## Thursday Afternoon, May 25, 2023

Coatings for Use at High Temperatures

**Room Golden State Ballroom - Session AP-ThP** 

# Coatings for Use at High Temperatures (Symposium A) Poster Session

#### AP-ThP-1 Thermal Stability of Thick $\alpha$ - and $\gamma$ -Al<sub>2</sub>O<sub>3</sub> Coatings Deposited by High Speed PVD, K. Bobzin, Christian Kalscheuer, M. Moebius, P. Hassanzadegan Aghdam, RWTH Aachen University, Germany

Metal oxide coatings such as  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> are state of the art for different high-temperature applications T > 800 °C. However, synthesis of stable  $\alpha$ alumina coatings is limited to chemical vapor deposition (CVD), due to its high formation temperature T >1000 °C. Physical vapor deposition (PVD) can realize deposition of aluminium oxide coatings at lower process temperatures T  $\leq$  600 °C. Nevertheless, metastable or amorphous Al<sub>2</sub>O<sub>3</sub> phases can be formed in this case. This leads to a lower thermal stability and limits the application of these coatings at higher temperature. However, previous studies showed that High Speed PVD can realize the synthesis of cristalline  $\alpha\text{-}$  and  $\gamma\text{-}Al_2O_3$  coatings with high coating thickness of s > 10  $\mu$ m at T  $\approx$  780 °C. In this study the thermal stability of  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> coatings deposited by means of HS-PVD was investigated. High temperature (HT)X-ray diffraction (XRD) measurements were performed in order to study the phase transformations of the coatings as a function of temperature between T = 700 °C to T = 1,200 °C in vacuum and in atmosphere. Moreover, morphology, indentation hardness  $H_{\text{IT}}$  and indentation modulus EIT of the coatings were investigated after HT-XRD. HT nanoindentation (HT-NI) measurements were performed to in-situ analyze the indentation hardness  $H_{IT}$  and indentation modulus  $E_{IT}$  at T  $\approx$  650 °C and the highest device measurement temperature of T<sub>max,NI</sub>≈ 750 °C. The HT-XRD results show that up to a temperature of T = 1,200 °C neither in vacuum nor in atmosphere any phase transformations of coatings are recognizable. This confirms phase stability of the coatings up to this temperature. Moreover, the morphology of the coatings did not show any changes after HT-XRD, indicating a high structural stability. In addition, the morphology did not show any cracks detectable by scanning electron microscopy after HT-XRD. In situ HT-NI measurements showed a reduction of indentation hardness and indentation modulus compared to measurements at room temperature. However, the indentation hardness and indentation modulus after HT-XRD do not vary from the as-deposited state. The results of the conducted research reveal a high thermal stabillity of crystalline  $\alpha$ - and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>coatings deposited by HS-PVD up to T = 1.200 °C. This can extend the application of these coatings to higher tempertaures.

AP-ThP-3 e-Poster Presentation: High-Temperature Stability and Mechanical Properties of Non-Reactive PVD-Synthesized MoSi<sub>2</sub> Coatings, Sophie Richter, A. Bahr, T. Wojcik, Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria; O. Hunold, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; S. Kolozsvári, P. Polcik, Plansee Composite Materials GmbH, Germany; J. Ramm, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; H. Riedl, TU Wien, Institute of Materials Science and Technology, Austria Molybdenum disilicide belongs to the group of refractory transition metal silicides, which are highly attractive as oxidation-resistant materials to be applied in high-temperature regimes (i.e., above 1000 °C). The unique strength of  $MoSi_2$  is based on its high melting temperature and the formation of a highly protective silicon-based oxide scale. In relation to the metallic and covalent bonding nature, MoSi<sub>2</sub> obtains unique mechanical properties, suggesting this disilicide as a promising candidate for future protective coatings.

