

Hard Coatings and Vapor Deposition Technologies Room On Demand - Session B2

CVD Coatings and Technologies

B2-1 *In-situ* Investigation of the Oxidation Behaviour of Chemical Vapour Deposited Zr(C,N) Hard Coatings Using Synchrotron X-ray Diffraction, Florian Frank (florian.frank@unileoben.ac.at), M. Tkadletz, C. Saringer, Montanuniversität Leoben, Austria; A. Stark, N. Schell, Helmholtz-Zentrum Geesthacht, Germany; C. Czettel, CERATIZIT Austria GmbH, Austria; N. Schalk, Montanuniversität Leoben, Austria

ZrN, ZrC and ZrC_{0.4}N_{0.6} coatings were successfully deposited via chemical vapour deposition (CVD) in an industrial scale CVD plant on cemented carbide substrates and steel foils. The microstructure and the mechanical properties of the as-deposited coatings were investigated by scanning electron microscopy, X-ray diffraction (XRD) and nanoindentation tests. To gain insight into the high temperature oxidation behaviour, *in-situ* synchrotron XRD experiments were performed. Powdered samples were annealed in air between 100 °C and 1000 °C, while 2D XRD patterns were recorded. Subsequently, the 2D XRD patterns were azimuthally integrated and the resulting 1D diffractograms were evaluated by sequential and parametric Rietveld refinement. Applying these techniques, the phase evolution during oxidation could be determined. The results were then correlated with differential scanning calorimetry measurements, in order to further illuminate the oxidation mechanism of each coating system. It was shown that all Zr(C,N) samples form tetragonal, cubic and monoclinic ZrO₂ phases, whereas the onset temperature of the individual phases depends on the chemical composition. The investigated ZrCN coating exhibits the highest oxidation resistance, followed by ZrC and ZrN.

B2-3 Ti-Si-B-C-N PECVD Nanocomposite Coatings for Tribological Applications at Elevated Temperatures, Alexander Nienhaus (alexander.nienhaus@ist-extern.fraunhofer.de), TU Braunschweig, Institute for Surface Technology, Germany

With increased demands for service lifetime of tools in hot forming applications, e.g. extrusion molding and die-casting, surface modifications of hot working steels play an important role to improve its tribological properties under thermal load conditions. The machining of aluminum (Al) and copper (Cu) is especially challenging, considering its tendency to stick at the tools surface, which is increasingly impactful at elevated temperatures. Developing Ti-Si-B-C-N PECVD nanocomposite coatings is a promising approach, because, with an adequate Si-content, thermal stability and oxidation resistance can be increased by forming a thin, amorphous (a-) Si₃N₄ tissue layer between the nanocrystalline (nc-) grains, mostly nc-TiN, ncTiC, and nc-TiCN. In this study, the influence of nitrogen on its thermal and mechanical properties is under investigation. The N-content ranged from 0.0..14.6 at.-%. Chemically stable TiB₂ phases are formed by adding BCl₃ to the PECVD coating deposition process. These phases are not observed in XRD-diffraction patterns, which indicates a-TiB₂ rather than ncTiB₂, e.g. in contrast to Ti-B-N nanocomposite coatings. With C-contents up to 32 at.-%, formation of a-C particles in the a-matrix is likely. High temperature (T = 750-900 °C) *in-situ* XRD-measurements in air atmosphere provided by synchrotron radiation showed different behavior in oxidation resistance, with dependence of N-content. Furthermore, tempering in air atmosphere at 850 and 900 °C for 30 and 60 min was carried out to gain additional information on the oxidation resistance. In contact with molten or close to molten Al or Cu, B-containing nanocomposites are expected to reduce the adhesive wear on the tools surfaces. The multiphase coatings form compositionally complex nanostructures, leading to a universal hardness of up to 39 GPa, close to the so called 'superhardness' (> 40 GPa). The starting point of oxidation was determined to be in the range of 850-900 °C, underlining the possible application as protective coating for hot forming tools. Further work will focus on the nanocomposite structure, the mechanical properties, and pin-on-disc tests at T = 750 °C with Al₂O₃ counterparts.

