ALD for Manufacturing
Room Plaza Exhibit - Session AM-MoP
ALD for Manufacturing Poster Session

AM-MoP-1 Anhydrous Hydrogen Peroxide Gas Delivery for Semiconductor Manufacturing: Optimal Delivery Conditions for ALD Processes*, D Alvarez, J Spiegelman,* ***Keisuke Andachi****, R Holmes, Z Shamsi,* RASIRC

Introduction

H_2O_2 gas is a novel oxidant for ALD that improves passivation and nucleation density at semiconductor interfaces, potentially leading to reduced interfacial defect density.[1,2] We have previously reported a new technology capable of generating and delivering stable anhydrous H_2O_2 gas.[3] The method utilizes a solvent based H_2O_2 solution, a carrier gas and membrane pervaporator in order to deliver anhydrous H_2O_2. Several studies have reported on the advantages of hydrogen peroxide as an ALD oxidant versus water and ozone.[4,5]

Heat and Materials Compatibility Challenges

In practice, ALD process limitations have been found due to Manufacturing tool configuration and the heating of delivery lines as well as other components. H_2O_2 is thermodynamically unstable and can decompose into H_2O and O_2 on metal and other material surfaces. The decomposition rate increases with temperature and/or concentration. Most ALD manufacturing tools incorporate long heated lines and/or incompatible materials, and thus are not readily suitable for H_2O_2 use.

Materials Decomposition Study

The decomposition rates of various tube materials have been examined at elevated temperature. These materials included: Pre-conditioned SS; [https://en.wikipedia.org/wiki/Fluorinated_ethylene_propylene] (FEP) - coated SS; and SS with a metal oxide coating. The electropolished SS316L tubing (1/2"x3m) was passivated with H_2O_2 gas at 140C for 8 hours. FEP and the metal oxide coating were applied on the inner wall of the SS tubing by solvent based methods. H_2O_2 concentration vs temperature was monitored by FT-IR. H_2O_2 decomposition rates are significant at T> 90C for the Preconditioned SS and FEP materials. In contrast, the metal oxide coating mitigates decomposition from 60–140C, thus can enhance process performance. Additional material surface results for Hasteloy and Aluminium oxide will be presented.

References

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AM-MoP-2 OpenALD - A Framework for an Open Source ALD Reactor*,* ***Vivek Dwivedi****,* NASA

In general terms the equipment that is used for a fully functioning Atomic Layer Deposition reactor is constant from reactor to reactor with specialized preprocessing, post processing and in situ characterization tools such as RGA's, QCM's and ellipsometers. In all reactor builds the following are constants: precursors are pulsed into a chamber volume utilizing high speed pneumatically actuated valves, reactor pressure is monitored using a pressure transducer/manometer, purge gas flow rates and constant reactor baseline pressures are set via mass flow controllers and thermocouples are used to monitor chamber temperature. Specialized commercial software is typically used to preprogram a desired recipe and for *in situ* diagnostics. This specialized software can be cost prohibited, lack expansion functionality and be a deterrent to fully understanding the inner workings of the deposition tool. This talk will introduce an OpenALD framework for reactor control and monitoring that utilizes the open source software Python and it's extension libraries to control multiple ALD reactors while provide flexibility and cost savings measures that can be is easily implemented. Various algorithms, techniques and code examples will be discussed along with implementation success stories.

AM-MoP-3 Multilayer ALD Metal Oxide Films Deposited by Spatially Resolved ALD Processes for Moisture Barrier Films*,* ***Sang Heon Yong****, S Kim, H Chae,* Sungkyunkwan University (SKKU), Republic of Korea

Organic light emitting diodes (OLEDs) are considered as flexible future panel displays due to many advantages such as high color quality, thin thickness. Thin film encapsulation (TFE) is one of essential technologies required flexible display to protect OLEDs from moisture and oxygen.[1] Inorganic films deposited by atomic layer deposition (ALD) process have demonstrated high barrier performance. But, extremely low throughput of ALD process is a major weakness for commercialization. To overcome the low throughput of ALD, high throughput 'spatial ALD' processes have been studied.[2] To achieve high flexibility of barrier layers, multilayer structure has been studied with various inorganic and organic layers [3]. In the multilayer structure, the inorganic layers typically function for the permeation barrier and organic layer for stress relief with increased diffusion pathway of moisture and oxygen.

In this study, Al_2O_3 films were deposited by a spatial ALD process. We characterized various process conditions such as the flowrate of Al precursor and oxygen precursor, scanning speed. We also investigated carbon-rich Al_2O_3 films by controlling the deposition conditions and observed the improved flexibility of the carbon-rich Al_2O_3 films as carbon contents increases in the barrier films. This carbon-rich Al_2O_3 films potentially replace to organic layer without losing flexibility much as shown in Figure 1. Water vapor transmission rate (WVTR) was determined by a calcium resistance test and optical test, and less than 10^{-4} g/m^2 ·day was achieved. Samples were analyzed by x-ray photoelectron spectroscopy (XPS) and field emission scanning electron microscope (FE-SEM).

