ALD Applications
Room Hall 3D - Session AA2-WeA
Emerging: Optics/Optoelectronics

Moderators: Tero Pilvi, Picosun Oy, Dr. Tania Sandoval, Technical University Federico Santa Maria

4:00pm **AA2-WeA-11 Deposition and Characterization of Electro-Optic ALD $K(Ta_x,Nb_{1-x})O_3$ Films for Photonics***, **Eric Martin,*** Ohio State University*; J. Bickford,* Army Research Laboratory*; H. Sønsteby,* University of Oslo, Norway*; R. Hoffman,* Army Research Laboratory*; R. Reano,* Ohio State University

Electrooptic (EO) materials are critical for optics and photonics, enabling fast and dynamic control propagating light.In particular $K(Ta_x,Nb_{1-x})O_3$ (KTN) stands as a unique EO material in that it possesses an extremely high quadratic (Kerr) EO coefficient. KTN is a solid solution of $KTaO_3$ and $KNbO_3$ perovskites with a Curie temperature (T_c) that is defined by the Ta:Nb ratio.Bulk KTN crystals have shown record high Kerr nonlinearity of greater than 2.2 x 10^{-14} m^2/V^2 when thermally tuned near T_c.However, difficultly in fabrication of KTN crystals greater than 1cm^2 in area has limited applications to small varifocal lens and beam deflectors.Recent advancements in ALD of KTN utilizing alkoxide precursors and O_3 as the oxidant has shown precise composition control and excellent crystallinity on both $(LaAlO_3)_{0.3}(Sr_2TaAlO_6)_{0.7}$ (LSAT) and $MgAl_2O_4$ (MAO) substrates.ALD KTN films open exciting new possibilities for both bulk and integrated optical building blocks, including tunable filters, reflectors, modulators, phase shifters, and grating emitters.In this work, we investigate the deposition of crystalline KTN on a variety of substrates including LSAT, MAO, MgO, and Si. Our approach encompasses several facets. We begin by depositing KTN thin films using alkoxide precursors and O_3 as the oxidant. The deposited films are characterized for their optical properties using spectroscopic ellipsometry. We determine the crystallinity of the films through x-ray diffraction (XRD) and evaluate the composition via x-ray fluorescence (XRF). We further probe the dielectric response of the films and determine T_c the through capacitance measurements. This includes utilizing interdigitated capacitors (IDCs) and metal-oxide-semiconductor (MOS) capacitors for precise temperature-dependent characterization. Additionally, we investigate the EO properties of KTN films as a function of temperature and composition. This is achieved using specialized silicon Photonic Integrated Circuit (PIC) test chips, comprised of a Mach-Zehnder interferometer trenched for KTN deposition with an integrated photodetector. For the first time, we report the optical, dielectric, and electrooptic properties of ALD KTN thin films.

4:15pm **AA2-WeA-12 Advances in Plasma-based Atomic Layer Processing of AlF_3 for the Passivation of FUV Mirrors***, **Virginia Wheeler**, M. Sales, D. Boris,* Naval Research Laboratory*; L. Rodriguez de Marcos,* Catholic University of America and NASA Goddard Space Flight Center*; J. del Hoyo,* NASA Goddard Space Flight Center*; A. Lang, S. Walton,* Naval Research Laboratory*; E. Wollack, M. Quijada,* NASA Goddard Space Flight Center

Efficient ultraviolet mirrors are essential components for UV astronomy. While aluminum mirrors with stable and reliable fluoride-based passivation layers are commonly used, the optical performance is still insufficient for systems where several reflections are required. We previously demonstrated the feasibility of a new, room temperature plasma process based on a benign SF_6 electron beam (e-beam)-generated plasma to simultaneously remove the native oxide and form an AlF_3 layer with tunable thickness [1]. This produces Al-mirrors with high FUV reflectivity (R≈90% at λ=121nm) and improved durability. Plasma-enhanced atomic layer deposition (PEALD) is a known low temperature, highly conformal coating process which has previously been shown to produce AlF_3 films [2], though little has been reported on their performance in FUV applications. In this work, we focus on optimizing a PEALD AlF_3 process using a remote ICP plasma and developing a new hybrid approach combining the e-beam-generated plasma and ICP processes. We will provide a detailed analysis of AlF_3 film materials properties and FUV optical performance produced by each approach individually and combined.

