ALD Applications
Room On Demand - Session AA16
Emerging: Others (Protective Coatings, Hardness, MEMs...)

AA16-1 Capacitance Modulation by Light and Mechanical Stimuli in ALD-deposited ZnO Thin Films Integrated in Piezotronic MEMS Strain Microsensors*, **Raoul Joly**, S. Girod, N. Adjeroud, P. Grysan, J. Polesel,* Luxembourg Institute of Science and Technology (LIST), Luxembourg

By the means of Atomic Layer Deposition (ALD), we developed piezotronic strain sensitive sensors integrated in polyimide cantilevers, where a zinc oxide (ZnO) thin film is deposited on top of patterned interdigitated platinum electrodes (Figure 1(a)). The rapidly spreading Internet-of-Things is accelerating MEMS (Micro-ElectroMechanical Systems) industry to deliver highly sensitive and miniaturized self-sensors with low consumption and cost-effective production process. Due to its high film conformality, low temperature processing, self-limiting nature and thickness control at the nanoscale level, ALD technique has emerged as an ideal technique to add new functionalities in MEMS.

We propose to rationalize the ALD processing deposition parameters on the sensor's electrical properties and the sensitivity of detection for mechanical strain and light. We report on the evidence of negative capacitance phenomena occurring at the interface of Schottky junctions over a wide frequency range (between 20 Hz and 100 kHz). We demonstrate an original way to modulate the sensors capacitance characteristics in the presence of a light source as well as by applying a mechanical strain to the devices (Figures 1(b), (c) and (d)). The rationale behind these observations will be discussed. The ALD thin film is made of wurtzite polycrystalline zinc oxide with a privileged (002) orientation. We optimized the deposition temperature to be compatible with microfabrication processing on polymer and photoresists by thin film growth below 100 °C. Hence, Schottky junctions are realized by microstructuring interdigitated micro-combs at the interface of high work function platinum metal electrodes and a semiconducting piezoelectric ZnO thin film. The obtained piezotronic junction has the particularity of an exponential dependence of the flowing diode current as a function of the applied mechanical strain. The sensitivity is thus greatly improved with gauge factor higher than 100.

In the last stage of this work, we will present the strain sensors size miniaturization for integration in microcantilevers in a full polymer body, compatible with AFM (Atomic Force Microscopy) scanning probe operations to highlight the very high sensitivity of detection. These results open up new perspectives and applications towards the miniaturization of highly sensitive and low power consumption environmental sensors, as well as for broadband impedance matching in radio frequency applications by the means of negative capacitance devices.

AA16-2 Applications of Atomic-Scale Processing for the Next Decade of MEMS Technology*, **Daniel Potrepka**, N. Strnad, R. Rudy,* U.S. Army Research Laboratory

Atomic-scale processing has had a major impact in the fields of microelectronics and CMOS fabrication technology, building upon the significant advances in academia. Now the field of micro-electomechanical systems (MEMS) is poised to reap the benefits of atomic-scale fabrication, as gains achieved with standard process technologies remain limited to low-cost devices. To achieve this breakthrough, ALD atomic-scale techniques will play a vital role, driving MEMS deep into the nanoscale regime by engineering innovative designs to take advantage of the scaled piezo- and ferroelectric properties encountered therein. Using a combination of ALD, ALE, and pre- and post- treatments for area-selective growth of function-enhancing features and layers, ALD can hermetically seal off devices from harsh in-process or working environments, eliminate stiction, tailor conformal multilayer geometries to provide new functionalities such as phonon crystals and metamaterials, control stress, enhance polarization and structural integrity, lower operating voltage, increase chip work density by an order of magnitude, and meet thermal, mechanical, geometrical, barrier, interface, and ferroic materials design requirements for competitive commercial MEMS technologies and devices. These breakthroughs will be enabled by a wide array of viable precursors, providing new metal oxides and metals of increasing diversity and complexity from throughout the periodic table, including the recently developed nitrides, sulfides, and tellurides [1-3]. The resulting new materials, along with rapidly occurring growth and integration of computer modeling for precursor-surface chemical reactions [4] and hardware improvements [3] will enhance the scale and pace of MEMS modernization. ALD commercial infrastructure and equipment sales, currently predicted to increase to about USD $2 billion in 2026 at a compound annual growth rate from 2020 of 26.3% can leverage off the larger MEMS global market. Impacts of the key atomic-scale processes that can fuel this exciting expansion of the MEMS technology arena will be reviewed, emphasizing the benefits for the future of prototyping and scaled fabrication in commercial, industrial, and defense applications.

