

Thin Films Division

Room 102A - Session TF+EM+MI-WeM

Thin Film Processes for Electronics and Optics I

Moderators: Virginia Wheeler, U.S. Naval Research Laboratory, Mark Losego, Georgia Institute of Technology

8:00am **TF+EM+MI-WeM1 Crystalline Conductors: Transition Metal Nitride Materials and Device Applications**, *David Meyer, D.S. Katzer, N. Nepal, B.P. Downey, M.T. Hardy, D.F. Storm*, U.S. Naval Research Laboratory

INVITED

Development of gallium nitride and related III-N materials thin film growth technology has been instrumental in realizing high performance light-emitting, RF, and power electronic devices for both commercial and military applications. To continue pushing the frontiers of nitride materials research, we have recently incorporated a multi-pocket electron-beam evaporator into our nitride molecular beam epitaxy growth system to enable the evaporation of refractory transition metals. In spite of the complexity of the equilibrium phase diagrams of transition metal nitride (TMN) compounds, we have found that it is possible to grow single-phase TMN thin films epitaxially on SiC and AlN by carefully managing growth kinetics. This talk will discuss our latest results involving TMNs, such as NbN_x and TaN_x, which can be nearly lattice-matched and integrated within III-N heterostructures. Cross-sectional transmission electron microscopy indicates that TMN/III-N interfaces are atomically abrupt with no evidence of interdiffusion of the host elements. We anticipate that the device applications of these metallic films are quite broad ranging from selective etching of sacrificial layers for epitaxial lift-off of processed devices, to buried metallic or superconducting electrodes for quantum computation circuit elements, to optical mirrors and waveguides for improved optoelectronics.

8:40am **TF+EM+MI-WeM3 Growth Mechanism and Characteristics of Hf-Si-O Film by PE-ALD using TDMAS and TDMAH Precursors and Oxygen Plasma Gas**, *Toshihide Nabatame*, National Institute for Materials Science (NIMS), Japan; *M. Inoue*, National Institute for Materials Science (NIMS); *E. Maeda, K. Yuge, M. Hirose*, Shibaura Institute of Technology, Japan; *M. Takahashi, K. Ito*, Joining and Welding Research Institute, Osaka University, Japan; *N. Ikeda*, National Institute for Materials Science (NIMS), Japan; *T. Ohishi*, Shibaura Institute of Technology, Japan; *A. Ohi*, National Institute for Materials Science (NIMS), Japan

GaN-based power devices with metal-oxide-semiconductor (MOS) structure have been widely investigated for next-generation power devices. To reduce the leakage current, Hf-Si-O has one candidate material among various gate insulators. To fabricate Hf-Si-O film, a HfO₂/SiO₂ laminate film was generally deposited by ALD. The growth mechanism of SiO₂ films was greatly changed by the oxidant gas when Tris(dimethylamino)silane (TDMAS) was as precursor [1]. By using ozone gas, the stable growth rate and high quality SiO₂ films could be obtained while no growth using H₂O gas. We expect that similar data can be also obtained by using oxygen plasma gas. However, the growth mechanism of the SiO₂ film during HfO₂/SiO₂ laminate film deposition and its characteristics have not been fully understood.

In this study, we systematically investigate growth rate of HfO₂/SiO₂ laminate film by plasma-enhanced ALD (PE-ALD) using TDMAS and Tetrakis(dimethylamino)hafnium (TDMAH) precursors and oxygen plasma gas, and also examine characteristics of the Hf-Si-O films.