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AM-MoP-4 A Green Precleaning Process in Wettability Improvement for Thinner and Uniform ALD Al_2O_3 Film Deposition on Layered MoS_2 Film*,* ***Cheng-Ying Wang****,* National Taiwan Normal University, Taiwan*; Y Ho, Y Chu,* National Chiao Tung University, Republic of China*; H Hsu,* ITRI, Republic of China*; B Chen, P Chen,* Minghsin University of Science & Technology, Taiwan*; M Lee,* National Taiwan Normal University, Taiwan*; C Jong,* NARLabs, Republic of China

Layered-transition metal dichalcogenides (TMDCs) with tunable energy band gap is promising for many electrical or optical devices application. However, the progress for the device fabrication and characterization are limited because of some integration issues which have not been overcome yet. Up to date, better device data was obtained from the exfoliated film transferred onto a dielectric substrate and source/drain contact metal processed by lift-off process. Furthermore, most of the studied transistor is operated by back gate structure because of poor wettability for ALD HK gate dielectric deposition. It was reported that an oxygen plasma treatment[1], UV-O_3 atmosphere treatment[2] or bi-layer dielectric stacking structure[3] were studied to form a thinner and continuous dielectric layer on TMDCs. Even that, a continuous film thicker than 10 nm and a defective interface is expected.

In this study, we focus on the clean process development by dipping TMDCs film into an alkaline solution (patented by Chu Chi Industrial Co. Ltd) for surface energy tuning. The process was carried out at room temperature. The PH value of the solution is above 10. Previous works[4-5] shown that the alkaline solution could effectively create a hydrophilic surface on bare Si wafer at lower temperature and in shorter time than conventional SPM process (using H_2SO_4+H_2O_2 >120C for 10mins) in current Si ULSI process. The C-V electrical data shown a defect-free interface between HK and Si substrate. Herein, a PLD direct growth MoS_2 film on sapphire and a MoS_2 film on SiO_2/Si substrate formed through sulfidation of tiny sputtering MoO_x were studied. The film thickness were around 5-6 nm and confirmed by cross section TEM. The surface energy of TMDCs films were examined by contact angle observation. The contact angle reduced and then kept from 49 to 38 degree for the non-treated and 10

second treated sample. The high k dielectric layer Al_2O_3 film was deposited by atomic layer deposition after the solution precleaning process. The film thickness measured by ellipsometry and confirmed by TEM is 10 nm. A clean and uniform interface between the Al_2O_3 film and the MoS_2 film was observed. The electrical properties of the dielectric layer will also be discussed. This work proposes a green and promising precleaning process for TMDCs before high k film deposition.

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AM-MoP-5 Highly Sensitive Ion Trap Mass Spectrometer for Inline Process Control*, **Ruediger Reuter**, V Derpmann, G Fedosenko, A Laue, T Graber, M Aliman, H Chung,* Carl Zeiss SMT GmbH, Germany

Real-time inline control of process gas compositions with high sensitivity has been of particular importance in recent years in the semiconductor industry and beyond. Most of the real-time process gas analysis was carried out with differentially pumped Residual Gas Analyzer (RGA) which are based on a linear quadrupole mass filtering technique. The sensitivity of this technology is limited by the dynamic range of the secondary electron multiplying detector and its strong mass discrimination with increasing m/z–ratio. To generate a complete mass spectrum, a RGA usually needs a few minutes which is often too slow for real-time inline process control. A new mass spectrometer, based on Fourier-Transform 3D-Quadrupole Ion Trap technology, is more appropriate for real-time process control and will be presented in this work.

The 3D-Quadrupole Ion Trap mass spectrometer (*iTrap*) by ZEISS is installed in a vacuum chamber (~120mm x 120mm x 120mm) with a fast sampling valve for pulsed gas injection (pulse duration ~ 20ms). An electron gun is used to ionize the gas. The Ion Trap achieves ion trapping and accumulation by means of a radio frequency voltage applied to the ring electrode of the trap. With the aid of advanced electronic amplifiers and selective ion excitation technique, a very small current, generated by the ion oscillations, is induced on the upper and lower electrodes of the ion trap and can be measured electrically without using any separate particle detector. A mass spectrum is finally obtained by a Fourier Transform of the recorded electrode current signal in less than one second.

Real-time measurements of the hydrogen plasma cleaning process of Sn contaminated samples were performed with the *iTrap* mass spectrometer. The working pressure of the plasma cleaning process was 0.5 mbar. Decreasing signal of SnH_4 and other contaminations from the samples which are directly correlated to the cleaning process were observed with *iTrap*. This result is extremely useful for the process control of plasma processes and inline real-time contaminations control for high-end applications.

Measurements were also performed on a deposition chamber monitoring the deposition process and the cleaning steps in between. Fast dynamic changes in process gas (C_2F_4, O_2, NF_3, H_2) and reaction gas composition could be detected with a repetition rate of ~1 Hz.

