

## Energy Transition Focus Topic

Room A212 - Session TL+MS+VT-TuM

### Implications of Implementation: Making Energy Transition a Reality (ALL INVITED SESSION)

**Moderators:** Margaret Fitzgerald, Colorado School of Mines, Natalie Seitzman, Colorado School of Mines

8:00am **TL+MS+VT-TuM-1 The Energy Transition: Science and Technology Development Aspects, Richard M.C.M. van de Sanden**, DIFFER, Eindhoven University, The Netherlands, Netherlands **INVITED**

The Paris climate agreement requires a decarbonization of our energy infrastructure leading to a CO<sub>2</sub> neutrality by 2050. Therefore renewable energy generation by means of wind or from solar radiation through photovoltaics or concentrated solar power will continue to increase its share in the energy mix. Intermittency (due to e.g. day/night cycle), the regional variation of these energy sources, and penetration of renewable energy into other sectors than electricity (e.g. the chemical industry) requires means to store, transport and convert energy on a large scale. A promising option is the synthesis of chemicals and synthetic fuels (easily deployable within the present fossil fuels infrastructure) from raw feedstock using renewable energy. A truly circular economy requires that the raw materials are the thermodynamically most stable molecules such as water (H<sub>2</sub>O), carbon dioxide (CO<sub>2</sub>) and nitrogen (N<sub>2</sub>) to produce base chemical feedstock, such as e.g. hydrogen, hydrocarbons and ammonia. In this talk I will discuss the opportunities this transformation of the chemical industry provides. Furthermore, I will highlight the science and technology challenges, the catalytic materials, processes and systems developments needed that can provide compatibility of renewable energy driven chemistry with e.g. intermittency and localized production.

8:40am **TL+MS+VT-TuM-3 Electrochemical CO<sub>2</sub> Reduction Across Scales: From Fundamental Mechanisms to Practical Applications, Wilson Smith**, Delft University of Technology The Netherlands, The Netherlands **INVITED**

Electrocatalytic CO<sub>2</sub> reduction has the dual-promise of neutralizing carbon emissions in the near future, while providing a long-term pathway to create energy-dense chemicals and fuels from atmospheric CO<sub>2</sub>. The field has advanced immensely in recent years, taking significant strides towards commercial realization. While catalyst innovations have played a pivotal role in increasing the product selectivity and activity of both C<sub>1</sub> and C<sub>2</sub> products, slowing advancements indicate that electrocatalytic performance may be approaching a hard cap. Meanwhile, innovations at the systems level have resulted in the intensification of CO<sub>2</sub> reduction processes to industrially-relevant current densities by using pressurized electrolytes, gas-diffusion electrodes and membrane-electrode assemblies to provide ample CO<sub>2</sub> to the catalyst. The immediate gains in performance metrics offered by operating under excess CO<sub>2</sub> conditions goes beyond a reduction of system losses and high current densities, however, with even simple catalysts outperforming many of their H-cell counterparts. Using recent literature as a guidepost, this talk will focus on some of the underlying reasons for the observed changes in catalytic activity, and proposes that further advances can be made by shifting additional efforts in catalyst discovery and fundamental studies to system-integrated testing platforms.

9:20am **TL+MS+VT-TuM-5 Perspectives on the Research and Development of Nanomaterials for Hydrogen Production, Marcelo Carmo**, Forschungszentrum Jülich, Germany **INVITED**

Hydrogen is often considered the best means by which to store energy coming from renewable and intermittent power sources. With the growing capacity of localized renewable energy sources surpassing the gigawatt range, a storage system of equal magnitude is required, such as the production of electrolytic hydrogen by water electrolysis. Despite of more than 100 years of experience in alkaline electrolysis systems, and thousands of plants installed all over the world, only a few systems or industries remain, providing the state-of-the-art of this technology today. This is due to the fact that the cost of electrical energy has always remained as an uncomfortable barrier, with electrolytic hydrogen costs not being able to compete with the costs for the production of hydrogen by conventional steam reforming of fossil fuels. Nevertheless, today, increased interest can be observed for PEM water electrolysis technology, and over the past 20 years, new companies and projects have appeared, with new leaders being consequently established in this growing niche. The reason is that PEM electrolysis provides a sustainable solution for the production of hydrogen, and is well suited to couple with energy sources

such as wind and solar. The advantages of PEM electrolysis over alkaline electrolysis together with novel R&D approaches can potentially reduce the investment costs of PEM electrolyzers. We expect that in the following years, frontier advancements on PEM electrolysis systems will appear, demonstrating a true capacity to ultimately establish hydrogen as a key player in the energy market, and contribute to a future hydrogen economy.

11:00am **TL+MS+VT-TuM-10 Impacts and Adaptation Strategies in Ethiopia, Aschale Dagnachew Siyoum**, Xavier University of Louisiana

This paper highlights climate change and variability and its impact and adaptation strategies in Ethiopia. Due to low adaptive capacity and high sensitivity of socio economic systems, climate vulnerability is worsening over the last few decades in Ethiopia. Available evidences showed that since 1960, the mean annual temperature of the country has risen by about 1.3°C with an average rate of 0.28°C per decade imposing a significant challenge on food security, water availability, energy supply, poverty reduction and sustainable development efforts of the nation. Ethiopia has responded to the increasing impact of climate change and variability through developing relevant adaptation strategies, plans and policies largely focused on decreasing vulnerability in many different sectors including agriculture and food security, water resources, forestry, and health. To tackle the impact of climate change, the government has approved the National Adaptation Program of Action (NAPA) in 2007 which includes projects that focused on promoting drought insurance program, strengthening drought and flood early warning systems, developing small-scale irrigation and water harvesting schemes in arid, semi-arid, and dry sub-humid areas, and realizing food security through a multi-purpose large-scale water development project. Results, however, shows that although some progress has been made in addressing the impacts of climate change and variability, adaptation measures implemented over the last few decades were generally ineffective resulting in increasing losses as more and more people occupy vulnerable areas. This requires a sustained effort to further plan and implement the right mix of climate change adaptation strategies to address vulnerability to biodiversity and humanity to the increasing impacts of climate change. Addressing the impact of climate change requires a good understanding of the nexus between climate change adaptation measures and sustainable development as well as knowledge of climate change adaptation tools and techniques, which when used properly can minimize the total damage to life and property.

11:20am **TL+MS+VT-TuM-11 Developing and Scaling Up the Manufacturing of Thin Film Materials for the Future of Energy Production, Storage, and Reduction, Ken Nauman**, Von Ardenne North America **INVITED**

The world, and thus the economy, are clearly dependent on energy and in particular electricity. Production of electricity is ever increasing while the desire to create cleaner sources becomes a higher priority to reduce the environmental impact. The transition to electricity for mobility in transportation and communication relies on new technology to improve market penetration. Thus, the three key aspects of electricity in our modern society are: generation, storage, and saving. In order to make the energy transition a profitable reality, companies that develop technology will have to reduce the cost of energy production and storage while also considering how to lower energy usage. This talk will cover these key aspects from the perspective of an equipment and process technology company. Companies such as Von Ardenne that develop thin film materials have focused on these topics to reduce the CapEx and CoO for our customer's factories. This includes processes for Thin Film Photovoltaics and Crystalline Photovoltaics, along with emerging cell architectures, to reduce the cost of electricity generation while reducing overall CO<sub>2</sub> production. Our company also works on leading material science in battery and fuel cell technology to increase storage capacity and cost of storing energy. Finally, other technology development is focused on saving energy with low-e coatings for glass and plastics. This presentation will review the history of the technological development as well as the latest trends, economics, and status of market leading performance in manufacturing products related to generating, storing, and saving electricity.

## Atomic Scale Processing Focus Topic

Room B130 - Session AP+EL+MS+PS+SS+TF-TuA

### Advancing Metrology and Characterization to enable Atomic Layer Processing

**Moderators:** Eric A. Joseph, IBM Research Division, T.J. Watson Research Center, Jessica Kachian, Intel Corporation

2:20pm **AP+EL+MS+PS+SS+TF-TuA-1 In Situ Ellipsometry Characterization Of Atomic Layer Processes: A Review, James Hilfiker, G Pribil, J VanDerslice, J.A. Woollam Co., Inc.** **INVITED**

Atomic layer processes such as atomic layer deposition (ALD) and atomic layer etch (ALE) provide monolayer-level thin film deposition or etch. Spectroscopic ellipsometry (SE) is ideally suited for the characterization requirements of such very thin layers. In situ SE provides real-time feedback, which is invaluable for establishing new atomic layer processes. In situ SE characterization has been adopted by many researchers due to its versatility. SE measurements are sensitive to deposition or etch at the (sub)monolayer level. The real-time evolution of film thickness provides details on nucleation periods or delays, the growth or etch rates per cycle, and verifies the self-limiting nature of a process. Multiple experiments can be performed within a single run by modifying the process conditions, allowing quick qualification of deposition temperatures, chemical exposure times, plasma influences, and purge times. In this paper, we will review the areas where in situ SE has been applied to both atomic layer deposition and etch.

