Tuesday Morning, April 25, 2017

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

PVD Coatings and Technologies

Moderators: Joerg Vetter, Oerlikon Balzers Coating Germany GmbH, Jyh-Ming Ting, National Cheng Kung University

8:00am B1-3-1 Synthesis and Applications of High-precision Thin Film Multilayers, Andreas Leson, S Braun, P Gawlitza, C Gruhne, A Kubec, M Menzel, Fraunhofer Institute for Material and Beam Technology, Germany INVITED

Nowadays thin film coating technologies like magnetron or ion beam sputter deposition are successfully used for the fabrication of extremely precise and smooth nanometer thin films, which are needed for optical coatings especially in the short wavelength range. In most of the applications coatings with thickness gradients on curved substrates are needed. Using optimized coating parameters thickness deviations of only a few picometers from the target profile can be obtained. Another important parameter for the performance of the coatings is roughness. Recent improvements of the deposition process resulted in HSFR values of < 0.1 nm rms (HSRF = high spatial frequency roughness, usually measured with atomic force microscopy).

One of the current main drivers for the development of nanometer multilayers is extreme ultraviolet lithography. For this application Mo/Si multilayers are needed that have to have highest possible reflectances, precise thickness distributions, low film stress as well as high long-term and thermal stability. With dedicated improvements of our coating processes and by applying diffusion barrier layers we have succeeded to increase the EUV reflectance to world record values of > 70.5 % (photon wavelength I = 13.5 nm, incidence angle a = 5 degree). Additionally, the amount of diffuse scattered EUV light could be reduced by decreasing the roughness of the coatings down to Rq < 0.1 nm rms. In addition to Mo/Si multilayers numerous other material combinations of absorber (Ni, Cr, Mo, La, W) and spacer (B4C, C, Si, Sc) layers have been developed during the last years and applied for mirrors and monochromators.

Nanometer multilayers are not only used as reflectors for X-rays. It is also possible to use depth-graded multilayers as diffractive elements. In this case multilayer lamellas have to be fabricated and the individual layer thicknesses have to be chosen according to the zone plate law of Fresnel zone plates. Such kind of diffractive optics is called multilayer Laue lens (MLL). MLLs are promising complementary optics to zone plates particularly in the hard X-ray range where the small aspect ratios of zone plates limit the efficiency. One of the main challenges with the coating of MLL is that several thousand of nanometer layers are necessary in order to obtain lenses with apertures in the range of 20...100 μ m. Only if all these layers have the right thickness, the diffraction at the artificial grating results in efficient X-ray focusing. With the latest developments MLLs with apertures of 50 μ m x 50 μ m have been developed that show X-ray spot sizes in the range of 30 nm x 30 nm at a photon energy of E = 10.5 keV.

8:40am B1-3-3 Influences of Frequency and Duty Cycle on the Mechanical Properties of TiCrBN Thin Films Grown by a Hybrid Superimposed High Power Impulse Magnetron Sputtering and Radio Frequency sputtering technique, *ChiYu Lu*, *J Lee*, *W Diyatmika*, Ming Chi University of Technology, Taiwan

The high power impulse magnetron sputtering (HIPIMS) technique has been studied intensively due to its extremely high peak power density to grow thin films with dense microstructure and excellent mechanical properties. Lots of efforts have been made to improve the low deposition rate of HIPIMS technique. In this study, a hybrid coating system consisting of a radio frequency power supply and a superimposed HIPIMS system was used to deposit the TiCrBN coatings with higher deposition rate. The phase of each coating was studied by means of the X-ray diffractometer. The microstructures of thin films were examined by the field-emission scanning electron microscopy. Atomic force microscopy was used to characterize the surface morphology. The nanoindentation and scratch tests were used to evaluate the hardness and adhesion properties of thin films, respectively. It can be found that the deposition rate increased greatly due to the superimposed module and also the addition of RF sputtering. Influences of the frequency and the duty cycle of HIPIMS on the microstructure, chemical composition and mechanical properties were studied in this work.

9:00am **B1-3-4 Controllably Manipulating Adatom Mobility during PVD Deposition through Surface Acoustic Waves**, *Piyush Shah*, *A Reed*, *A Waite*, *B Howe*, *M McConney*, Air Force Research Laboratory, USA

In this work we explore the ability to controllably manipulate adatom mobility in a spatially defined anisotropic way using standard physical vapor deposition (PVD) technique. Here we investigate the nucleation, growth, and microstructural evolution of PVD-based thin film growth under the influence of electrically induced surface acoustic waves (E-SAWs). Thin films made from classical and next generation resilient plasmonic materials are deposited on SAW grade lithium niobate substrate with inter-digitated electrode pattern to generate SAWs. Increase in adatom mobility and surface diffusion is primarily attributed to SAW-adatom coupling during the early stage nucleation process. As a result, we demonstrate improved crystallinity in thin films deposited under the influence of E-SAWs. Films are characterized using XRD, SEM and AFM techniques. We believe this technique of growing films is complementary to elevating the substrate temperature during deposition in a conventional PVD deposition system. These results are very promising for future work in the area of thin films deposition onto substrates coupled with SAWs.