AM-MoP-6 STD-PEALD Equipment Design and Evaluation of Nano Thin-Film Characteristics*, M Jeong,* Korea Electronics Technology Institute, Republic of Korea*; T Ryu,* Sung Kyun Kwan University*; K Hong,* Korea Electronics Technology Institute, Republic of Korea*; J Choi,* Sung Kyun Kwan University*; C Song, M Koo,* LEED Corp.*; **Yekyung Kim**, S Chang,* Korea Electronics Technology Institute, Republic of Korea*; I Jeong,* LEED Corp.*; H Kim,* Korea Electronics Technology Institute, Republic of Korea

The spatial atomic layer deposition (ALD) technique has been developed to complement the drawbacks of the time-dependent conventional ALD technique. However, the spatial ALD technique still has some weaknesses of low productivity, large equipment size which results in high developing cost and so on. The plasma-enhanced ALD (PEALD) which is controlled by time and space in a device, named space and time divided PEALD (STD-PEALD), was developed, in this study. The fast reciprocating motion of substrate with short distance as much of an injector pitch in the proposed STD-PEALD enables to reduce the size of the equipment and to ensure high productivity compared with the spatial ALD constructed so far. Also, the sequential exposure of precursor and reactive gas decreases the dust problem, generally occurs in spatial ALD process. The detailed structure of the proposed STD-PEALD was designed based on the results of thermal-structural coupled field analysis considering both the effect of heat and load produced during operation. Thermal stress was examined by the existence of a heat source. Also, load stress and deformation of the equipment were analyzed to determine the optimum design of the moving parts for the substrate which were made of electromagnetic coils. Additional analysis of fatigue life for the developed equipment was performed regarding of operation parameters--time, speed, etc. Finally, the performance of the developed STD-PEALD was verified by deposition of Al_2O_3 on various films. The high deposition speed and quality of deposition layer of the STD-PEALD were confirmed in this study.

AM-MoP-7 Demonstration of a Correlation between Barrier Property and Defect Visualization of ALD(Al_2O_3)/Graphene Film*, K Hong, M Jeong, D Lee, J Seo, S Lee,* Korea Electronics Technology Institute, Republic of Korea*; J Choi,* Sung Kyun Kwan University*; I Jeong,* LEED Corp.*; S Chang, **Yekyung Kim**, H Kim,* Korea Electronics Technology Institute, Republic of Korea

Graphene, which shows excellence in mechanical and electrical properties, has been widely researched in various fields. Due to the increase of research areas and demand of usage, commercialization of graphene with its outstanding properties is required. Quality control of mass-produced graphene is a crucial factor for large-scale production. However, the defects which degrade mechanical and electrical properties of graphene occur during synthesis and transfer process. To examine the quality of graphene, we developed an inspection system which can visualize the graphene grain boundary and defects precisely and fast. The visualization system consists an optical microscope with long working distance, a heating stage and gas supply system. Moreover, in this study, the Al_2O_3 layer was deposited by atomic layer deposition (ALD) method on the graphene prepared by chemical vapor deposition (CVD) method. The Al_2O_3/graphene/Cu film was then applied to the developed visualization stage to evaluate the effect of the existence of Al_2O_3 layer by ALD. The visualization strategy is based on the oxidation behavior of graphene and substrate Cu under the temperature and atmosphere condition, especially humidity of the air. Defect visualization of various thicknesses of ALD layer on graphene/Cu were carried out together with the water vapor transmission rate (WVTR). The effect of barrier property of Al_2O_3/graphene film on defect visualization was researched in this study.

AM-MoP-8 Transport and Kinetics of a Remote DBD Plasma for ALD Processing of Metal Oxides*, T Beekman, **Yves Creyghton**, J Emmelkamp,* Solliance/TNO, Netherlands*; A Sobota,* Eindhoven University of Technology, Netherlands

Different types of dielectric barrier discharge (DBD) plasma sources are being used in ALD platforms at Solliance. Replacement of H_2O by O_2/N_2 plasma in the thermal TMA-H_2O process offers various benefits such as increased speed (shortening purge periods) and low temperature deposition of alumina (100-150^oC). Another example is improved composition control in mixed metal oxide ALD such as Indium zinc oxide. We use linear plasma sources with a thin plasma volume (~0.1 mm) ending short above the moving substrate (~0.2 mm). Flow velocities at the plasma slit nozzle are in the 1-20 m/s range. The DBD plasma is generated with alternating pulses with 5 kV amplitude, 50 kHz repetition frequency and 300 W/cm^3 energy density. In order to optimize the geometry and plasma operating conditions for high radical flux homogeneity at moderate gas flows, an integrated fluid dynamics and chemical kinetics model has been set-up using Comsol modelling software. The model assumes constant densities of atomic radicals (O, N) and excited states of N_2 derived from published experimental data. The chemical kinetic reaction rates are obtained from validated models of O_3 production in atmospheric air. The model includes temperature dependent diffusion of radical and molecular gas species, surface recombination of radicals on metals and dielectric source materials, as well as heat transfer by conduction and flow. Both radical and heat production are taken into account as a boundary condition on the dielectric barrier. The calculated oxygen radical flux towards the substrate is compared with the oxygen content in a monolayer of Al_2O_3, based on the measured growth per cycle of 0.16 nm. In order to further validate our model a series of dedicated experiments with the plasma source has been performed. Stationary etching of a 40 nm thick amorphous

carbon layer provides detailed information on the spatial distribution of reactive nitrogen species arriving at the substrate. Both temperature measurements within the plasma source and in the process gas directly downstream the nozzle slit have been performed. Finally the influence of the O_2/N_2 ratio on measured O_3 has been determined. Since the model predicts a lower O radical flux from the gas towards the substrate than needed for saturated growth we assume that dissociative recombination of O_3 into O_2 and O at the CH terminated surface contributes to layer growth. In-depth understanding of the influence of the flow distribution and surface recombination of radicals on plasma source and substrate materials has been gained and used for optimization of process settings and geometry.

