MEMS and NEMS Group
Room A210 - Session MN-TuM

MEMS, BioMEMS, and MEMS for Energy: Processes, Materials, and Devices II

Moderators: Robert Davis, Brigham Young University, Zenghui Wang, Case Western Reserve University

8:00am **MN-TuM1 Near-Zero Power Integrated Microsystems for the IoT**, *M. Rinaldi,* Northeastern University*; **Zhenyun Qian,*** Unaffiliated **INVITED**

The recent advancements in terms of sensor miniaturization, low power consumption and low cost allow envisioning a new era for sensing in which the data collected from multiple individual smart sensor systems are combined to get information about the environment that is more accurate and reliable than the individual sensor data. By leveraging such sensor fusion, it will be possible to acquire complete and accurate information about the context in which human beings live, which has huge potential for the development of the Internet of Things (IoT). To address the growing demand of such large wireless sensor networks, there is a need for wireless sensors with dimensions and power consumption that are orders of magnitude smaller than the state-of-the-art. Energy is the key challenge. Batteries have limited capacity, and existing sensors are not "smart" enough to identify targets of interest. Therefore, they consume power continuously to monitor the environment even when there is no relevant data to be detected. This talk presents a new class of zero-power microsystems that fundamentally brake this paradigm, remaining dormant, with zero-power consumption, until awakened by a specific physical signature associated with an event of interest. In particular, a zero-power infrared (IR) digitizing sensor microsystem consisting of plasmonically-enhanced micromechanical photoswitches is presented. Such a passive IR digitizer is capable of producing a wake-up bit when exposed to a specific IR spectral signature associated to a target of interest (such as the exhaust plume of a car, a forest fire, or a human body) while rejecting background interference. The capability of these zero-power sensors of consuming power only when useful information is present results in a nearly unlimited duration of operation, with a groundbreaking impact on the proliferation of the IoT.

8:40am **MN-TuM3 Development of Inorganic Metal Salt Inks for Printable Sensor Applications**, *Y. Sui,* Case Western Reserve University*; A. Hess-Dunning,* Louis Stokes Cleveland VA Medical Center*; R.M. Sankaran, **Christian Zorman,*** Case Western Reserve University

The rapid advancement of flexible and stretchable electronics has stimulated the development of printing approaches as a means to fabricate metallic interconnects, antenna and other essential conducting structures. Unfortunately, metal inks have been limited to silver, copper, and gold, due to the complexity of nanoparticle synthesis and metallo-organic compound design. While sufficient for interconnects, the development of printed sensors is significantly limited by the small selection of printable metals. Recently, we reported a new class of inks that are based on inorganic metal salts that are converted to metallic structures by exposure to a low-temperature inert gas plasma. This approach to ink design opens up a much wider range of printable metals than currently available from conventional inks. We found that chemical, biological and mechanical sensors fabricated using this printing approach significantly outperformed the same sensors fabricated using conventional approaches, presumably due to the surface morphology of the printed sensors.

In this paper, we describe a method of controlling the surface morphology for metal structures fabricated using plasma activation of inorganic metal salt-based inks. We have found that the ink solvent plays a key role in the nucleation and crystal growth of metal nanostructures during the plasma reduction process. Using solvents of different vapor pressures, we were able to control the duration of plasma-induced liquid-phase nucleation and crystallization, thereby tuning the surface morphology, conversion depth, and resistivity of the printed metal structures. Silver nitrate-based ink was used for this study and the ink solvents in order of decreasing vapor pressure were ethylene glycol (EG), di-ethylene glycol (di-EG), and tri-ethylene glycol (tri-EG). The structural, morphological, and electrical properties of metals printed with different ink solvents were characterized by cross-sectional scanning electron microscopy (SEM), optical profilometry, and sheet resistance measurements, respectively. To show that the tunable morphology can be used to enhance the sensitivity of printed sensors, we fabricated and tested a silver-based hydrogen peroxide sensor using inks made from the three solvents.

