Tuesday Afternoon, May 23, 2023

Hard Coatings and Vapor Deposition Technologies
Room Town & Country D - Session B4-4-TuA

Properties and Characterization of Hard Coatings and Surfaces IV

Moderators: Dr. Naureen Ghafoor, Linköping University, Sweden, **Dr. Marcus Günther**, Robert Bosch GmbH, Germany, **Dr. Fan-Yi Ouyang**, National Tsing Hua University, Taiwan

1:40pm **B4-4-TuA-1 Magnetron Sputter Deposition of Ultrathick Boron Carbide Coatings on Spherical Substrates for Inertial Confinement Fusion***, **J. B. Merlo**, G. Taylor, S. Shin, L. Bayu Aji, J. Bae, L. Sohngen, S. Kucheyev,* Lawrence Livermore National Laboratory, USA

Boron carbide has attractive properties for use as ablator capsules for inertial confinement fusion (ICF). Creating an ultrathick, uniform, defect-free film on a rolling spherical substrate has many challenges, including delamination and fracture due toresidual stress and nodular growth defects, which are believed to originate from particulates deposited onto the film surface during growth. We have systematically studied effects of direct-current (DC) versus radiofrequency (RF) driven magnetron sputter deposition, substrate temperature, chamber pressure, and the target to substrate distance for amorphous boron carbide films deposited on stationary planar substrates. Here, we describe how these deposition parameters are optimized when the process is transferred to coating rolling spherical substrates.

This work was performed under the auspices of the U.S. DOE by LLNL under Contract DE-AC52-07NA27344 and by General Atomics under Contract 89233119CNA000063.

2:00pm **B4-4-TuA-2 Corrosion and Electrical Insulation Properties of SiO_x Thin Films Deposited by Microwave PECVD***, **Atreya Danturthi**, R. Drummond Brydson,* University of Leeds, UK*; I. Kolev,* Hauzer, Netherlands*; L. Yang, A. Bell, G. Wu,* University of Leeds, UK

Smart sensor technology is currently being developed and incorporated into several industries as part of the Industry 4.0 scheme to optimise energy input to industrial equipment thereby reducing carbon emissions.

These smart sensors applied on wheel bearings of EVs are prone to premature electrical failure due to current passage from dc/ac motors and also corrosion because of exposure to extreme environments. One of the solutions to this problem is developing electrically insulating and corrosion-resistant coatings that could be applied to the existing system. Current research is focussing on Al_2O_3-based coatings for these applications deposited using plasma spray/ electrolytic oxidation. However, Al_2O_3 coatings produced using the above methods were porous requiring additional sealing to be useful in insulation and corrosion-resistant applications. Therefore, Si-based coatings using µ-plasma enhanced chemical vapour deposition (PECVD) are being explored in this current study as the properties of silicon-based coatings can be varied through variations of nitrogen, oxygen, and silicon composition and the fact that PECVD is capable of producing denser coatings than PVD. In addition, higher deposition rates can be achieved using precursors such as hexamethyldisiloxane (HMDSO)/ hexamethyldisilazane (HMDSN) due to their high vapour pressures thereby reducing manufacturing cost.

In this study, HMDSO and O_2 gases were used to deposit SiO_x coatings at varying gas flow ratios (1:12, 1:16, 1:20), µ-powers (2 and 4kW) and coating thicknesses (3 - 10 µm) to study their impact on corrosion & insulation properties. Characterisation techniques such as Calo test, scratch testing, Rockwell C adhesion test, Fourier Transform Infrared spectroscopy, Scanning Electron Microscopy, X-ray Photoelectron Spectroscopy, X-ray diffraction were used for characterisation. In addition, cyclic polarisation and electrochemical impedance spectroscopy were used for corrosion behaviour characterisation, and finally I-V measurements to ascertain insulation behaviour. Preliminary corrosion results of all the coated samples showed significant improvement in corrosion resistance compared to bare high-speed steel substrate. In particular, coatings deposited at 4kW (µ-power) and 1:12 (HMDSO:O_2) gas flow ratio had very low corrosion currents (a few pA) indicating high corrosion resistance.