AM-MoP-9 Effects of Sealing Components on ALD Film Quality*, Fred Pourmirzaie,* Flodynamix

Atomic layer deposition (ALD) of high quality thin films has recently penetrated manufacturing lines of several major memory and logic manufacturers due to the promise of unprecedented control of thickness, uniformity, quality and material properties. ALD tools were designed around the anticipation that future ultrathin materials are likely to be binary, ternary or quaternary alloys or nanolaminate composites. A unique chemical delivery system enables synergy between traditional, production-proven low pressure chemical vapor deposition (LPCVD) technology and atomic layer deposition (ALD) controlled by sequential surface reactions. Source chemicals from gas, liquid or solid precursors are delivered to arrive on reactive surfaces where self-limiting surface reactions yield film growth with layer-by-layer control. Surfaces are made reactive by the self-limiting reactions, by surface species manipulation, or both. The substrate is exposed to one reactant at a time to suppress possible chemical vapor deposition (CVD) contribution to the film. Precisely controlled composite materials with multiple-component dielectric and metal–nitride films can be deposited by ALD techniques. The research community has demonstrated these capabilities during the past decade. Accordingly, ALD equipment for semiconductor processing is unanimously in high demand. Sealing parts like O-rings and lip-seals used to isolate chamber from outside world play a critical role, more so in ALD processes than any other Semiconductor manufacturing process. This is because any minute outgassing or permeation through O-ring polymer poses grave risk to quality of film deposited. Conventional Perfluoroelastomer (FFKM) O-rings with inherent porosity are not the ideal material of choice for ALD processes. In this article, we will demonstrate the effect of outgassing and permeation from sealing parts on ALD film uniformity, stoichiometry and overall thin film quality. Flodynamix LLC has developed a unique fluoropolymer called Kratos® ideal for ALD and PVD processes.

Viton® Kratos® FFKM

Permeation of O_2 1.8 1.3 7.4

Permeation of He 15.5 13.2 72.0

Permeation of N_2 0.06 0.05 9.5

Unit in 10^{-8} sccm-cm/sec-cm^2-atm @ 25°C

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ALD for Manufacturing
Room Plaza F - Session AM+EM-TuA
ALD for Manufacturing (1:30-3:30 pm)/MLD II (4:00-5:00 pm)

Moderators: Maarit Karppinen, Aalto University, Paul Ma, Applied Materials, Inc., Mike McSwiney, Intel, USA, Sean Smith, Sandia National Laboratories

1:30pm **AM+EM-TuA-1 Large Area Spatial Atmospheric ALD**, ***Corné Frijters***, *F van den Bruele, F Grob, A Illiberi, P Poodt,* TNO/Holst Center, Netherlands

Atmospheric spatial ALD (sALD) is able to deliver high deposition rates while maintaining the advantages of conventional ALD, such as low defect density, high conformality and thickness uniformity. First industrial applications of spatial ALD include passivation of c-Si solar cells and roll-to-roll manufacturing of flexible barrier foils. An emerging application for Spatial ALD are flat panel (OLED) displays. We have developed spatial ALD processes for making high mobility oxide semiconductors for the TFT backplane as well as thin-film encapsulation for the OLED front plane.

As today's displays are fabricated using glass panels in the order of several square meters, a remaining challenge is the development of large-area sALD deposition technology that is able to combine high throughput with uniform performance across large areas.

As an intermediate step between the lab and the display fab, we have installed a large area sALD sheet-to-sheet tool which can handle up to 400x325 mm^2 sized substrates able to deposit uniform films across a deposition width of 300 mm. This large area sALD tool is comprised of an RTP oven to quickly heat up the substrate and an ALD reactor with a maximum deposition temperature of 350 ^oC. The whole system is operated under an inert N_2 atmosphere. The substrate is transported into the tool by an automatic conveyor system to the ALD reactor and placed on a substrate carrier which is operated by an air bearing to allow for fast and uniform motion underneath the injector head where the deposition takes place.

We will present the initial results achieved with this tool where layer thickness non-uniformities of <1% have been achieved for AlOx layers deposited at 125 ^oC and a substrate speed up to 21 m/min, equivalent to 30 ms exposure times as well as excellent compositional uniformities for binary oxides. Furthermore we will demonstrate excellent multilayer thin-film encapsulation results on polymer foil-on-carrier by the large area sheet-to-sheet tool.

1:45pm **AM+EM-TuA-2 Low Resistivity Titanium Nitride ALD: Low Temperature Enabled by the Use of Ultra-High Purity Hydrazine**, ***Daniel Alvarez***, *J Spiegelman, K Andachi, R Holmes,* RASIRC; *A Kummel, S Wolf, M Kavrik,* UCSD; *M Raynor, H Shimizu,* Matheson Tri-Gas

Introduction

Next generation channel materials (SiGe, Ge and InGaAs) place very difficult thermal constraints (<400°C) on metal nitride deposition methods. New low temperature methods are needed for ALD of TiN_x metal gate electrodes. Previous studies have shown viable reactivity for hydrazine but have been limited by hydrazine purity, specifically water contamination [1-3]. This has been attributed to the low purity of commercially available 'Anhydrous' Hydrazine, which has water concentration ranging from 0.2-2.0%. Also, high concentrations of oxygen found in the resulting Metal-Nitride films range from 4-15% for SiN_x and TiN_x.

We have previously reported the safe delivery of gaseous hydrazine from a solvent-based formulation and membrane delivery system. [4] Here we present our studies on water measurement and removal, as well as hydrazine-based low temperature TiN_x ALD to obtain films with low resistivity.

