Wednesday Morning, April 25, 2018

Hard Coatings and Vapor Deposition Technologies Room Golden West - Session B1-3

PVD Coatings and Technologies

Moderators: Joerg Vetter, Oerlikon Balzers Coating Germany GmbH, Qi Yang, National Research Council of Canada, Jyh-Ming Ting, National Cheng Kung University

8:20am B1-3-2 High Quality Oxide Films Deposited at Room Temperature by Ion Beam Sputtering, Gerard Henein, National Institute of Standards

and Technology, USA; *J Topolancik*, Roche Sequencing Solutions, USA The highest quality oxides such as SiO₂, Al₂O₃ and Indium Tin Oxide (ITO) require high temperature processing either during the growth of the film or annealing post-growth. Thermal SiO₂ is grown at \approx 1000 °C , ALD Al₂O₃ is deposited at \approx 300 °C , and magnetron-sputtered ITO must be annealed above 350 °C in order to turn the film conductive . These elevated temperature requirements are not compatible with polymer substrates used for flexible electronics .

We have deposited dense and pinhole-free thin films of SiO₂, Al₂O₃ and ITO at room temperature via ion beam sputtering. The deposition system consists of a 3-grid 14 cm RF ion gun directed at 200 mm targets of SiO₂, Al and ITO. All three processes require a small flow of O₂ to achieve stoichiometry. Typical conditions were: argon flow rate 3.3×10^{-7} m³/s, beam voltage 600 V, beam current 220 mA and acceleration voltage 150 V. The substrate wafers were kept at 20 °C. The base vacuum prior to deposition was 2.6x10⁻⁶ Pa.

The SiO₂ films were 100 nm thick and measured by the mercury probe technique to obtain the C-V and I-V characteristics. The films were found to be of similar quality as thermal oxide with a resistivity of $10^{14} \Omega \cdot m$, breakdown field in excess of 7x10⁸ V/m, and pinhole-free with an etch rate in 6:1 Buffered Oxide Etch (BOE) of 1.6 nm/s.

The Al₂O₃ films were part of a Pt- Al₂O₃-Pt vertical tunnel junction and were kept extremely thin, from 3 nm to 4 nm. The current-voltage characteristics of these junctions indicated a breakdown field roughly twice that achieved by ALD films for the same structure. This breakdown voltage was found to be independent of junction area, strongly suggesting the absence of pinholes in the film.

The ITO films were 50 nm to 100 nm thick. As deposited, they are fully transparent with an electrical resistivity of $5x10^{-6} \Omega \cdot m$.

In conclusion, the ion beam deposition technique has proven to be a powerful tool for the room temperature production of very high quality oxides, as thin as 3 nm. In addition, this process allows for a sub-nanometer control over the film thickness.

8:40am B1-3-3 van der Waals Oxide Heteroepitaxy, Ying-Hao Chu, National Chiao Tung University, Taiwan INVITED

The research field of oxide heteroepitaxy suffers from the characteristics of misfit strain and substrate clamping, hampering the optimization of performance and the gain of fundamental understanding of oxide systems. Recently, there are demonstrations on functional oxides epitaxially fabricated on layered muscovite substrate. In these heterostructures, due to the weak interaction between substrate and film, they show the lattice of films close to bulk with excellent strictive properties, suggesting that these critical problems can be potentially solved by van der Waals oxide heteroepitaxy. In addition, by exploiting the transparent and flexible features of muscovite, such a heteroepitaxy can deliver new material solutions to transparent soft technology. In this paper, the history, development, and current status of van der Waals oxide heteroepitaxy are addressed and discussed. In the end, new research directions in terms of fundamental study and practical application are proposed to highlight the importance of this research field.

9:20am **B1-3-5 Color Controllable TiO_xN_y Coatings Deposited by Magnetron Sputtering,** *Tun-Yi Chang, J Ting,* National Cheng Kung University, Taiwan

 TiO_xN_y -based decorative coatings were deposited using a reactive DC magnetron sputter deposition method. The substrates included AI, stainless steel, and Ti. Optical properties of single-layered and double-layered TiO_xN_y coatings were both investigated. The color of the coating was controlled by manipulating the sputter deposition conditions, including gas pressure and composition, DC power, electrode distance, and substrate type. Selected coatings were also deposited with an anti-reflective layer.

