Hard Coatings and Vapor Deposition Technology
Room: Royal Palm 1-3 - Session B1-2

PVD Coatings and Technologies
Moderator: P. Eklund, Linköping University,
J. Vetter, Sulzer Metaplas GmbH,
J.-H. Huang, National Tsing Hua University

1:30pm **B1-2-1 Growth Morphology and Corrosion Resistance of Magnetron Sputtered Cr Films**, ***K.-T. Chiang*** *(kchiang@swri.org), R. Wei*, Southwest Research Institute

Chromium films are commonly used as corrosion resistant coatings because they form a passive, protective scale. The film morphology and microstructure are important aspects of corrosion resitance. In this paper, we presented grwoth mechanisms and morphologies of these films to provide insight into the microstructural properties of corrosion resistance. Thin films of Cr were deposited on silicon wafer, copper and carbon steel substrates using a plasma-enhanced magnetron sputtering technique. A filament was utilized to produce a plasma that effects an ion bombardment on the workpiece during the magnetron sputter deposition process. The deposited fims were characterized by x-ray diffraction, scanning electron microscopy and atomic force microscopy. The film growth morphology and microstructure are correlated with sample orientation (with respect to the magnetron) and the deposition parameters. On important deposition parameter affecting Cr film properties is the level of plasma ion bambardment. It has been demonstrated that at a low level of ion bombardment, a columnar structure is formed and film is discontiguous. As the level of ion bombardment increases, the film become denser and contiguous. Eventually the film becomes fully dense without indication of columnar structure. The corrosion behavior of the deposited films was studied using potentiodynamic polarization techniques. It has been demonstrated that adequate ion bombardment is necessary to achieve the required corrosion resistance.

1:50pm **B1-2-2 Fundamental Studies on the Deposition of Nanocrystalline Diamond (n-D) Films by Means of Pulsed Laser Deposition**, ***H. Grüttner*** *(hgruettn@hs-mittweida.de)*, Hochschule Mittweida - University of Applied Sciences, Germany

The results of fundamental studies on the deposition of nanocrystalline diamond (n-D) films by means of pulsed laser deposition (PLD) will be present. The n-D films were deposited on silicon (111) and hard metal by excimer laser ablation from a graphite target at elevated substrate temperatures and in hydrogen background gas. The variation of the microstructure and the properties of the films with temperature was investigated in the range of 100°C to 660°C and with hydrogen pressure in the range of 1 mbar to 7 mbar. With diamond and / or ion bombardment pretreated and non-pretreated substrates were used and the influence of the pretreatment process on the microstructure of the films was investigated. The films were produced at laser fluences between 10 J/cm² and 15 J/cm². The thickness of the films was varied from 100 nm up to 2 microns. The influence of deposition parameters on the n-D growth and the sp^2/sp^3 bonding ratio was determined by Raman spectroscopy and TEM / EELS analysis and it will be shown that n-D films of good quality can be prepared using proper parameters. The hardness and Young´s modulus were determined using nanoindentation and the optical properties in the UV / VIS range was measured by using photospectrometry. The variation of these properties with deposition parameters and their correlation with the microstructure of the n-D films will also be presented.

2:10pm **B1-2-3 Oxidation and Degradation of Nitride Thin Films at High Temperature under Controlled Atmosphere**, ***F.-H. Lu*** *(fhlu@dragon.nchu.edu.tw)*, National Chung Hsing University, Taiwan **INVITED**

Oxidation and degradation of nitride thin films, such as TiN, CrN, ZrN, AlN, prepared by magnetron sputtering were investigated over various temperatures in different atmospheres by analyzing changes in appearance and crystalline phases, as well as microstructures. The atmospheres contained air, nitrogen, and forming gas ($N_2/H_2 = 9$), which exhibited drastically different nitrogen and oxygen partial pressures. Oxidation of the nitride films was driven by the Gibbs free-energy changes between the nitrides and the formed oxides, and could be tailored by controlling the annealing temperature and nitrogen/oxygen partial pressures. Both internal stresses resulted from sputtering and thermally-induced stresses were responsible for many types of degradation in the films, which would be further discussed.

