Surface Engineering - Applied Research and Industrial Applications
Room Sunrise - Session G4
Pre-/Post-Treatment and Duplex Technology

Moderators: Hiroshi Tamagaki, NIRO (The New Industry Research Organization), Wan-Yu Wu, Da-Yeh University, Chris Stoessel, Eastman Chemical Company, Inc., USA

8:40am **G4-3 Nitriding and DLC Coating of Aluminum Alloy Using High Current Pressure-Gradient-Type Plasma Source**, ***Akio Nishimoto,*** Kansai University, Japan*; E Furuya, K Kousaka,* Chugai Ro Co., Ltd., Japan

The low hardness and poor tribological performance of aluminum alloys restrict their wide applications in automotive fields. However, protective hard coatings deposited onto aluminum alloys are effective for overcoming their poor wear properties. In this study, a diamond-like carbon (DLC) film followed by a nitriding layer was deposited onto an aluminum alloy via a pressure-gradient-type plasma source using nitrogen and acetylene gases. This pressure-gradient-type plasma source is operated at a low discharge voltage of 60-100 V and a high current of 60-130 A. An aluminum alloy EN AW-5052 sample was plasma nitrided for 4 h at 520 ºC under 0.09-1.1 Pa. DLC was then coated with an acetylene gas after plasma nitriding using the same apparatus. The Vickers microhardness of the surface nitrided at 0.51 Pa reached approximately 340 HV from 125 HV. In addition, glow discharge optical emission spectrometry (GD-OES) revealed that nitrogen was concentrated at the surface region. After the DLC coating, the sample was reddish brown in color. GD-OES results demonstrated that a carbon-rich region formed in the top surface region (DLC film), followed by the formation of a nitrogen-rich region (nitriding layer). Nanoindentation test showed that the hardness of the top surface (DLC film) was 10.3 GPa. The DLC coating also exhibited good tribological performance in a ball-on-disk wear test, with friction coefficients of approximately 0.17, which was characterized as low value of DLC. In addition, aluminum nitride (AlN) interlayer was deposited on the nitriding layer by ion plating method in order to enhance adhesion between the DLC film and the substrate. Rockwell indentation indicated good adhesion. Hardness, roughness, and structure of the DLC film deposited on the AlN interlayer was investigated.

9:00am **G4-4 Towards Hard yet Tough Ceramic Coatings**, ***Sam Zhang,*** Nanyang Technical University, Singapore

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

9:20am **G4-5 Flash Lamp Annealing (FLA) for Post-deposition Treatment at High Throughput**, ***Thoralf Gebel,*** University of Applied Sciences Mittweida, Germany*; M Neubert,* ROVAK GmbH, Germany*; W Skorupa,* Helmholtz Zentrum Dresden-Rossendorf, Germany **INVITED**

Today's deposition processes for large area / large volume applications are strongly influenced by cost saving issues, by environmental regulations and by aspects of improved throughput. Especially in the case of thermal processing steps (e.g. for transparent conductive oxide (TCO) layers for displays on flexible glass) this becomes a very important aspect: substrates which need to be heated during or after the deposition process require sophisticated handling systems, and the moving substrates heated to elevated temperatures require cooling zones which may limit the throughput. A promising approach to overcome these problems are novel ultra-short time thermal processes with treatment times in the (sub)millisecond range. By using pulsed photonic treatment (e.g. by means of laser or flash lamp annealing) only the surface layers of the substrate materials are heated and therewith the thermal budget is strongly reduced. The work presented here focuses on investigations of such new thermal processes by using simulations and providing energy & cost saving models. Aspects of process design and overlapping issues and their influence to homogeneity will be discussed.

10:00am **G4-7 Evaluating the Effect of Titanium-Based PVD Metallic Thin Films on Nitrogen Diffusion Efficiency in Duplex Plasma Diffusion/Coating Systems**, ***Gorkem Yumusak,*** *A Leyland,* University of Sheffield, UK*; A Matthews,* University of Manchester, UK

Titanium is a very popular engineering metal due to its outstanding properties, such as low density and high specific strength. However, the wear resistance of titanium is very poor in many industrial environments. Wear resistant hard coatings can be used to increase the service lifetime of manufactured products but the effectiveness of these coatings on titanium is sometimes weak due to poor load bearing capacity of the substrate. Therefore, titanium alloys need certain pre-treatments before the Physical Vapour Deposition (PVD) of ceramic hard coatings.

In this work, triode plasma nitriding (TPN) has been applied in order to increase the load bearing capacity of titanium alloys. It is known that the adhesion between titanium alloy substrates and PVD hard coatings can be increased significantly after substrate diffusion treatment [1]. TPN treatments were used in this work because the diffusion of the nitrogen can be achieved more easily at lower temperatures and shorter times, without the need for hydrogen in the gas mixture.

The efficiency and effectiveness of triode-plasma diffusion treatment can be increased by applying a thin PVD metallic layer on titanium alloy substrates, before plasma nitriding [2, 3]. In this context, different compositions of β-titanium coating (stabilized by addition of Nb) were produced on α+β Ti-6Al-4V and β Ti-15Mo substrate materials; the formation of β phase in Ti-Nb coatings before nitriding (and of nitride phases after TPN treatment at 500-700^{o}C) was analysed.

[1] G. Cassar, S. Banfield, J.C. Avelar-Batista Wilson, J. Housden, A. Matthews, A. Leyland, Wear 274 (2012) 377.

[2] G. Cassar, A. Matthews, A. Leyland, Surf Coat Technol 212 (2012) 20.

[3] B. Attard, A. Matthews, A. Leyland, G. Cassar, Surf Coat Technol 257 (2014) 154.