We will also discuss the applications of in situ SE that benefit from a broad wavelength range. SE is best known for determining film thickness and optical constants. This characterization can be accomplished for many types of materials – dielectrics, semiconductors, organics, and even metals – provided the layer remains semi-transparent. Other material properties affect the optical constants and can be determined via this relationship. In situ SE has been used to estimate the crystal structure, composition, and even conductivity of thin films. We will discuss the advantages and limitations of in situ SE, which in many ways has proven to be an ideal partner for atomic layer processes.

3:00pm **AP+EL+MS+PS+SS+TF-TuA-3 Elucidating the Mechanisms for Atomic Layer Growth through In Situ Studies, Jeffrey Elam, Argonne National Laboratory** **INVITED**

Atomic Layer Deposition (ALD) provides exquisite control over film thickness and composition and yields excellent conformality over large areas and within nanostructures. These desirable attributes derive from self-limiting surface chemistry, and can disappear if the self-limitation is removed. Understanding the surface chemical reactions, i.e. the ALD mechanism, can provide insight into the limits of self-limitation allowing better control, successful scale up, and the invention of new processes. In situ measurements are very effective for elucidating ALD growth mechanisms. In this presentation, I will describe our recent investigations into the growth mechanisms of ALD nanocomposite films comprised of conducting (e.g. W, Mo and Re) and insulating (e.g. Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> and TiO<sub>2</sub>) components using in situ measurements. These ALD nanocomposites have applications in particle detection, energy storage, and solar power. We have performed extensive in situ studies using quartz crystal microbalance (QCM), quadrupole mass spectrometry (QMS), Fourier transform infrared (FTIR) absorption spectroscopy, and current-voltage measurements. These measurements reveal unusual ALD chemistry occurring upon transitioning between the ALD processes for the two components. This results in unique reaction products that affect the properties of the films in beneficial ways. The knowledge gained from our in situ studies of the ALD nanocomposite films has helped us to solve problems encountered when we scaled up the ALD processes to large area substrates.

4:20pm **AP+EL+MS+PS+SS+TF-TuA-7 Surface, Interface, or Film: A Discussion of the Metrology of ALD Materials in Semiconductor Applications, G. Andrew Antonelli, N Keller, Nanometrics** **INVITED**

Atomic layer deposition, etching, and interface engineering are enabling technologies for semiconductor manufacturing. These processes have led to an explosion in the use of laboratory techniques such as transmission electron microscopy and the need to bring such instruments closer to or into the fab itself. However, there remains a need for in-line, non-destructive, non-contact metrology capable of quickly characterizing and monitoring these extremely thin films on test structures, on product, or in

device as these data are the only meaningful method for monitoring of ultimate device performance. Indeed, in cases such as the use of selective deposition or etching, no test vehicle other than the ultimate product may be relevant. A variety of measurement techniques with a focus on x-ray and optical probes as applied to this class of problems will be reviewed. Examples will be provided on relevant logic such as the Gat-All-Around FET and memory devices such as 3D NAND.

5:00pm **AP+EL+MS+PS+SS+TF-TuA-9 In Line and Ex Situ Metrology and Characterization to Enable Area Selective Deposition, Christophe Vallee, M Bonvalot, B Pelissier, J Tortai, S David, S belahcen, V Pesce, M Jaffal, A Bsiesy, LTM, Univ. Grenoble Alpes, CEA-LETI, France; R Gassilloud, N Posseme, CEA-LETI, France; T Grehl, P Bruner, IONTOF GmbH, Germany; A Uedono, University of Tsukuba, Japan** **INVITED**

Innovation in materials, architectures (3D), gap filling technologies, lithography and etch processes are mandatory at every node of CMOS or memory devices. These challenging integration issues can be facilitated by the use of an integration scheme currently being intensively investigated known as area selective deposition (ASD). Criteria for an adequate area selective deposition process are: growth only on specific regions, high throughput compatible with industrial demands, no so-called mushroom profiles into adjacent features as well as no nuclei defectivity on undesired sites. Several routes can be developed to achieve an ASD process with ALD. The one discussed here concerns the deposition/etch approach which takes benefit from an *in situ* etching step inserted in a standard ALD cycle [1]. By incorporation of anisotropic or isotropic etching steps in the ALD process, “surface” selective deposition, as well as topographically selective deposition (TSD) have been obtained [2, 3]. The major current shortcoming of this approach lies in the deep insight which is required regarding elementary atomic-scale reaction mechanisms. Indeed, in the case of an ALD/ALE Area Selective Deposition process, a highly precise control of etching and its selectivity at the atomic scale is needed. Controlling the nature and density of defects induced by etching or passivation steps and understanding their impact on the physical and electrical properties of selectively deposited films are of course also required. Moreover, in order to optimize these processes, an accurate understanding of the underlying reasons why passivation after a low number of ALD cycles, is no more effective. Thus, *in situ* as well as *ex situ* monitoring and metrology are mandatory.

In this presentation, we will discuss how to optimize and understand atomic-scale reaction mechanisms in an ALD/ALE ASD process using combined *in situ* or *ex situ* measurements, such as ellipsometry, XPS, XRR, LEIS, FIB-STEM, and positron annihilation. We will show that when crosslinked, these technics are very effective to perform atomic scale metrology and characterization. As an example, we will discuss F atom localization and density in selectively deposited oxides thanks to a F-based ALE chemistry incorporated in the ALD process. In the case of a topographically selective deposition (TSD) process attempts will be presented to understand ion/surface interactions when low energetic ions are extracted from the plasma of the PEALD reactor both during deposition and plasma-ALE steps.

[1] R. Vallat et al, JVSTA **35** (2017) 01B104

[2] R. Vallat et al, JVSTA **37** (2019) 020918

[3] A. Chacker et al, APL **114** (2019)

5:20pm **AP+EL+MS+PS+SS+TF-TuA-10 Recent Progress in Thin Film Conformality Analysis with Microscopic Lateral High-aspect-ratio Test Structures, Riikka Puurunen, Aalto University, Finland** **INVITED**

Conformal thin films which cover complex 3D shapes with a film of uniform properties (thickness, composition, etc.) are increasingly demanded applications such as semiconductor devices, microelectromechanical systems, energy conversion/storage and catalysis. Atomic layer deposition (ALD) and its counterpart atomic layer etching (ALE) [together known as atomic layer processing (ALP)], are increasing in usage largely thanks to their known conformal character.

A question that needs to be asked in the R&D of 3D applications using conformal ALD/ALE processes is: how conformal is conformal; is the conformality sufficient to meet the specs? In semicon industry, vertical vias and cross-sectional transmission electron microscopy (TEM) are standardly used for conformality analysis. Recently, microscopic lateral high-aspect-ratio (LHAR) test structures have been developed to improve the conformality analytics capabilities. LHAR structures e.g. enable detailed conformality analysis at arbitrarily high aspect ratios (e.g., >5000:1), where no film can coat the 3D structure fully, thereby exposing the saturation

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profile characteristic for the process. This, in turn enables the kinetic analysis of the process and e.g. extraction of the sticking coefficients related to the growth reactions.

This invited talk will address recent progress related to the fabrication and the use of microscopic LHAR conformality test structures. After the breakthrough with the first prototypes (PillarHall LHAR1; Gao et al. 2015, Mattinen et al. 2016; reviewed in Cremers et al., 2019), third and fourth generation prototypes have been developed (PillarHall LHAR3 and LHAR4). This work will review the conformality analysis progress enabled by the microscopic LHAR structures and discuss the benefits and challenges of this approach. Recent published progress includes the conformality modelling by Ylilammi et al. (2018) and experimental extraction of sticking coefficient by Arts et al. (2019). In addition, several other ongoing conformality analysis cases will be presented.

