Monday Afternoon, November 6, 2023

Nanoscale Science and Technology Division
Room B113 - Session NS+EM+MN-MoA

Nanoscale Devices, Structures and Materials

Moderators: Aubrey Hanbicki, Laboratory for Physical Sciences, **Deep Jariwala**, University of Pennsylvania

1:40pm **NS+EM+MN-MoA-1 Integrated Nanophotonics Temperature Metrology Platform***, **Nikolai N. Klimov**, K. Douglass, D. Barker, T. Bui, S. Robinson, T. Herman, K. Quelhas,* National Institute of Standards and Technology (NIST)

Temperature, being, perhaps, the second most measured physical property after time and frequency plays a crucial role in various aspects of modern technology ranging from medicine and Earth's climate to semiconductor industry and advanced manufacturing process control. While there have been great strides in developing novel thermometry approaches, resistance-based thermometry remains the standard method for disseminating the SI unit of temperature at the highest level of precision. The fundamental limitations of resistance thermometry, as well as the desire to reduce sensor ownership cost, have ignited a substantial interest in the development of alternative technologies such as photonics-based temperature sensors.

At the National Institute of Standards and Technology (NIST), we are developing a new photonics-based temperature measurement solution that has the potential to revolutionize how temperature is realized and disseminated to customers. One of the key elements of our Photonic Thermometry program is an ultra-Sensitive Photonic Thermometer (SPoT) – an on-chip integrated silicon nanophotonic resonator, whose optical resonance frequency shifts with temperature due to high thermo-optic coefficient of silicon and can be used to trace temperature variations with high precision. Our goal is to evolve SPoT into a robust, field-deployable device that is on par or better than the state-of-the-art resistance thermometer.

In this work, we describe the performance of SPoT thermometer, as well as a new photonic readout for SPoT. In our new read-out scheme, we employ a novel offset-locking technique for reading out the resonance wavelength of the SPoT. This method provides extremely high accuracy for relative temperature changes on a short time scale (<< 1s).Our results indicated that the packaged on-chip integrated SPoT can detect temperature fluctuations as small 2 µK over 200 ms integration time. This methodology as well as other proposed methods will be discussed.We also show a benchmark comparison of SPoT thermometer to Standard Platinum Resistance Thermometer (SPRT) – the best-in-class resistance thermometer, in various fixed-point cells of ITS-90, evaluating temperature resolution and repeatability

2:00pm **NS+EM+MN-MoA-2 AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient Talk: Breaking the Efficiency Bottleneck of Micro-LEDs Through Nanoscale and Excitonic Engineering***, **Yixin Xiao**[1], R. maddaka, Y. Wu, Y. Malholtra, Y. Guo, S. Yang, K. Sun, A. Pandey, J. Min, Z. Mi,* University of Michigan, Ann Arbor

The performance of conventional optoelectronic devices, such as LEDs and laser diodes, is extremely sensitive to the presence of defects and dislocations. For these reasons, it has remained challenging to achieve high efficiency nanoscale LEDs and laser diodes. For example, while conventional broad area LEDs can exhibit external quantum efficiency (EQE) in the range of 80-100%, the EQE of submicron scale LEDs is often <1%, due to the dominant nonradiative surface recombination. The operation of conventional LEDs involves the radiative recombination of free electrons and holes in the active region. It is known that an exciton, a bound state of an electron and hole through strong Coulomb interaction, can drastically enhance the radiative recombination efficiency, which can be potentially exploited to make micro and nanoscale LEDs relatively immune from the presence of defects/traps. Recent studies have shown that the exciton oscillator strength can be increased by nearly two orders of magnitude in small size InGaN nanowires, due to efficient strain relaxation. Here we demonstrate that the efficiency bottleneck of µLEDs can be fundamentally addressed by utilizing bottom-up III-nitride nanostructures. We report on the demonstration of micrometer scale green and red LEDs with an external quantum efficiency of 25% and 8%, respectively, which are the highest values ever reported to the best of our knowledge. We employ selective area plasma-assisted molecular beam epitaxy as the material synthesis platform. Due to efficient strain relaxation, such bottom-up nanostructures are largely free of dislocations. By exploiting the large exciton binding energy and oscillator strength of quantum-confined InGaN nanostructures, we show that the external quantum efficiency of a green-emitting micrometer scale LED can be dramatically improved from ~4% to >25%. The dramatically improved efficiency is attributed to the utilization of semipolar planes in strain-relaxed nanostructures to minimize polarization and quantum-confined Stark effect and the formation of nanoscale quantum-confinement to enhance electron-hole wavefunction overlap. We have further developed a new approach that included an InGaN/GaN short period superlattice together with an InGaN quantum dot active region to achieve high efficiency red emission. A maximum quantum efficiency of >7% was measured. Our studies offer a viable path to achieve high efficiency micrometer scale LEDs for a broad range of applications including mobile displays, virtual/augmented reality, biomedical sensing, and high-speed optical interconnects, that were difficult for conventional quantum well based LEDs.