10:20am **G4-8 Properties of Surface Passivation at Si/Al_2O_3 Interface Annealed in Different Gas Ambient**, *C Yang,* National Chung-Hsing University, Taiwan*;* ***Chun-Wei Huang****, C Hsu,* Da-Yeh University, Taiwan*; C Kung,* National Chung-Hsing University, Taiwan*; S Lien,* Da-Yeh University, Taiwan*; W Zhu, X Meng, X Zhang,* Xiamen University of Technology, China

Efficient surface passivation is crucial in most electronic devices, especially in solar cells, where the generated electron-hole pairs need to be collected by contacts before recombining at the surfaces. Aluminum oxide (Al_2O_3) film synthesized by atomic layer deposition system (ALD) offers a high level of surface passivation for p-type passivation emitter and rear cells (PERC). The high passivation quality of Al_2O_3 is related to high negative charge combined with low interface density. The post-annealing process after the deposition of Al_2O_3 films can effectively activate the passivation. The objective of this work is to optimize the passivation performance by hiring different annealing temperature and atmosphere containing air, nitrogen, oxygen, and forming gas (95 % N_2+5 % H_2) inside the furnace.

The silicon wafers used in this study is mono-crystalline p-type commercial-grade CZ silicon wafers with a thickness of 200±20 µm and a resistivity of 0.5 to 5 Ω-cm. The original lifetime of bare wafer is under 5 µs. Initially silicon wafers are cleaned through a standard Radio Corporation of America (RCA) cleaning process and textured using 6 % KOH solution. After that, the uniform 25 nm Al_2O_3 films are prepared by non-vacuum spatial atomic layer deposition on double sides of wafers. Post annealing process in air, nitrogen, oxygen, and forming gas ambient are performed to the samples at 350°C to 650°C for 30 min using a furnace. To characterize the passivation quality of Al_2O_3 films, effective carrier lifetime (τ_{eff}), negative fixed charge (Q_f) and interface trap density (D_{it}) are measured and determined. The result shows that as the annealing temperature increasers, the τ_{eff} increases first and then decreases after peak τ_{eff} of around 152 µs at 450°C. It indicates that appropriate annealing temperature can activate the passivation effect of Al_2O_3, but τ_{eff} may decline rapidly when the temperature over 600°C due to the crystallization of Al_2O_3. The trend of τ_{eff} value can be further explained by the fixed negative charge and D_{it}. The highest Q_f of -1.07×10^{12} cm^{-2} and lowest D_{it} of 7.5×10^{12} eV^{-1}cm^{-2} that can effectively reduce recombination on the Si/Al_2O_3 interface are determined from the capacitance-voltage curve at 450°C, hence increasing the lifetime of carriers before trapped by defects. For gaining higher τ_{eff}, the samples are annealed in various atmosphere for 450°C. The better τ_{eff} of around 180 µs is acquired while annealed in forming gas. This is probably attributed to the extra chemical passivation effect via filling dangling bonds on the Si surface by hydrogen atom. The results of this study can able be used for high efficiency PERC cells.

10:40am **G4-9 High Performance Solar Selective Coatings based on TiNxOy***, **Cho-Yen Lee**, J Ting,* National Cheng Kung University, Taiwan

In this work, a series of TiNxOy films have been investigated for used as solar selective absorbers due to their remarkable optical, mechanical, and electronic properties. The films were deposited using a reactive magnetron sputtering technique. A pure titanium target was used and the deposition took place in different mixtures of oxygen, nitrogen, and argon. The obtained TiNxOy was then coated with an anti-reflection layer, consisting of metal or non-metal oxides with desirable refractive index. The TiNxOy films and the resulting multilayer coatings were analyzed for the material characteristics. Effects of the material characteristics on the optical performance is presented and discussed. We demonstrate that the resulting multilayer coatings make a breakthrough on the limitation of traditional absorbers' monotonous color appearance which is expected to be desirable in many applications.

11:00am **G4-10 Diagnostics of Surface Roughness during Electrolytic Plasma Polishing Pre-treatment for Stainless Steels***, V Mukaeva, E Parfenov, R Farrakhov, M Gromova,* Ufa State Aviation Technical University, Russian Federation*; **Aleksey Yerokhin**,* The University of Manchester, UK

Pre-treatments of steel surface constitute an important step for successful physical vapor deposition of commercially used protective coatings such as TiN, Ti and other. Electrolytic plasma polishing (EPPo) provides several effective pre-treatment operations such as deburring, cleaning and polishing which can be combined in one process. The EPPo has found its industrial scale applications in treatment of stainless and high carbon steels, nickel, copper, titanium and aluminum alloys. This process has high efficiency, and it meets modern environmental requirements, but its drawbacks include process non-linearity and high power consumption.

To investigate into the process mechanism and to overcome its drawbacks, a novel acoustic emission study has been performed, and a diagnostic approach providing estimation of the surface roughness during the treatment has been proposed. A source of the acoustic emission during the EPPo is a vapor gaseous envelope (VGE) boiling around the workpiece connected as an anode. The EPPo is usually carried out at high voltages in the range from 200 to 500 V applied between the anode and cathode. These conditions promote an appearance of the VGE with a glow discharge intensively modifying the surface. The experimental study has been dedicated to the EPPo of stainless steel BS420S29. The acoustic emission oscillations were measured by a waterproof piezoelectric hydrophone which was *immersed* into the electrolyte and located 6…12 cm away from the workpiece. A data acquisition system consisted of a computer and an analog-to-digital converter, which were controlled by a Labview based program. It was shown that an informative frequency range from 500 to 2000 Hz has characteristic spectral features which help to estimate the surface roughness during the process.

Finally, a new method of the surface roughness diagnostics was designed and introduced into automated EPPo equipment, increasing the process efficiency by 5-7%

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