## References

Arts, Vandalon, Puurunen, Utriainen, Gao, Kessels, Knoops, J. Vac. Sci. Technol. A 37, 030908 (2019); <https://doi.org/10.1116/1.5093620>

Cremers, Puurunen, Dendooven, Appl. Phys. Rev. 6, 021302 (2019); <https://doi.org/10.1063/1.5060967>

Gao, Arpiainen, Puurunen, J. Vac. Sci. Technol. A 33, 010601 (2015); <https://doi.org/10.1116/1.4903941>

Mattinen, Hämäläinen, Gao, Jalkanen, Mizohata, Räisänen, Puurunen, Ritala, Leskelä, Langmuir, 32, 10559 (2016); <http://doi.org/10.1021/acs.langmuir.6b03007>

Ylilammi, Ylivaara, Puurunen, J. Appl. Phys. 123, 205301 (2018); <https://doi.org/10.1063/1.5028178>

6:00pm **AP+EL+MS+PS+SS+TF-TuA-12 *In operando*XPS Study on Atomic Layer Etching of Fe and Co Using Cl<sub>2</sub> and Acetylacetone or Hexafluoroacetylacetone, Zijian Wang, O Melton, D Angel, B Yuan, R Opila, University of Delaware**

Etching of transition metals is one of the major challenges in magnetoresistive random-access memory (MRAM) device fabrication. In this work, atomic layer etching of iron and cobalt surfaces with halogen and an organic molecule was studied. We successfully performed etching of Fe and Co thin films via forming volatile metal complexes at low temperature with cyclic sequential reactions of Cl<sub>2</sub> and acetylacetone (acac) or hexafluoroacetylacetone (hfac). The etching reaction mechanism of acac and hfac reacting with Chlorine-modified Fe and Co surfaces was investigated: the surface was first activated with Cl<sub>2</sub> gas, and subsequently, the top layer of chlorinated metal was removed by reaction with a diketone (acac/hfac). The extent of Cl<sub>2</sub> reaction determines the etching rate of the metal. At substrate temperatures lower than 135°C, acac could remove the chlorinated Fe metal layer from Fe surfaces, but not chlorinated Co from Co surfaces. *In-operando* x-ray photoelectron spectroscopy (XPS) and density functional theory (DFT) simulation shows that the reaction of acac or hfac with Chlorinated Fe or Co surfaces is likely following a complex reaction pathway instead of simple diketone substitution for the metal chloride. Diketone decomposition may play an important role in the etching process.

## Manufacturing Science and Technology Group Room A226 - Session MS-WeA

### Science and Technology for Manufacturing: Solid State Batteries (ALL INVITED SESSION)

**Moderators:** Kelsy Hatzell, Vanderbilt University, Gary Rubloff, University of Maryland, College Park

2:20pm **MS-WeA-1 The Importance of Modifying the Nothing Within 3D Electrode Architectures for Solid-State Energy Storage**, *Debra Rolison, M Sassin, C Chervin, J Parker, J Long*, U.S. Naval Research Laboratory **INVITED**  
Our team has found that an architectural design metaphor serves as a powerful guide in re-imagining materials and electrodes in electrochemical energy science [1,2]. Key consumer and military portable power sources (e.g., batteries, fuel cells, supercapacitors) must balance multiple functions (molecular mass transport, ionic/electronic/thermal conductivity, and electron-transfer kinetics) even though these functions often require contradictory structures [2]. The design and fabrication of size- and energy-scalable three-dimensional multifunctional architectures from the appropriate nanoscale building blocks for charge storage seamlessly embodies all of the requisite functions. A critical knob to turn to amplify performance—or move to a new performance curve, such as a 3D solid-state battery with interpenetrating components [2,3]—is the ability to “paint blind,” to modify interiors with functional materials that do not block the internal porosity through which reactants enter and products depart. Architecture also matters with the electrocatalysts under exploration to improve oxygen redox (higher activity and lower potential energy costs to drive the reaction) in air cathodes in aqueous metal-air batteries. Expressing oxygen reduction or evolution electrocatalysts in ultraporos aerogel form allows us to extract higher activity at lower overpotentials [4–6], further underscoring the importance of nothing and the unimportance of periodicity in energy-relevant nanoarchitectures [7].

[1] J.W. Long, D.R. Rolison, *Acc. Chem. Res.* 2007, 40, 854–862.

[2] D.R. Rolison, J.W. Long, J.C. Lytle, A.E. Fischer, C.P. Rhodes, T.M. McEvoy, M.E. Bourg, A.M. Lubers, *Chem. Soc. Rev.* 2009, 38, 226–252.

[3] J.W. Long, B. Dunn, D.R. Rolison, and H.S. White, *Chemical Reviews* 2004, 104, 4463–4492.

[4] C.N. Chervin, P.A. DeSario, J.F. Parker, E.S. Nelson, D.R. Rolison, J.W. Long, *ChemElectroChem* 2016, 3, 1369–1375.

[5] J. S. Ko, C. N. Chervin, M. N. Vila, P. A. DeSario, J. F. Parker, J. W. Long, D. R. Rolison, *Langmuir* 2017, 33, 9390–9397.

[6] J. S. Ko, J. F. Parker, M. N. Vila, M. A. Wolak, D. R. Rolison, and J. W. Long, *J. Electrochem. Soc.* 2018, 165, H777–H783.

[7] D.R. Rolison, *Science* 2003, 299, 1698–1701.

3:00pm **MS-WeA-3 Precision 3D Solid State Battery Architectures: Science, Challenges and Manufacturing Opportunity**, *Sang Bok Lee Lee, G Rubloff*, University of Maryland, College Park **INVITED**

This presentation describes recent findings related to the design and architectures of thin electrode materials synthesized by thin layer deposition techniques. Throughout the presentation I will describe how these techniques enable us to synthesize electrodes of interest with precise control over the structure and composition of the material. The electrochemical response of these thin electrodes will be discussed in the aspects of structural parameters, ion storage mechanism, interfacial electrochemical issues related to electrode degradation. While it is important to identify and understand mechanisms in performance and degradation, it is even more critical to design strategies and to mitigate challenging technical hurdles for developing means to implement and validate the strategies in the aspect of future manufacturing opportunity. For example, the design and the development process of precision 3D solid state battery architectures on a Si wafer will be discussed. In a nut shell, this talk illustrates how careful design of thin materials architecture can facilitate desirable electrochemical activity, resolve or shed light on mechanistic limitations of electrochemical performance in solid electrolyte systems, and eventually try to convince audience that the thin film processes using primarily existing semiconductor fabrication facilities may provide a new paradigm changing opportunity in solid state battery manufacturing technology.

4:20pm **MS-WeA-7 Understanding the Electronic and Mechanical Properties of High Energy Density Anodes on 3D Structures**, *Amy Prieto, J Ma, M Schulze*, Colorado State University **INVITED**

We are interested in mitigating mechanical failure in high energy density alloy anodes used for rechargeable Li-ion and Na-ion batteries by incorporating 3D architectures. We will present the use of direct electrodeposition of inter metallic alloys onto 3D current collectors, and their cycling in half cell and full cell batteries. A 3D architecture is critical for reasonable power densities in solid state batteries, and we will present our efforts moving toward a fully integrated solid state battery.

5:00pm **MS-WeA-9 Enabling High Cycle Life Alkali Metal Anodes through Imposed Thermal Gradients**, *R Atkinson III, EXCET, Inc.; R Carter, Corey Love*, U.S. Naval Research Laboratory **INVITED**

Solid state batteries promise a number of advantages over liquid electrolyte alternatives. The solid state battery will significantly improve safety by eliminating flammable electrolytes, enable high energy density by utilizing alkali metal anodes, and eliminate the weight and volume contribution from a host or alloying element at the anode. However, significant challenges remain in stabilizing metal anodes over many cycles and at high rates. Significant efforts in interfacial design and current collector structure have aimed to demonstrate the viability of the solid state battery, but these strategies often involve complex and costly manufacturing. Herein, we demonstrate the advantage of simply externally warming the anode (40 °C) and cooling the cathode (0°C) to stabilize charging or the plating of metal compared to isothermal controls (20 °C). This technique enables the high rate and long cycle-life desired for viability of the solid state configuration. Our results reveal remarkable stability over many hours (32% lower voltage hysteresis after 400 hr) of operation and fast charging with current densities up to 10 mA/cm<sup>2</sup> (competitive with 2C in conventional Li-ion). Further, a thermal gradient is easily implemented in the thermal management strategies commonly used in battery modules making the strategy commercially viable. Finally, it is likely that the thermal gradient will not only assist in realization of the metal anode but also the solid electrolyte. Solid electrolytes are challenged by low ionic conductivity which is often enhanced by heating the material up to ~80 °C. The operational benefit observed in the liquid cells and the directionality of ion movement provided suggest that application of an external thermal gradient will provide better performance than isothermal heating alone.

Carter, R.; Love, C. T., Modulation of Lithium Plating in Li-Ion Batteries with External Thermal Gradient. *ACS Applied Materials & Interfaces* 2018, 10 (31), 26328–26334.

Mistry, A.; Fear, C.; Carter, R.; Love, C. T.; Mukherjee, P. P., Electrolyte Confinement Alters Lithium Electrodeposition. *ACS Energy Letters* 2018, 156–162.

