

Friday Morning, May 26, 2023

Hard Coatings and Vapor Deposition Technologies

Room Town & Country C - Session B1-3-FrM

PVD Coatings and Technologies III

Moderators: Dr. Christian Kalscheuer, RWTH Aachen University, Germany, Dr. Vladimir Pankov, National Research Council of Canada

8:00am B1-3-FrM-1 Effect of Wettability Modification of Ti-Al-Based Thin Films on Heat Transfer Exchange During Water Drop Cooling, Alexis Carlos Garcia Wong, G. Marcos, Institut Jean Lamour - Université de Lorraine, France; G. Castanet, O. Caballina, F. Lemoine, Laboratoire d'Energétique et de Mécanique Théorique et Appliquée, France; J. Pierson, T. Czerwicz, Institut Jean Lamour - Université de Lorraine, France

Heat production constitutes about 70% of the world's primary energy consumption. Better thermal management of industrial processes would be a step toward the energy transition process. The development of sufficiently compact and affordable extractors and heat exchangers is an urgent industrial challenge. Spray cooling on a hot surface is one of the most effective heat extraction techniques. When a droplet is in contact with a wall, the liquid heats up and boils, extracting a high energy amount during this process. However, no dedicated exchange surfaces have yet been designed to exploit the full potential of this cooling technique. The study of heat transfers associated with the impact of a water drop on hot surfaces with controlled properties (wettability, roughness) is essential to achieve this goal. We used time-resolved infrared thermography (TR-IRT) to measure the contact temperature and determine the heat extracted by water drops. For this purpose, the upper surface of a sapphire substrate (transparent in the IR) must be covered with a highly IR-emissive layer. The method requires an opaque surface, with a micrometer thickness to neglect the thermal resistance of the deposit, and it must also withstand the impact of the drops while being thermally stable. The temperature reduction produced by the drop impact is observed through the sapphire without being disturbed by the drop presence [1].

We revealed that opaque TiAlN and TiAl thin films are an attractive choice for this application. All the studied films were deposited by magnetron sputtering. Scanning electron microscopy, optical profilometry, and X-ray diffraction were performed for morphological and structural characterizations of the films. In addition, the wettability of the films was investigated by contact angle measurements and the emissivity by Fourier transform infrared spectroscopy. The effect of different wettability of the Ti-Al-based films on the heat transfer during droplet impact at different temperatures from 80 to 300°C was analyzed by TR-IRT. The heat flux and energy extracted by the drop projection onto hot walls of different wettability were determined. Superhydrophilic TiAlN displays better results than hydrophobic TiAl alloys due to the larger contact area between the drop and the surface. Biphilic surfaces with spatially variable wetting properties are supposed to improve the heat transfer of the boiling system [2]. We are currently exploring the effect of different sizes of hydrophobic patches in a hydrophilic matrix to assess the impacts on the cooling efficiency.

[1] G. Castanet *et al.*, *Phys. Fluids*, 30, 12, (2018).

[2] H. Cho *et al.*, *Nat. Rev. Mater.*, 2, 2, (2016).

8:20am B1-3-FrM-2 Rf-Bias Assisted, Combinatorial Sputtering of Conductive (TiZr)N Hard Coatings on Insulating Substrates, Kerstin Thorwarth, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland; M. Watroba, Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland; J. Sommerhaeuser, S. Zhuk, J. Patidar, A. Wiecek, S. Siol, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

Ternary transition metal nitrides are promising materials for many applications as they offer advantages in microhardness and higher oxidation resistance compared to binary counterparts. A common challenge in the deposition of these materials is oxygen contamination during the sputtering process. This oxygen contamination adversely affects the functional properties of the coatings, especially their hardness and electrical properties. Here we present a practical approach to grow virtually oxygen-free (Ti, Zr)N coatings, even on insulating substrates.

To cover the completely compositional range of (Ti, Zr)N we employ combinatorial reactive co-sputtering from Ti and Zr targets in Ar/N₂ atmosphere. The depositions are carried out with or without applying a low-power RF substrate bias to the substrate holder with the goal to reduce the oxygen contamination the growing film. The compositional gradients are complemented by orthogonal deposition temperature

gradients to cover entire regions of the synthesis diagram in individual depositions. Automated mapping characterization (XPS, XRF, XRD, 4 point probe) is used to evaluate the structure and composition as well as electrical properties of the libraries. Nano-indentation mapping for evaluation of the mechanical properties is performed on selected combinatorial libraries. For selected parameter sets, UHV-transfer XPS is performed. Comparison of oxygen contamination in UHV and ambient conditions linked to changes in the films' microstructure depending on the synthesis conditions.