The HfO₂/SiO₂ laminate films were deposited on SiO₂/Si substrates by PE-ALD at 300 °C with TDMAH and TDMAS precursors and oxygen plasma gas. The composition Hf/Si ratio of the HfO₂/SiO₂ laminate films were varied by changing each number of ALD cycle. The SiO₂ film was also deposited on SiO₂/Si and HfO₂/Si substrates by the same PE-ALD condition. The Hf-Si-O films were formed by post-deposition annealing (PDA) at 700 – 900 °C in a N₂ atmosphere. The growth per cycle (GPC) of the HfO₂/SiO₂ laminate, HfO₂, and SiO₂ films was estimated by spectroscopic ellipsometry, XPS, and cross-sectional TEM measurements. The structure of Hf-Si-O films was identified by XRD analysis, and electrical characteristics of capacitors with Hf-Si-O films were examined using capacitance – voltage measurement.

The GPC of the HfO₂/SiO₂ laminate, HfO₂, and SiO₂ films on SiO₂/Si substrates were found to be 0.08, 0.08, and 0.04 nm/cycle, respectively, regardless of Hf/Si composition ratio. Considering to the GPC of the HfO₂ (0.08 nm/cycle), the estimated GPC of a SiO₂ layer during HfO₂/SiO₂ laminate film deposition was 0.08 nm/cycle, which was unexpected, but

increased by about 2 times compared to that of SiO₂ film on SiO₂/Si substrate. This suggest that an initial adsorption of TDMAS precursor on the SiO₂ and HfO₂ films must be different and strongly affects to the GPC. The Hf-Si-O films (Hf/Si=2/1) maintains amorphous structure even after PDA temperature at 900 °C. The Hf-Si-O films (Hf/Si=2/1) exhibited high dielectric constant of around 14 as expected.

Reference

[1] L. Han, and Z. Chen, ECS J. of Solid State Sci. and Technol., 2 N228 (2013).

9:00am **TF+EM+MI-WeM4 Atomic Layer Epitaxy of Ultra-wide Bandgap Ga₂O₃ Films**, *Virginia Wheeler, N. Nepal*, U.S. Naval Research Laboratory; *L.O. Nyakiti*, Texas A&M University; *D.R. Boris, S.G. Walton, B.P. Downey, D.J. Meyer*, U.S. Naval Research Laboratory; *C.R. Eddy, Jr.*, U. S. Naval Research Laboratory

Ga₂O₃ has emerged as a promising material for next generation power electronics and UV photodetectors applications due to its large bandgap (4.9 eV) and the availability of affordable native substrates from melt-grown bulk crystals. While β-Ga₂O₃ (monoclinic) is the most stable and studied of five polymorphs, the slightly less energetically favorable α- and ε-Ga₂O₃ phases have unique characteristics that can be exploited. The α-Ga₂O₃ (rhombohedral corundum) has the largest bandgap of 5.3 eV and can be alloyed with α-Al₂O₃ and α-In₂O₃ for bandgap engineering. The ε-Ga₂O₃ phase (hexagonal wurtzite) is a polar phase, with a calculated polarization strength that is 10 and 3 times larger than that of GaN and AlN, respectively. Like the III-N system, polarization induced charges can lead to higher charge densities and mobilities in two-dimensional electron gases formed at heterojunctions, which would improve the viability of Ga₂O₃ electronic devices. In this work, we use atomic layer epitaxy (ALEP) to produce high-quality homo- and heteroepitaxial Ga₂O₃ films and investigate phase selectivity as a function of substrate type and orientation, growth temperature (T_g), plasma gas phase chemistry and gas pressure.

