

## MEMS and NEMS Group

### Room On Demand - Session MN-Contributed On Demand

#### MEMS and NEMS Contributed On Demand Session

**MN-Contributed On Demand-1 Observation of Tunable Opto-Mechanical Responsivity in Two-Dimensional Semiconducting Nanoelectromechanical Resonators, Jiankai Zhu,** University of Electronic Science and Technology of China; *P. Zhang,* Shanghai Jiao Tong University, China; *J. Li, B. Xu, S. Wu, F. Xiao, Y. Liang, T. Wen, F. Wang,* University of Electronic Science and Technology of China; *R. Yang,* Shanghai Jiao Tong University, China; *Z. Wang,* University of Electronic Science and Technology of China

#### Introduction

Nanoelectromechanical systems (NEMS) based on two-dimensional (2D) materials typically have motional parts that are atomically-thin, with displacement in the picometer range or even smaller<sup>[1]</sup>. When measuring the nanomechanical device motion, a key challenge is to optimize the signal transduction efficiency (responsivity) in order to best detect the infinitesimal motion<sup>[2][3]</sup>. Here we demonstrate tuning of the responsivity in optically-transduced atomically-thin NEMS resonators, by controlling the device deflection and thus profiles, and demonstrate the importance of optimizing the measurement condition.

#### Methods

In this work, we study the tuning of opto-mechanical responsivity with laser position and gate voltage, by using a custom-built 2D NEMS resonator measurement system, including optical components for laser interferometry, a vacuum chamber to keep the device in  $\sim 1 \times 10^{-6}$  Torr environment, an x-y stage with precise position control, and electrical connections for gate tuning and driving (Fig. 1a). The MoS<sub>2</sub> NEMS resonator (Fig. 1b & 1c) is fabricated using a dry transfer method<sup>[4]</sup>, and actuated capacitively through its gate electrode. As the laser is incident on the device, the reflected light intensity (incident on the photodetector) varies with the position of the MoS<sub>2</sub> flake, resulting from optical interferometry (Fig. 2a).

#### Results

When DC gate voltage ( $V_g$ ) is applied, the electrostatic force pulls down the MoS<sub>2</sub>, which changes the interferometry condition and tunes the reflected light intensity. For the same MoS<sub>2</sub> resonator under  $V_g = 0.5$  V and 7 V, the resonance signal amplitudes are clearly different, indicating a change in responsivity (Fig. 2). When the gate voltage varies from -10 V to 10 V, we observe the striking feature of 0 responsivity: at certain gate voltage ( $\sim \pm 6$  V), the resonant signal becomes unmeasurable, as the responsivity crosses 0 point (Fig. 3). We confirm this is due to the change in interferometric condition and thus responsivity, by measuring the same device, under the exact same excitation amplitude and gate voltages, but at different laser spot positions. Fig. 4 clearly shows that as the laser spot moves, the interferometric condition changes due to different vacuum gap size, and the zero responsivity condition is met at different gate voltages (orange arrows). We further estimate the profile of the MoS<sub>2</sub> NEMS resonator using finite element simulation (Fig. 5). These results clearly demonstrate the importance of laser spot position and gate voltage on the responsivity of 2D NEMS resonators, and provide important guidelines for optimizing the transduction efficiency of resonances.

#### Reference

- [1] *Sci. Rep.*, 4, p. 3919, 2014.
- [2] *Nat. Commun.*, 5, p. 5158, 2014.
- [3] *Sci. Adv.*, 4, p. eaao6653, 2018.
- [4] *Nano Lett.*, 16, p. 5394, 2016.

**MN-Contributed On Demand-4 Improving Signal-to-Noise Ratio and Noise Matching in AlN NEMS Resonators Using Parametric Amplification, Tahmid Kaisar,** *J. Lee, P. X.-L. Feng,* University of Florida

Parametric amplification has been widely employed in various fields such as in optics, nano-mechanics, and electronic circuits to increase signal-to-noise ratio (SNR) of low-level signals through modulating system parameters. Due to very small mechanical motions of resonant nano/microelectromechanical systems (NEMS/MEMS), it has been challenging to have an efficient displacement signal transduction method, and displacement signals are often superposed on the much larger electrical background and noise from readout electronics. Using parametric

amplification, it is possible to amplify the signal directly in the mechanical domain first, before electrical transduction, allowing us to alleviate excess amplifier noise. Parametric effects have been achieved in NEMS/MEMS by changing certain device parameter at twice the resonance frequency [1]. Besides parametric gain, it can enhance quality ( $Q$ ) factors of the devices [2], improve sensitivity of atomic force microscopy (AFM), enhance SNR in gyroscopes, etc.