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AA16-3 Texture Control of Piezoelectric Aluminum Nitride Grown by Atomic Layer Deposition for 3D Microelectromechanical Systems*, **Nicholas Strnad**, W. Sarney,* CCDC Army Research Laboratory*; G. Rayner,* Kurt J. Lesker Company, Inc.*; G. Fox,* Fox Materials Consulting, LLC*; R. Rudy, J. Pulskamp,* CCDC Army Research Laboratory

3D microelectromechanical systems (3D-MEMS) are an emerging application space for piezoelectric films grown using ALD. ALD provides an ideal solution for the deposition of piezoelectric materials on trench sidewall or 3D fin structures which may be used to improve the size, weight, power and frequency tunability of MEMS devices. Aluminum nitride is a technologically relevant material for piezoelectric MEMS due to its significant piezoelectric response, high breakdown voltage and large mechanical quality factor. AlN is amenable to film growth by ALD, although there are relatively few reports of ALD AlN used for MEMS due to the need for additional process development to meet stringent crystal structure, film purity and grain orientation requirements for device performance. We explore several strategies for controlling the grain orientation of ALD-grown AlN on planar substrates, which includes the use of {111}-textured Pt as a growth template, precursor chemistry and dose variation, stress-engineered substrates, inductively-coupled plasma conditions for film bombardment during growth, and ALD equipment modifications. For select cases, we report the mechanical Q, determined from measurements of MEMS resonator structures, and piezoelectric coefficients, determined from measurements on MEMS cantilevers, of ALD deposited AlN . We analyze the Pt-AlN interface properties primarily by using TEM with EDS. The baseline ALD AlN process yielded completely c-axis oriented aluminum nitride as determined by x-ray diffraction, and a rocking curve full-width half max of 2.9° was achieved. The relative dielectric constant was measured to be 8.1 < K < 8.6 and an average dielectric loss of < 1% was observed within the an applied electric field range of +/- 3350 kV/cm (+/- 35 V across 104 nm thick AlN) at 10 kHz. The leakage current of the textured AlN was quite low at 1.5 x 10^{-6} A/cm^2 over the applied field range of +/- 1820 kV/cm (+/- 19 V across 104 nm thick AlN).

AA16-4 Electrically-Conductive Kevlar Fabrics for Multi-Functional Fiber Reinforced Composites Enabled by Atomic Layer Deposition*, **Robin E. Rodríguez**, T. Lee, Y. Chen, T. Cho, C. Huang, E. Kazyak, A. Poli,* University of Michigan, Ann Arbor*; W. LePage,* University of Michigan - Ann Arbor*; M. Thouless, M. Banu, N. Dasgupta,* University of Michigan, Ann Arbor

Multi-functional composites have wide-ranging applications from structural batteries to electronic devices, which are of increasing interest in the aerospace community. To operate as a device, at least one constituent of the composite needs to be electrically conductive. Polymer-matrix composites (PMCs) are generally electrically insulating and often necessitate the inclusion of electrically-conductive additives, but such additives tend to affect the bulk mechanical properties of the final product. A method to impart electrical conductivity without affecting the bulk mechanical properties of the composite is to modify the surface of the reinforcement by adding a thin, conductive coating. Among the coating techniques that can be utilized, atomic layer deposition (ALD) provides unparalleled conformality in coating of the 3-D fiber template, as well as sub-nanometer resolution in film thickness and composition.

In this work we demonstrate the fabrication of electrically-conductive Kevlar-reinforced PMCs without measurably affecting the bulk material properties, by coating Kevlar fabrics with aluminum-doped zinc oxide (AZO) via ALD. The core-shell fabric morphology and structure were characterized by SEM, XPS, and XRD. The conductive properties of the AZO-coated Kevlar fabric were measured using the four-point probe method, which were taken at the single-fiber, single-tow, and fabric level, after varying the thickness of AZO from 80-200 nm. For all the cases, the electrical resistivity decreases as the film thickness increases. The thickness-dependence of the electrical resistivity is well described by an analytical model, which allows for predictive design. These fabrics are sufficiently conductive to serve as an electrode material in textile-based devices ranging from thin-film electronics to energy storage devices.

