

## Live Session

### Room Live - Session LI-ThM1

#### Hard Coatings and Vapor Deposition Technologies Live Session

**Moderators:** Dr. Tiberiu Minea, Université Paris-Sud, France, Dr. Farwah Nahif, eifeler-Vacotec GmbH, Germany

10:00am **LI-ThM1-1 General Chair's Welcome, Chris Muratore (cmuratore1@udayton.edu)**, University of Dayton, USA; **F. Nahif**, voestalpine eifeler-Vacotec GmbH, Düsseldorf, Germany

Welcome to the ICMCTF 2021 Virtual Conference! We hope you will enjoy the Exhibition Keynote Lecture and our invited talks!

10:15am **LI-ThM1-2 Exhibition Keynote Lecture: Carbon based Coatings in Industrial Scale for Sustainable Surface Solutions, Jörg Vetter (joerg.vetter@oerlikon.com)**, Oerlikon Balzers Coating Germany GmbH, Bergisch Gladbach, Germany **INVITED**

The attractive properties of carbon based hard coatings include high hardness, chemical inertness, tuneable electrical resistivity and optical properties, biocompatibility, excellent tribological behaviour in many engineering applications, show a high potential for use in anti-corrosion and electrochemical applications, and have a potential for sensory applications and for fuel cell applications. The main coatings in use are amorphous carbon coatings consisting of a disordered network of carbon atoms with sp<sup>2</sup> and sp<sup>3</sup> coordinated C-C bonds. The family of amorphous carbon films is called "diamond like carbon": DLC. However also diamond coatings with nearly 100% sp<sup>3</sup> carbon bond hybridization are in application. Oerlikon Balzers develops and applies industrial solutions to deposit amorphous carbon coatings based on PACVD processes, vacuum arc evaporation (direct and filtered), magnetron sputtering including newer developments of HiPIMS (e.g. S3p®). The diamond coatings are deposited by a special PACVD process or by a hot filament process. Tailored batch coating systems with different sizes are used both for large scale and small lot applications. Selected industrial coating systems will be briefly described (a-C:H:Me, a-C:H, a-C:H:X, a-C, ta-C, diamond). Typical dedicated applications of the carbon based coatings and diamond coatings including surface solutions for green car developments (e.g. ICEV, HEV, FCEV) and green manufacturing are presented.

11:15am **LI-ThM1-6 Impact of Nitrogen Deficiency on the Phase Transformation of (Ti,Al)N Thin Films at Elevated Temperatures, Isabella Schramm (isabella.schramm@sandvik.com)**, Sandvik Coromant R&D, Sweden **INVITED**

The work presented here contributes to the understanding of the effect of nitrogen vacancies (nitrogen deficiency) on the phase transformations of cathodic arc-evaporated cubic (Ti,Al)N thin films at elevated temperatures. It experimentally confirms theoretical predictions by Alling *et al.* on the effect of N vacancies on the decomposition pathway of c-(Ti,Al)N<sub>y</sub> (y < 1) [1]. For the low/medium N deficient alloys (1 > y > 0.74), special attention is paid to the evolution of the beneficial spinodal decomposition into c-TiN and c-AlN, the detrimental formation of wurtzite AlN, and the potential application as hard coating in cutting tools [2, 3]. For the highly nitrogen-deficient solid solution cubic (Ti<sub>1-x</sub>Al<sub>x</sub>)N<sub>y</sub> (0.58 ≥ y ≥ 0.40) alloys, the decomposition pathway was investigated with an emphasis on the formation of Ti<sub>4</sub>AlN<sub>3</sub> (MAX phase) in thin films via solid state reactions [4].

Solid solution cubic(Ti<sub>0.52</sub>Al<sub>0.48</sub>)N<sub>y</sub> thin films with low/medium N content (y = 0.93 to 0.75) show a substantial improvement of the thermal stability with lower nitrogen content. This results in a significant delay in the spinodal decomposition when increasing the amount of N vacancies, and consequently in a 300 °C increase in the age hardening temperature maximum [2, 3]. Highly N-deficient (Ti<sub>1-x</sub>Al<sub>x</sub>)N<sub>y</sub> thin films (y = 0.58 to 0.40) showed formation of c-TiN, w-AlN, and additional phases such as intermetallic and MAX phases (Ti<sub>4</sub>AlN<sub>3</sub>, Ti<sub>2</sub>AlN, Al<sub>5</sub>Ti<sub>2</sub>, and Al<sub>3</sub>Ti) during annealing. This is the first study showing the formation of Ti<sub>4</sub>AlN<sub>3</sub> (MAX phase) in thin films via solid state reaction in nitrogen deficient cubic(Ti<sub>1-x</sub>Al<sub>x</sub>)N<sub>y</sub> alloys. A transformation mechanism from Ti<sub>2</sub>AlN to Ti<sub>4</sub>AlN<sub>3</sub> via intercalation of Al layers for N layers along Ti<sub>2</sub>AlN basal plane is proposed [4].