B2-4 INVITED TALK: Atomic Layer Deposition for Complex-Shape and Temperature Sensitive Objects: Towards New Functions and Products, Frédéric Mercier (frederic.mercier@simap.grenoble-inp.fr), Univ. Grenoble Alpes, CNRS, France

INVITED

Atomic Layer Deposition (ALD) technique finds many applications today in the fields of microelectronic, batteries and catalysts. Indeed, the intrinsic advantages of ALD like conformality, uniformity and precise control of the thickness at the atomic scale can meet the requirements of the increasing

complexity and the variety of objects to be coated. Besides the aforementioned fields, other emerging fields can benefit from the advantages of the ALD technique to provide objects with enhanced functionalities or new products. To illustrate the opportunities and challenges of depositing conformal layers on either complex-shape or temperature sensitive objects or both, the talk will focus on ALD coatings on additive manufactured metallic structures and on biopolymers like cellulose matrices. The talk will include a discussion on their potential applications in energy and packaging industry. Our recent results on enhanced functionalities provided by ALD like surface finishing (color, surface smoothing), high temperature oxidation resistance and gas diffusion barrier among others will be presented. The understanding and improvement of the chemical/thermal compatibility between the object to be coated and the coating will be discussed based on a comprehensive evaluation of the structure and composition.

B2-6 INVITED TALK: Plasma-assisted Deposition using Microdroplets, Tsuyohito Ito (tsuyohito@k.u-tokyo.ac.jp), K. Nitta, K. Terashima, The University of Tokyo, Japan

INVITED

With recent development of atmospheric-pressure technologies, various plasma applications with liquid have been extensively studied. In this presentations, we are demonstrating spherical particle deposition as well as pattern drawing via atmospheric-pressure non-equilibrium plasma using microdroplets. By using microdroplets, we can apply more various raw materials hopefully obtaining certain controllability, which are difficult only with gas phase processing.

The first part of the presentation will be demonstration of sub-micrometer spherical particles deposition [1]. Here, we apply microdroplets as semi-closed micro-reactors to control size distribution of synthesized particles. A mist atomizer was used to generate microdroplets with diameter of approximately 5 µm. Such microdroplets were carried by He gas to the plasma reactor. Zinc acetate (Zn(Ac)₂) solution was used as a raw material for ZnO particles synthesis and the concentration was regulated at 0.5, 1, and 2 mM (mol/L). The generated particles are deposited on a silicon substrate locating under the plasma generator. The size distributions of the generated particles agree well with the ones expected by the distribution of microdroplets and the concentrations of the raw materials; demonstrating that one particle is generated from one microdroplet in conditions tested here. Thus microdroplets could be used as semi-closed micro-reactors at least for controlling particle sizes.

The later part of the presentation will be about plasma-assisted inkjet printing (PIP), where a microdroplet is injected through plasma by an inkjet system. By using an inkjet system, the controllability of a microdroplet in time and space can be significantly improved, developing a new printing technique, PIP. So far, we have successfully demonstrated silver line drawing [2] as well as the simultaneous polymerization of 3,4-ethylenedioxythiophene (EDOT) monomer stock solution ink and printing of the resulting poly(3,4-ethylenedioxythiophene) (PEDOT) [3]. With silver line fabrications, compared with heat treatment, the line with lower electrical resistivity and a narrower width could be achieved with a much shorter treatment time. As for PEDOT line fabrications, it was demonstrated that plasma-assisted chemical reactions could be combined with inkjet printing method.

Details will be presented at the conference.

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[2] M. Tsumaki, K. Nitta, S. Jeon, K. Terashima, T. Ito, J. Phys. D: Appl. Phys. **51**, 30LT01 (2018)

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B2-8 High Throughput Deposition of Hydrogenated Amorphous Carbon Films using High-Pressure Ar+CH₄ Plasmas, Kazunori Koga (koga@ed.kyushu-u.ac.jp), S. Hwang, K. Kamataki, N. Itagaki, M. Shiratani, Kyushu University, Japan

Plasma chemical vapor deposition (CVD) method has attracted much attention for fabricating hydrogenated amorphous carbon (a-C:H) films because it can realize to deposit large area films with a good uniformity [1]. In the conventional plasma CVD, the working gas pressure was the range between 0.05 Torr and 1 Torr. The lifetime of carbon-related radicals tends to be shorter for larger gas pressure resulting in a low deposition rate. Here we found a high rate deposition of a-C:H films with high-pressure Ar+CH₄ plasmas.