2:20pm **NS+EM+MN-MoA-3 Modeling Gas Phase Etching in High Aspect Ratio Stacked Nanostructures for Semiconductor Processing: Stacked SiGe Layer Etching***, **Zach Zajo**,* Stanford University*; D. Mui, J. Zhu, M. Kawaguchi,* Lam Research Corp.*; E. Shaqfeh,* Stanford University

Gate all around (GAA) nano-transistors offer better channel control and increased current carrying capacity compared to FinFETs (Field Effect Transistor) which are currently the standard in the semiconductor industry. However, the need for precise control of their nanoscale features poses a challenge in manufacturing such GAA nano-transistors. The high material selectivity required in fabricating these transistors makes gas phase etching much more appealing in comparison to liquid phase and plasma-based etching techniques. An etching configuration that is of particular interest is one consisting of alternating layers of Si and SiGe from which the SiGe layers are selectively etched by fluorine gas. In the etching of these structures, it is important to have a uniform etch-rate for SiGe layers from top to bottom, to maintain consistency of the etched features. This consistency is essential for the superior performance of the GAA devices. The key gas phase processing challenge then is to determine and maximize the number of SiGe-Si layers that can be stacked in a single structure while still maintaining a nearly uniform etch-rate from top to bottom in the stack. While experiments have offered insights in terms of the effect of layer thickness, number of layers, gas pressure etc. on the viability of the process, such experiments are quite expensive and tedious. We propose and develop computer simulations as a tool to predict the etch profile evolution over time in a gas phase etching process. The tool is based on a mathematical model which considers the transport processes and surface interactions involved in the gas phase etching process – which at the nanoscale is primarily governed by Knudsen diffusion in the free molecular flow regime. Thus, the transport model is formulated as a boundary integral equation which takes into account the direct flux of etchant molecules that any given point on the exposed surface receives from the bulk gas phase as well as the re-emission flux from other parts of the structure itself. We compared the applicability of two different surface reaction models - a model where the local etch rate is linear in the flux at a point and a Langmuir adsorption/reaction model- to connect the net flux received at a point on the surface to the local etch rate. Our results show that the re-emission of etchants at the etching interface plays a vital role in determining the differential etch rates observed across layers at different depths in the stacked feature. In addition, we have characterized the effect of layer thickness and the spacing between adjacent stacks as these impact the etch rates observed from layer to layer.

2:40pm **NS+EM+MN-MoA-4 Fabrication of Silicon Microfluidic Gratings for Neutron Imaging***, S. Robinson, R. Murphy,* National Institute of Standards and Technology (NIST)*; Y. Kim,* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park*; J. LaManna, C. Wolf, K. Weigandt, D. Hussey, **Nikolai N. Klimov**,* National Institute of Standards and Technology (NIST)

In this work, we describe the development of a spatial modulator for x-ray and neutron beams. Current technology for x-ray and neutron phase imaging uses individual source gratings with a fixed period to modulate the beams. Each fixed-period, or "static" source grating, has a limited range in spatial resolution at a specific length scale, thus, a relatively larger set of individual static source gratings is required to achieve high-resolution imaging. Our team is developing a neutron imaging technique that will probe heterogeneous samples across multiple length scales. To address the need for a large variability of source grating periods, we are building a