The resulting coatings were characterized using SEM, XRD, XPS, TEM, UVvis spectroscopy, a-step, ellipsometry, and colorimetry. We show that at least 6 different colors were obtained on each type of the substrate, either single- or double-layered.

9:40am **B1-3-6** SiO₂/Sc_{0.31}Al_{0.69}N/LiNbO₃ Multilayer Structure for SAW Device Applications, *Chun-Ting Shen*, National Cheng Kung University, Taiwan; *S Wu*, Tung-Fang Design University, Taiwan; *J Huang*, National Cheng Kung University, Taiwan

We report a temperature compensated surface acoustic wave (SAW) device on a SiO₂/ Sc_{0.31}Al_{0.69}N /LiNbO₃ structure. The Sc_{*}Al_{1-x}N films (x=0.23, 0.24, 0.26, 0.29, 0.31, 0.34, 0.39) are deposited on lithium niobate substrate by reactive magnetron co-sputtering using Al and Sc as targets. The films have (002) texture and different degree of lattice strain caused by Sc atoms replacing specific Al sites. The highest piezoelectric coefficient (d₃₃) value 22.2 pm/V is achieved at x=0.31, which corresponds to the largest lattice distortion presented in 2D-XRD data. The electromechanical coupling coefficient (K²) and temperature coefficient of frequency (TCF) of SAW devices with different SiO₂ thickness on Sc_{0.31}Al_{0.69}N /LiNbO₃ are compared. The SiO₂/ Sc_{0.31}Al_{0.69}N /LiNbO₃ structure have a great potential in high bandwidth, high temperature stability SAW device application.

10:00am **B1-3-7 Self-lubricant CrO-Ag Coatings for Machining Tools**, *Filipe Fernandes*, University of Minho, Portugal; *A Cavaleiro*, University of Coimbra, Portugal

High performance dry machining is one of the major trends in modern manufacturing. This is a very hot topic inside the tribology community; PVD tool-coaters and cutting tools costumers are seeking innovative coating solutions which could improve the performance and lifetime of tools, as well as, increase the material volume removal rates through increasing cutting speeds; this could be achieved by eliminating the use of harmful liquid lubrication. Dry machining cutting conditions generate severe shear stresses and high temperature harsh conditions on the cutting zone which, consequently, lead to a premature degradation of the tool. Thus, coating solutions should exhibit, simultaneously, high toughness, low friction coefficient, low wear rate and thermal stability at high temperature. Selflubricant coating systems, with control release of the lubricious species. have enormous potential to be used in the protection of these tools. However, solutions developed up to now do not allow the control of the lubricious phase release leading to the easy degradation of the coating after a short period of time. In this investigation, we propose the development of self-lubricant coating system based on a matrix of Cr-O, well known by their good high temperature performance and antidiffusion properties, alloyed with Ag, which has lubricious properties. The goal is to observe if Cr-O coating can work as an efficient barrier to Ag ions diffusion. The deposition rate of Cr-O coating is 1.6 times higher than a single Cr coating, taken as reference, deposited with identical conditions. Incorporation of Ag in the Cr-O coating increases the deposition rate. Transition from a dominant columnar to a very compact morphology, as well as from crystalline to amorphous structure, was observed with the O addition. However, no morphological changes were observed with Ag additions. Annealing at temperature≥500 °C promotes the crystallization of Cr-O and Cr-O-Ag coatings, i.e. the formation of a crystalline composite structure, formed by: Cr, Cr2O3 and Ag phases, the later in the case of Cr-OAg coatings. Thermogravimetric analysis showed that O and Ag additions to Cr have no influence on the onset point of oxidation. The oxidation kinetics of the Cr-based coatings is controlled by the outward diffusion of Cr ions through a top Cr2O3 layer. A dual oxide layer was formed for Cr-O and Cr-O-Ag coatings, when exposed to a continuous increase of the temperature up to 1200 °C. Cr2O3 layer controls the Ag ions diffusion to the surface.

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