## Electronic Materials and Photonics Division Room A214 - Session EM+AP+MS+NS+TF-ThM

### Advanced Processes for Interconnects and Devices

**Moderators:** Andy Antonelli, Nanometrics, Bryan Wiggins, Intel Corporation

8:00am **EM+AP+MS+NS+TF-ThM-1 High-density Plasma for Soft Etching of Noble Metals**, *Gerhard Franz, V Sushkov*, Munich University of Applied Sciences, Germany; *W Oberhausen, R Meyer*, Technische Universität München, Germany

During our research to define a contact which can be serve as thin hard mask in III/V semiconductor processing, we focused on the Bell contact which consists of Ti/Pt(Mo)/Au and chlorine-based plasmas generated by electron cyclotron resonance. For platinum, we identified  $\text{PF}_3$  as main component which acts comparable to CO [1]. This fact triggered our search for suited etchants for gold and copper. For Au, the best ambient is a mixture of  $\text{CH}_4$ ,  $\text{Cl}_2$ , and  $\text{O}_2$  which is stabilized by Ar [2]. This mixture generates residual-free etching of metal films which are clearly free of "fencing" and "hear's ears."

The etching process has been established up to thicknesses of half a micron which is the typical thickness of metal films on the p-side of laser devices. With the aid of optical emission spectroscopy, the generation of CO could be proven [3]. This reagent seems to be the main component for real etching without residual fencing.

[1] G. Franz, R. Kachel, and St. Sotier, *Mat. Sci. Semicond. Proc.* **5**, 45 (2002)

[2] G. Franz, R. Meyer, and M.-C. Amann, *Plasma Sci. Technol.* **19**, 125503 (2017)

[3] G. Franz, W. Oberhausen, R. Meyer, and M.-C. Amann, *AIP Advances* **8**, 075026 (2018)

8:20am **EM+AP+MS+NS+TF-ThM-2 Crystalline InP Growth and Device Fabrication Directly on Amorphous Dielectrics at Temperatures below 400°C for Future 3D Integrated Circuits**, *Debarghya Sarkar, Y Xu, S Weng, R Kapadia*, University of Southern California

A fundamental requirement to realize 3D integrated circuits is the ability to integrate single crystal semiconductor devices on the back-end of functional layers within a thermal budget of  $\sim 400^\circ\text{C}$ . Present state-of-the-art methods involve wafer bonding or epitaxial growth and transfer, since directly growing on amorphous materials by traditional epitaxial growth processes like MOCVD and MBE would give polycrystalline films with submicron-scale grains. To that end, a newly introduced and actively developing growth method called Templated Liquid Phase (TLP) has demonstrated the ability to achieve single crystal compound semiconductor mesas of areal dimension  $\sim 10\mu\text{m}$  diameter on diverse amorphous substrates. While previous demonstrations of TLP growth were at temperatures around  $500\text{--}600^\circ\text{C}$ , in this presentation we would discuss some of the recent material characteristics and device results achieved and insights obtained, for crystalline InP mesas grown on amorphous dielectrics at temperatures below  $400^\circ\text{C}$ . InP nucleation and growth was obtained for temperatures  $360^\circ\text{C}$  down to  $200^\circ\text{C}$ . Morphological variations of the grown crystals observed under different growth conditions (temperature, pressure, precursor flux) and strategies to obtain compact macro-defect free crystal growth would be presented. Contrary to general expectation of poor optoelectronic quality at these lower temperatures, the room temperature steady-state photoluminescence shows peak position and full width at half maximum comparable to that of commercial InP wafer. External quantum efficiency is within an order of magnitude of single crystal commercial wafer at optimal growth conditions. Back-gated phototransistor was fabricated using low temperature InP grown directly on the amorphous gate oxide, and with all processing steps below the thermal budget of  $400^\circ\text{C}$ . A typical device showed reasonable ON-OFF ratio of about 3 orders of magnitude, with peak responsivity of  $20\text{ A/W}$  at  $V_{\text{gs}}=3.2\text{V}$  and  $V_{\text{ds}}=2.1\text{V}$  under an irradiance of  $4\text{ mW/cm}^2$  of broadband light. In summary, this technology could potentially open up a viable avenue to realize 3D integrated circuits by enabling integration of high performance electronic and optoelectronic devices on the back-end of functional layers within the acceptable thermal budget of  $400^\circ\text{C}$ .

8:40am **EM+AP+MS+NS+TF-ThM-3 The Role and Requirements of Selective Deposition in Advanced Patterning**, *Charles Wallace*, Intel Corporation **INVITED**

The edge placement error (EPE) margin on features patterned at tight pitches presents a difficult integrated challenge. Area selective deposition, chemically selective etches and the design of thin films for selectivity have risen to the top priorities in advanced patterning. The EPE control requirement creates a complex interaction between many integrated modules such as thin film deposition, etch (wet and dry), chemical-mechanical polish and lithography. The introduction of EUV lithography into the semiconductor patterning process has enabled some simplification of process architecture; however, has not decreased EPE margin enough to keep up with the pitch scaling requirements. Chemical selectivity is the most effective way to avoid EPE-caused failures on devices which lead to poor yield. Some of the limits to achieving selective growth solutions include development of self-assembled monolayers (SAMs), selective ALD/CVD growth and the metrology required to prove success. The development of manufacturable deposition chambers by the industry is a key requirement in order to adequately test the capability of these new process options.

9:20am **EM+AP+MS+NS+TF-ThM-5 Graphene-Template Assisted Selective Epitaxy (G-TASE) of Group IV Semiconductors**, *M. Arslan Shehzad, A T. Mohabir, M Filler*, Georgia Institute of Technology

As conventional 2-D transistor scaling approaches its limits, 3-D architectures promise to increase the number of devices and reduce interconnect congestion. A process able to monolithically integrate single-crystalline group IV materials into the back-end-of-line (BEOL) may enable such designs. Here, we demonstrate the graphene-template assisted selective epitaxy (G-TASE) of single-crystal Ge on amorphous substrates at temperatures as low as  $250^\circ\text{C}$ . This work represents a significant step forward for TASE methods, which have been largely limited to III-V and II-VI materials, bulk crystal templates, as well as higher temperatures. We specifically grow Ge nanostructures on graphene-on-oxide at the bottom of nanometer-scale oxide trenches by leveraging differences in group IV atom sticking probability between graphene and oxide surfaces. Raman mapping confirms the single crystallinity of as-grown Ge crystals. Time-dependent studies show a linear increase in Ge crystal height even after emerging from the oxide trench, indicating Ge atoms preferentially adsorb to the top facet under our growth conditions. Our studies also reveal that G-TASE is sensitive to the plasma process used to expose graphene in the oxide trenches. This work extends TASE to a new, technologically-relevant materials system and provides fundamental insight into the underlying physicochemistry.

**KEY WORDS:** silicon, germanium, epitaxy, graphene, selective deposition

9:40am **EM+AP+MS+NS+TF-ThM-6 Resistivity and Surface Scattering Specularity at (0001) Ru/dielectric Interfaces**, *S Ezzat*, University of Central Florida; *P Mani*, View Dynamic Glass, Inc.; *A Khaniya, W Kaden*, University of Central Florida; *D Gall*, Rensselaer Polytechnic Institute; *K Barmak*, Columbia University; *Kevin Coffey*, University of Central Florida

In this work we report the variation of resistivity with film thickness and with changes in surface characteristics for ex-situ annealed single crystal (0001) Ru thin films grown on c-axis sapphire single crystal substrates. The room temperature deposition of  $\text{SiO}_2$  on the Ru surface increased the resistivity of the annealed films and is interpreted as an increase in diffuse scattering of the upper surface from a primarily specular previous condition in the context of the Fuchs-Sondheimer model of surface scattering. The characterization of the films and upper Ru surface by low energy electron diffraction (prior to  $\text{SiO}_2$  deposition), x-ray reflectivity, x-ray diffraction, and sheet resistance measurements is reported. The film resistivity and specularity of the Ru/ $\text{SiO}_2$  interface is observed to reversibly transition between high resistivity (low specularity) and low resistivity (high specularity) states.