The experiments were carried out using a capacitively coupled plasma reactor [2, 3]. Ar diluted CH₄ gas was introduced to the chamber. The total

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gas flow rate and CH₄ concentration were at 98.8 sccm and 3.8 %, respectively. A 1cm x 1cm Si substrate was placed on a substrate holder. 28 MHz voltage of 170 V was applied to the powered electrode. The substrate temperature was room one. To analyze the deposition rate and the mass density, a scanning electron microscopy (JEOL JIB-4600F) and microbalance (Mettler Toledo) were used.

We have examined dependence of substrate position from the powered electrode on deposition rate as a parameter of the gas pressure. For the pressure below 2 Torr, the deposition rate monotonically decreases with increasing the distance d between the powered electrode and the substrate from 20 nm/min to 10 nm/min. In contrast, for the pressure above 5 Torr, the deposition rate decreases from around 60 nm/min for $d=15$ mm to about 30 nm/min for $d=30$ mm. Photos of the plasmas suggest that the radical generation tends to be localized near the powered electrode and the rate increases with the gas pressure in the region. Therefore, the high deposition rate realizes for $d=15$ mm for 5 and 7 Torr. The mass density for 7 Torr and $d=15$ mm is 1.41 g/cm³. To further improve the film characteristics, we studied the effects of dc pulse bias on the substrates. A dc pulse bias voltage V_{dc} with 1 μs in the pulse duration and 25 kHz in the repetition frequency was applied to the substrates. We found high mass density film of 1.67 g/cm³ is deposited at 66.7 nm/min for V_{dc}= -202 V. This indicates that the impinging ions can modify the newly deposited films and generate the dangling bonds at the surface, leading to the determination of the mass density and deposition rate [4].

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B2-9 Influence of Co-enriched Surface Zones in WC-Co Cemented Carbides on the Microstructure and Mechanical Properties of TiC_{0.6}N_{0.4}/α-Al₂O₃ Coatings, Fabian Konstantiniuk (fabian.konstantiniuk@unileoben.ac.at), M. Tkadletz, Montanuniversität Leoben, Austria; C. Zettl, CERATIZIT Austria GmbH, Austria; N. Schalk, Montanuniversität Leoben, Austria

In metal cutting applications functionally graded near surface zones in WC-Co cemented carbide substrates are applied to optimize their properties, in particular toughness and hardness. Thus, the present work focuses on the influence of Co-enriched substrate surface zones and their thickness on the microstructure and mechanical properties of state-of-the-art TiC_{0.6}N_{0.4}/α-Al₂O₃ coatings synthesized using chemical vapor deposition. Complementary cross-sectional energy dispersive X-ray spectroscopy and electron back-scatter diffraction maps provided insight into the grain size, preferred orientation and phase composition of coatings and substrates. While the hardness and Young's modulus of the coatings were hardly affected by the Co-enriched surface zone and its thickness, nanoindentation maps performed on the cross-sections of the substrates confirmed a lower hardness and Young's modulus in zones with higher Co content. Since the tensile residual stress in both, the TiC_{0.6}N_{0.4} and α-Al₂O₃ decreased with increasing thickness of the Co-enriched surface zone, as determined by X-Ray diffraction, it is suggested that stress relaxation occurs through plastic deformation of the soft Co-enriched surface zone. Despite the influence on the residual stress, the Co-enriched surface zone and its thickness was found to have no effect on the thermal crack networks of the coatings. However, Rockwell-indentation tests demonstrated a reduction of the coating adhesion with increasing thickness of the Co-enriched surface zone. The results obtained within this work contribute to a better understanding of the influence of a Co-enriched surface zone and its thickness on the performance of TiC_{0.6}N_{0.4}/α-Al₂O₃ coated cutting tools.

