Electronic Materials and Photonics Division
Room 304 - Session EM1+MN+NS-FrM

Piezoelectric, Ferroelectric, and Multiferroic Devices & Microelectronics

Moderators: M. David Henry, Sandia National Labs, **Stephen McDonnell**, University of Virginia

8:20am **EM1+MN+NS-FrM-1 Piezoelectric Adjustable X-ray Optics***, **Susan Trolier-McKinstry**,* Penn State University **INVITED**

Next generation X-ray observatories require lightweight, high throughput optics that maintain a < 0.5 arcsecond resolution. Thin adjustable X-ray mirrors can correct deformations generated from fabrication errors, gravity release, mounting stresses, and thermal variations, maintaining the high angular resolution (< 0.5 arcsecond) and large effective area (> 2 m^2) required for future X-ray missions. This paper describes fabrication of adjustable mirrors for the Lynx X-ray observatory mission concept.Prototype X-ray mirrors were built on either a 400 µm thick curved *Corning EAGLE XG*®glass substrate or on polished Si.In both cases, a Cr/Ir X-ray mirror coating was deposited on the front (concave) side, and an array of 1.5 µm thick radio frequency (RF) sputtered $Pb_{0.995}(Zr_{0.52}Ti_{0.48})_{0.99}Nb_{0.01}O_3$(PZT) piezoelectric thin film actuators on the back (convex) side to enable correction of figure errors.A two-layer metal routing scheme with a polymeric insulator was used to independently address 288 actuators on the mirror. The two-layer metal allows narrow kerfs between actuators and increased actuator density. A chrome-iridium layer was deposited on the concave side to function as the X-ray reflective coating for the films deposited on the convex side.Anisotropic conductive film was used to bond thin flexible copper cables to flat edges of the mirror to interface with external control electronics. Improved stress balancing process was achieved using compressively stressed SiO_2 films deposited on the convex side of the mirror to balance the tensile integrated stress of the actuator array while also matching the film thickness distribution.Finite element methods were used to assess the impact of film thickness distributions on the convex and concave substrate surfaces.The resulting models show peak-to-valley figure errors of 105 nm, well within the 1 µm peak-to-valley dynamic range of the piezoelectric adjusters. In contrast, when stress compensation was done with an iridium mirror film deposited on the front side, the mismatched thickness distribution results in peak-to-valley figure errors over 3 µm.

9:00am **EM1+MN+NS-FrM-3 Oxide and Nitride Ferroelectric Wurtzite Crystals***, **Jon-Paul Maria**,* Penn State University

In the past three years, the demonstration of ferroelectricity in wurtzite-based crystals introduced exciting opportunities to explore and discover new structure-property relationships in novel formulation spaces, and to investigate new integration and device implementations given new process compatibilities. The seminal discovery of ferroelectric $Al_{1-x}Sc_xN$ by Fichtner *et al.* initiated this excitement and was followed by comparable observations of polarization reversal in the structurally similar $Al_{1-x}B_xN^2$ and the $Zn_{1-x}Mg_xO^3$ systems.

In this presentation our group will present recent results that demonstrate the structure-process-property relationships in the B-substituted AlN and Mg-substituted ZnO nitride and oxide systems. The B-substituted materials exhibit square hysteresis loops with polarization values between 150 $\mu C/cm^2$ and 120 $\mu C/cm^2$ when boron concentrations range between 2% and 15% respectively. Coercive field values fall with additional boron, from 5.5MV/cm to about 5 MV/cm at B saturation. Bandgap values are approximately 5 eV or above in all cases. Material can be prepared between 100 °C and 350 °C with very little difference in electrical properties. W bottom and top electrodes are used in all cases. Capacitors can be prepared down to 50 nm thick before leakage current becomes problematic during low frequency hysteresis measurements. First principles calculations that rationalize the unit cell volume, bond angle distribution, and remanent polarization will be presented.

Comparable results are found in the $Zn_{1-x}Mg_xO$ system. Between 25% and 35% Mg substitution, square hysteresis loops with remanent polarization values above 100 $\mu C/cm^2$ are readily achieved. Transmission measurements show bandgap values between 4.0 eV and 4.2 eV in this range. In comparison to AlBN, coercive field values for ZMO are as low as 1.7 MV/cm. As is the case with AlBN and AlScN, sustaining high insulation resistance to arbitrarily low thickness is challenging, the current thinness limit for low-leakage switching is ~ 125 nm. SHG analysis will also be presented for the ZMO system – preliminary measurements suggest values comparable to ferroelectric niobates.