9:00am **MN-TuM4 Void-Free Copper Electrodeposition in Full Wafer Thickness Through-Silicon Vias with 10:1 Aspect Ratios**, ***Rebecca Schmitt**, L. Menk, C. Sadler, E. Baca, A.E. Hollowell,* Sandia National Laboratories

Copper-filled through-silicon vias (TSVs) are incorporated in microelectronic devices as a 3D integration technique to increase I/O per unit volume. Industry has incentivized thinning wafers to increase TSV density, but certain MEMS applications require full thickness substrates, thus creating a demand for mesoscale TSVs. Using a full thickness silicon wafer helps preserve wafer flatness during multi-layer device fabrication and conserves mass, often required in MEMS applications. Traditionally, a three-additive Cu deposition chemistry is used for TSV filling; however, in this work, a single-additive chemistry has been established to achieve bottom-up superfilling in high-aspect ratio features. This electroplating chemistry involves a mixture of $CuSO_4$, CH_4O_3S or H_2SO_4, chloride, and a poloxamine suppressor additive. Cyclic voltammetry (CV) can be used to characterize the electrolyte and identify a hysteretic region, which is caused by suppressor breakdown at the cathode surface. This hysteresis corresponds to an operating window where void-free Cu filling of high-aspect ratio features can be achieved.

Previously, potentiostatic and galvanostatic deposition conditions for void-free filling were developed for nominally 100 µm diameter and 600 µm deep vias. Copper electrodeposition in TSVs with a 10:1 aspect ratio is currently under investigation. These TSVs have a 62.5 µm diameter etched into a 625 µm thick silicon-on-insulator (SOI) wafer. However, the conditions that resulted in void-free, bottom-up filling in 100 µm diameter TSVs have not translated to fill 62.5 µm geometries. In this work, electrolyte constituent concentrations, applied potential, and applied current were varied to analyze their effect on fill profile in 62.5 µm TSVs. Fill profiles were analyzed through cross sectioning and optical microscopy, as well as through X-ray CT scans. This work details the experimental approach associated with determining electrodeposition conditions for 62.5 µm diameter TSVs and presents the resulting fill profiles of copper in these vias.

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9:20am **MN-TuM5 Ion-Conducting Materials and Devices for Cold Atom Microsystems**, ***Christopher Roper**,* HRL Laboratories, LLC*; S. Kang,* NIST*; R.P. Mott, A.V. Mis,* HRL Laboratories, LLC*; E.A. Donley, J. Kitching,* NIST

Atomic instruments using laser-cooled atoms in ultra-high vacuum enable highly precise measurements of time, acceleration, and rotation [1]. Use of such devices outside the laboratory requires control of the atomic vapor density to prevent warm atoms from prematurely disturbing the cold atoms prior to measurement. Portable, miniature cold atom devices require a low-power, scalable method for controlling atomic vapor density.

Recently, solid-state electrochemical devices based on the solid electrolyte beta"-alumina have been used to change Rb vapor density on the scale of first 100s [2] and then 10s [3] of seconds. Reduction in vapor density up to 7X has been reported. Furthermore, these devices have been used to stabilize Rb vapor density using a feedback loop [4].

We present a solid-state electrochemical device consisting of a fine Pt grid top-electrode with submicron lithographically patterned features, beta"-alumina solid electrolyte, patterned Pt bottom electrode, and graphite reservoir. For relatively slow actuation cycle frequencies (17 mHz), this device exhibits a 100X increase in Rb vapor density with -30 V sourcing voltage and 20X decrease in the Rb vapor density with +30V sinking voltage. Rb vapor density can be modulated at up to 50 Hz, although at lower Rb vapor density dynamic range. An inverse relationship is found between Rb vapor density dynamic range and actuation cycle frequency. The high vapor density dynamic range and fast cycling rates demonstrated with this device are attributed to the fine, submicron top electrode features compared to coarse top electrode features (>100 µm) used in prior works. These devices are expected to be key components in future cold atom microsystems.

[1] J. Kitching, et al., IEEE Sensors J 11 (9), 1749 (2011).

[2] J. Bernstein, et al., Hilton Head 2016, pp. 180-184.

[3] S. Kang et al., Applied Physics Letters 110, 244101 (2017).

[4] S. Kang et al., Optics Express 26 (3) pp. 3696-3701 (2018).

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9:40am **MN-TuM6 Determining the Material Properties of Carbon Nanotube Structures Through Cantilever Resonances**, ***Richard Cass,*** Brigham Young University*; E. Eion Hindsman-Curry,* University of Alabama*; R. Vanfleet, R.C. Davis, D.D. Allred, B. Anderson, R.R. Vanfleet,* Brigham Young University

Nanotube Templated Microfabrication (CNT-M) processes use nanotube forest growth from a 2-D pattern to form 3-D structures. The resulting structure is then infiltrated with a second material to form the final device. Materials properties of these structures with different infiltration materials and varying degrees of infiltration is of interest.