B2-10 CVD Alumina-based Nanocomposite Coatings, Zhenyu Liu (Zhenyu.Liu@kennametal.com), Kennametal Inc., USA

Nanocomposite is a multiphase solid material where one of the phases has the size of less than 100 nanometers (nm) in at least one dimension, or structures having nano-scale repeat distances between the different phases that make up the material. Nanocomposite coating represent a new generation of materials exhibiting completely new properties with respect to the conventional used materials. The superior mechanical properties of nanocomposites originate from their peculiar nanostructures (size effects) and high density of interfaces. The unique structure and exceptional properties make nanocomposite materials a possible alternative to traditional polycrystalline materials, which have met their limits in many recent engineering applications.

Inspired by nanolayer coatings of PVD and multilayer CVD coatings development, we demonstrate a couple of potential Al₂O₃-based nanocomposite systems deposited by CVD process directly using multilayer concepts with well-controlled deposition conditions to maintain the deposited "thin film" at early stage, nucleation regime. As a consequence, the "thin film" would maintain at the island forms or particles/nanoparticles states with the size smaller than 100 nm at least in one dimension, whilst the alumina matrix would keep depositing to form a continuous matrix. Ultimately, a nanocomposite coating can be formed with improved wear resistance and metal-cutting performance. The ability to process nanocomposite by direct nucleation and growth of ceramic materials via CVD technique should provide new technical opportunity on the advanced materials and application development.

Keywords: CVD, Al₂O₃-based nanocomposite, thin films, nucleation, crystal growth

B2-11 Compatibility of a CoCrFeNi Multi-principal Element Alloy Substrate with Halide-based Thermal CVD Processes for TiN Deposition, Katalin Böör (katalin.boor@kemi.uu.se), Uppsala University, Sweden; R. Qiu, Chalmers University of Technology, Sweden; A. Forslund, KTH Royal Institute of Technology, Sweden; O. Bäcke, Chalmers University of Technology, Sweden; H. Larsson, KTH Royal Institute of Technology, Sweden; E. Lindahl, Sandvik Coromant R&D, Sweden; M. Halvarsson, Chalmers University of Technology, Sweden; M. Boman, Uppsala University, Sweden; L. von Fieandt, Sandvik Coromant R&D, Sweden

Multi-principal element alloys (MPEAs) are materials consisting of at least four metallic elements in near-equimolar amounts. These alloys can exhibit new combinations of material properties, either deriving from the new phases formed or from the individual components. Their applications may require a protective coating, it is therefore crucial to investigate their compatibility with conventional coating technologies. Titanium nitride is a widely used corrosion- and wear resistant coating, which is frequently deposited by CVD. This presentation assesses the compatibility of the CoCrFeNi substrate, an important MPEA, with a conventional CVD process for TiN deposition using TiCl₄/H₂/N₂ as precursors [1]. Different reactions between the substrate, the coating and the precursors will be discussed. These include substrate etching by Cl-containing species and intermetallic compound or nitride building between the substrate elements and Ti or N, respectively. Substrate etching can result in voids in the substrate and the substrate elements may be redeposited and incorporated in the coating. Intermetallic compounds and nitrides can be formed by diffusion of the substrate elements into the coatings or Ti/N diffusion into the substrate. SEM, XRD and (S)TEM combined with EDS were used to determine which of the mentioned processes take place in the CoCrFeNi-TiCl₄/H₂/N₂ system between 850-950 °C. Thermodynamic calculations were carried out using the Thermo-Calc software to determine the stable compounds that can be formed during the process.

The substrate was stable under the process conditions. Only Cr was reactive towards the N₂ precursor and appeared in the coating grain boundaries, shown by EDS in TEM. Thermodynamic calculations predicted that Cr-containing nitride phases could form, explaining the driving force for Cr diffusion in the grain boundaries. XRD results only indicated the presence of a TiN and a CoCrFeNi phase. No intermetallic phases were formed between the substrate elements and titanium and no signs of etching were observed.