9:20am **EM1+MN+NS-FrM-4 Development and Processing of $Al_{1-x}Sc_xN$ (x<0.40) Films for Resonator and Filter Applications***, **Giovanni Esteves**, S. Yen, T. Young,* Sandia National Laboratories*; Z. Tang,* The University of Pennsylvania*; E. Schmidt, L. Gastian, M. Henry, T. Bauer, C. Nordquist,* Sandia National Laboratories*; R. Olsson,* The University of Pennsylvania

As the development of aluminum scandium nitride ($Al_{1-x}Sc_xN$/AlScN) films continues to be pushed towards higher Sc content, fabricated devices yield insight into the challenges associated with processing while demonstrating increased electromechanical coupling coefficients (k_t^2) over AlN. The addition of Sc into AlN presents film development and fabrication challenges that increase with higher Sc content such as the reduction of abnormal grains (AG), higher compressive stress, and etching. The development of $Al_{0.6}Sc_{0.4}N$ films using a single-alloyed target poses a significant challenge in terms of managing stress and the density of AG. Compressive stress help in reducing the amount of AG density through tuning the Ar/N_2 flow and pressure, but the magnitude of stress needed to achieve a low density of AG exceed -600 MPa. The use of certain metal templates aid in reducing AG density but are not sufficient to achieve AG-free films. Etching AlScN leads to long etch times due to slow etch rates of 25 nm/min and result in sidewall angles of ~74°. Nevertheless, AlScN lamb wave resonators (LWR) have been fabricated to demonstrate k_t^2 over 10%. Additionally, LWR with varying k_t^2 were interconnected to fabricate ladder filter configurations to determine that amount of bandwidth increase that can be achieved over AlN. Though AlScN demonstrates higher k_t^2, that lead to higher bandwidth, pushing this current technology to achieve more desirable metrics requires more stringent process quality.

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9:40am **EM1+MN+NS-FrM-5 Formation of Aluminum Scandium Nitride Microelectromechanical Systems Via Etching in Aqueous Potassium Hydroxide (KOH)***, **Zichen Tang**, M. D'Agati, R. Beaucejour, S. Sofronici, J. Zheng, K. Kaylan,* University of Pennsylvania*; G. Esteves,* Sandia National Laboratories*; R. Olsson,* University of Pennsylvania

We report on the etch rate of sputter deposited piezoelectric and ferroelectric Aluminum Scandium Nitride ($Al_{1-x}Sc_xN$) thin films in aqueous potassium hydroxide (KOH). Specifically, we report on the vertical etch rate, lateral etch rate, and sidewall angle as a function of the scandium alloying ratio (x), temperature, and KOH concentration. As the scandium alloying ratio is increased, the vertical etch rate in 30% KOH at 45 °C is reduced from > 100 nm/s for AlN to < 4 nm/s for $Al_{64}Sc_{36}N$. The lateral etch rate, however, follows a very different trend, arriving at a minimum values of 0.05 nm/sec for $Al_{88}Sc_{12}N$. This is in contrast to the much higher lateral etch rates observed for both AlN and $Al_{64}Sc_{36}N$ of 2 nm/s. These trends in vertical and lateral etch rate are shown to hold for KOH concentrations from 10 to 30% and etch temperatures from 45 to 65 °C. We show that the etched sidewall angle can be predicted from a combination of the crystal structure and the vertical and lateral etch rates. We report a technique that utilizes the crystal structure and the vertical and lateral etch rates to form vertical (i.e. 90°) sidewalls solely from aqueous KOH etching. The ability to control the sidewall angle is vitally important in the formation of microelectromechanical systems (MEMS). We report on several piezoelectric MEMS devices fabricated utilizing the KOH etching processes. Finally, we report on the etching of AlScN as a function of ferroelectric polarization.