Additionally, we studied the relationships between electrical conductivity and mechanical deformation of AZO-coated Kevlar fabrics as used in a PMC. The electrical resistance of the composite was continually monitored *in situ,* while loading the sample to failure in a tensile load frame. The ultimate tensile strength and Young's modulus of the Kevlar-reinforced PMC was not significantly affected by AZO interface coatings, illustrating the advantage of this approach over bulk conductive additives. Furthermore, changes in the electrical resistance at higher strains were observed, which is attributed to the cracking of the AZO coating. These results demonstrate the potential of AZO-coated Kevlar to make electrically conductive composites with applications such as monitoring the state-of-health of the composite within the linear elastic regime.

AA16-5 COO Reduction for Semiconductor Parts via ALD Coatings and Recycling of Parts*, **Russell Parise**, I. Iordanov,* QuantumClean, A Business Unit of UCT*; G. Osoro,* Inficon

The semiconductor industry has considerable experience with thermal spray protective coatings of parts in the etch and deposition areas to modify surface chemistry, provide corrosion resistance, or provide a barrier layer.These coatings are effective but are generally thick (150 - 200 µm) and may not provide sufficiently low porosity for parts exposed to reactive gases or plasmas.The ALD process creates high purity thin-films that are dense, highly conformal and defect free. Materials such as Al_2O_3 and other metal oxides are resistant to the reactive halogens that part surfaces see during semiconductor processing.

To address these challenges, ALD coatings have been developed to uniformly coat showerheads, pedestals, and other parts with high aspect ratios with 100 – 500 nm thin-films such as Al_2O_3, SiO_2, or other metal oxides.These films protect the part from the reactive halogen radicals and extend green to green time. Besides protecting the parts from reactive gases, the high aspect ratio ALD coatings on chamber components can serve as a diffusion barrier to avoid metal migration from the part itself and reduce conditioning times.

Additionally, parts with these ALD coatings can be recycled. Using Selective Coating Removal, the ALD protective coating is removed with minimum damage to the substrate, including the high aspect ratio features, before the part is prepared for ALD recoating. The loop essentially extends part life almost indefinitely which decreases cost of ownership.

This paper addresses the recent advances in the use of ALD thin films as a functional, protective coating that enhances part performance and reduces process costs.It will also cover the technology to selectively remove the deposition layer and ALD coating without damage to the part.Surface preparation, final cleaning, metrology and analytical testing for validation will also be discussed.

Keywords: ALD, Al_2O_3, corrosion, diffusion barrier, SiO_2, aspect ratio, etch, CVD

AA16-8 Aqueous Degradation and Nanoscale Coatings of Al_2O_3 via Atomic Layer Deposition (ALD) of $BaAl_2O_4$: Eu^{2+}, Dy^{3+} Long Afterglow Phosphors*, **Erkul Karacaoglu**,* Georgia Institute of Technology, USA and Karamanoglu Mehmetbey University, Turkey*; E. Öztürk,* Karamanoglu Mehmetbey University, Turkey*; M. Uyaner,* Necmettin Erbakan University, Turkey*; A. Okyay,* OkyayTechALD Okyay Technnologies, Turkey and Stanford University*; M. Losego,* Georgia Institute of Technology, USA

In this presentation aqueous degradation studies of $BaAl_2O_4$:Eu^{2+}, Dy^{3+} phosphors synthesized from solid-state reaction methods and coated with nanoscale Al_2O_3 protective layers via atomic layer deposition (ALD) will be presented. The uncoated phosphor powders degrade in water within just 30 minutes of immersion. This degradation directly affects the bluish-green phosphorescence (497 nm), creating both blue- and red-shift which are maximized at 429 nm and 687 nm, respectively. Hydration and decomposition of the $BaAl_2O_4$ phase reveals a continuous change in the phase assemblage over 30 days. ALD coatings of ~10 nm Al_2O_3 protects the phosphor from aqueous degradation upon long-term immersion in water. ALD Al_2O_3 coated $BaAl_2O_4$: Eu^{2+}, Dy^{3+} phosphors retain their phosphorescence for at least 7 days of water immersion. Successful encapsulation of $BaAl_2O_4$-based phosphors will make them possible to store in humid environments or use in applications directly requiring aqueous solution.

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