We have used force-displacement data (in fixed-free and 3-point bending configurations) to determine the Ultimate Strength, the Young's Modulus, and the Maximum Strain of CNT-M structures using various infiltration materials. However, in the case of tungsten infiltration processes and typical test beams (~250 um in width), the infiltration was not sufficiently uniform for high confidence results. Smaller beams (< 50 um width) are difficult to handle using the conventional 3-point bending processes. We report resonance frequency testing, using a Laser Doppler Vibrometer (LDV), of thin CNT-M cantilevers to find the Young's Modulus of these CNT structures.

11:00am **MN-TuM10 Nanoporous Titanium Nitride Electrodes for Biosensing**, ***Mark Ming-Cheng Cheng,*** *G. Chen,* Wayne State University

We report corrosion-resistant and high-capacity implantable nanoporous titanium nitride (TiN) electrodes for neural probes applications Traditionally, the TiN electrodes are prepared using reactive sputtering techniques and have limited surface areas. To research smaller electrodes to minimize tissue damage, high aspect ratio TiN nanotube structures were fabricated using electrochemical anodization of Ti wires followed by high-temperature nitration. The specific charge capacity of nanoporous TiN correlates proportional with the surface area and pore size.

According to Shannon criteria, an empirical rule in neural engineering for possibility of tissue damage from electrical stimulation, the recommended limit density of a stimulation pulse is 30μCcm^{-2}for a geometric surface area of 0.06cm^2. Nevertheless, the charge injection capacity of chronically implanted electrodes has shown degraded over the time (within one month to a year), including sputtered iridium oxide (SIROF), porous platinum and tungsten. One of the challenges for these implantable electrodes involves irreversible reduction and oxidation reactions occurring at the electrode surface through faradic or pseudocapacitive charge transfer. On the other hand, TiN has different mechanism of charge injection (through capacitive double layers). TiN has been shown promising electrode material in neural implants thanks to its super electrical conductivity, biocompatibility and chemical stability. TiN is also known for physiologically inert and corrosion resistant. To increase the spatial resolution of neural stimulation, small electrodes with high surface areas are more desirable. To the best of our knowledge, TiN nanotubes electrodes have not been studied in the literature for neural implants. Compared to tungsten electrode, the impedance and morphology of nanoporous TiN was found stable over a long-term in stress tests (at an elevated temperatures in phosphorous buffered solution).

11:20am **MN-TuM11 Toward a Simple Process for Fabricating Multi-channel Neural Probes on Optical Fiber Substrates**, ***Md Ashiqur Khan,*** *M. Gheewala, V.S. Jonnalagadda, T.A. Tisa, M. Rao, A. Awale, P. Motwani, N.S. Randhawa, H. Sajedi, W.-C. Shih, J.C. Wolfe,* University of Houston*; J.A. Dani,* University of Pennsylvania*; P. Mauger,* No Matching Affiliation

Electrical probes are used to stimulate spiking activity within a target population of neurons and monitor how these electrical signals propagate through the brain. This paper describes a simple fabrication process for multi-electrode neural probes on optical fiber substrates. It relies on neutral particle proximity lithography to achieve the required depth-of-field and freedom from charging artifacts but circumvents the complexity of membrane masks (complementary exposures, radiation resistant coatings, and fragility) and on-fiber alignment.

Fig. 1 of the supplementary document shows, conceptually, a probe with 4-channel thin-film sensor, a tetrode, on each of four sides of a fiber. It requires two masks; the first for the interconnect traces. The other for vias in the dielectric overcoat where the metal lines contact the brain.

As shown in Fig. 2, optical fibers are held in V-grooves etched into the top surface of a (100) Si wafer. A second set of V-grooves, etched from the opposite side of the wafer, forms open windows at the bottom of the upper grooves. When this *mask* is illuminated by 50 keV He atoms, transmitted beamlets transfer the stencil pattern to resist on the fibers. A negative--tone, plasma-deposited, resist is used to mask the gold interconnects. The vias are similar, but require a tone-reversal step. Rotational alignment of the 2 masks uses a high precision cubic bead glued to the end of the fiber to reference the rotational angle of the fibers to a precision-ground aluminum platform on the jig. Longitudinal alignment is achieved using a fiber-stop. These are high precision (Grade 5) 440C stainless steel ball bearings which are held in an anisotropically etched pocket at the tip-end of a V-groove by a rare earth magnet. Longitudinal and transverse positional errors of 1.0$\pm$0.6 mm and 0.3$\pm$.15 mm, respectively. A single-interconnect mask can be printed multiple times to build the probe of Fig.1. The offset is produced by tilting the mask relative to the beam. Fig. 3 shows two lines printed on a 300 mm fiber with 21.5 and 29.5 mm offsets in the longitudinal and transverse directions, respectively.