In this work, we demonstrate parametric amplification of the thermomechanical noise in an optically and piezoelectrically transduced AlN NEMS resonator, which has a two-dimensional (2D) square diaphragm with a side length of 26  $\mu$ m and thickness of 140 nm. The device shows a resonance frequency of  $\sim 7$  MHz and a  $Q$  factor of 8800 in the linear regime. Duffing nonlinearity and dynamic range (DR) of the device are characterized. Next, we introduce  $2\omega$  parametric pumping voltage ( $v_p$ ) to the device and measure displacement of thermomechanical noise in both optical and electrical domains, simultaneously. We find that the thermomechanical noise is gradually amplified as  $v_p$  increases from 1 mV to 30 mV, and it goes into self-oscillation regime when  $v_p \geq 25.3$  mV (*i.e.*, parametric threshold) in the optical domain. While in the electrical domain, noise from the measurement system is much higher than the piezoelectrically transduced thermomechanical noise. Once the device is parametrically pumped, electrically transduced thermomechanical noise is greatly amplified and gradually appears above the electronic noise level near the parametric pumping threshold. Above this level, we have achieved noise matching. Based on the comparison between the parametric pumping in the optical and electrical domain, we find that although the piezoelectrically transduced thermomechanical noise is  $\sim 46$  dB below the noise level of the typical electronic measurement system, the parametric pumping can greatly amplify the signal to emerge above the electronic noise level, allowing us to electrically detect the thermomechanical resonance and enhance SNR, suggesting the possibilities of improving performance of NEMS/MEMS devices for building sensors and oscillators.

[1] R. B. Karabalin, *et al.*, *Nano Lett.* 9, 3116-3123 (2009).

[2] P. Prasad, *et al.*, *Nanoscale* 9, 18299-18304 (2017).

**MN-Contributed On Demand-7 MoTe<sub>2</sub> NEMS Resonator for Near-Infrared Light Detection, S M Enamul Hoque Yousuf, X. Zheng, P. Feng,** University of Florida, Gainesville

In recent years, molybdenum ditelluride (MoTe<sub>2</sub>) has received significant attention [1] due to its unique semiconducting and phase transition properties [2]. Semiconducting 2H-MoTe<sub>2</sub> is known to have thickness-dependent bandgap from 0.83 eV (bulk) to 1.1 eV (monolayer) where bandgap also changes from indirect to direct. Ultrasensitive near-infrared (NIR) light detection is desirable in NIR imaging, surveillance, communication, and security applications. High-end commercial NIR detectors require cryogenic cooling to reduce the thermal noise, which limits their applications in broader fields. As an alternative solution, transducers enabled by nanomechanical resonators have been proposed [3]. The bandgap of monolayer 2H-MoTe<sub>2</sub>, which matches that of silicon (1.1 eV), is narrower than the bandgaps of other transition metal dichalcogenides and makes it a suitable candidate for visible and NIR light detection.

In this work, we fabricate MoTe<sub>2</sub> drumhead resonators on sapphire substrate with local electrostatic gates. After forming Au/Ti top electrodes, we transfer a MoTe<sub>2</sub> flake with desired shape and thickness on to the predefined sapphire substrate using all-dry transfer method through a custom-built transfer stage. Since the tellurides are more prone to ambient degradation than selenides or sulfides, the MoTe<sub>2</sub> flake is encapsulated by hexagonal boron nitride (h-BN) thin layer and is stored in vacuum all the time. To confirm the identity of the constituent crystal of the resonator, we use Raman spectroscopy with a laser at  $\lambda = 532$  nm. The measured data clearly shows characteristics of Raman modes confirming the high quality of the suspended 2D MoTe<sub>2</sub> crystal. We then measure the transistor properties of the fabricated device. The transfer curve shows a p-type behavior with  $I_{on}/I_{off} > 10^4$ ; and the transport curve shows an ohmic contact between the MoTe<sub>2</sub> and the Au electrodes. We measure the resonance of the device by using an optical interferometry system [4] and obtain a fundamental-mode resonance at 12.24 MHz. We further measure the NIR response of the drumhead resonator by illuminating the device using a 785 nm laser. This study provides an initial exploration in building MoTe<sub>2</sub> NIR detectors.

#### References:

- [1] X. Liu, *et al.*, *ACS Nano* 14, 1457 (2020).
- [2] A. Roy, *et al.*, *ACS Appl. Mater. Interfaces* 8, 7396 (2016).

# On Demand available October 25-November 30, 2021

- [3] A. Islam, *et al.*, *Proceedings of IEEE MEMS* 2020, 826 (2020).  
[4] X. Liu, *et al.*, *Transducers* 2019, 2408 (2019).