10:00am **EM1+MN+NS-FrM-6 Interface Reactions During the Ferroelectric Switching of HfZrO Thin Films on InAs***, A. Irish, Y. Liu, R. Atle, A. Persson, R. Yadav, M. Borg, L. Wernersson, **Rainer Timm**,* Lund University, Sweden

Traditional MOSFET-based electronic components have reached severe bottlenecks regarding data handling speed and power dissipation. A very promising alternative approach builds on MOS material stacks with thin ferroelectric oxide films in novel device architectures for e.g. steep-slope transistors, neuromorphic networks, or in-memory computation [1]. $Hf_{1-x}Zr_xO_2$ (HZO) films grown by atomic layer deposition are widely used in this context, due to their excellent film quality and conformity with existing semiconductor technology. Ferroelectric MOS devices based on III-V semiconductors are especially promising for high-speed applications due to the high charge carrier mobility of e.g. InAs. Furthermore, InAs/HZO/TiN devices have shown an unexpectedly high remanent polarization of the ferroelectric film [2]. In spite of the excellent electrical performance, only little is known about the structure, chemical composition, and switching dynamics of the semiconductor-ferroelectric oxide interface. We have previously used X-ray photoemission spectroscopy (XPS) to investigate interfaces of ferroelectric HZO [3], but *in situ* structural characterization obtained during the ferroelectric switching has been lacking until now.

Here, we present operando hard X-ray photoelectron spectroscopy (HAXPES) results from ferroelectric InAs/HZO/TiN MOS devices obtained during electrical biasing and switching. We observe an interface layer consisting of In- and As-oxides at the InAs/HZO interface. As 2p and In 3d core level spectra were obtained after subsequent switching processes of a "positive-up-negative-down" (PUND) series, showing a reproducible increase of the amount of interface oxide upon upward polarization and a decrease upon downward polarization. Thereby, electrical PUND cycles confirm the ferroelectric nature of the MOS device. Such a redox reaction at the semiconductor-oxide interface upon ferroelectric switching has – to our knowledge – not been reported before. Furthermore, we observe that the major fraction of the applied bias does not drop over the 10 nm thin HZO layer, but instead over the thin InAs-oxide interface layer.

These observations are challenging the established understanding of ferroelectric behavior in thin oxide films and are a key to understanding the superior performance of III-V/HZO based devices.

[1] M. Park et al., MRS Commun. **8,** 795 (2018).
[2] A. Persson et al., Appl. Phys. Lett. **116**, 062902 (2020).
[3] R. Athle et al., ACS Appl. Mat. Int. **13**, 11089 (2021).

10:20am **EM1+MN+NS-FrM-7 The Effect of Hf Doping on Piezomagnetic Properties of FeCo for Magnetoelectric Heterostructure Devices***, **Thomas Mion**, K. Bussmann, M. Staruch, P. Finkel,* US Naval Research Laboratory

New developments in magnetoelectric devices have demonstrated increased energy efficiency and temperature stability with reduced size compared to current technologies. Artificial magnetoelectrics, built on the combination of ferromagnetic magnetostrictive materials structurally coupled to piezoelectric and ferroelectric materials, display the ability to control magnetic properties of the ferromagnet with electric voltage across the piezo/ferroelectric layer. The best performance requires the implementation of soft magnetic materials with large magnetostriction and large voltage-induced strain in the piezo/ferroelectric layer. Processing requirements for device fabrication often complicate the realization of these combined qualities as inherent stresses from the deposition technique are often detrimental to the magnetoelectric functionality. Solutions to these problems are rarely reported though alloying of FeCo and subsequent metalloid substitutions such as $(Fe_{0.5}Co_{0.5})_{1-x}C_x$, and $(Fe_{0.5}Co_{0.5})_{1-x}B_x$, have proven successful in reducing the coercive field while retaining high magnetostriction and piezomagnetic properties **[1,2]**.

In this work we present the systematic study of sputter-deposited Hf-doped $Fe_{50}Co_{50}$ alloy thin films with a focus on the correlation between film stress and magnetic softness and find an inflection point from tensile to compressive stress with increasing Hf composition. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) of the $(Fe_{0.5}Co_{0.5})_{1-x}Hf_x$ system reveal the magnetic softening is also correlated to emergence of an amorphous phase with reduced grain size for these sputter-deposited films. We will show the utilization of this new alloy in a multiferroic MEMS resonator device demonstrating a high magnetoelectric response required for magnetic sensors.

[1] Phys. Rev. Applied 12, 034011 (2019)

[2] Appl. Phys. Lett. 91, 182504 (2007)

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