9:20am B1-3-5 Effects of Processing Parameters on the Fabrication of TiCrSiN Thin Films Deposited by a Hybrid HiPIMS and RF Sputtering System, *Yi-Xiang Qiu, Y Yang*, National Taipei University of Technology, Taiwan; *J Lee*, Ming Chi University of Technology, Taiwan

High power impulse magnetron sputtering (HiPIMS) is a relatively new physical vapor deposition technology, which is characterized for its ultrahigh peak current, peak power density and high-density plasma to achieve unique thin film mechanical properties, such as high hardness, good adhesion and good wear resistance. In this work, a radio frequency (RF) and HiPIMS hybrid coating system was used to deposit TiCrSiN coatings with higher deposition rate. The TiCr and Si targets were connected to the HiPIMS and RF power supplies, respectively. The phase of each coating was studied by means of the X-ray diffractometer. The microstructures of thin films were examined by the field-emission scanning electron microscopy. Atomic force microscopy was used to characterize the surface morphology. The nanoindentation and scratch tests were used to evaluate the hardness and adhesion properties of thin films, respectively. The pin-on-disk wear test was employed to study the tribological property of coating. Effects of processing parameters, including duty cycle and pulse frequency of HiPIMS power on the microstructure, mechanical and tribological properties of TiCrSiN coatings were further discussed in this work.

9:40am **B1-3-6 Exploring the High-temperature Hardness of Nanocrystalline W-Ti Coatings**, *Yip-Wah Chung*, *C Gross*, *X He*, Northwestern University, USA

Nanocrystalline coatings are harder than their bulk or microcrystalline counterparts because of the Hall-Petch effect. Their hardness tends to degrade with increasing temperature due to grain coarsening. Previous studies by Weissmüller, Kirchheim, and Schuh suggest that introduction of proper alloying elements can provide thermodynamic stabilization of the grain size, even at elevated temperatures. In this work, we have synthesized a series of W-Ti coatings by magnetron co-sputter-deposition of W and Ti. The coatings range in thickness from 450 to 525 nm and in composition from 85W-15Ti to 67W-33Ti. These coatings show high hardness at room temperature, with peak hardness values near 28 GPa. The hardness values are almost unchanged after 1h exposure at 600°C, remaining near 27 GPa. These results suggest the validity of a general strategy for designing thermally stable hard coatings that can maintain high hardness even after extended exposure to elevated temperatures.

10:00am B1-3-7 Growth Mechanism of Sputter Deposited Self-assembled Alternating Layered Metal Containing Hydrogenated Amorphous Carbon Film, J Ting, National Cheng Kung University, Taiwan; *Wan-Yu Wu*, Da-Yeh University, Taiwan

The growth and characteristics of metal containing amorphous hydrogenated carbon thin films (a-C:H/Me) were studied in this research. a-C:H/Me thin films were synthesized using one single target, a rotating but not revolving substrate, and constant feed gas compositions in a conventional reactive sputter deposition chamber. The metals used include AI, Si, Cu, Pt, Fe and Ni. Various mixtures of methane and argon having fixed total flow rates were used as the feeds tocks. A number of growth parameters, including methane concentration, working pressure, electrode distance, dc power, substrate bias, and substrate temperature were used. Among the three distinct structures formed, self-assembled, alternating nano-layered structure is of particular interest. In order to understand the formation of these three distinct structures, correlations were first made

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among the deposition rate, the composition, the crystallinity, the surface chemistry, and the microstructure of a-C:H/Me thin films. It was found that the self-assembled, alternating nano-layered structures can be obtained under controlled growth parameters for selected metals. A growth mechanism based on the considerations of clustering of carbon and metal, segregation of carbon, catalytic effects of metal, formation of carbide, energy of adatoms, and surface diffusion of metal and carbon, has been developed. Further data analysis was also performed to verify the validity of the mechanism.

10:20am **B1-3-8 Phase Formation in Sputter Deposited Tantalum Coatings,** *Glen West, M Ratova, P Kelly,* Manchester Metropolitan University, UK

Tantalum is a refractory metal with many applications. In bulk form, Ta generally has a bcc crystal structure, referred to as the α -phase. However, in thin film form, the metastable β -phase commonly forms. The β -phase is harder, but also more brittle than the α -phase and has other less desirable physical properties.

Control of phase formation in Ta films has been the subject of a number of recent studies. Several process/operating parameters have been put forward as influencing the final structure of the films, including choice of substrate material, use of a metallic interlayer between the substrate and film, substrate bias voltage, operating pressure, substrate temperature, mass of bombarding species and type of power delivery mode. These parameters can essentially be divided into those which influence the nucleation and growth of the film (e.g. substrate parameters) and those which control the energy delivered to the growing film (e.g. pressure, power delivery mode, temperature, etc.).

Of the thin film deposition processes available, magnetron sputtering is the process of choice for many commercial applications. It is a highly versatile, scalable technique capable of depositing high quality coatings of a very wide range of materials. The design of the magnetron(s) and the choice of power delivery mode allow a very wide process window to be readily explored to enable the determination of optimised deposition conditions for specific film/substrate combinations. A range of deposition parameters, magnetic field designs, power delivery modes, and biasing regimes, combined with a suite of surface analysis techniques has been utilized in a detailed study of the phase formation in tantalum films. This has enabled the conditions necessary for the deposition of α -tantalum to be identified.

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