PEALD AlF_3 films were deposited using trimethylaluminum and SF_6 plasma precursors in a Veeco Fiji G2 reactor custom fitted with an on-axis cylindrical e-beam-generated plasma to replicate the self-fluorination process directly on an ALD reactor. ALD windows were optimized using *in situ* ellipsometery directly on Al substrates and supplemented with post-deposition x-ray photoelectron spectroscopy, atomic force microscopy, transmission electron microscopy and FUV measurements to elucidate process-structure-property relationships. Plasma diagnostics, including optical emission spectroscopy and Langmuir probe measurements, were also conducted on the reactor to correlate ion fluence and ion energy to resulting film properties. PEALD AlF_3 films with F/Al ratios of 2.92-2.97, < 2 at% oxygen, and surface roughness similar to starting Al-mirrors were attained at lower plasma power (100W), high SF_6/Ar ratios (≥ 0.5) and gas flows (> 30sccm). However, the FUV properties of these films are still inhibited by the native oxide interface that cannot be adequately treated with remote ICP plasma alone. Initial films combining an *in situ* e-beam-generated plasma for fluorine passivation of the Al mirror interface with the optimum PEALD produced better FUV performance in the 100-190 nm region. Full detailed characterization of this hybrid approach will be discussed.

[1] L.V. Rodriguez de Marcos, et al. Opt. Mater. Express 11, 740-756 (2021)

[2] M.F.J. Vos, Appl. Phys. Lett. 111, 113105 (2017)

4:30pm **AA2-WeA-13 Plasma-Enhanced Atomic Layer Deposition with RF Substrate Biasing to Tune the Performance of Superconducting Nanowire Single-Photon Detectors in the Mid-Infrared***, **Ciaran Lennon,*** Oxford Instruments Plasma Technology, University of Glasgow, UK*; D. Morozov,* University of Glasgow, UK*; Y. Shu,* Oxford Instruments Plasma Technology, UK*; H. Knoops,* Oxford Instruments Plasma Technology, UK, Eindhoven University of Technology, Netherlands*; K. Hore,* Oxford Instruments Plasma Technology, UK*; R. Hadfield,* University of Glasgow, UK

Superconducting nanowire single-photon detectors (SNSPDs) offer field-leading time-correlated single photon detection in the infrared, with ultrafast timing jitter, near-unity internal detection efficiency and low dark count rates [1]. Extending the performance in the mid-infrared has been a focus in the field, potentially expanding the range of SNSPD applications in areas like exoplanet spectroscopy and LIDAR [2,3].

Previous work on mid-infrared SNSPDs has focused on amorphous superconducting materials (WSi) owing to their lower superconducting energy gap (Δ), demonstrating saturated internal detection efficiency up to 29 µm [4,5]. However, amorphous materials have lower T_c, requiring device operation at < 2K with bulky, energy-intensive cryocoolers. Crystalline metal nitrides, like NbN and NbTiN, are a promising alternative, with T_c > 10 K, enabling device operation at > 2 K, although with higher Δ. Lowering Δ has been shown to increase the detection efficiency in the mid-infrared [5]; therefore, tuning Δ for NbN and NbTiN could be a promising approach.

Controlling the ion energy in the plasma-enhanced atomic layer deposition (PEALD) process using RF substrate biasing can influence various material properties such as crystallinity, composition and stress [6]. Recent work has also shown that the superconducting properties of metal nitride thin films can be tuned using RF substrate biasing [7,8], as well as enhancing the uniformity, making it an ideal technique for the development of large-area SNSPD arrays. Consequently, we have used PEALD with RF substrate biasing to develop NbN and NbTiN thin films tuned for mid-infrared SNSPDs. We report on the electrical and superconducting properties of the ultrathin films and discuss the fabrication, electrical transport properties and optical testing of fabricated SNSPDs, with their performance benchmarked from 1.5-4 µm.

Overall, this study highlights the potential of PEALD with RF substrate biasing for developing NbN and NbTiN thin films for SNSPDs tuned for mid-infrared photon counting applications, with scope to develop large-area SNSPD arrays.

[1] Morozov D. V., *et al.*, *Contemp Phys* **62** 69-91

[2] Wollman E. E., *et al.*, J Astron Telesc Instrum Syst **7** 1-10

[3] Taylor G. G., *et al.*, Opt Express **27** 38147

[4] Verma V. B., *et al.*, APL Photonics **6**

[5] Taylor G. G., *et al.*, Optica **10** 1672

[6] Faraz T., *et al.*, ACS Appl Mater Interfaces **10** 13158-80

[7] Peeters S. A., *et al.*, Appl Phys Lett **123** 132603

[8] Lennon C. T., *et al.*, Materials for Quantum Technology **3** 045401

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