11:00am **EM+AP+MS+NS+TF-ThM-10 Electrochemical Atomic Layer Deposition and Etching of Metals for Atomically-Precise Fabrication of Semiconductor Interconnects**, *Y Gong, K Venkatraman, Rohan Akolkar*, Case Western Reserve University **INVITED**

Moore's law drives continued device miniaturization in nano-electronics circuits. As critical dimensions are approaching the single nanometer length scale, the semiconductor industry is seeking novel technologies for precisely tailoring materials and structures at the atomic scale. While vapor-phase, plasma-assisted techniques of atomic layer deposition (ALD) and etching (ALE) are capable of providing nano-scale control over metal

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deposition and etching, these processes may not provide the requisite atomic-scale precision. Additionally, ALD precursors are unstable and often expensive. Thus, alternative solution-phase electrochemical processes are being developed in our laboratory. In our electrochemical ALD (e-ALD) approach, a sacrificial monolayer of zinc is first deposited on the noble substrate via underpotential deposition (UPD). The zinc adlayer then undergoes spontaneous surface-limited redox replacement (SLRR) by the desired metal such as Cu or Co. Sequential UPD and SLRR steps enable fabrication of multi-layered deposits in a layer-by-layer fashion. An analogous approach for electrochemical ALE (e-ALE) is also being developed. In electrochemical ALE of Cu, surface-limited sulfidization of Cu forms a cuprous sulfide (Cu<sub>2</sub>S) monolayer. The sulfidized Cu monolayer is then selectively removed through spontaneous complexation of the Cu<sup>+1</sup> in a chloride-containing etchant medium. The sequence can be repeated to etch bulk metal films one atomic layer at a time. This talk will highlight numerous advantages and fundamental characteristics of e-ALD and e-ALE processes and describe opportunities for integrating them in wafer-scale metallization applications.

11:40am **EM+AP+MS+NS+TF-ThM-12 Mechanical Properties of Patterned low- $\kappa$  Films Measured by Brillouin Light Scattering**, *Jon Zizka, H Wijesinghe*, The Ohio State University; *S King, H Yoo*, Intel Corporation, USA; *R Sooryakumar*, The Ohio State University

In order to improve device performance of interconnects, the microelectronics industry utilizes low- $\kappa$  dielectric technology in place of traditional SiO<sub>2</sub>. Integration of these materials into circuits has, however, been challenging due to poor mechanical stability as a result of the increased dielectric porosity. Large thermal stresses may also build up during successive thermal cycling due to differences in the thermal expansion of component materials. These adverse features become more prominent as smaller dimensions are sought for improvement in device performance. While titanium nitride (TiN) is currently being widely used as a hard mask to pattern low- $\kappa$  materials such as SiOC:H into desired architectures with < 100 nm length scales, the high stress and stiffness of the TiN over-layer can influence the delicate underlying patterns and affect device performance.

In this study we utilize Brillouin light scattering (BLS) to probe the elastic properties of TiN/SiOC:H structures grown on Si that have been patterned into a series of parallel wires of rectangular cross-sections with sub 200 nm pitch and depths. In studying the influence of the hard mask on the mechanical properties of SiOC:H, BLS offers a non-invasive approach to detect thermally activated acoustic excitations and to measure their mode dispersions for incident light with wave-vector components parallel or perpendicular to the TiN wires. The widely-used technique of nanoindentation (NI) to measure mechanical properties has been shown, for ultra-thin soft films, to interact with the underlying substrate, thus skewing the results. On the other hand, the inelastic light scattering approach probes ambient modes allowing for the individual acoustic behavior of the film to be distinguished from the substrate.

The results of measurements performed on samples with a range of wire dimensions (width/depth) will be presented that include the dependence of the Brillouin peak intensities on the incident and scattered light polarization as well as a model of the mode profiles. The latter include finite element modeling that simulate the different mode frequencies and associated relative vertical and horizontal displacements for the non-dispersive cantilever type modes that characterize modes with wave-vector perpendicular to the TiN wires. The modes with wave-vector parallel to the wires followed a dispersive nature similar to blanket films. The relationship of such acoustic properties will be discussed in the context of the mechanical properties of the patterned structures and the influence of processing of the nanowires.

12:00pm **EM+AP+MS+NS+TF-ThM-13 Wafer-Scale Fabrication of Carbon-Based Electronic Devices**, *Zhigang Xiao*, *J Kimbrough*, *J Cooper*, *K Hartage*, *Q Yuan*, Alabama A&M University

In this research, we report the wafer-scale fabrication of carbon nanotube or graphene-based electronic device such as field-effect transistors (FETs). Carbon nanotube-based devices were fabricated with the alternating electric field-directed dielectrophoresis (DEP) method, and the graphene-based devices were fabricated with the carbon films grown with plasma-enhanced atomic layer deposition (PEALD) or e-beam evaporation. Semiconducting carbon nanotubes were dispersed ultrasonically in solutions, and were deposited and aligned onto a pair of gold electrodes in the fabrication of carbon nanotube-based electronic devices using the dielectrophoresis method. The DEP-aligned tubes were further fabricated

into carbon nanotube field-transistors (CNTFETs) and CNTFET-based electronic devices such as CNT-based inverters and ring oscillators using the microfabrication techniques. The fabricated devices were imaged using the scanning electron microscope (SEM) and high-resolution transmission electron microscope (HRTEM), and the electrical properties were measured from the fabricated devices using the semiconductor analyzer. The semiconducting CNTs achieved higher yield in the device fabrication, and the fabricated devices demonstrated excellent electrical properties.

## Manufacturing Science and Technology Group Room A226 - Session MS+EM+QS-ThM

### Science and Technology for Manufacturing: Neuromorphic and Quantum Computing (ALL INVITED SESSION)

**Moderators:** Nathaniel C. Cady, SUNY Polytechnic Institute, Albany, Alain C. Diebold, SUNY College of Nanoscale Science and Engineering

8:40am **MS+EM+QS-ThM-3 Materials and Fabrication Challenges for Neuromorphic and Quantum Computing Devices**, *S Olson*, *C Hobbs*, *H Chong*, *J Nalaskowski*, *H Stamper*, *J Mucci*, *B Martinick*, *M Zhu*, *K Beckmann*, *I Wells*, *C Johnson*, *V Kaushik*, *T Murray*, *S Novak*, *S Bennett*, *M Rodgers*, *C Borst*, *N Cady*, *M Liehr*, **Satyavolu Papa Rao**, SUNY Polytechnic Institute  
**INVITED**

Devices for quantum computing, quantum communications and quantum sensing share many challenges in terms of the materials, their interfaces, and fabrication technologies. This presentation will quickly review the broad swath of quantum technologies that are being actively studied, while identifying synergies among them that can be exploited for efficient development of integrated quantum computing systems. Advanced process tools capable of exquisite control of the processes, materials and interfaces at 300mm wafer scale have been utilized for the fabrication of structures for quantum computing. Examples of such efforts, including structures for superconducting transmon qubits, resonators, and superconducting nanowire single photon detectors, will be discussed – with an emphasis on the materials and process control issues that needed to be tackled, while keeping manufacturability considerations always in mind. The presentation will conclude with a discussion of how advances in the fabrication of such devices for quantum computing are being applied to ‘adjacent spaces’ such as neuromorphic computing using superconducting optoelectronics (in partnership with AFRL-Rome and NIST Boulder).

9:20am **MS+EM+QS-ThM-5 IBM Q: Quantum Computing in the 21st Century**, **Robert Sutor**, IBM Research  
**INVITED**

For almost 40 years, quantum computing has intrigued and amazed scientists and non-scientists in its future possibility for solving problems that are intractable using classical computing. Over the last three years, IBM has made real quantum computers available on the cloud so that clients, students, and researchers can begin to learn and experiment with this new way of computing. We'll see what use cases are being considered in industry, the state of quantum computing today, and how you can get on the right path to make the earliest use of this rapidly evolving technology.

11:00am **MS+EM+QS-ThM-10 Quantum Information Science at AFRL**, **Michael Hayduk**, Air Force Research Laboratory  
**INVITED**

Recent advances in Quantum Information Science (QIS) indicate that future applications of quantum mechanics will lead to disruptive advances in capabilities for the US Air Force. Controlling and exploiting quantum mechanical phenomena will enable inertial sensors and atomic clocks that provide GPS-like positioning and timing accuracy for extended periods of time in degraded environments, communications networks with information security based on physics principles, unprecedented sensor resolution, and computers that may be able to provide exponential speedup in processing speed. To ensure that the future Air Force warfighter maintains a technological advantage, the AF must implement a QIS strategy that leads to robust, deployable quantum systems. This invited talk will discuss the recently developed Air Force Research Laboratory QIS strategy that covers the areas of timing, sensing, communications and networking, and computing. Capability development across these four areas will also be discussed.