2:50pm **B1-2-5 Effects of Pulsed Laser Irradiation of As-Deposited c-BN-Films using Photons of 157 nm Wavelength**, ***R. Bertram*** *(rbertram@hs-mittweida.de)*, Hochschule Mittweida - University of Applied Sciences, Germany

We present the effects of pulsed laser irradiation of as-deposited c-BN-films using photons of 157 nm wavelength and 7.9 eV energy, respectively. The films were deposited by pulsed laser deposition (PLD) using a KrF excimer laser of 248 nm wavelength and up to 30 J/cm^2 laser fluence on the pyrolytic hexagonal boron nitride target with additional ion beam bombardment of the growing films using a mixture of nitrogen and argon ions produced in a r.f. ion source with 700 eV ion energy.

The irradiation of such coatings with a fluorine laser was found to influence the number and size of sp^2-bonded hBN particulates and thus the further cBN growth as well as the sp^3 / sp^2 ratio.

So, alternating deposition of sub-layers and irradiation directly affects the quality of the entire c-BN films.

Furthermore, calculations were done concerning the mean penetration depth of the photons in the c-BN films and, based on these evaluations of laser-induced temperature fields, experiments have been carried out using proper sub-layer thickness. The influence of the irradiation of the films with photons on the intrinsic shear stresses, which limited the film thickness so far, was investigated and will be presented.

3:10pm **B1-2-6 High Power Impulse Magnetron Sputtering of Niobium in Non-Reactive and Reactive Gas Environments**, ***R.J. Mendelsberg***, *S.H.N. Lim, K.M. Yu, A. Anders (aanders@lbl.gov)*, Lawrence Berkeley National Laboratory

High power impulse magnetron sputtering (HIPIMS) is a young technology whose opportunities, advantages and limitations are currently intensely investigated by a number of groups. Here we selected niobium as one of the most interesting materials since thin films of niobium and niobium compounds are used in a diverse range of applications. Pure niobium films are needed for the next generation of superconducting radio-frequency cavities not made from solid niobium. Niobium nitride is a material sometimes incorporated in hard, wear-resistant coatings and multilayers for its added corrosion resistance benefit. Niobium oxide is an attractive high index material for optical and photonics applications. The complex refractive index can be tuned by going to niobium oxynitride. We report on the dramatic changes of the HIPIMS discharge behavior when going from pure metal mode to reactive deposition, and correlate some plasma and film properties. One could expect that HIPIMS with other transition metals exhibit similar features.

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3:30pm **B1-2-7 Barrier Capability of Reactively Sputtered $Ta_xZr_{1-x}N$ Films with Slight Ta Addition Against Copper Diffusion**, ***J.-L. Ruan***, *J.-L. Huang*, National Cheng Kung University, Taiwan, *H.-H. Lu*, National Chin-Yi University of Technology, Taiwan, *J.-S. Chen*, National Cheng Kung University, Taiwan, *D.-F. Lii (dflii888@csu.edu.tw)*, Cheng Shiu University, Taiwan

The $Ta_xZr_{1-x}N$ films were prepared by reactive magnetron sputtering and the concentration of zirconium and tantalum was regulated by controlling the power of the sputtering guns. A sputter-prepared Cu (100 nm)/$Ta_xZr_{1-x}N$ (5 nm)/SiO_2 (100 nm)/Si stacked structure was fabricated for the evaluation of diffusion barrier performance of $Ta_xZr_{1-x}N$ films. The composition, microstructure, resistivity and diffusion barrier properties of $Ta_xZr_{1-x}N$ films were studied by x-ray diffraction, electron probe microanalyzer, Auger electron spectroscopy, and four point probe method. Results indicated that the slight addition of Ta (3.5 at. %) could effectively further decrease the electrical resistivity of films to a value of 78 μΩ-cm compared with pure ZrN films due to the extra d valence electron of Ta comparing with Zr. Auger electron spectroscopy and sheet resistance measurements showed that the slight incorporation of Ta (3.5 at. %) into the ZrN films significantly improved the barrier performance against Cu diffusion. In addition, the $Ta_xZr_{1-x}N$ films with 3.5 at. % of Ta could be successfully used as a diffusion barrier layer between Cu and SiO_2 even up to the high temperature of 800°C for 5 minutes in a vacuum, while the ZrN films failed at the same temperature.