Within this study, direct current magnetron sputtering (DCMS) as well as high-power pulsed magnetron sputtering (HPPMS) techniques have been deployed to grow MoSi<sub>2</sub> thin films. These coatings were non-reactively deposited in an in-house developed (laboratory-scaled) sputter system using 3" compound targets. The influence of the deposition parameters (e.g., substrate bias potential and deposition temperature) on the phase formation, morphology, chemical composition, and mechanical properties (e.g., hardness and indentation modulus) has been investigated systematically by high-resolution characterization methods such as X-ray diffractometry (XRD), scanning and transmission electron microscopy (SEM and TEM), energy-dispersive X-ray spectroscopy (EDS), as well as nanoindentation. Additionally, micro-cantilever bending tests have been performed to determine the fracture toughness of the as deposited thin films. Furthermore, we employed differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) to investigate the oxidation kinetics.

The formed oxide scales have been analysed for different temperature regimes up to 1500 °C (1 hour in synthetic air). The samples were subjected to long-term oxidation treatments in ambient air at 1200 °C for up to 100 hours. Based on these results, sputter deposited MoSi<sub>2</sub> thin films constitute a promising protective coating material applied for challenging environmental conditions.

**Keywords:** Disilicides; PVD; Protective coatings; Oxidation resistance; Phase stability;

#### AP-ThP-4 Thermal Stability of Atmospheric Pressure Plasma Jet Deposited YSZ Top Coats and Sputtered Al Bond Coats on Inconel 617, Yung-I Chen,

*L. Wang, X. Qiu,* National Taiwan Ocean University, Taiwan In this study, microstructure and oxidation behavior of YSZ-alumina thermal barrier coatings after high-temperature annealing were explored. Ni-based superalloy (Inconel 617) was used as the substrate material. Al bond-coat was deposited by direct current magnetron sputtering. YSZ topcoat were deposited through several iterations of atmospheric pressure plasma jet deposition and rapid thermal annealing at 600°C. Multilayered YSZ/Al coatings were fabricated with various stacking periods. The Al sublayer transformed into a dense Al<sub>2</sub>O<sub>3</sub> layer after annealing. The multilayered YSZ/Al<sub>2</sub>O<sub>3</sub> coatings were further annealed at 1100 °C in ambient air. The YSZ coatings maintained at a tetragonal phase after annealing at 1100 °C for hundreds of hours.

AP-ThP-5 Ti<sub>1-x</sub>Al<sub>x</sub>N PVD Coatings in Hot-Corrosion Environments, O. Hudak, Rainer Hahn, A. Scheiber, Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria; L. Shang, O. Hunold, Oerlikon Balzers, Oerlikon Surface Solutions AG, Liechtenstein; S. Kolozsvari, Plansee Composite Materials GmbH, Germany; H. Riedl, Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria

Hot corrosion is an accelerated oxidation process commonly observed in high-temperature settings (650-950 °C), such as gas turbines, coal gasification plants, and waste incinerators. It is a phenomenon where sulfur-rich atmospheres (exhaust gases) react with salt impurities to form high-melting sulfate salts that adhere to machining components. There, the salt deposits elicit an accelerated degradation of the operating parts by forming porous, non-protective oxide scales, drastically reducing the longevity of in-service parts. Ni-, Co-, and Fe-based superalloys, representing the backbone in the previously mentioned applications, are especially prone to hot-corrosion attacks.

This contribution presents  $Ti_{1-x}Al_xN$  as a potential candidate as a protective PVD coating for hot-corrosion environments.  $Ti_{1-x}Al_xN$  coatings with varying metal content ratios were arc-evaporated on a Ni-based superalloy and tested in an in-house built hot-corrosion testing rig. By applying a sulfate-salt mixture from the alkali and alkaline earth metal groups, we tested coated and uncoated samples in a SO<sub>x</sub>-rich atmosphere at 700 and 850°C for a maximum duration of 30 h and subsequently analyzed using a set of high-resolution characterization techniques.

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