All ALE Ga₂O₃ films were deposited in a Veeco Fiji G2 reactor equipped with a load lock and turbo pump using trimethylgallium and O₂ plasma precursors. Initial studies on c-plane sapphire substrates showed that decreasing chamber pressure an order of magnitude during the plasma step resulted in a shift from pure β-Ga₂O₃ to pure α-Ga₂O₃. Additionally, at 350°C and 8 mTorr, the phase could be altered by a varying the O₂ plasma flow from 5-100 sccm. Optical emission spectroscopy indicate that the ratio of O*/O₂ is critical for phase selectivity while the high ion flux to the surface can contribute to the crystallinity at low T_g. By varying T_g from 300 to 500°C at 8 mTorr, films went from mixed β/ε phase at <350°C, to pure α-Ga₂O₃ at 350°C, to pure β-Ga₂O₃ at 500°C. Using the optimum growth conditions for α-Ga₂O₃ on c-sapphire, the influence of substrate was explored using a variety of substrates including AlN, GaN (bulk and epilayers), SiC, diamond, and Si. Deposition on III-N and β-Ga₂O₃ substrates all resulted in crystalline β-Ga₂O₃ films, while amorphous films were deposited on both SiC and Si. This suggests that a clean crystalline substrate interface is critical to obtaining high quality films and promoting metastable phases is more dependent on growth parameters than underlying crystal symmetry. Finally, we will discuss simple electrical properties of optimum films of each phase to validate feasibility of the process in device applications.

9:20am **TF+EM+MI-WeM5 Effects of Process Gases and Gate TiN Electrode during the Post Deposition Anneal to ALD-Al₂O₃ Dielectric Film**, *Masaya Saito, A. Teramoto, T. Suwa, K. Nagumo, Y. Shiba, R. Kuroda, S. Sugawa*, Tohoku University, Japan

We investigated effects of a post deposition anneal(PDA) to Al₂O₃ film which was formed by the low temperature atomic layer deposition(ALD)⁽¹⁾. Al₂O₃ films were formed at 75°C by the ALD process using Al(CH₃)₃ as a precursor and H₂O as an oxidant, and these thicknesses were 40 nm. TiN films were formed as gate electrodes of MIS capacitors. We applied three kinds of PDAs. N₂ and O₂ annealing at 400°C were applied just after the ALD(PDA I(a), (b)), and N₂ annealing at 400°C was applied after the gate electrode formation (PDA II). We measured C-V and I-V characteristics.

The negative V_{FB} compared to the ideal one(+0.54 V) was observed in the C-V curve without PDAs, and the fixed charge density calculated from V_{FB} was ~10¹³ cm⁻². In contrast, positive V_{FB} compared to the ideal one was observed in the C-V curve with PDA I(b), and the fixed charge density was ~10¹¹ cm⁻². These results indicate that PDA I(b) is effective to decrease the fixed charges. Moreover, the hysteresis in the C-V curve decreased by PDA I(b), and the leakage current also decreased within the voltage range of C-V measurement. It is considered that the decrease of hysteresis was caused by decreasing the electrons that were trapped in the Al₂O₃ film⁽²⁾. However, a few samples broke down at low voltage after PDA I(b). In contrast, we

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couldn't measure the C-V characteristics of almost capacitors with PDA I(a) because the capacitors broke down at low voltage. In the case of PDA II which means N₂ annealing after the gate electrode formation, the leakage current decreased same as PDA I(b) and the production yield was still good even after PDA II. This was different tendency from PDA I(a) even as the same annealing. However, V_{FB} shifted to the positive voltage by the PDA II and the fixed charge density was ~10¹² cm⁻². This was the same phenomenon as the PDA I(b).

We considered these phenomena as follows; the oxidizing species were desorbed by PDA just after the ALD, and then the film quality was bad after the N₂ annealing. The oxygen for improving the film was supplied by the O₂ annealing with the desorption. When PDA was carried out after the gate electrode formation, the desorption did not occur because of capping by the gate electrode, and then the oxidizing species were sufficient for improving the film by the annealing.

Reference:

- (1) Y. Koda, et al., *ECS Trans.*, **72**(4), 91-100 (2016)
- (2) L. Sambuco Salomone, et al., *J. Appl. Phys.*, **123**, 085304 (2018)

Acknowledgement:

This work was carried out at fluctuation free facility of New Industry Creation Hatchery Center, Tohoku University.

