposited for passivation and antireflection. The experimental results show that the $AgNO_3$ concentration of 0.06 M produces the most prominent nanostructures, but the silicon nitride cannot well-deposited on the surface. The silicon wafer etched at the $AgNO_3$ concentration of 0.03 M exhibits the lowest average reflectance of 1.6% while not compromising on passivation quality. Solar cell simulation reveals that PERC cells with the optimal black silicon nanostructure can have short-circuit current two percent higher than that of traditional PERC cells, and reach a conversion efficiency of 22.04%.

Keywords: Nanostructure, Black silicon, Single crystalline, PERC, Passivation

GP-ThP-24 Optical Performances of Antireflective Moth-Eye Structures under Thermal and Humid Stress – Application to Outdoor Lighting LEDs., *C Ducros,* ***Agathe Brodu***, *G Lorin, F Emieux, A Pereira,* Univ. Grenoble Alpes, CEA, France

Antireflective moth-eye structures were studied in order to increase optical properties of protection windows for outdoor LED lighting. This antireflective structure was compared to "standard" antireflective coatings elaborated by electron-beam evaporation: MgF_2 single layer and broadband SiO_2/ZrO_2 multilayers. Two types of substrates, glass and polycarbonate (PC) have been treated in order to determine the influence of substrates on failure mechanisms generated during ageing tests.

In a first time, effective refractive index and surface morphology were respectively determined by ellipsometry and scanning electron microscopy (SEM). Reactive ion etching process was then optimised on both substrates materials for maximising transmission in normal and angular incidence in the wavelength range of white LEDs (400-750nm). Best results with moth-eye structure were achieved on glass substrate treated on both sides: we measured an increase of 7% of white LED light transmission comparatively to untreated substrate.

In a second time, three standard ageing tests, normative for outdoor applications, were then enforced in climatic chamber on different antireflective treatments in order to estimate their optical properties degradation. First one is an extreme heat test at 130°C with 50% of relative humidity (RH) during 1 hours. Humid heat test consists in testing coated samples during 4 days at 70°C and 95% RH. Third test is a high humidity heat test at 40°C, 98% RH with condensation during 2 days. Surface energy measurement, ellipsometry and SEM were performed on aged surfaces in order to highlight the main degradation mechanisms. The influence of environmental stress on optical properties were then described for all antireflective surfaces. Three main observations can be pointed out from this study: First, degradation of MgF_2 coating under high humidity conditions was mainly due to its high hydrophilic property: a lowering of 3% of light transmission is measured on PC after high humidity heat test. The second main observation is surface cracking of SiO_2/ZrO_2 multilayer antireflective coating on PC leading to losses of 2% in light transmission under extreme heat test. Finally, only moth-eye structures gives high environmental stability as they maintain their white LED light transmission after ageing tests.

Author Index

Bold page numbers indicate presenter