The results provide an understanding on the processes involved in the substrate-precursor interaction and the driving forces behind. They explain why CoCrFeNi outperforms elemental Ni, Fe [2] and alloys of its components, giving a basis for determining which multi-component alloys can be compatible with conventional CVD processes.

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B2-12 Silicon Carbide Coatings for High Temperature Receiver of Concentrated Solar Power Plants, Michel Pons (michel.pons@grenoble-inp.fr), D. Chen, University of Grenoble Alpes, France; J. Colas, PROMES-CNRS, France; F. Mercier, University Grenoble-Alpes, France; L. Charpentier, M. Balat-Michelin, PROMES-CNRS, France

There is a growing interest in concentrating solar power plants as electricity generation systems. Mirrors concentrate the sun's energy to drive traditional steam turbines or engines that create electricity. The thermal energy concentrated in a CSP plant can be stored and used to produce electricity when it is needed, day or night. One of the challenges is to build the solar receiver which can work at temperatures near or higher than

On Demand available April 26 - June 30, 2021

1000 °C for optimizing the yield. Current candidate materials are metallic alloys such as Inconel, or bulk ceramics like silicon carbide, but their operating temperatures may be limited due to oxidation or mechanical problems. Silicon carbide coatings, deposited by chemical vapor deposition technology at 1100 °C, are selected for their high thermal conductivity, low thermal expansion coefficient, high temperature stability and oxidation resistance. They forms stable and protective silica scales at temperatures higher than 1000°C. Oxide dispersion strengthened (ODS) FeCrAl alloys (Kanthal APMT), are alumina-forming alloys which can resist to high temperature oxidation. They are chosen as model substrates to study the potential of SiC coatings. Accelerated cyclic oxidation and high temperature emissivity measurements are performed in Promes solar furnace facilities (France), confirming the potential of silicon carbide coatings as materials for high temperature central receivers. The SiC based multilayered system exhibits low degradation after 1500 h of oxidation at 1000 °C in air. The modelling and simulation of stresses during thermal cycles taking into account the creep and growth of the oxide layer are used to show the limits of use of these materials.

B2-13 Hot Filament CVD Diamond Coatings for Hard-to-machine Materials, Michael Woda (michael.woda@cemecon.de), W. Puetz, M. Frank, W. Koelker, C. Schiffers, O. Lemmer, CemeCon AG, Germany

In the group of carbon-based coatings, polycrystalline CVD diamond thin films reveal some extraordinary material properties. Applying the very high hardness of up to 10000HV onto cutting tools enables economically feasible tool usage when machining very high abrasive materials. CVD diamond thin films are typically coated by either microwave or hot filament CVD techniques. On cutting tools with cemented carbide substrates and complex geometries hot filament CVD is well established on an industrial scale nowadays. The basics of hot filament CVD diamond coating technology are briefly introduced in the scope of this presentation. The films coated by this method can be utilized to address cutting of work piece materials including Carbon fiber reinforced plastics (CFRP), ceramics, graphite, Aluminum-Silicon alloys or even sintered carbide. This work presents results of case studies revealing the benefits of CVD diamond coatings upon cutting operations on these very demanding work piece materials.

B2-14 Ald-Pvd Multilayers: Deposition, Thermal Stability And Mechanical Properties, Thomas Edwards (thomas.edwards@empa.ch), T. Xie, L. Petho, S. Büchel, X. Maeder, B. Putz, J. Michler, Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland

The extent of the embrittlement in ductile-brittle multilayers often depends on the modulation period ($t_{\text{brittle}} + t_{\text{ductile}}$) as well as on the modulation ratio ($t_{\text{brittle}}/t_{\text{ductile}}$) [1]. In this work, ductile-brittle multilayers of Al / Al₂O₃ / Al... and Ti / TiO₂ / Ti... were produced on Si substrates by a unique combination of atomic layer (ALD, Al₂O₃, TiO₂) and physical vapor deposition (PVD, Al, Ti) within a single deposition system. Using this ALD/PVD combination, neighbouring layer thicknesses can easily differ by one order of magnitude or more. In particular, the ability to deposit continuous sub-nm layers with ALD opens up a wide range of otherwise unachievable modulation and thickness ratios. The thicknesses and structures of the ALD layers were verified by HR-TEM imaging of lift-outs. Further depositions on flexible substrates have also been performed with thinner Al layer thicknesses to minimize residual stresses. The Al₂O₃ or TiO₂ layer thickness is varied across the multilayer cross-section (0.1 nm – 10 nm) to study the effect on strength of the film as determined by microcompression, and on crack onset and propagation as a function of oxide layer thickness in tensile tested multilayer films. Single layered films (Al or Al₂O₃, etc.) are used as reference materials. Further the thermal stability of such multilayer films was studied up to 0.9 T/T_m, considering the stability and crystallinity of the ALD interlayers and the texture of the PVD layers. Grain growth of Al is limited by the Al₂O₃ layer, allowing for easy discrimination of individual Al layers necessary for locating onset of cracks, and for cross-sectional fragmentation analysis by focused ion beam (FIB) cross-sectioning under tension which avoids crack closure upon unloading. The multilayer structure has good adhesion between individual layers as well as to the polymer substrate and the oxide layers show increasing stretchability with decreasing film thickness, as a result of being extremely well defined and practically defect free. This study helps improve the understanding of deformation mechanisms in flexible thin film structures and can give useful guidelines for strong and damage tolerant thin film metallic systems.

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B2-15 Investigation of Diamond Coating Characteristics on Chrome-Plated AISI 4140 Steel by Hot-Filament Chemical Vapour Deposition Process, R. Vignesh, S. Bhooinatha Sellarajan (rayarajan2005@gmail.com), J. Rajaguru, N. Arunachalam, M. Ramachandra Rao, Indian Institute of Technology Madras, India

In the modern engineering world, extensive research has led to the development of certain unique grades of steel, mostly suitable for enhanced functions. AISI 4140 steel is one such grade, having major applications in power plants, automobile and aerospace industry. HFCVD process on these steel substrates have attracted significantly to improve the mechanical performances due to the superior properties of diamond films. However, CVD diamond films on steel are a great challenge due to the formation of interfacial graphite layers; intrinsic stresses and high stiffness due to considerable differences in thermal expansion coefficients between substrate and diamond. This may lead to premature failure of diamond films. To overcome these limitations, interlayers such as Cr, Ti, Mo, W, etc., were demonstrated for an improved adhesion by different techniques (sputtering and CVD). In this work, Cr interlayer made by hard-chrome plating technique was utilized. Then, diamond film was deposited over Cr interlayer by HFCVD process. High substrate temperature (~750°C) generated during the diamond deposition can significantly affect the mechanical properties of the steel substrate as the material undergoes phase transformation. This leads to material failure in real-time applications. Despite this, the necessary investigation about the influence of diamond coating deposition temperature on the microstructure and mechanical properties of the steel substrate has not been covered yet. Hence, this work aims at analyzing the substrate properties at varying temperature (450-750°C) by adjusting the hot-filament to substrate distance (d: 20-40 mm) and the filament temperature (1800-2200°C) and deposition time (soaking time: 1-8 hrs). Tensile strength, ductility, XRD and microstructure via SEM were carried out. The results indicate that the mechanical properties and microstructural features of the steel are affected significantly by deposition temperature and time. The strength and hardness of AISI 4140 steel drop as the deposition temperature and time are increased. However, the ductility increased with increasing deposition temperature and holding time. Microstructural observations reveal that the carbide precipitates have a plate-like structure at lower temperatures, but are spheroid-like at high temperatures. Carbide formations at high temperature were also confirmed by the XRD analysis. These results suggest that it is essential to select an optimal depositing temperature and time by considering the material high-temperature behaviour. Further, a case study on diamond film formation as a function of filament-substrate gap is discussed.

B2-16 The Challenge and Strategy of a-Si CVD Coating on Aluminum Alloys, Min Yuan (min.yuan@silcotek.com), SilcoTek Corporation, USA

Aluminum alloys in general present a particular challenge in thermal CVD of amorphous silicon coating.