9:40am **TF+EM+MI-WeM6 Controlling the NbO_x Materials System for Neuromorphic Computing**, *Alexander C. Kozen*, U.S. Naval Research Laboratory; *Z.R. Robinson, A.H. Rowley*, The College at Brockport - SUNY; *T.J. Larrabee, M.E. Twigg, H.S. Cho, S.M. Prokes*, U.S. Naval Research Laboratory

The niobium oxide system has seen recent interest, particularly due to its potential use as both a non-volatile, in the case of Nb₂O₅, or volatile, in the case of NbO₂, memristor material. Nb₂O₅ is a high k dielectric (k~41) with a high refractive index (n~2.2) and a wide bandgap (3.5 eV), while NbO₂ is a thermochromic material with a lower bandgap (1.2 eV) that undergoes a first order crystalline phase transition at a critical temperature (T_c) of 800°C. Both of these phases, along with their intermediate sub-oxides, can serve as material components of memristors to facilitate low-power neural computing hardware.

We will examine the impact of annealing temperature, duration, and atmospheric chemistry on the phase selectivity of the NbO_x thin films fabricated using atomic layer deposition. Anneals of stoichiometric amorphous Nb₂O₅ films are kinetically limited by both Nb⁵⁺ to Nb⁴⁺ reduction and crystallization steps, and we find that the addition of H₂ during annealing is critical for the Nb⁵⁺ to Nb⁴⁺ reduction. To remove this kinetic-limiting step, we instead deposit reduced amorphous NbO₂ films, which crystallize at lower temperatures. We investigate the optical and electrical properties of both amorphous and crystalline NbO_x films and discuss the utility of the NbO_x materials system to the fabrication of memristor materials.

11:00am **TF+EM+MI-WeM10 Sputtering Power Dependent on Switching Characteristics of ZnO-based Transparent Resistive Memory Devices**, *Firman Mangasa Simanjuntak*, Tohoku University, Japan; *T. Ohno*, Oita University, Japan; *S. Samukawa*, Tohoku University, Japan

Transparent resistive random access memory (T-RRAM) devices have great potential as data storage for invisible electronics.¹ ZnO material is one of the promising candidates for T-RRAM application due to its high transparency to the visible light.² However, ZnO is an *n*-type semiconducting material which the intrinsic donor defects exist abundantly; consequently, the ZnO-based switching devices require high current compliance to operate as compared to another oxide system.^{1,3} The employment of the high current compliance is to facilitate the formation of conducting filament. Various efforts have been made to improve the ZnO-based switching memories, such as: by controlling the thickness, introducing a dopant(s), adjusting the gas ambient during deposition, stacking with various material(s) and structure(s).¹ However, modulating the switching characteristics by controlling the sputtering power is still less overlooked. In this work, we investigated the influence of sputtering power in resistive switching characteristics of ZnO-based T-RRAM devices.

An approximately 50 nm thickness of ZnO films were deposited onto Indium Tin Oxide (ITO)-coated transparent substrates using conventional RF sputtering. Various sputtering power was used for the deposition while Ar/O₂ ambient is maintained at 2/1 ratio. The deposition rate was measured using a surface profilometer. In order to fabricate sandwich

device structure, circular gallium-doped ZnO (GZO) top electrodes were deposited onto the ZnO/ITO structure; patterned using a metal shadow mask with 150 μm in diameter. X-ray diffraction and X-ray photoelectron spectroscopy were used to investigate the crystal structure and the defect concentration of the ZnO films, respectively. A semiconductor device analyzer was used to measure the electrical characteristics of the devices.

XRD pattern indicated that all ZnO films show (002) oriented crystals. The devices made with higher sputtering power showed lower leakage current for OFF state and lowered current compliance to form the conducting filament (for ON state). We also found that the donor defects (oxygen vacancies and zinc interstitials) decrease as the sputtering power increases. These findings indicate that the sputtering power parameter cannot be simply overlooked in the T-RRAM fabrication.