Hydrazine Measurement and Purification

New purification methods have led to water contamination levels of <50 parts-per-million (ppm) in the hydrazine source formulation as measured by Karl-Fischer and GC-MS methods. Gas phase moisture measurements by FT-IR have been developed to a lower detection limit of 0.83ppm. The gas phase output of the ultra-dry material has been measured to below this detection limit (Figure 1). This compares to 31ppm for a standard commercially available material.

TiN_x Film Deposition and Characterization

Atomic layer deposition of near stoichiometric TiN_x was achieved on an SiON substrate by sequential pulsing of $TiCl_4$ and N_2H_4 precursors at a substrate temperatures of 275°C-350°C, where growth rate is approximately 0.5A per cycle. Films have been characterized by XPS (Figure 2), AFM, KPFM, STM and Four-point sheet resistance measurements. A TiN_x film with near stoichiometric ratio of Ti/N and the absence of added oxygen was achieved. Most significant is an initial measured Resistivity of $R_{average}$ =130 ohm, R_{sheet} = 50 ohm. Additional optimization of these initial results will be presented.

References

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2:00pm **AM+EM-TuA-3 Modeling Ampoule Performance for Low Vapor Pressure Precursor Delivery**, ***James Maslar***, *W Kimes, B Sperling,* National Institute of Standards and Technology; *W Kimmerle, K Kimmerle,* NSI

Low vapor pressure organometallic compounds are widely utilized as precursors for ALD processes. Such precursors are often delivered using an inert carrier gas that is passed through either a bubbler (an ampoule with a dip tube) or a vapor draw ampoule (no dip tube: the gas in and gas out ports open directly into the ampoule headspace), depending on the precursor properties and process requirements. However, it can be difficult to predict how much precursor will be delivered for a given carrier gas flow rate, or even the relationship between precursor and carrier gas flow rate. A number of factors may contribute to this difficulty, including a pressure drop between the ampoule and the pressure control location, sublimative/evaporative cooling of the precursor (i.e., cooling reduces the precursor vapor pressure and hence the amount of material entrained in the carrier gas), incomplete saturation of the carrier gas (i.e., the precursor sublimation rate is too low to permit saturation of the carrier gas for a particular residence time in the ampoule), or slow mass transport processes in the ampoule. Furthermore, the relative importance of the factors depends on the precursor, ampoule design, and process conditions. The goal of this work is to develop analytical and numerical models that can be utilized to characterize ampoule performance, including establishing the precursor and carrier gas flow rate relationship. Models for both bubblers and vapor draw ampoules were developed. A variety of low vapor pressure precursors, e.g., CCTBA and PDMAT, and precursor simulants, e.g., hexadecane, were employed in this investigation. The focus of this investigation was on commercial 1.5 L-volume ampoules (with a maximum fill of 1.2 L). All ampoule models were experimentally validated using data that was obtained with custom-designed optical mass flow meters located downstream of the ampoule. For selected ampoules, resistance temperature detector arrays were employed in order to characterize the liquid temperature distribution in the ampoule and level sensors were employed to confirm liquid level in the ampoule. It is expected that the development of such models can facilitate both process optimization and development of improved ampoule designs.

2:15pm **AM+EM-TuA-4 An innovative chamber designed for ALD, PECVD and FAST® SiO_2 processes: towards high throughput and conformal deposition at low temperature**, ***Laetitia Bonnet***, *F Piallat, J Vitiello,* KOBUS, France

Since transistors are employed into integrated circuits, miniaturization of the components is the driver of their evolutions. 3D packaging is already used to stack and interconnect the components thanks to vertical vias driving through the silicon substrate. To prevent important energy loss by leakage, a liner is used in these vias, thanks to its great electrical properties silicon oxide is the most used dielectric material. However, SiO_2 deposition in Through Silicon Via is currently facing a challenge: a thick and conformal layer is needed, with a deposition temperature as low as possible for integration in thermally sensitive devices.

The use of a high power plasma, in the plasma-enhanced CVD method (PECVD), allows to achieve suitable electrical properties, while decreasing the substrate temperature down to temperatures as low as 350°C[1]. Nevertheless, with sensitive substrates the plasma power generated is high enough to damage the devices. In an ALD mode, with or without plasma assistance, the substrate temperature can be down to room temperature when a catalyst is added to the deposition cycle[2]. However, the low

deposition rate, characteristic of ALD, is not compatible with the production demands, where thick layers (> 100 nm) are used.

In this context and to merge CVD and ALD advantages, the Fast Atomic Sequential Technique (F.A.S.T.®) was developed. Dedicated hardware solutions were found to allow a complete separation of the reactant and precursor until the substrate[3]. FAST tool can be used in continuous mode, in pulse-purge mode or in pulse only mode (fig.1). Similar approaches were presented in the past and referred to "purge-less ALD" or "pulsed-CVD", the novelty resides here in the possibility to process all three techniques: CVD (fig.1a), ALD (fig.1b) and FAST (fig.1c) in the same chamber. Moreover, the process window is now extended with a new parameter: the pulse position; i.e. pulses can overlap for a CVD-like process or be well separated for ALD-like process.