The structural analyses indicate solid solution formation over the entire compositional range as described by Vegard's Law. The oxygen contamination of the films is evaluated using combinatorial sputter-depth profiles. Irrespective of the composition of the films, the RF-bias leads to a dramatic reduction of the oxygen-contamination, which is reflected in a significant improvement in the films' conductivity as well as hardness. In addition, the RF-bias leads to a denser microstructure and improved oxidation resistance in ambient conditions as evidenced by XPS oxidation studies. The approach presented here provides a practical route to synthesize nitrides with improved phase purity that can be applied to many different material systems.

8:40am B1-3-FrM-3 The Microstructure and Properties of Highly (111)-Oriented Nano-Twinned Cu-Ag Thin Film Prepared by DC Sputtering System, Ko-Chieh Hsueh, National Tsing Hua University, Taiwan; J. Lee, F. Ouyang, National Tsing Hua University, Taiwan

The Moore's Law pushes the size of electronic devices continue to shrink. Thus, the traditional solder joint technology cannot be used as interconnect any more. To solve this problem, metal-to-metal direct bonding is regarded as the promising replacement technique. In this study, we used the Direct Current sputtering technology to deposit highly (111)-oriented nano-twinned Cu-Ag thin films with doped Cu concentration from 2.3 at% to 6.6 at%. The results show that higher Cu concentration in Ag facilitates the reduction of fine nanocrystalline region and enhancement of nanotwinned region. The X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) analysis revealed that the (111) texture is up to 99% that offered the fastest diffusivity among different crystalline planes of Cu and Ag, effectively shortening bonding time. Due to increased nucleation sites, the grain sizes of films decreased with higher Cu concentration. The surfaces roughness of films is below 8 nm, providing an excellent property for three dimensions-Integrated Circuit (3D-IC) metal to metal direct bonding. The highest hardness of nanotwinned Cu-Ag thin films can reach 2.87 GPa, but the resistivity is still low. The correspond mechanisms will be discussed in this talk. The finding in this study demonstrate nanotwinned Ag-Cu thin film is a promising new material as a substitute for traditional solder in 3D-IC packaging.

9:00am B1-3-FrM-4 High Gain CMOS inverter with Vertically-Stacked Hybrid PVD-Formed IWZO TFT and Monolithic FinFET, Yu-Hsin Chen, National Tsing Hua University, Taiwan; D. Ruan, Fuzhou University, China; K. Chang-Liao, National Tsing Hua University, Taiwan

The monolithic three dimension integrated circuit (3D-IC) is promising technology in overcoming the area limitation in realizing the more than Moore's law. However, it is hard to fabricate the second layer high performance device without single crystal Si material. Recently, thin film transistor (TFT) is applicable to the back-end-of-line due to its low thermal budget fabrication process. In this work, a vertically stacked complementary p-type Si FinFET and n-type amorphous indium tungsten zinc oxide TFT, which have symmetric electrical characteristics, have been demonstrated. The vertically-stacked hybrid inverter could be operated at the low voltage and exhibit the high voltage gain. The excellent performance exhibited in the proposed hybrid complementary FinFET/TFT based inverter has the great potential for monolithic 3D-IC circuits in the future.

9:20am B1-3-FrM-5 Development of New Magnetron Sputter Deposition Processes for Inertial Confinement Fusion Targets, S. O. Kucheyev, S. Shin, L. Bayu Aji, G. Taylor, A. Engwall, J. Merlo, L. Sohngen, Lawrence Livermore National Laboratory, USA; J. Bae, General Atomics, USA

The demonstration of fusion ignition at Lawrence Livermore National Laboratory in December 2022 has opened up new opportunities for fundamental and applied research. All inertial confinement fusion (ICF) experiments require laser targets. Magnetron sputter deposition is an enabling technology for laser target fabrication. Solutions are readily available for the deposition of most sub-micron-thick elemental films on planar substrates. However, major challenges still remain for the development of robust deposition processes in regimes of ultrathick (over about 10 microns) coatings and non-planar substrates. These challenging

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deposition regimes are directly relevant to laser target applications, including both spherocylindrical hohlraums and spherical ablators for ICF targets. Understanding underlying physical mechanisms for a specific material system is crucial for process development, given the overall complexity of the deposition process, its nonlinear dependence on deposition parameters, and a very large process space, often precluding conventional process optimization approaches. Here, we describe our approach to developing new deposition processes with examples from our ongoing studies of glassy boron carbide ceramics for next generation ICF ablators and non-equilibrium gold-tantalum alloys for hohlraums for magnetized ICF schemes. Emphasis is given to two major challenges of ultrathick coatings related to achieving process stability and reducing residual stress.

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