References

- ¹ F.M. Simanjuntak, D. Panda, K. Wei, and T. Tseng, *Nanoscale Res. Lett.* **11**, 368 (2016).
- ² F.M. Simanjuntak, O.K. Prasad, D. Panda, C.-A. Lin, T.-L. Tsai, K.-H. Wei, and T.-Y. Tseng, *Appl. Phys. Lett.* **108**, 183506 (2016).
- ³ T. Ohno and S. Samukawa, *Appl. Phys. Lett.* **106**, (2015).

11:20am **TF+EM+MI-WeM11 Influence of Intrinsic and Extrinsic Dopants in HfO_x Films for Resistive Switching Memory**, *SungYeon Ryu, Y. Kim*, Seoul National University of Science and Technology, Republic of Korea; *W.Y. Park, S.G. Kim*, SK Hynix Inc., Republic of Korea; *B.J. Choi*, Seoul National University of Science and Technology, Republic of Korea

Bipolar type resistive switching random access memories (RRAMs) utilized by TaO_x and HfO_x as switching materials, have been demonstrated superior to unipolar type RRAMs owing to the low switching current and better reliability. Bias-polarity-dependent valence change mechanism in HfO_x is attributed to the repeated generation and rupture of conducting filaments (CF), which could be the clusters of oxygen vacancies (V_o). As the device size scales down to 2x nm, the dimension of CF and the whole device area become comparable, and thus the involvement of oxygen loss or unintended V_o incorporation, that is, intrinsic defects may significantly degrade the device reliability. On the other hand, extrinsic defects by doping metallic elements may also change the switching characteristics and device variability depending on the fabrication methods, such as, co-sputtering, implantation, thermal and photo-assisted diffusion, etc.

In this study, influence of both intrinsic and extrinsic defects was investigated systematically. The device having TiN contact-plug with 28 to 2000-nm-diameter formed in SiO₂ inter-layer was fabricated. 1.2-nm-thick HfO_x layer was deposited by atomic layer deposition (ALD). At first, for the control of intrinsic defect, the fabricated RRAM devices were post-annealed in both air and vacuum (~10mTorr) at a wafer temperature of 150 to 250 °C for 20 min, respectively. Electrical property is measured by semiconductor parameter analyzer (HP-4155) and function generator (AFG-3102) for DC and AC measurements. More reliable switching and free-from abnormal switching behavior, such as reset-failure, were observed in the vacuum-annealed device.

Secondly, Al- and Ga-doped HfO_x films are grown by using thermal ALD to change the structural and chemical properties of HfO₂ film via incorporating extrinsic defects. Dopant concentration and location were controlled by the ALD cycle ratio and sequence. Through Al doping, pristine resistance and forming voltage of the device were increased with increasing Al cycle ratio. On the other hand, Ga-doped HfO₂ device showed reverse switching polarity. The resistive switching characteristics caused by intrinsic and extrinsic defects in HfO_x films will be presented in detail.

Acknowledgment

This paper was result of the research project supported by SK hynix Inc.

11:40am **TF+EM+MI-WeM12 Scaling up of an Electrochemical Atomic Layer Deposition of Copper**, *D. Dictus*, Lam Research Corporation, Belgium; *Aniruddha Joi*, Lam Research Corporation; *G. Alessio Verni*, Lam Research Corporation, Belgium; *K. Vandersmissen*, Imec, Belgium; *B. Frees*, Lam Research Corporation, Belgium; *Y. Yezdi*, Lam Research Corporation

Just like atomic layer deposition in the gas phase, electrochemical atomic layer deposition in the liquid phase holds the promise of delivering very good conformality and uniformity for nm-thick film deposition by using surface limited reactions. Up to today however, there is no industry in which such process has been scaled up from cm-size coupons to industrial-scale substrates. In this paper, the scale up of electrochemical ALD of Cu is reported and it is demonstrated that this process can be used to fill <20 nm Cu interconnect lines as required for future microchip technology nodes.