Fig. 4 is an in-vitro recording from a brain slice (mouse) after a battery of bench tests, including a) a 3-week soak phosphate buffered, b) repeated insertion in agar and a stainless steel cannula, d) disinfection in MetriCide-2.6% glutaraldehyde, and a 6 hour implantation in mouse brain). Impedance spectra were the same within the measurement error of the impedance bridge before and after these bench tests.

At this time the tone reversal process has not been fully optimized.

11:40am **MN-TuM12 A Low-Temperature Packaging Process for Mechanically-Adaptive Neural Interfaces for Microfluidic-Aided Drug Delivery**, *E. Szabo, L. Greenwood,* Case Western Reserve University*;* ***Allison Hess-Dunning,*** Louis Stokes Cleveland VA Medical Center

Advances in polymer-based materials development have led to an array of environmentally-responsive materials that are uniquely suited for biomedical implant applications. Compatible microfabrication processes continue to be developed to integrate these responsive materials into biomedical microdevices. Progressing beyond proof-of-concept devices into functional implants for long-term use requires additional development of compatible and reliable packaging strategies to facilitate interfacing the microdevices with peripheral components. Our group has previously developed a mechanically-adaptive, polymer nanocomposite-based (NC) intracortical implant with a microfluidic channel for diffusion-based drug delivery. Interfacing the device with fluid pumps requires a means for secure attachment of polyethylene tubing to the inlet and outlet ports of the neural microdevice that will remain stable under physiological conditions. Packaging process considerations include incompatibilities of NC with organic solvents and temperatures exceeding 80°C. Additionally, out-of-plane forces on the devices must be minimized in order to maintain the integrity of the microfluidic channels.

We designed connectors to interface polyethylene tubing with the NC-based neural microdevices, to be built using a multi-jet 3D printer. Components produced using multi-jet printers typically have a plastic structural material and a wax-based support material that is removed during post-processing operations. However, our approach involves using a controlled, thin layer of the wax support material as a functional adhesive between the plastic connector and the NC device. With a melting point of 60°C, the wax is stable at body temperature (37°C) and can be melted and reformed at temperatures below the processing limit for the device. Because the wax is printed as part of the connector, the tubing ports can be aligned to the microfluidic inlet and outlet in a single step. Through this approach, we established a leak-free connector design and packaging process to facilitate a fluidic connection between syringe or osmotic pumps and NC-based microdevices. The adhesive bonding strength provided by the wax to the NC exceeded 37 MPa, much higher than the 0.75 MPa required to pump fluid through the connector and microfluidic channel. Packaged devices remained functional in phosphate buffered saline heated to 37°C, even after soaking for 24 hours. The potential for scaling the packaging process and for applying to other materials will also be discussed.

12:00pm **MN-TuM13 Vascular Graft Pressure-Flow Monitoring Using Nanocomposite Carbon Black/PDMS Based Strain Sensors**, ***Hao Chong,*** Case Western Reserve University*; S.J.A. Majerus,* Louis Stokes Cleveland VA Medical Center*; J. Liu, C.A. Zorman,* Case Western Reserve University

A vascular graft is commonly used to bypass damaged blood vessels or to form an arteriovenous shunt for vascular access (e.g. for hemodialysis). Real-time monitoring of blood flow in synthetic grafts would provide early warning of graft failure to permit interventions such as angioplasty or graft replacement to avoid catastrophic failure. Based on biocompatible materials, we have developed a new type of flexible pulsation sensor (FPS) which is wrapped around a graft to monitor blood pressure and flow. The FPS uses Carbon Black (CB) dispersed in polydimethylsiloxane (PDMS) as a piezoresistive sensor layer, which is stencil printed on a structural PDMS layer. In this study, we analyze the strain transducer mechanisms on a vascular graft and show the linear and stable strain response of CB-PDMS composites from 0-50% strain. The material has a broader strain range than graft materials and a gauge factor of 5. In vitro testing of the FPS on a vascular graft phantom showed a robust, linear sensor output to pulsatile flows and pressures. The composite material shows excellent potential in biologic strain sensing applications where a flexible sensor with large maximum strain range is needed.

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