2:20pm **B4-4-TuA-3 High-Throughput Methodology for The Realization of High-Entropy High-Dielectric-Constant Ba(Ti,Zr,Ta,Hf,Mo)O_3 Film-Based Metal-Oxide-Semiconductor-Related Devices***, **Kao-Shuo Chang**,* National Cheng Kung University (NCKU), Taiwan*; V. Nguyen,* No.1, University Road, Taiwan*; T. NAGATA,* National Institute for Materials Science, Japan **INVITED**

The use of a high-throughput sputtering technique for the fabrication of high-entropy high-dielectric-constant (high-k) Ba(Ti,Zr,Ta,Hf,Mo)O_3 film libraries on Si substrates and sub-nm equivalent-oxide-thickness (EOT) metal-oxide-semiconductor (MOS) devices and metal-oxide-semiconductor field-effect transistors (MOSFETs) will be presented. The elemental variations and amorphous microstructures were characterized using high-throughput X-ray fluorescence (XRF) and X-ray diffraction, respectively. The film library was patterned into 100 MOS configurations, and their dielectric constants and losses were systemically mapped. The MOSFETs after rapid thermal annealing (RTA) exhibited excellent characteristics, including an on/off current ratio of »10^6 and a saturated field-effect mobility of 288 $cm^2 \times V^{-1} \times s^{-1}$. Small variations in the threshold voltage and negligible changes in the maximum drain current were also under various positive and negative gate-bias stress conditions before and after the RTA. Our results indicated the potential of the Ba(Ti,Zr,Ta,Hf,Mo)O_3 films for usein a gate-first process for advanced gate stack-related devices.

3:00pm **B4-4-TuA-5 Effects of Nitrogen Flow Ratio on the Mechanical and Anticorrosive Properties of Co-sputtered (TiZrHfTa)N_x Films***, **Tzu-Yu Ou**,* National Taiwan Ocean University, Taiwan*; L. Chang,* Ming Chi University of Technology, Taiwan*; Y. Chen,* National Taiwan Ocean University, Taiwan

In this study, (TiZrHfTa)N_x films were prepared through co-sputtering with four sputter guns. The stoichiometric ratio x of (TiZrHfTa)N_x films was varied by adjusting the reactive gas ratio of f_{N2} (N_2/(N_2 + Ar)) at 0, 0.4, and 0.7. With an f_{N2} of 0, the fabricated metallic $Ti_{0.23}Zr_{0.22}Hf_{0.30}Ta_{0.25}$ film, namely N00, exhibited a valence electron concentration of 4.25, a bcc phase with lattice constants of 0.3395 nm, a hardness of 8.0 GPa, and a Young's modulus of 148 GPa. The introduction of N into the TiZrHfTa crystallites transformed the phase from bcc to fcc. N04 [$(Ti_{0.22}Zr_{0.30}Hf_{0.17}Ta_{0.31})N_{0.83}$] and N07 [$(Ti_{0.33}Zr_{0.34}Hf_{0.13}Ta_{0.20})N_{0.88}$] films were prepared whenf_{N2} was set at 0.4 and 0.7, respectively. The N04 and N07 films exhibited a common fcc phase with lattice constants of 0.4490 and 0.4464 nm, respectively. The N04 and N07 films exhibited hardness values of 33.2 and 32.2 GPa and Young's modulus values of 379 and 363 GPa, respectively. The corrosion resistance of (TiZrHfTa)N_x films was investigated using potentiodynamic polarization and electrochemical impedance spectroscopy.

4:00pm **B4-4-TuA-8 Magnetron Sputter Deposition of Boron Carbide in Ne and Ar Plasmas***, **Liam Sohngen**, S. Shin, L. Bayu Aji, G. Taylor, J. Bae, A. Engwall, J. Hammons, S. Kucheyev,* Lawrence Livermore National Laboratory, USA

Boron carbide ceramic films have many applications, including wear resistant coatings and fuel capsules for inertial confinement fusion (ICF). However, the deposition of boron carbide films by conventional magnetron sputtering with an Ar plasma suffers from relatively low deposition rates. Here, we explore the deposition of boron carbide with a Ne plasma, which is ballistically better matched to B and C and is expected to exhibit larger sputtering yields than Ar. We study plasma discharge characteristics, ion and atom energy distributions, and properties of films deposited with different substrate tilt angles in both direct-current (DC) or radio-frequency (RF) mode in Ar or Ne plasmas. Results show that film properties are dominated by the effect of the working gas on the plasma discharge and gas phase scattering of depositing species flux rather than by sputtering ballistics and energetics.

Author Index

Bold page numbers indicate presenter