11:40am **MS+EM+QS-ThM-12 Neuromorphic Computing: From Emerging Devices to Neuromorphic System-on-a-Chip**, **Vishal Saxena**, University of Idaho  
**INVITED**

Several classes of emerging non-volatile memory (NVM) devices are currently being investigated for their application in analog implementation

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of artificial neural networks (ANN) hardware. The device can be two- or three-terminal and employ a wide range of material systems and associated physical mechanisms to achieve two or more non-volatile memory states. ANN hardware realizations include vector matrix multipliers (VMMs) and neural-inspired or Neuromorphic computing circuits. The NVM devices are employed in the form of crossbar or crosspoint arrays with or without selectors. In order to exploit the high-density and potential low-power operation of these devices, Analog circuit designers need to accommodate non-ideal behavior of these devices. This is particularly important for optimizing transistor-level circuit design for layout area, reliability, and static and dynamic power consumption. NVM nonidealities include device variability, low resistances offered by the two-terminal devices, finite resolution, relaxation of incremental states, limited dynamic range, and read/write endurance. This talk will provide an overview of Neuromorphic System-on-a-Chip (NeuSoC) that can be realized using emerging NVM arrays, expected device characteristics, associated circuit design challenges, and potential strategies for their mitigation. The talk will also include energy-efficiency estimation and benchmarking for NeuSoCs and provide pathways for future work in this area.

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## Manufacturing Science and Technology Group Room Union Station AB - Session MS-ThP

### Manufacturing Science and Technology Poster Session

#### **MS-ThP-1 Evaluation of Mechanical Properties of Infill Structures Change during 3D Modeling, *Seita Ogawa, A Matsumuro*, Aichi Institute of Technology, Japan**

By products with a 3D printer, modeling of complex shapes is possible based on the data obtained by 3D CAD and 3D scanner. In recently, 3D printers are actively used for products from the stage of being used for prototyping. Therefore, we have focused on the filling rates and infill structures for 3D printing products. There are cavities in the shaped object, and the mechanical properties of the object change depending on infill structures and filling rates. Our purpose of this study is to evaluate mechanical characteristics for infill formation conditions due to create an important database in future. we considered it to be important creating a database of their mechanical properties for products used 3D printer. Conventional tensile test and three-point bending test were performed using typical types of materials, ABS resin, PLA resin, and Primalloy ( Mitsubishi Chemical Co., Ltd. ). In this research, the infill structures of several kinds of default set with slicer software used, i.e. hatching, zigzag, honeycomb and 3D honeycomb. The filling rates were increased to 50% every 10%. The test pieces used the dumbbell shape based on JIS.K7161. I used a MUTOH 3D printer MF-2200D as a device, and used an Imada measuring stand and a force gauge for a tensile test and a three-point bending test. In the three-point bending test, we tested from both side of flat surface and edge side to the stacking direction. As a result, the ABS test piece of the filling rate 30% and 50% increased 60% and 70% higher than the filling rate 10% and 50% respectively in the tensile strength. It was found that an increasing of the filling rates leads to an increasing of mechanical properties. Summarizing results of all test conditions, it was cleared that the tendency of the mechanical properties of bending from two directions was different in the all materials. The tendency of mechanical properties was different depending on different kinds of material. From our results, we considered it to these databases will be great important for products of large field using 3D printers. Furthermore, we try to investigate several kinds of infill structures that have higher strength. Finally, we hope that our study contributes to innovative industry in the near future.

#### **MS-ThP-2 Development of Innovative CNT/Extra Super Duralumin Composite Materials, *Chihiro Fujiwara*, Aichi institute of Technology; *A Matsumuro*, Aichi Institute of Technology, Japan**

Current science and technology should serve as it overcoming the issues of global environment and realization of the new industrial revolution immediately. To solve these important problems, drastic challenge from every field is accomplished. We focus on materials development with innovative characteristics in this study. In late years technology development about the space utilization become much active. Importance of the aerospace apparatus will increase more and more. Due to contribute to current technological development, we should design creatively an innovative high specific strength material. As our research objective, we came up with the idea of the development of an Extra Super Duralumin (ESD) based composite material with Carbon Nanotube (CNT), which has been well known as unprecedented excellent characteristics. ESD is an aluminum base alloy with high tensile strength and pressure resistance. The basic fabrication process of metal based composite material with nano-carbon materials have been established in various materials system. We established original successful uniform dispersion of CNT with in Al based materials, and we demonstrated already that Vickers hardness of CNT/Al composite pellet-formed sintered materials showed several times up in comparison with that of conventional Al bulk material.

In this study, pellet-plate type specimens consisted of our CNT/ESD composite materials were fabricated using compression sintering method with commercial single wall carbon nanotube and ESD powder of dozens of micrometers of particle size powder. The heat-treatment after sintering specimen was done under the conventional method. The obtained main optimum conditions as follows: CNT composite ratio of 1wt. %, ultrasonic dispersion time of 4 h and sintered temperature of 723 K for 4 h.

It has already been demonstrated that Vickers hardness of CNT/ESD composite showed about 6 % increase in comparison with sintered ESD bulk material, and the density decreased down to 1.5 %. So, the specific

strength improved up to 10 % just as expected. This result shows clearly possibility to develop a product with innovative characteristic by the effect of composition with nano-carbon materials. These results would suggest bringing a change in the concept of manufacturing process. From now on, we will estimate tensile and bending characteristics with quantity evaluation. Furthermore we would intend challenge to develop CNT/ESD and graphene/ESD composites bulk materials made using the melting process due to lead the innovative materials to practical use. We would like to present charming results at the conference.

#### **MS-ThP-3 Development of Composite Resin Materials with High Dispersion Cellulose Nanofibers, *Naoki Iwanaga*, *A Matsumuro*, Aichi Institute of Technology, Japan; *K Osawa*, Aichi Institute of Technology, Japn, Japan**

Progress of science and technology should serve as it overcoming the problem of global environment immediately. The challenge from every field is accomplished to solve this important problem. In terms of material issues, high strength and environmentally friendly objects are required. So, we strongly focused on cellulose nanofibers(CNF).

CNF show extraordinary mechanical, physical and chemical properties. Furthermore, CNF is a biodegradable material with low environmental impact and excellent recyclability. Therefore, it is possible to suppress the occurrence of micro plastics, which is a problem in global issues.

In addition, development of the advanced composite resin filaments with CNF for uses 3D printing leads to extend especially application field, due to excellent environmental problem and mechanical properties as one of core of current technology Industry revolution.

In this study, we try to development an innovative high strength resin based composite materials reinforced by CNF with characteristics of eco-friendly and material characteristics.

At first, we researched the possibility of the application of CNF composite materials with ABS base resin, in order to investigate possibility of fabrication of composite material with CNF and improvement of characteristics in comparison with conventional resin materials. The fabrication method with uniform dispensation of CNF in ABS resin powder was applied with the ultrasonic method established on our own. Specimen were made by the die molding method at 250K for 30minutes.

In our results, the optimum dispersion time was determined 6 hours by surface observation.

FT-IR measurement showed that CNF and ABS resin could be dispersed while maintaining its structure. We should remarkable results of representative mechanical property. Tensile strength and young's modulus for specimen excellently increased up to about 70 % for 1.0 wt% CNF and 97 % for 0.5 wt% CNF in comparison with the value of pure ABS resin specimen. Furthermore, strength improvement using nylon 6 was clarified as the same trend in the case of ABS resin.

These results described above should clarified the development important guidelines for fabrication process, of an innovative composite resin materials with enough specific characteristics of CNF.

Furthermore, in order to expand the possibility of practical realization, we are now challenging, to apply to biodegradable PLA resin.

#### **MS-ThP-4 Improvement of Laminated Interface Strength of Printed Objects by FDM 3D Printer, *Li Song*, Aichi institute of technology, Japan**

Indispensable 3D printing technique in manufacturing has made us an important unavoidable problem until now. It is unquestionably laminated interfacial existence in molded products. The problem become obviously that the strength of printed products in a vertical direction for printing direction is extremely weaker than the strength with along direction about almost same strength of original filament. The reason is absolutely clear for existence of laminated interfaces with a heterogeneous structure.

In this study, we tried to challenge an innovative improvement method of the excellent laminated interface strength which is the ultimate purpose to obtain conventional products with strength of the bonding force with uniformization of the upper layer and the structure. We tried to create a new 3D printer by installing a compact halogen spot heater attached to one direction at the extruder of the FDM 3D printer with commercially black PLA filament as our first step. We constructed our original system in conjunction with the extruder with spot optical heating on fabricating lower layer interfaces due to change semi-melted state of already fabricated solid satate molded object during modeling. This effect contributes to the homogenization of the interface organization that is improvement of the interface strength.



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In this study, it is great important to optimize optical radiation conditions of halogen spot heater due to change lower layer state to semi-melt again while maintaining the shape of the solidified lower layer part, while maintaining the shape of a product. Through trial and error, Optimum conditions of geometric placement of the devices and the irradiation temperature of 393 K estimated using a thermocouple. In order to evaluate a change of the interface strength by a tension test more definitely, we loaded perpendicularly in the laminated direction.