[1] B. Alling *et al.*, *Phys. Rev. B* **75** (2007) 45123.

[2] I. C. Schramm *et al.*, *Acta Mater.* **119** (2016) 218.

[3] I. C. Schramm *et al.*, *Surf. Coatings Technol.* **330** (2017) 77.

[4] I. C. Schramm *et al.*, *Acta Mater.* **129** (2017) 268.

11:45am **LI-ThM1-8 Air-based Sputtering Deposition of Gradient Oxynitride Coatings, Fu-Hsing Lu (fhl@nchu.edu.tw)**, National Chung Hsing University, Taiwan; **Y. Liou, Y. Lee, M. Chan**, National Chung-Hsing University, Taiwan **INVITED**

Oxynitride coatings exhibiting characteristics mainly between nitride and oxide coatings have many technological applications. In the literature, oxynitride coatings have often been prepared by using N<sub>2</sub> and O<sub>2</sub> as reactive gases in sputtering. We previously reported that air could be employed to replace conventionally used N<sub>2</sub>/O<sub>2</sub> in producing oxynitride coatings with various O/N compositions. Hence, high vacuum is not required, which can reduce substantially the processing time and cost. Gradient coatings with different O/N compositions were further made by sequentially changing the air/Ar ratio during sputtering in this research. Gradient TiN<sub>x</sub>O<sub>y</sub> and TaN<sub>x</sub>O<sub>y</sub> thin films have been selected as model systems. The gradient coatings were designed with the structures varying from crystalline to amorphous, corresponding to the coatings with conductive, semiconductive, to insulating and opaque, translucent, to transparent behaviors. High efficient selective solar absorbers and electrochemical photocurrents were achieved by employing the gradient oxynitride coatings. The facilely air-based sputtering deposition of gradient oxynitride coatings may bring in more technological applications.

12:15pm **LI-ThM1-10 Optimizing Ionization and Deposition Rate in High Power Impulse Magnetron Sputtering, Daniel Lundin (daniel.lundin@liu.se)**, Linköping University, Sweden **INVITED**

Quantification and control of the fraction of ionization of the sputtered species are crucial in magnetron sputtering. This is especially important in high power impulse magnetron sputtering (HiPIMS), since the presence of significant amounts of material ions during film growth has resulted in very smooth and dense films, control over their phase composition and microstructure, as well as enhanced mechanical and electrical properties. Yet proper definitions of the various concepts of ionization are still lacking. In this contribution, we distinguish between three approaches to describe the degree (or fraction) of ionization: the ionized flux fraction  $F_{flux}$ , the ionized density fraction  $F_{density}$ , and the fraction  $\alpha$  of the sputtered metal atoms that become ionized in the plasma (sometimes referred to as probability of ionization). By studying a reference HiPIMS discharge with a titanium target, we show how to extract absolute values of these three parameters and how they vary with peak discharge current. Using a simple model, we also identify the physical mechanisms that determine  $F_{flux}$ ,  $F_{density}$ , and  $\alpha$  as well as how these three concepts of ionization are related. This analysis identifies ion back-attraction  $\beta$  as a key parameter in the ion balance between the target and the bulk plasma and finally explains why a high ionization probability does not necessarily lead to an equally high ionized flux fraction or ionized density fraction. In the second part of this contribution we seek to decrease ion back-attraction by exploring the effect of magnetic field strength  $|B|$  and geometry (degree of balancing) on the deposition rate and ionized flux fraction in dc magnetron sputtering and HiPIMS when depositing titanium. By investigating HiPIMS discharges operated in fixed voltage mode as well as fixed peak current mode in seven different magnetic field configurations, we relate the measured quantities, the deposition rate and ionized flux fraction, to the ionization probability  $\alpha$  and the back-attraction probability  $\beta$  of the sputtered species and show that it is indeed possible to simultaneously increase both deposition rate (up to 40%) and ionized flux fraction (up to 50%).

12:45pm **LI-ThM1-12 Closing Remarks & Thank You's, Tiberiu Minea (tiberiu.minea@u-psud.fr)**, Université Paris-Saclay, France

Thank you for attending today's session! Please just us for the Post-Session Discussion and additional Q&A opportunities! We will see you tomorrow!

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