Aluminum is known to catalyze the crystallization of amorphous silicon and induce nanowire growth under thermal CVD conditions. The deposited thin film usually contains a mixture of amorphous and microcrystalline silicon, which not only manifest as undesirable cosmetic defects on finished products, but also compromise other coating properties such as coating adhesion, corrosion resistance and chemical inertness.

Here we present a modified thermal CVD process that is designed to prime the aluminum substrate at the beginning of CVD, so that subsequent a-Si deposition can proceed smoothly. The priming step acts to prevent aluminum from catalyzing the amorphous-to-crystalline transition and stop the undesirable silicon nanowire growth. The coated products from this process deliver more appealing cosmetic finish, as well as superior coating properties that will be discussed in the presentation.

The priming effect is achieved by exposing the aluminum substrate to a mixture gas at the initial stage of the CVD process. The synergistic reaction of the gas mixture deposits a thin barrier layer at the substrate/coating interface, which serves to cut off the negative influence of aluminum on the coating. The barrier layer itself has strong adhesion to both the aluminum substrate and the a-Si coating deposited on top of it, thus improving the overall adhesion properties.

This modification is easily incorporated as part of the CVD process and does not require separate wet chemistry or vacuum break. This makes it straightforward to implement and scale up in a manufacturing setting, as has been demonstrated in the facility at SilcoTek Corporation.

Author Index

Bold page numbers indicate presenter

— A —

Arunachalam, N.: B2-15, **3**

— B —

Bäcke, O.: B2-11, **2**

Balat-Michelin, M.: B2-12, **2**

Boman, M.: B2-11, **2**

Boominatha Sellarajan, S.: B2-15, **3**

Böör, K.: B2-11, **2**

Büchel, S.: B2-14, **3**

— C —

Charpentier, L.: B2-12, **2**

Chen, D.: B2-12, **2**

Colas, J.: B2-12, **2**

Czettel, C.: B2-1, **1**; B2-9, **2**

— E —

Edwards, T.: B2-14, **3**

— F —

Forslund, A.: B2-11, **2**

Frank, F.: B2-1, **1**

Frank, M.: B2-13, **3**

— H —

Halvarsson, M.: B2-11, **2**

Hwang, S.: B2-8, **1**

— I —

Itagaki, N.: B2-8, **1**

Ito, T.: B2-6, **1**

— K —

Kamataki, K.: B2-8, **1**

Koelker, W.: B2-13, **3**

Koga, K.: B2-8, **1**

Konstantiniuk, F.: B2-9, **2**

— L —

Larsson, H.: B2-11, **2**

Lemmer, O.: B2-13, **3**

Lindahl, E.: B2-11, **2**

Liu, Z.: B2-10, **2**

— M —

Maeder, X.: B2-14, **3**

Mercier, F.: B2-12, **2**; B2-4, **1**

Michler, J.: B2-14, **3**

— N —

Nienhaus, A.: B2-3, **1**

Nitta, K.: B2-6, **1**

— P —

Petho, L.: B2-14, **3**

Pons, M.: B2-12, **2**

Puetz, W.: B2-13, **3**

Putz, B.: B2-14, **3**

— Q —

Qiu, R.: B2-11, **2**

— R —

Rajaguru, J.: B2-15, **3**

Ramachandra Rao, M.: B2-15, **3**

— S —

Saringer, C.: B2-1, **1**

Schalk, N.: B2-1, **1**; B2-9, **2**

Schell, N.: B2-1, **1**

Schiffers, C.: B2-13, **3**

Shiratani, M.: B2-8, **1**

Stark, A.: B2-1, **1**

— T —

Terashima, K.: B2-6, **1**

Tkadletz, M.: B2-1, **1**; B2-9, **2**

— V —

Vignesh, R.: B2-15, **3**

von Fieandt, L.: B2-11, **2**

— W —

Woda, M.: B2-13, **3**

— X —

Xie, T.: B2-14, **3**

— Y —

Yuan, M.: B2-16, **3**