We showed great results that the tensile strength of typical specimen increased from 27 MPa to 34 MPa, and the surface smoothness drastic increase from Ra of 24 to 11 simultaneously by a mild fusion effect by the heat. Both surprisingly results was supported by the fracture surface observations. Using laser microscope, the microstructure of the unheated fracture surface was broken at the specific layer, but that of heated product was observed at complex layers. These results were attributed to the interfacial texture homonigeton because of optimal semi-meltirized under layers.

The above-mentioned result makes the great effectiveness of the laminate spot heating method at the same time of molding heating remarkable clear.

**MS-Thp-5 Investigation of Multi-Level ReRAM in 65nm CMOS for Logic-in-Memory Applications, Sarah Rafiq, K Beckmann, J Hazra, M Liehr, SUNY Polytechnic Institute; S Jha, University of Central Florida; N Cady, SUNY Polytechnic Institute**

Resistive Random Access Memory (ReRAM) has been extensively investigated as a non-volatile memory due to its low energy consumption and scalability. Bipolar ReRAM devices integrated in array architectures with selector devices is a prime candidate for high density memory arrays, novel logic-in-memory applications, and neuromorphic computation. Using a 65nm CMOS process technology, we have integrated 100 x 100 nm<sup>2</sup> HfO<sub>2</sub>-based ReRAM devices at the metal 1 / via 1 (M1/V1) interface in a 1 transistor – 1 ReRAM (1T1R) configuration. Arrays of 1T1R cells were evaluated for binary switching between high resistance states (HRS) and low resistance states (LRS), exhibiting excellent yield and performance across a full 300mm wafer. Multi-level switching of 1T1R cells was then investigated by adjusting the gate voltage of the control transistor, which in turn, modulates the current compliance during programming (set operation) of the ReRAM device. Individual 1T1R cells within 8 x 8 arrays were programmed using increasing compliance current from 20 uA to 0.14 mA, which resulted in a 5-fold change in resistance level from 36 kOhm to 6.6 kOhm respectively. Multiple arrays from multiple 300 mm wafers have been evaluated to determine the variability within arrays, and the effects of changing processing conditions between wafers. Our results show that within a single wafer, 1T1R performance is consistent, but that variation in processing conditions for the HfO<sub>2</sub> switching layer can dramatically affect resistance levels and endurance of 1T1R. When comparing arrays on a single wafer, the standard deviation of the resistance state (for 100 switching cycles) decreased from 15 kOhm when programmed with current compliance of 20uA, to less than 500 Ohm at higher current compliance. Therefore, multiple distinguishable resistance states were achieved with higher current compliance. Using the two states (LRS and HRS), a 2 x 2 sub-array of 1T1R cells was then used to implement XOR logic functionality in a logic-in-memory configuration. Despite one of the cells having a low HRS not exceeding 20 kOhm, the output of the XOR logic was still unaffected. This demonstrates the robustness of logic-in-memory applications. The distinct binary state based logical computations, enabled by the appropriate selection of current compliance, also paves the way for ternary state logic and memory. Ongoing efforts are focused on higher precision control of the multi-level memory performance for 1T1R arrays up to 512 x 512 cells, and understanding the effects of wafer processing conditions on stochasticity of multi-level memory states, with the ultimate goal of full analog operation.

**MS-Thp-6 III-V NanoWires for Junctionless Transistors Fabricated by Focused Ion Beam (FIB) System with Silicon Nitride Passivation, Cássio Almeida, University of Campinas, Brazil; P Souza, PUC-Rio, Brazil; M Pires, Federal University of Rio de Janeiro, Brazil; J Diniz, University of Campinas, Brazil**

III-V Junctionless semiconductors devices were fabricated on InGaP and GaAs substrates using Gallium (Ga<sup>+</sup>) Focused Ion Beam (FIB) System. Two groups of samples, with n<sup>+</sup>-InGaP (10nm)/GaAs-buffer layer (300nm) and n<sup>+</sup>-GaAs (10nm)/GaAs-buffer layer (300nm), both on semi-insulating GaAs (001) nominal orientation substrates, were studied. The samples were grown by Metalorganic Vapor Phase Epitaxy (MOCVD) in an Aixtron AIX 200

horizontal reactor at 100mbar, where the samples are heated by infra-red (IR) lamps. The total hydrogen carrier gas flow rate was 8L/min. The precursors used for the GaAs growth were trimethylgallium (TMGa) and arsine (AsH<sub>3</sub>). Silane (SiH<sub>4</sub>) was used for the n doping. The layers were grown at 630°C with a growth rate of 0,36nm/s and a V/III ratio of 70 for the n doped layer. A pre-growth treatment for de-oxidation at 700°C was applied to the GaAs substrates for 15minutes under AsH<sub>3</sub> over pressure. Furthermore, for the first time, the silicon nitride layer (SiN<sub>x</sub>), thickness of 10nm, deposited by ECR-CVD, was used as gate dielectric of Junctionless and as passivation layer of the surfaces of structures. The morphology of the samples was observed by Atomic Force Microscopy (AFM). X-Ray diffraction (XRD) analysis was used in order to determine the InGaP lattice parameter and mismatch to the GaAs substrate. Hall measurements provided silicon doping levels of 10<sup>19</sup>cm<sup>-3</sup> for both groups of samples, indicating the formation of n<sup>+</sup>-type layers. These samples were used for MOS Junctionless (JL) Transistors applications, since III-V semiconductors present higher electron mobility values than silicon. These JL transistors (with three terminals: gate, source and drain) are fabricated using a Focused Ion Beam (FIB) System. Thus, Gallium (Ga<sup>+</sup>) Focused Ion Beam (FIB) is used to define the III-V (InGaP or GaAs) nanowires (III-V NWs), which are the electron conduction channel between source and drain and Pt deposition (as gate, drain and source electrodes) layers. Finally, drain-source current (I<sub>DS</sub>) versus drain-source voltage (V<sub>DS</sub>) and drain-source current (I<sub>DS</sub>) versus gate-source voltage (V<sub>GS</sub>) measurements of JL devices will be extracted and will be able to indicate if these InGaP or GaAs nanowires and the passivation, with the SiN<sub>x</sub> deposited by ECR-CVD are of high quality and suitable for Junctionless technology.

**MS-Thp-7 The Development of High Efficiency X-ray Tube with Carbon Nanotube Yarn based-cold Cathode, Hyun Suk Kim, C Lee, Wonkwang University, Korea**

It is development high-efficiency X-ray tube using carbon nanotube yarn as an electronic source of field emission. It is inevitable to secure durability for uniform electronic emission characteristics of materials and to improve the stable structure of the emitter due to the miniaturization of the X-ray tube. Aimed at a high-efficiency X-ray generator with a new concept of cold-polar emission e-meter structure that can control the gap of uneven field discharge by a Chaos of uncontrolled faults in the process. The effects of various structures on the beam focusing performances and emission currents were simulated and fabricated. In the design of the X-ray sources, it is important to ensure that the fine beam focus and efficient electron emission can be simultaneously obtained. Therefore, the geometrical parameters, such as electrode shape and the gaps between parts should be optimized. Owing to the unique design of the cathode, the electron beam emitted from the cathode was focused onto the anode without using electric lenses or extra biased electrodes. It was indicated that the beam spot sizes on the anode plate different with the changing electrode shape design. It will be studied that the optimum x-ray yield condition and focusing electrical shape effect in the CNT micro-focus CNT x-ray tube.

**MS-Thp-8 High Aspect Ratio Carbon Nanotube Optical Collimator, Tyler Westover, R Davis, R Vanfleet, Brigham Young University**

Patterned carbon nanotube forests are finding an increasing number of applications due to their high aspect ratios and the characteristics of the nanotubes themselves. For example carbon nanotubes are highly absorptive of light in various wavelengths including visible and near infrared. Due to the high absorption of light that comes into contact with a carbon nanotube forest these structures work as sidewalls for an optical collimator. Here we will present our results using a carbon nanotube collimator in the visible and the IR.