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[1] **AVS Dorothy M. and Earl S. Hoffman Scholarship Recipient**

silicon microfluidic-based device, DynAmic ReconfIgUrable Source grating (DARIUS), that itself is capable to adjust, with high resolution, the grating period from 20 µm to 20,000 µm. With such on-demand tunability, a single DARIUS has the potential to replace more than 500 static source gratings, minimizing the need of fabricating, installing, and aligning a new source grating for the required period. To achieve such functionality, DARIUS features over 5,000 microfluidic grating channels that are etched on both the front and back sides of a 100 mm silicon wafer. Each of these grating channels can be selectively infilled with a neutron and/or x-ray absorbing fluid, and provide real-time reconfigurable spatial modulations for neutron and x-ray beams. In this work, we present updates on the double-sided deep silicon etch (on the front and the back of the wafer), as well as the front-to-back alignment of the silicon channels. We also describe the progress towards wafer bonding to seal the front and back sides of DARIUS device. Due to the challenges of both the microfluidic control and tight fabrication tolerances, we are evaluating the design specifics to address larger grating periods.

3:00pm **NS+EM+MN-MoA-5 The Small Shift Matters – Submilliradian Tilt Goniometry in Scanning Electron Microscopy***, **Andrew Madison**, J. Villarrubia, D. Westly, R. Dixson, C. Copeland,* National Institute of Standards and Technology (NIST)*; J. Gerling, K. Cochrane, A. Brodie, L. Muray,* KLA-Tencor*; J. Liddle, S. Stavis,* National Institute of Standards and Technology (NIST)

Electron optical aberrations degrade the accuracy and reliability of scanning electron microscopy. Among multiple aberrations of potential concern, an axial tilt of the electron beam shifts the apparent positions and deforms the intensity profiles of features in scanning electron micrographs. Measurement of the beam tilt can enable either a physical correction of beam deflection or an analytical correction in a measurement function. In this study, we report a novel reference structure and image analysis method to measure such shifts, among other key effects. Our new concept has the potential to improve accuracy in scanning electron microscopy, with multifunctional standards enabling integrative calibrations of beam tilt and beyond. Such advances will be of particular interest in semiconductor manufacturing metrology, where even the small shift matters.

We explore conical frustum arrays as multifunctional reference structures, using physical theory to guide ongoing experiments. For a tilt inclination ϑ, a centroid shift s between the top and bottom edges of a conical frustum shows the effect of tilt. For a frustum height h, the measurement function is simply $\vartheta = \sin^{-1}(s/h) \approx s/h$, yielding a null-tilt sensor and self-calibrating goniometer. To understand the limiting random effect of shot noise, we simulate frustum images using a physical model of electron scattering and emission. At a dose of 60 electrons per nm^2, model shifts show the possibility of submilliradian accuracy for sidewall angles greater than approximately 40 mrad. In experimental measurements, charge accumulation and hydrocarbon contamination may limit the achievable electron dose, while conical asymmetry among other systematic effects will ultimately limit accuracy. In initial experiments, we fabricate submicrometer frustum arrays in silicon using electron-beam lithography and reactive ion etching and demonstrate use of the reference structure in calibrations of a typical scanning electron microscope.

In an integrative calibration, frustum arrays are optimal structures for correlative atomic force, super-resolution optical, and scanning electron microscopy. This workflow yields reference heights and positions that allow calibration of scale factor and correction of scanfield distortion, improving the accuracy of centroid shifts to show electron beam tilt and spatial variation thereof across the imaging field.

4:00pm **NS+EM+MN-MoA-8 On Point – Accurate Integration of Quantum Dots and Bullseye Cavities***, **Craig Copeland**, A. Pintar, R. Dixson, A. Chanana, K. Srinivasan, D. Westly, B. Ilic, M. Davanco, S. Stavis,* NIST-Gaithersburg

