ALD Applications
Room Van Rysselberghe - Session AA2-WeA
Emerging Applications of ALD

Moderators: Giuseppe Alessio Verni, ASM, Adrie Mackus, Eindhoven University, Netherlands

4:00pm **AA2-WeA-11 Mechanical Properties of ALD Coatings***, **Aile Tamm**,* University of Tartu, Estonia*; H. Piirsoo,* University of Tartu,, Estonia*; J. Kozlova, T. Jõgiaas,* University of Tartu, Estonia **INVITED**

Demand in using films grown by atomic layer deposition (ALD) for the fabrication of microelectromechanical systems has significantly increased during the past decades [1]. ALD films can be used as supporting layers (seed layers, masks, etc) as well as device's functional layers. Depending on the application, the ALD film thicknesses used may vary from nanometer-level to several hundreds of nanometers, while composition can vary from pure metal or metal oxide to several metals together in one film. For the preparation of the devices, it is often required to obtain a film that combines several specific physical properties in one film.

Mechanical properties (elastic modulus and hardness) of ALD films with a thickness from 30 up to 150 nm were measured by nanoindentation using Hysitron TriboIndenter TI980 (Bruker). The properties (thickness, morphology, composition and crystallinity) of pure oxide films, like Cr_2O_3, Al_2O_3, Ta_2O_3, HfO_2 and ZrO_2 as well as layered nanostructures, like ZrO_2-SnO_2 [2], HfO_2-ZrO_2 [3], Al_2O_3-ZrO_2 [4] Al_2O_3-Ta_2O_5, HfO_2-Al_2O_3 will be discussed.

A few of the results of the layered nanostructures studies were follows:

1. the thickness and sequence of oxide layers of the two-and three-layered laminates influenced the hardness and elastic modulus of the laminates.
2. plasma ALD processes do not give advantages over thermal ALD.

This work overviews the results obtained by studying a quite wide range of materials where the mechanical and also magnetic or optical properties of the same ALD films were explored. Using alternating layers of different metal oxides allows fine-tuning of the film properties and even obtaining a film that overcomes the performance of the pure oxide film.

[1] M. Nasim, Y. Li, M. Wen, and C. Wen, “A review of high-strength nanolaminates and evaluation of their properties,” *J. Mater. Sci. Technol.*,50 (2020) 215–244.

[2] A. Tamm, H.-M. Piirsoo, T. Jõgiaas, et. al , Mechanical and Magnetic Properties of Double Layered Nanostructures of Tin and Zirconium Oxides Grown by Atomic Layer Deposition,*Nanomaterials, 11* (2021) 1633.

[3] T. Jõgiaas, M. Kull, H. Seemen, P. Ritslaid, K. Kukli, A. Tamm, Optical and mechanical properties of nanolaminates of zirconium and hafnium oxides grown by atomic layer deposition, *J. Vac. Sci. Technol.* A 38 (2020) 022406.

[4] T Jõgiaas, R. Zabels, A. Tarre, A. Tamm, Hardness and modulus of elasticity of atomic layer deposited Al2O3-ZrO2 nanolaminates and mixtures, *Mat Chem Phys.*240 (2020) 122270.

4:30pm **AA2-WeA-13 Superconducting Tantalum Nitride Prepared by Plasma ALD With RF Biasing for Quantum Applications***, **Silke Peeters**,* Eindhoven University of Technology, Netherlands*; C. Lennon, R. Hadfield,* University of Glasgow, UK*; E. Kessels, H. Knoops,* Eindhoven University of Technology, Netherlands

This contribution reports the first critical temperatures (T_c) for tantalum nitride prepared by plasma ALD and outlines the promising electrical and structural properties of this material for application as a low-loss superconducting material in quantum devices. The motivation is to further improve quantum device performance for which a reduction of material-related decoherence sources is critical. The recent demonstration of Ta superconducting transmon qubit coherence times above 0.3 milliseconds[1] calls for further exploration of Ta-based materials for quantum applications.