**MS-Thp-9 Development of a Fabrication Process for Integrated inductors on Flexible Substrate, Wilson Freitas, State University of Campinas, Brasil; M Oliveira Piazzetta, Brazilian Nanotechnology National Laboratory, Brazil; L Manera, UNICAMP, Brazil; A Gobbi, Brazilian Nanotechnology National Laboratory, Brazil**

This paper presents the development of a fabrication process for integrated inductors on flexible substrate. The process consists of two metal and one dielectric layer on a polyimide flexible substrate. Kapton was the choice for the substrate due to its attractive characteristics such as high dimensional stability and thermal and electrical insulation. The first metal layer was 0.5 μm thick gold deposited by electroplating on a nickel film. Nickel was deposited by sputtering and used both as a seed for the electroplating deposition process and to improve the adhesion of gold to the substrate. Gold lines were patterned by lift-off and serve as mask for nickel wet etching. The second metal layer was made through the same

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process, with thickness of 1  $\mu\text{m}$ . Silicon dioxide was selected as dielectric, also patterned by lift-off and preceded by a chrome e-beam deposition step to enhance adhesion. Structures were formed by conventional photolithography process, with minimum line and space dimensions of 10  $\mu\text{m}$ . With the use of intermediate layers of nickel there was no need for substrate modification to improve metal layers adhesion. The first metal layer thickness was optimized to improve dielectric step coverage, and the second layer was made thicker to reduce series resistance and provide better performance in both quality factor and frequency response. With this technology, it was possible to fabricate integrated inductors with 1 to 5 nH, maximum quality factor of 8, and self-resonant frequency in excess of 20 GHz, with values compatible with those from simulation with Keysight ADS, allowing for the implementation of VCOs and others RF circuits for hybrid flexible electronic structures.

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Bruner, P: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Bsiesy, A: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
— C —  
Cady, N: MS+EM+QS-ThM-3, **6**; MS-ThP-5, **9**  
Carmo, M: TL+MS+VT-TuM-5, **1**  
Carter, R: MS-WeA-9, **4**  
Chervin, C: MS-WeA-1, **4**  
Chong, H: MS+EM+QS-ThM-3, **6**  
Coffey, K: EM+AP+MS+NS+TF-ThM-6, **5**  
Cooper, J: EM+AP+MS+NS+TF-ThM-13, **6**  
— D —  
David, S: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Davis, R: MS-ThP-8, **9**  
Diniz, J: MS-ThP-6, **9**  
— E —  
Elam, J: AP+EL+MS+PS+SS+TF-TuA-3, **2**  
Ezzat, S: EM+AP+MS+NS+TF-ThM-6, **5**  
— F —  
Filler, M: EM+AP+MS+NS+TF-ThM-5, **5**  
Franz, G: EM+AP+MS+NS+TF-ThM-1, **5**  
Freitas, W: MS-ThP-9, **9**  
Fujiwara, C: MS-ThP-2, **8**  
— G —  
Gall, D: EM+AP+MS+NS+TF-ThM-6, **5**  
Gassilloud, R: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Gobbi, A: MS-ThP-9, **9**  
Gong, Y: EM+AP+MS+NS+TF-ThM-10, **5**  
Grehl, T: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
— H —  
Hartage, K: EM+AP+MS+NS+TF-ThM-13, **3**  
Hayduk, M: MS+EM+QS-ThM-10, **6**  
Hazra, J: MS-ThP-5, **9**  
Hilfiker, J: AP+EL+MS+PS+SS+TF-TuA-1, **2**  
Hobbs, C: MS+EM+QS-ThM-3, **6**  
— I —  
Iwanaga, N: MS-ThP-3, **8**  
— J —  
Jaffal, M: AP+EL+MS+PS+SS+TF-TuA-9, **2**

Jha, S: MS-ThP-5, **9**  
Johnson, C: MS+EM+QS-ThM-3, **6**  
— K —  
Kaden, W: EM+AP+MS+NS+TF-ThM-6, **5**  
Kapadia, R: EM+AP+MS+NS+TF-ThM-2, **5**  
Kaushik, V: MS+EM+QS-ThM-3, **6**  
Keller, N: AP+EL+MS+PS+SS+TF-TuA-7, **2**  
Khaniya, A: EM+AP+MS+NS+TF-ThM-6, **5**  
Kim, H: MS-ThP-7, **9**  
Kimbrough, J: EM+AP+MS+NS+TF-ThM-13, **6**  
King, S: EM+AP+MS+NS+TF-ThM-12, **6**  
— L —  
Lee, C: MS-ThP-7, **9**  
Lee, S: MS-WeA-3, **4**  
Liehr, M: MS+EM+QS-ThM-3, **6**; MS-ThP-5, **9**  
Long, J: MS-WeA-1, **4**  
Love, C: MS-WeA-9, **4**  
— M —  
Ma, J: MS-WeA-7, **4**  
Manera, L: MS-ThP-9, **9**  
Mani, P: EM+AP+MS+NS+TF-ThM-6, **5**  
Martinick, B: MS+EM+QS-ThM-3, **6**  
Matsumuro, A: MS-ThP-1, **8**; MS-ThP-2, **8**;  
MS-ThP-3, **8**  
Melton, O: AP+EL+MS+PS+SS+TF-TuA-12, **3**  
Meyer, R: EM+AP+MS+NS+TF-ThM-1, **5**  
Mucci, J: MS+EM+QS-ThM-3, **6**  
Murray, T: MS+EM+QS-ThM-3, **6**  
— N —  
Nalaskowski, J: MS+EM+QS-ThM-3, **6**  
Nauman, K: TL+MS+VT-TuM-11, **1**  
Novak, S: MS+EM+QS-ThM-3, **6**  
— O —  
Oberhausen, W: EM+AP+MS+NS+TF-ThM-1,  
**5**  
Ogawa, S: MS-ThP-1, **8**  
Oliveira Piazzetta, M: MS-ThP-9, **9**  
Olson, S: MS+EM+QS-ThM-3, **6**  
Opila, R: AP+EL+MS+PS+SS+TF-TuA-12, **3**  
Osawa, K: MS-ThP-3, **8**  
— P —  
Papa Rao, S: MS+EM+QS-ThM-3, **6**  
Parker, J: MS-WeA-1, **4**  
Pelissier, B: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Pesce, V: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Pires, M: MS-ThP-6, **9**  
Posseme, N: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
Pribil, G: AP+EL+MS+PS+SS+TF-TuA-1, **2**  
Prieto, A: MS-WeA-7, **4**  
Puurunen, R: AP+EL+MS+PS+SS+TF-TuA-10,  
**2**

— R —

Rafiq, S: MS-ThP-5, **9**  
Rodgers, M: MS+EM+QS-ThM-3, **6**  
Rolison, D: MS-WeA-1, **4**  
Rubloff, G: MS-WeA-3, **4**  
— S —  
Sarkar, D: EM+AP+MS+NS+TF-ThM-2, **5**  
Sassin, M: MS-WeA-1, **4**  
Saxena, V: MS+EM+QS-ThM-12, **6**  
Schulze, M: MS-WeA-7, **4**  
Shehzad, M: EM+AP+MS+NS+TF-ThM-5, **5**  
Siyoum, A: TL+MS+VT-TuM-10, **1**  
Smith, W: TL+MS+VT-TuM-3, **1**  
Song, L: MS-ThP-4, **8**  
Sooryakumar, R: EM+AP+MS+NS+TF-ThM-  
12, **6**  
Souza, P: MS-ThP-6, **9**  
Stamper, H: MS+EM+QS-ThM-3, **6**  
Sushkov, V: EM+AP+MS+NS+TF-ThM-1, **5**  
Sutor, R: MS+EM+QS-ThM-5, **6**  
— T —  
T. Mohabir, A: EM+AP+MS+NS+TF-ThM-5, **5**  
Tortai, J: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
— U —  
Uedono, A: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
— V —  
Vallee, C: AP+EL+MS+PS+SS+TF-TuA-9, **2**  
van de Sanden, R: TL+MS+VT-TuM-1, **1**  
VanDerslice, J: AP+EL+MS+PS+SS+TF-TuA-1,  
**2**  
Vanfleet, R: MS-ThP-8, **9**  
Venkatraman, K: EM+AP+MS+NS+TF-ThM-  
10, **5**  
— W —  
Wallace, C: EM+AP+MS+NS+TF-ThM-3, **5**  
Wang, Z: AP+EL+MS+PS+SS+TF-TuA-12, **3**  
Wells, I: MS+EM+QS-ThM-3, **6**  
Weng, S: EM+AP+MS+NS+TF-ThM-2, **5**  
Westover, T: MS-ThP-8, **9**  
Wijesinghe, H: EM+AP+MS+NS+TF-ThM-12,  
**6**  
— X —  
Xiao, Z: EM+AP+MS+NS+TF-ThM-13, **6**  
Xu, Y: EM+AP+MS+NS+TF-ThM-2, **5**  
— Y —  
Yoo, H: EM+AP+MS+NS+TF-ThM-12, **6**  
Yuan, B: AP+EL+MS+PS+SS+TF-TuA-12, **3**  
Yuan, Q: EM+AP+MS+NS+TF-ThM-13, **6**  
— Z —  
Zhu, M: MS+EM+QS-ThM-3, **6**  
Zizka, J: EM+AP+MS+NS+TF-ThM-12, **6**