Self-assembled quantum dots are promising light sources for quantum networks and sensors. These emerging technologies require the accurate integration of quantum dots and photonic structures, but epitaxial growth forms quantum dots at random positions in semiconductor substrates. Optical localization of these random positions can guide the placement of photonic structures by electron-beam lithography. This integration process requires the reliable registration of position data across microscopy and lithography systems. However, large errors can result from multiple sources, including lithographic and cryogenic variation of reference dimensions for microscope calibration, as well as localization errors from optical distortion. Such errors tend to increase across an imaging field, presenting a critical impediment to exploiting the throughput and scalability of widefield microscopy. In this study, we target this problem and show how our solution enables accurate integration to improve device performance and process yield. We develop our methods of traceable localization to calibrate a cryogenic localization microscope – an optical microscope with the sample and objective lens inside of a cryostat, and custom optics outside of the cryostat. We fabricate and characterize arrays of submicrometer pillars in silicon, creating microscopy standards with both traceable reference positions and traceable reference data for thermal expansion coefficient. We image these arrays with the cryogenic microscope at approximately 1.8 K, localize the pillar positions, and use the reference data to calibrate the microscope. Our calibration determines the scale factor of the imaging system and corrects position errors due to complex distortion, among other aberration effects. We combine the results of this cryogenic calibration with our previous assessment of fabrication accuracy by electron-beam lithography, introducing a comprehensive model of the effects of registration errors on Purcell factor. This performance metric quantifies the radiative enhancement that occurs upon integration of a quantum dot into a bullseye resonator. The Purcell factor reaches a maximum value of approximately 11 for error-free registration of the quantum dot and resonator center. Our model demonstrates the possibility of greatly improving Purcell factor across a wide field. Depending on the Purcell factor threshold, on-point integration can increase yield by one to two orders of magnitude. This foundation of accuracy will enable a transition from demonstration devices to efficient processes, leading to the reliable production and statistical characterization of quantum information systems.

4:20pm **NS+EM+MN-MoA-9 Nanostructured Gas Sensors for the Detection of Meat Spoilage***, **Ken Bosnick**,* National Research Council of Canada

The sustainability of the food industry will remain a key societal challenge in the decades ahead. In North America, over 20% of meat produced is wasted, mostly at the later stages in the supply chain [1]. When meat begins to degrade, it releases biogenic amines (e.g., putrescine, $H_2N\text{-}(CH_2)_4\text{-}NH_2$) for which early detection provides a means to sense the onset of spoilage [2]. Early detection of meat spoilage through the sensing of such amines can proactively assure quality in the food production process and potentially eliminate the need for food recalls and other waste. At the heart of these envisioned quality assurance strategies are portable devices that are capable of rapid detection of low levels of biogenic amines and that can be easily deployed at various stages in the production process. New material technologies with a selective response to low levels of biogenic amines are needed to enable these envisioned devices.

The use of MOS-type gas sensing technology represents a promising avenue for portable gas sensors with many advantages over competing sensing technologies and over more traditional analysis methods. We have investigated the application of Pd-decorated ZnO nanoflowers in a chemiresistive sensing mechanism and found an excellent response of 99.5% at 250 °C towards 400 ppm methylamine [3]. The device also shows a promising response of 45% at room temperature, making it a candidate sensing material for early detection of spoilage in meat-based products. Towards improved performance at room temperature, ZnO nanocantilevers [4] are being investigated for amine sensing and will also be discussed.

[1]FAO. 2011. “Global food losses and food waste – extent, causes and prevention”

[2]Fernanda Galgano, Fabio Favati, Malvina Bonadio, Vitina Lorusso, Patrizia Romano, “Role of biogenic amines as index of freshness in beef meat packed with different biopolymeric materials”, Food Res. Int., 42 (2009) 1147

[3]Jennifer Bruce, Ken Bosnick, Elham Kamali Heidari, “Pd-decorated ZnO nanoflowers as a promising gas sensor for the detection of meat spoilage”, Sens. Actuators B Chem. 355 (2022) 131316

[4]Kissan Mistry, Viet Huong Nguyen, Mohamed Arabi, Khaled H. Ibrahim, Hatameh Asgarimoghaddam, Mustafa Yavuz, David Muñoz-Rojas, Eihab Abdel-Rahman, and Kevin P. Musselman, “Highly Sensitive Self-Actuated Zinc Oxide Resonant Microcantilever Humidity Sensor”, Nano Lett. 22 (2022) 3196

4:40pm **NS+EM+MN-MoA-10 From Natural to Fabricated Gas Sensing Photonic Nanostructures: Unexpected Discoveries and Societal Impact***, **Baokai Cheng**, J. Brewer, B. Scherer, R. Potyrailo,* GE Research Center

Existing gas sensors often degrade their performance in complex backgrounds. Thus, new sensing approaches are required with improved sensor selectivity, accuracy, and stability for demanding applications ranging from homeland security, to industrial process monitoring and safety, and to monitoring of outdoor and indoor pollutants and volatile biomarkers.