For this study, the well known silicon precursor TetraEthyl OrthoSilicate (TEOS) was employed in all three deposition modes. A comparison of the growth rate (fig.2), conformality (fig.3) and films properties of the SiO_2 deposited by ALD, PECVD and FAST in the same reactor and with similar process conditions will be discussed. For example, SiO_2 deposited by FAST mode shows a growth rate 10 times faster than in ALD mode while the conformality reaches at least 70% for vias with aspect ratios up to 8:1 (compared to only 30% obtained in CVD). Additionally, optical and electrical properties were investigated and will be presented, as optical index, breakdown voltage and leakage current.

2:30pm **AM+EM-TuA-5 Growth Rates During Silicon Spatial Electron-Enhanced Atomic Layer Deposition: Role of Dangling Bond Lifetime*,* *Andrew Cavanagh,*** *S George,* University of Colorado

Electron enhancement can dramatically reduce the temperatures required for ALD. Electrons can desorb surface species, such as hydrogen, by electron stimulated desorption (ESD) and create "dangling bonds". These dangling bonds can then facilitate precursor adsorption leading to low temperature thin film growth. However, other residual species can also consume the dangling bonds and reduce the dangling bond lifetime. Film growth rates may then be reduced after longer times between the generation of the dangling bonds and their consumption by precursor adsorption.

Silicon films were grown using spatial electron-enhanced ALD (SEE-ALD). There was linear motion of the 6" wafer back and forth between the electron source and the disilane (Si_2H_6) precursors (see supplemental Figure S1). A DC glow discharge plasma was employed as the electron source. Si SEE-ALD growth rates were measured versus the electron dose time and the time between the electron dose and the subsequent Si_2H_6 exposure. In one experiment, Si thicknesses were measured versus position on the 6" wafer after 200 SEE-ALD cycles using two different wafer velocities under the electron source (see supplemental Figure S2).

Spectroscopic ellipsometry measurements revealed that the Si growth rate increased with electron dose time. The Si growth rate also decreased versus time between the electron dose and the subsequent Si_2H_6 exposure. A model was developed to explain the Si growth rates based on number of dangling bonds formed during the electron dose time, t_1, and the dangling bond loss during the time, t_2, between the electron dose and the subsequent Si_2H_6 exposure. The surface coverage of dangling bonds from hydrogen ESD is $\Theta = 1-\exp[-t_1\Phi\sigma]$, where Φ is the electron flux and σ is the hydrogen desorption cross-section. The dangling bond coverage is proportional to $\exp[-t_2/\tau]$ where τ is the dangling bond lifetime.

A dangling bond lifetime of τ = 113 s and hydrogen desorption cross-section of $\sigma = 8 \times 10^{-20}$ cm² were determined by fitting the model to the growth rates versus t_1 and t_2. The results indicate that a higher wafer speed to lower t_2 is needed to minimize the effect of the dangling bond lifetime. This higher wafer speed will not produce a lower growth rate if the electron flux is also higher. We are currently working to increase the electron flux by using a hollow cathode plasma electron source.

2:45pm **AM+EM-TuA-6 Spatial Atomic Layer Deposition of Gate Encapsulation Silicon Nitride for Self-Aligned Contact Enablement*,* *Jiehui Shu*,** *S Mehta, J Chang, X Qiu, J Liu,* GLOBALFOUNDRIES U.S. Inc.

Aggressive gate pitch scaling and shrinking contact CDs beyond 14nm node require precise control of contact to gate overlay to avoid S/D contact to gate shorts [1]. Therefore, Self-Aligned Contact (SAC) process is widely used for 14nm node and beyond for gate to contact overlay margin improvement. This is achieved by recessing the gate (W) and work function (TiN) metals post W CMP selective to oxide, and filling the space above W with a robust dielectric capping material. The dielectric fill is polished to be coplanar with oxide. The contact plugs are then opened by high selective SiO2 etch to Si3N4. Enough Si3N4 thickness is needed to prevent gate to contact shorts due to Si3N4 erosion during contacts open etch [2]. The key requirements for such a gate capping application are, a) defect free fill, b) adhesion to the gate metal and low-k spacer, c) low deposition temp (< 500C) with reduced in-film hydrogen for gate Vt stability, d) compatibility with downstream wet and dry etch processes e) selectivity in contact etch (selective in oxide dry etch process) and f) low leakage and high Vbd (>6MVcm). In this paper, we present the results of low temperature spatial atomic layer deposition (ALD) gate encapsulation Si3N4 process for 7nm self-aligned contact enablement. Blanket Si3N4 film properties at different process temperatures were characterized, such as etch selectivity, wet etch rate, growth rate, uniformity, etc. In-film Hydrogen content is associated with various film properties such as etch selectivity, wet etch rate. The lower the hydrogen content in Si3N4 film, higher its etch selectivity and lower the wet etch rate. HFS and FTIR are used for hydrogen content analysis in this work. By changing different precursor exposure sequence in spatial ALD process, we are able to modulate the hydrogen content in Si3N4 film. I-V curves and Vbd were obtained using MOS structures.