**MN-Contributed On Demand-10 Interposer Fabrication with Heterogeneous Integration of Multi-Project Wafer Die for Mid-Volume RF Microsystems, Mieko Hirabayashi, S. Lepkowski, S. Herrera, C. Nordquist, C. Gibson, A. Ruyack, J. McDow, A. Hollowell, M. Jordan, Sandia National Labs**

We demonstrate a radio frequency (RF) interposer with built in passive components and the ability to accept 2.5D heterogeneously integrated die. 2.5D heterogeneous microsystems allow for the use of modular, commercial off the shelf or multi-project wafer components and/or disparate technologies like Si and GaN based semiconductors. The RF interposer is formed using glass wafers to prevent substrate coupling. The passive components are produced utilizing patterned copper electroplating followed by a spin-on interlayer dielectric.

High density flip-chip interconnects can be cost prohibitive during prototyping and for low volume applications. For example, if a multi-project wafer component is used, the die are delivered singulated and often with Al as the top metal, which by itself is not compatible with flip-chip interconnects. This then leads to labor intensive patterning and under bump metallization processes at the die level. We demonstrate a lithography-free, batch level under bump metallization process utilizing electroless plating to form ENEPIG terminated connections. We are able to show that the process is compatible with gold pillar and SnAg capped Cu pillar interconnects.

Gold interconnects are of interest for this application because of their high ductility and compatibility with III-nitride semiconductors. However, the mechanisms of gold-gold bonding are not well studied in microelectronics. Gold-to gold bonding and gold-to-sputtered gold nickel studs have been demonstrated, however the lack of a suitable model for gold-to-ENEPIG bonding led us to investigate the process more closely. This led us to interesting conclusions regarding the relationship between the bonding force, aspect ratio, and overall deformation of the gold pillars required to achieve acceptable bonds.

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**MN-Contributed On Demand-13 Scaling Copper Deposition in Through-silicon Vias from Die Level to Wafer Level Plating, Jessica McDow, R. Schmitt, M. Hirabayashi, E. Baca, J. McClain, L. Menk, A. Hollowell, M. Jordan, Sandia National Laboratories**

Copper-filled through-silicon vias (TSVs) are a key technology for 3D integration of microelectronic devices. 3D integration enables system miniaturization, increases bandwidth per volume, and improves device performance. Full wafer thickness vias are utilized for microelectromechanical systems (MEMS) devices where additional mass of the full wafer is advantageous. For high-power devices, large scale vias or via arrays can be used both to supply power and for thermal management. Utilizing the s-shaped negative differential resistance (S-NDR) mechanism<sup>2</sup>, Cu deposition for full wafer thickness TSV geometries in a suppressor-based electrolyte consisting of copper sulfate, sulfuric acid, potassium chloride, and Tetric 701 suppressor has been achieved.<sup>1</sup> Typically, void-free bottom-up filling with these additive-based electrolytes is performed through potentiostatic deposition with a reference electrode. However, to transition this process to a production scale wafer plating tool, a current-controlled plating regime is required because these tools are not equipped with reference electrodes. Voltage-controlled electrochemical deposition (ECD) parameters have been developed through the S-NDR mechanism for bottom up filling of vias, where potential stepping was performed from -500 mV (MSE) to -560 mV (MSE) in -10 mV increments, and each potential was held for 2 to 5 hours to ensure a void-free fill.<sup>1</sup> This process was used in die level scaling experiments to derive a current-controlled ECD procedure and understand the relationship between applied current density, total conductive surface area, and active via area.

Various applications for TSVs and downstream processes can occur at elevated temperatures. Understanding deformation and stress of Cu-filled TSVs that occurs during thermal cycling is important to the TSV reliability. Experiments were performed to characterize the protrusion of 30 um diameter - 100 um depth Cu-filled TSVs, where an optimized annealing profile was proposed to suppress grain growth during thermal cycling.<sup>3</sup>

On Demand available October 25-November 30, 2021

Annealing studies are currently being conducted on full wafer thickness Cu-filled TSVs to determine an optimal temperature cycling profile for the protrusion behavior of mesoscale TSVs. This work presents the results of a current-controlled wafer level Cu deposition process and thermal characterization of high aspect ratio mesoscale TSVs.

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<sup>1</sup>Rebecca P. Schmitt et al 2020 J. Electrochem. Soc. 167 162517.

<sup>2</sup>D. Josell and T. P. Moffat 2018 J. Electrochem. Soc. 165 D23.

<sup>3</sup>Si Chen et al 2016 Microelectronics Reliability 63 183-193.