In this talk, first, we will demonstrate and analyze capabilities of natural photonic nanostructures as sensors for detection of different gases and the origins of these capabilities. Next, we will demonstrate that this new acquired knowledge from studies of natural nanostructures allowed us to develop design rules to fabricate nanostructures for needed gas selectivity and stability for numerous gas monitoring scenarios at room and high temperatures. These design rules for selective gas sensors bring a multivariable perspective for sensing, where selectivity is achieved within a single nanostructured sensing unit, rather than from an array of separate sensors. We fabricated bioinspired nanostructures using several contemporary technologies and have achieved several new functionalities beyond Nature.By utilizing individual nanostructured sensors rather than sensor arrays we also have improved sensor stability by eliminating independent aging factors in separate sensors and their arrays.The use of existing and our new machine learning (a.k.a. multivariate analysis, chemometrics) tools further advanced our sensor designs and performance in detection of multiple gaseous species. The achieved performance capabilities of our developed bio-inspired photonic gas sensors complete with the capabilities of existing commercially available gas sensors and their arrays. These colorimetric sensors can be tuned for numerous gas sensing scenarios in ambient and high temperatures, in confined areas or as individual nodes for distributed monitoring.

5:00pm **NS+EM+MN-MoA-11 Argon-Plasma Dry Etch of sub-Micron Feature-Size Waveguides in Thin-Film Lithium Niobate***, **Sesha Challa**, N. Klimov, P. Kuo,* NIST-Gaithersburg

Lithium Niobate (LN) possesses exceptional properties such as a wide transparency range, large non-linear coefficient, and high-electro-optic efficiency. These properties along with successful implementation of periodic poling in LN have spurred the development of devices finding applications in spectroscopy, remote sensing, and quantum communications.

Despite these valuable properties and possessing large second-order non-linearity, LN has taken a backseat to compete integrated photonic platforms, such as silicon, which has no second-order nonlinearity.This was due to difficulties in fabrication of a low-loss LN waveguides (WGs), integration, as well as processing on a wafer-scale.

With commercial TFLN wafers now being accessible, in parallel with the rapid advancement of scalable micro-/nano fabrication techniques, TFLN photonic devices are steadily emerging. By offering tighter conifinement compared to ion-exchanged WGs, TFLN WGs boost the performance of devices such as EOMs to have bandwidth with smaller power consumption.

Etching LN is however challenging, hence, making it difficult to fabricate low-loss WGs. In particular poor etch induces substantial roughness and non-vertical side-wall angles contributing to high propagation losses in the WGs. Low-loss LN WGs have been demonstrated over the past several years. Most of these WGs were fabricated using argon gas inductively coupled reactive ion plasma (ICP-RIE) etching. Etch recipe optimization is of utmost importance to reduce optical losses. Initial demonstrations have shown that TFLN devices can match or exceed the performance of traditional silicon or indium phosphide devices. However, low-loss TFLN waveguides are not widely available.

As a part of the effort to develop low-loss TFLN devices, we perform a systematic study of fabrication high-quality LN WGs. The main goal of this investigation is to reduce the WG’s surface roughness while keeping an optimum side-wall angle profile that minimizes light propagation loss. In particular, our efforts are focused on three criteria: (1) selecting appropriate mask materials to reduce the transfer of mask defects into LN WGs, (2) establishing the optimal plasma chemistry by detailed study of various ICP-RIE etch parameters, and, (3) determining the optimum chemical cleaning protocol to remove the redeposited during ICP-RIE etch material on the WG’s side-wall. In this presentation, we discuss the details of all three fabrication aspects to make high-quality TFLN devices and structures.

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