[1] S. Demuynck et al., IITC 2014

[2] C. Auth et al., VLSI2012

3:00pm **AM+EM-TuA-7 Fast Atomic Layer Deposition Process for Thin-Film Encapsulation of Organic Light-Emitting Diodes*,* *Tony Maindron,*** *C Lopez, S Meunier Della-Gatta, M Tournaire, B Caulfield, M Gontier,* CEA-Leti, France*; J Kools, L Baril,* Encapsulix, France

Thin-film encapsulation Organic Light-Emitting Diodes (OLED) consists in the addition of vacuum deposited thin mineral barrier like oxides, nitrides or oxi-nitrides layers directly onto the organic pixel. The main challenge today is to achieve high encapsulation levels with WVTR ~ 10^{-6} g/m²/day. The ALD technology is well-suited to reach that goal because the technology allows the deposition of defect-free oxide barrier films. The main limitation of the ALD technology is the stability of the oxide barriers, mainly aluminum oxide, to humid atmospheres. Due to its composition, as AlxOy:H, resulting from the low temperature deposition used to deposit onto the fragile OLED circuits (< 100 °C), an additional passivation is mandatory in order to protect the aluminum oxide from water condensation onto its surface. Different works have illustrated the benefits of depositing an additional ALD layer [i], or using metalcone solutions [ii] or PVD-deposited passivating SiO layers [iii]. In each case, the stability of the aluminum oxide is improved because the sensitive film is not exposed directly to moisture condensation. Another limitation with ALD technology is the low deposition rate of the materials. Former applications of ALD were dedicated to the microelectronics world, where very thin layers of oxides (< 5 nm) were deposited as gate insulating materials. For encapsulation purposes, thicker films (typ. > 30 nm) are mandatory, leading to very high deposition times in standard reactors at low temperatures (typ. 30 s/cycle with 1 Å/cycle leads to ~ 2.5 h processing time).

The CEA-Leti has acquired in 2016 an Infinity 200 ALD system from the company Encapsulix SAS. The reactor is a parallel precursor wave architecture [iv] which allows to deposit thin film barriers at high speed (1.5 s/cycle), at temperatures below 100 °C. In the framework of our OLED developments for microdisplay applications, we have qualified the Infinity 200 reactor for OLED circuits. The materials that have been deposited were Al_2O_3 and TiO_2. In this work a systematic study of the material deposition parameters onto 8'' Si wafers and film characteristics will be presented. Performances of these materials deposited onto OLED as thin-film barriers will also be evaluated, for single Al_2O_3 and TiO_2 layers as well as bilayers Al_2O_3/TiO_2.

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[i] A. A. Dameron et al. J. Phys. Chem. C 112 (2008) 4573-4580

[ii] P. Minwoo et al. Thin Solid Films 546 (2013) 153-156

[iii] T. Maindron et al. J. Vac. Sci. Technol. A 34 (2016) 031513

[iv] J. C. S. Kools, US patent 8,721,835 (2014)

3:15pm **AM+EM-TuA-8 Flexible Functional Devices at Mass Production Level with the FLEx R2R sALD Platform**, ***Diederick Spee***, *W Boonen, E Clerkx, D Borsa,* Meyer Burger B.V., Netherlands

Atomic layer deposition has been around for quite some time. Only recently however, the link to mass production has been made, with the introduction of spatial atomic layer deposition (sALD), which allows for deposition rates in a range similar to established coating techniques like plasma enhanced chemical vapor deposition.

Simultaneously, flexible devices have gained increased popularity due to a huge diversity of applications. For such devices and their market introduction, sALD offers a unique opportunity: it combines high quality materials, from protective to functional coatings or active layers, with competitive costs and high throughput. At Meyer Burger (Netherlands) B.V. we have paved the road to sALD mass production of flexible devices with the introduction of the FLEx R2R sALD platform.

Our equipment is designed as a fully modular platform, allowing for integration of a sALD coating step with pre-and-post sALD steps (eg. surface treatment and activation, planarization and protective layers) for the production of high quality flexible functional foils. The design of the R2R platform, which is the result of dedicated gas flow modeling and web transport simulations, allows for a significant throughput of over 40 m^2/hour of 20 nm AlO_x coating on foil and already takes into account the scalability to larger web widths (currently 600 mm). Extensive process testing proves that layer growth corresponds to pure ALD both in terms of growth per cycle and saturation curves, even at such a high throughput. Furthermore, layer thickness uniformity is excellent: over a width of 500 mm, deviations are less than 1%. A 20 nm AlO_x moisture barrier deposited on 125 micron PET foil, without any additional surface treatment, has a water vapour transmission rate (WVTR) of 10^{-5}g/m^2/day at 20°C and 50% relative humidity, proving its high quality.

A detailed introduction of the R2R platform and the performance of our functional foils as well as a brief discussion on the scalability will be presented.

4:00pm **AM+EM-TuA-11 All-Organic Spatial MLD: Troubleshooting Deposition onto Porous Substrates**, ***Daniel Higgs***, ALD NanoSolutions, Inc.; *Y Wang,* GE; *E Chan,* National Institute of Standards and Technology; *H Wang,* GE; *C Stafford,* National Institute of Standards and Technology; *S George,* University of Colorado - Boulder

Thin film polymers are used in many applications including sacrificial layers, passive and active biosurfaces, separation membranes, lubrication and friction modification, and sensors. Several of these applications require thin membranes on porous mechanical supports. As these membranes become thinner, polymer deposition techniques are being pushed to their limits. Current state of the art techniques for producing thin film membranes, such as interfacial polymerization, can produce films down to ~50-100nm, but new techniques are required for thinner membranes. MLD can produce polymer thin films at thickness <50nm, but gas-phase deposition of these thin MLD films onto porous supports is very challenging as unwanted coating occurs inside the pores. This talk will describe the various attempts to produce a uniform, thin MLD film on a porous support.