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The e-ALD process is a cyclic process that consists of a step in which a monolayer of Zn is deposited at a potential below the one, at which, Zn would grow multilayers (underpotential deposition), followed by a step in which the zinc is spontaneously displaced by Cu when the substrate is allowed to drift to the open circuit potential (surface limited replacement reaction, SLRR). By cycling between potentiostatic Zn deposition and open-circuit steps, Cu films can be grown in layer-by-layer fashion.

In contrast to classical electroplating processes, e-ALD processes do not require significant overpotential to create uniform nucleation. Also, current densities are small since the deposited amount of material per cycle is low. Therefore, the e-ALD process can be used to deposit Cu with atomic layer thickness control on substrates with high resistivity which gives the advantage of being able to plate on very thin (sub-20 Å) liner materials.

Development of the e-ALD process is done on a Sabre electroplating tool from Lam Research on 300 mm substrates. These substrates contain a thin layer of Ru or Co on which the copper is deposited. The e-ALD process forms the seed layer for further metallization of the interconnect or can by itself fill the narrow interconnect lines.

We will demonstrate that good thickness uniformity can be achieved on 300 mm substrates with initial sheet resistance up to 1000 Ohm/sq and that filling of interconnect lines with dimensions smaller than 14 nm is achieved. Test chips containing our new Cu deposition process show high yield and the Cu interconnect lines have low resistance. This demonstrates the potential use of this new production method in future technology nodes.

12:00pm TF+EM+MI-WeM13 A Novel High-deposition-rate PECVD Process based on Hollow Cathode Plasma Technique, S. Shayestehaminzadeh, N. Rivolta, AGC Glass Europe, Belgium; M. Datz, Interpane E&B GmbH; John Chambers, AGC North America; H. Wiame, AGC Glass Europe, Belgium

AGC Plasma has recently developed a hollow cathode plasma device as part of its activity as the industrial vacuum equipment manufacturing segment of AGC. This device has been successfully scaled up in order to perform the Plasma Enhanced Chemical Vapor Deposition (PECVD) processes for the coatings on the glass sizes ranging from a few square-centimeters up to a few square-meters (jumbo-sized glass plates 3.2 x 6.0 m²). Plasma generation is undergone between multiple linear hollow cathode cavities, alternately driven by commercially available mid-frequency AC or pulsed power. This enables the generation of a dense and uniform plasma inside the cavities as well as outside of the cavities through the linearly located holes where a less dense plasma (as compared to inside the cavity) but still dense enough, can be utilized in order to activate the reaction between precursor and reactive gases in the depositing chambers.

This geometry and condition allow the system to be scalable to relevant sizes for any typical vacuum processes.

The selected plasma generation method does not require magnets incorporated into the source. This will allow a simplified cooling system within the plasma source, as well as the possibility for adding other magnets to be positioned within the process chamber for modifying the plasma geometry. Based on plasma and vacuum simulations, the electrode-cavity design has been dramatically improved and can be driven by various gases, such as oxygen, nitrogen, argon, helium, and hydrogen. Plasma operation is also possible in an extremely wide range of pressures from 1 mTorr to 1 Torr, while the most useful process pressures for PECVD are generally in the 1 mTorr to 30 mTorr range. The wide hollow cathode pressure operation range allows for the deposition of multilayer coating stacks by magnetron sputtering and PECVD in the same vacuum coating lines.

Electrode faces have also been enhanced using surface coatings to avoid erosion during high power operation, also reducing subsequent debris and extending source lifetime. Depending on the choice of precursor materials, the desired film properties, and in-chamber magnetic field geometries, dynamic deposition rates for inline PECVD coatings of 200-500 nm³/min have been demonstrated for SiO₂ on flat glass products however by selecting different gases and precursors various materials can be deposited using the same source line. This unique process opens a new range of possibilities to be investigated for a variety of substrates. These plasma sources are now commercially available in either lab or industrial sizes to explore new applications.

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