

Protective and High-temperature Coatings Room Town & Country C - Session MA4-1-MoA

Boron-containing Coatings I

Moderator: Martin Dahlgqvist, Linköping University, Sweden

2:40pm **MA4-1-MoA-4 Charge Trapping Behavior in BN Films Fabricated by a Reactive Plasma-Assisted Coating Technique and Their Design Strategies**, *Koji Eriguchi [eriguchi.koji.8e@kyoto-u.ac.jp]*, Kyoto University, Japan
INVITED

Boron nitride (BN) possesses highly desirable properties for a wide variety of industrial applications [1]. Its properties strongly depend on its microscopic structure: sp^2 -bonded hexagonal (h-BN) and sp^3 -bonded cubic (c-BN). For example, h-BN films are expected to be superior dielectric materials for electronic devices owing to their high dielectric breakdown field [2], whereas c-BN films have attracted considerable attention because of their high hardness [3]. Historically, using plasma-based technologies, these microscopic structures have been controlled predominantly by the energy of incident ions and the fluxes of B and other species [4]. However, crucial issues remain to be solved—namely, the degradation of dielectric breakdown lifetime for h-BN films and delamination due to residual stress in c-BN films. A fundamental understanding of the various degradation mechanisms in BN films is therefore required.

In this study, we formed BN films and their stacked structures with various bonding phases on Si substrates using a reactive plasma-assisted coating (RePAC) method [5]. After confirming their bonding networks by Fourier-transform infrared spectroscopy and optical properties by spectroscopic ellipsometry [6], we investigated the dielectric degradation of h-BN and the delamination behavior of c-BN in terms of charge trapping dynamics using Al/BN/Si devices. Characteristic charge trapping behaviors during time-dependent dielectric breakdown measurements were enhanced by the bombardment of higher-energy ions and the incorporation of impurities during h-BN film formation [7]. The presence of trapped charges was identified at the c-BN/Si interface and even within stacked BN films (h-/c-BN or c-/h-BN) [8]. In addition, film delamination was found to occur preferentially at the stacked c-/h-BN interface. Moreover, we found that time-dependent film delamination was closely correlated with the trapped charge density. These findings indicate that controlling charge trapping behaviors is key to improving the properties of BN films for various industrial applications.

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- [1] I. Levchenko et al., *Nat. Commun.* **9**, 879 (2018).
- [2] Y. Hattori et al., *Phys. Rev. B* **97**, 045425 (2018).
- [3] V. L. Solozhenko et al., *J. Appl. Phys.* **126**, 075107 (2019).
- [4] C. B. Samantaray and R. N. Singh: *Int. Mater. Rev.* **50**, 313 (2005).
- [5] T. Matsuda et al., *Jpn. J. Appl. Phys.* **61**, S11014 (2022).
- [6] T. Hamano et al., *Appl. Phys. Lett.* **120**, 031904 (2022).
- [7] Y. Asamoto et al., *J. Appl. Phys.* **137**, 105301 (2025).
- [8] N. Oguchi et al., to be presented at the 2025 Dry Process Symposium.

3:20pm **MA4-1-MoA-6 Development of TiB_2 :h-BN:a-C Based Nanocomposite Coatings with Enhanced Wear and Corrosion Resistance for Turbojet and Gas Turbine Components**, *Gokhan Gulten [gokhangulden@atauni.edu.tr]*, Banu Yaylali, Mustafa Yesilyurt, Ali Emre, Yasar Totik, Atatürk University, Turkey; Justyna Kulczyk-Malecka, Peter Kelly, Manchester Metropolitan University, UK; Ihsan Efeoglu, Atatürk University, Turkey

Turbojet and gas turbine engines operate under severe thermo-mechanical and chemically aggressive conditions where simultaneous control of friction, wear, and corrosion is essential. This study reports the design and synthesis of a solid-lubricating nanocomposite architecture based on TiB_2 :h-BN:a-C deposited by closed-field unbalanced magnetron sputtering (CFUBMS) driven by a hybrid HiPIMS + pulsed-DC power setup. The coating concept employs the synergistic combination of hard TiB_2 domains (load-bearing), hexagonal BN (lamellar solid lubricant, thermal stability), and amorphous carbon (low shear, transfer-film formation). To promote enhanced substrate adhesion and gradient stress accommodation on aerospace alloys (Inconel 718 and Ti-6Al-4V), a thin Cr adhesion layer and a CrN transition layer are incorporated. A Taguchi L9 experimental design is employed to map the influence of TiB_2 target voltage, N_2 flow, duty cycle,

and working pressure on structure–property relationships. Comprehensive characterization includes XRD, Raman and XPS, SEM cross-sections, nanoindentation, scratch testing, and pin-on-disk tribometry at room and elevated temperatures. Electrochemical performance is assessed by potentiodynamic polarization and EIS to quantify corrosion resistance. The hybrid power delivery enhances ionization and adatom mobility, producing dense microstructures and superior adhesion. Process–structure–property correlations reveal reduced friction, improved wear resistance, and enhanced corrosion protection, establishing TiB_2 :h-BN:a-C coatings as promising candidates for extending component life and reducing maintenance in advanced propulsion systems.