This work was part of a program funded under the DOE Innovative Manufacturing Initiative, to explore scale-up methods for producing model polyamide membranes for reverse osmosis water purification. As part of this project, we have developed a new spatial molecular layer deposition (MLD) reactor that is capable of sequentially depositing reactive monomers in the vapor phase on flexible substrates at speeds of up to 3 m/s. In this talk, we will describe the challenges and approaches for creating these thin film polymers. We will then compare the MLD films with solution-phase films. We will look at swelling behavior, internal structure, functional group density, and transport. We will also examine the various strategies employed to produce a thin MLD film on porous substrates. This understanding will enable companies to develop and manufacture next generation, energy-efficient membrane materials.

4:15pm **AM+EM-TuA-12 Graphene Oxide Functionalization by Molecular Layer Deposition**, ***Mercedes Vila Juarez***, Coating Technologies S.L., Spain; *A Jaggernauth, M Neto,* CICECO, University of Aveiro; *M Hortiguela, G Gonçalves, M Singh,* TEMA-NRD, University of Aveiro, Portugal; *F Oliveira, R Silva,* CICECO, University of Aveiro

The functionalization of graphene oxide (GO) with organic molecules is one of the investigated routes for increasing the interface compatibility of GO, thereby enabling its use in a variety of applications such as biomedical platforms, composite filler in conductive polymers or in energy storage solutions. Wet chemistry processes are commonly used to achieve this polymer functionalization of GO, but they have limitations of high heterogeneity, time consumption, and difficult purification processes implying a significant loss of material.

Two approaches for dry functionalization of the nano-GO surface are therefore proposed, utilizing an atomic layer deposition (ALD) reactor: (i) vaporization− condensation of polyethylene glycol amine (PEG-NH_2) and (ii) molecular layer deposition (MLD) of a polymer hybrid from trimethylaluminum (TMA) and ethylene glycol (EG).

The dry functionalization of GO−COOH was successfully accomplished by the MLD growth of a hybrid polymer using TMA and EG precursors. 100 °C is sufficient for this deposition with at least 20 TMA-EG cycles showing a change in the surface of GO and depositing a minimum thickness of 5.0 nm. FTIR confirmed the presence of −Al− O−, −C−H−, and −C−O− bonds being formed on GO- TMA-EG. In addition, XPS highlighted the existence of native oxide Al and the increasing thickness of the hybrid polymer layer with number of cycles, confirming bond formation between EG and −Al−. The resulting hybrid polymer also resulted in an increase in the thermal stability of GO−COOH with an increasing number of cycles, according to TGA data.

The route for dry functionalization of GO−COOH with PEG-NH_2, via pulsed depositions in an ALD reactor, proved to be more challenging. FTIR demonstrated a changing GO surface with increasing number of cycles, with the main result being the presence of amide and −N−H− bonds, suggesting the reaction between −COOH functional groups of GO and −NH-terminating groups of PEG-NH_2. A temperature of 130 °C resulted in pegylated GO within a relatively short time of about 5 h, although control over the formation of double reactions will have to be investigated if the resulting nanoparticles are to be further activated.

4:30pm **AM+EM-TuA-13 Contrast Enhancement of Biological Nanoporous Materials with Zinc Oxide Infiltration for Electron and X-ray Nanoscale Microscopy**, ***Leonidas Ocola***, Argonne National Laboratory; *V Sampathkumar,* University of Chicago; *N Kasthuri, R Winarski,* Argonne National Laboratory

Most biological materials have problems with charging when imaging with electrons and low contrast when imaging with hard X-rays. Standard protocols for sample preparation include coating techniques, using sputtering of a conductive metal such as gold, platinum, or gold/palladium alloy, or non-coating perfusion techniques using glutaraldehyde, tannic acid and osmium tetroxide.

In this paper we describe the use of infiltrated ZnO as a means to address both charge dissipation in electron microscopy and contrast enhancement for X-ray microscopy. With ZnO infiltration it is feasible to coat a 2 – 3 nm thick conductive metal oxide film throughout a porous biological sample, and image both the surface and the interior of a sample without any further sample recoating. ZnO is a conductive oxide and the precursors required for its synthesis (water and diethylzinc) easily penetrate nanoporous materials that exhibit water intake. The samples imaged by electron microscopy were a canine tooth from a dog, and brain tissue that was fixated and supercritically dried ahead of time. Both samples were pre-treated in a vacuum oven, initially at room temperature, ramped to 95 °C, and then baked at 95 °C for several hours. The temperature of the vacuum pre-treatment matches that of the infiltrated ZnO process used in our Arradiance Gemstar ALD tool. Both tooth and brain tissue samples were inspected by optical microscopy before and after the vacuum oven pre-treatment to determine if the samples were compatible with the infiltration process. Given that no sample changes were observable, we proceeded with running an infiltration ZnO process of 18 cycles of [H_2O:DEZ], which would be the equivalent of a 2-3 nm coating on a flat surface. No further processing was needed for both electron and X-ray microscopy imaging. We show that high resolution X-ray tomography can be performed on biological systems using Zn K edge (1s) absorption to enhance internal structures, and obtained the first nanoscale 10 KeV X-ray absorption images of the interior regions of a tooth.

- Use of the Center for Nanoscale Materials and the Advanced Photon Source, both Office of Science user facilities, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. Additional funding by IARPA, (ARIADNE) under Contract D16P00002

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