This work focuses on TaN, as reported T_c values for superconductivity exceed those of Ta[2]. Moreover, nitridation of superconductors has been shown to better protect against loss due to surface oxidation[3]. To our knowledge, no superconducting transition has been reported for TaN prepared by ALD. Literature on TaN prepared by techniques such as dc magnetron sputtering and pulsed laser deposition reveals that, together with resistivity, stoichiometry and crystal structure are critical film properties in achieving superconducting TaN films.

Thin TaN layers are prepared by plasma-enhanced ALD using TBTDMT and Ar-H_2 plasma at 250 °C with RF substrate biasing. A low room temperature resistivity of 221 μΩ cm of a 35 nm film is measured for a 20 W RF bias, which is a hundredfold improvement compared to films prepared without biasing. XPS measurements confirm that applying a substrate bias counteracts O incorporation during deposition. In addition, XPS reveals a significant increase in C content. Strikingly, these films are of (N+C):Ta ≈ 1 stoichiometry, and it is verified that C is present in Ta-C bonds. As tantalum carbonitrides display similar superconducting properties, this may provide additional opportunities of tuning layer properties. Moreover, XRD measurements show a (111) and (200) fcc crystal structure, where the cubic structure yields the highest T_c for TaN according to literature[4]. TEM analysis reveals an increase in crystal size due to substrate biasing. Preliminary liquid He four-point probe measurements on these samples indicate a T_c above 6 K.

Through substrate biasing stoichiometric fcc $Ta(C_x)N_y$ thin films of enhanced conductivity were obtained. The control of film properties and the relation with the superconducting transition will be discussed in this contribution.

References

1. Place *et al. Nat. Commun.***12**, 1779 (2021)

2. Thorwarth *et al. Solid State Commun.***20**, 870 (1976)

3. Wang *et al. Jpn. J. Appl. Phys.* **42**, 1843 (2003)

4. Reichelt *et al. J. Appl. Phys.* **49**, 5284 (1978)

4:45pm **AA2-WeA-14 Membrane Design by ALD for Hydrogen Purification***, L. Badouric, M. Drobek, A. Julbe, **Mikhael Bechelany**,* European Institute of Membranes, France

Hydrogen (H_2) is one of the energy vectors essential for the success of the energy transition. In less than twenty-five years, hydrogen is expected to represent 18% of the total energy consumed on the planet thus leading to possible CO_2 emissions decrease by 6 gigatonnes compared to current levels. At the same time, hydrogen energy technologies involve major environmental, research and industrial challenges. In this presentation, we will show our efforts in designing membranes by Atomic Layer Deposition (ALD) for hydrogen purification.

In view of the increasing use of hydrogen as "green energy carrier", the various aspects of its production must be considered and optimized. Indeed, the continuous separation/purification of H_2 is a key step in the production chain. When polymer (low selectivity and temperature resistance) or palladium-based membranes (more expensive and highly sensitive to sulfur compounds) cannot be used for H_2 purification, the application of inorganic or hybrid ultra-microporous membranes is a relevant option. In this context, we will present a preparation of highly selective ultra-thin prototype membranes deposited on the surface or inner porosity of commercial porous supports by Atomic Layer Deposition (ALD). The design and synthesis conditions of these membranes are optimized according to the constraints of the targeted application, in order to maximize the thermochemical stability, the abrasion resistance as well as the membranes performance (H_2 selectivity and permeability). Several examples including the development of Metal Organic Frameworks (MOFs) [1] and Palladium - based membranes [2] as well as their composites prepared by Atomic Layer Deposition (ALD) [3] will be highlighted in this presentation.

[1] Journal of Membrane Science, 2015, 475, 39–46

[2] Journal of membrane Science, 2020, 596, 117701

[3] Chemistry of Materials 2018, 30, 7368-7390

Author Index

Bold page numbers indicate presenter