4:00pm **MA4-1-MoA-8 Energy Efficiency in Pulsed-DC Powder-Pack Boriding: A Sustainable Approach to Surface Hardening of Metallic Materials**, *Ivan E Campos Silva [icampos@ipn.mx]*, Instituto Politecnico Nacional, Mexico
INVITED

Boriding has emerged as an efficient thermochemical treatment to enhance the wear and corrosion resistance of metallic materials. The resulting boride layer, characterized by its exceptional hardness and outstanding thermal and chemical stability, outperforms nitrided, carburized, and PVD-coated surfaces. However, conventional powder-pack boriding still faces critical challenges (mainly the need for long treatment durations and high temperatures ($\geq 850^\circ C$)) to achieve protective boride layers (50–75 μm thick). These conditions result in high energy consumption and increased production costs, limiting its industrial sustainability.

The pulsed-DC powder-pack boriding offers a sustainable alternative to conventional method by drastically reducing energy usage and processing time. This technique employs an electric field generated by a power source and a polarity-switching device connected to electrodes immersed in a powder mixture together with the metallic specimen. Remarkably, successful treatments have been performed at lower temperatures (600–750 $^\circ C$) and shorter durations (up to 1.5 h), producing boride layers with excellent wear and friction performance—an unprecedented advancement in the field of solid boriding media and aligned with the principles of sustainable manufacturing.

4:40pm **MA4-1-MoA-10 Investigation of Technologically Driven Compositional and Structural Changes, Mechanical Properties, and Alloying of Transition Metal Diboride Thin Films**, *Viktor Sraba [viktor.sraba@liu.se]*, Linköping University, Sweden, Slovakia
INVITED

This presentation focuses on the investigation of technologically driven compositional and structural changes, mechanical properties, and on alloying of transition metal diboride (TMB_2) thin films. The films were synthesized using state-of-the-art technological approaches, and modern analytical methods, including scanning transmission electron microscopy (STEM) and computational approaches based on density functional theory (DFT), were used for a complex characterization.

TMB_2 thin films grown by direct current magnetron sputtering (DCMS) are an exciting group of nanocomposite materials with excellent mechanical properties, making them a potential candidate for hard coatings in the cutting tools industry. But there are various structural drawbacks that limit their high-temperature application potential, brittle character, and low oxidation resistance. This work provides several pathways to mitigate these drawbacks.

The main topic is understanding the influence of deposition parameters of high power impulse magnetron sputtering (HiPIMS) on the growth of TMB_2 films. One of the benefits of HiPIMS is a high degree of ionization of the sputtered species, however accompanied by a lower deposition rate. These ions can then be attracted towards the growing film using synchronized bias. This high-energy bombardment results in compositional and structural changes in the film. Advanced hybrid HiPIMS/DCMS co-deposition combines a high degree of ionization provided by HiPIMS and a high deposition rate of DCMS.

In the first part, a study of cross-ionization in hybrid HiPIMS/DCMS co-deposition configuration was performed. It was demonstrated that the cross-ionization of the DCMS flux by HiPIMS is influenced by a relative ratio of two crucial parameters - ionization potentials and masses of the sputtered elements. In the second experimental part of the work, high ionization of the sputtered species during HiPIMS was used to suppress the formation of the amorphous boron-rich tissue phase of understoichiometric ZrB_x films while improving mechanical properties and oxidation resistance in the process. The next part of the research focused on the bombardment by the HiPIMS-generated W-ions showed significant densification of the TiB_2 films' structure and improved their mechanical

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properties, eliminating the need for external heating of the substrates during deposition. Lastly, on the basis of DFT predictions, alloying of TaB₂ and ZrB₂ films by aluminum and niobium, respectively, using conventional DCMS or hybrid DCMS/HiPIMS co-deposition, showed possible pathways to maintain or even improve mechanical properties at elevated temperatures and improvement in ductility.

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