**MN-Contributed On Demand-16 Device Fabrication Process for Controlled RF Plasma Breakdown, Sergio Herrera, A. Ruyack, M. Jordan, C. Moore, G. Hummel, M. Ballance, A. Bingham, A. Schiess, C. Gibson, C. Nordquist, Sandia National Labs**

Paschen's curve expresses the required voltage to create electric arcing between two electrodes (breakdown voltage) as a function of gas pressure, temperature, and the gap between them. At atmospheric pressure the Paschen relation fails at small (<5µm) gap distances due to small numbers of gas molecules between the electrodes (pseudo-vacuum condition) and the advent of electron tunneling phenomena which is enhanced by ions near the cathode. Extensive research has been conducted to characterize voltage breakdown behavior of large gaps, however little has been done to understand the high frequency breakdown of narrow gaps. In the world of microsystems, the gap size is approximately equal to the ionization mean free path of electrons (at atmospheric pressure), allowing for field emission, ion-enhanced field emission, and other mechanisms to exercise greater dominance.

Here we present on the breakdown characteristics of wafer fabricated vertical and horizontal micron-scale radiofrequency (RF) plasma discharge devices. Device breakdown gaps vary from 100 nm to several microns. Precision electroplating of high-stiffness gold combined with wet release of gold microbridges is used to produce discharge gaps for vertical devices. Dual sacrificial layers (allowing for more controlled spacing) and dry MEMS release techniques are incorporated to locally maximize the electric field. At these low discharge gaps, we expect to promote deviation from classical Paschen curve behavior with the potential for sub 10 V breakdown. Improved understanding of these relations will enable approaches for both mitigating high-frequency breakdown and the prospect for new classes of devices.

This work is supported by the Laboratory Directed Research and Development program at Sandia National Laboratories, a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

**MN-Contributed On Demand-19 Resonant Motion and Frequency Tuning in Nano-Electromechanical Devices Based on Two-Dimensional Semiconductor WSe<sub>2</sub>, Yachun Liang, J. Zhu, F. Xiao, S. Wu, C. Jiao, Z. Wang, University of Electronic Science and Technology of China**

## Introduction

Two-dimensional (2D) materials, like graphene and transition metal-dichalcogenides (TMDCs), have been extensively explored because of their intriguing physical properties. Nanoelectromechanical system (NEMS) resonators based on 2D materials hold great promises for transducers and signal processing devices<sup>[1,2]</sup>. Atomically-thin WSe<sub>2</sub> has demonstrated good potential towards high quality-factor NEMS resonators<sup>[3]</sup>. Here, we demonstrate WSe<sub>2</sub> resonators with tunable frequency at room temperature.

## Methods

In this work, WSe<sub>2</sub> resonators are fabricated using an all-dry transfer method with pre-patterned substrate, and the fabrication progress is shown in Fig. 1<sup>[4]</sup>. We investigate the frequency tuning in WSe<sub>2</sub> resonators, by using a custom-built 2D NEMS resonator measurement system, including laser interferometry readout, optical excitation (Fig. 2); electrical driving (Fig. 3); and the capability of measuring thermomechanical motion without external driving (Fig. 4).

## Results

We first explore the optical driving and optical readout measurement, where we connect the output of the Network Analyzer to the input of a 405 nm modulated laser (driving), which is aligned to share the same optical path with the 633 nm detection laser (Fig. 2a). Specifically, the output the RF signal controls the 405 nm laser power, which periodically drives the WSe<sub>2</sub> resonator through optothermal effect. The device shows a clear resonance frequency in MHz range (Fig. 2b), and the resonance frequency increase with the magnitude of the DC gate voltage  $V_g^{DC}$  (Fig. 2c).

We further investigate the gate tuning using electrical driving (Fig. 3a). The output of RF signal periodically drives the WSe<sub>2</sub> membrane, and resonance response detected by 633 nm laser can be observed when  $V_g^{DC}$  is 2 V (Fig. 3b). In this case, clear frequency tuning is also observed (Fig. 3c). We measure the thermomechanical resonant response with 633 nm laser detection as is shown in Fig. 4a. It shows a pronounced resonance peak in the noise spectrum, consistent with the optical and electrical driving schemes. The measured thermomechanical frequency can be tuned by  $V_g^{DC}$ .

## Summary

We have explored frequency tuning in WSe<sub>2</sub> NEMS resonators for optically driven resonances, electrically driven resonances, and completely undriven thermomechanical resonances, all under room temperature. Our results suggest that WSe<sub>2</sub> NEMS resonators can be explored for future frequency-tunable NEMS devices operating at room temperature.

## Reference

- [1] Science, 315, p. 490, 2007.
- [2] ACS Nano, 7, p. 6086, 2013.
- [3] Nano Lett., 16, p. 5102, 2016.
- [4] Nano Lett., 16, p. 5394, 2016.

## MN-Contributed On Demand-22 Observation of Temperature Coefficient of Frequency (TCf) Reversal in Bismuth Oxyiodide (BiOI) Vibrating Nanomechanical Resonators, Fei Xiao, S. Wu, J. Zhu, Y. Liang, C. Jiao, S. Pei, Z. Wang, University of Electronic Science and Technology of China

Atomically thin two-dimensional (2D) materials, like graphene and transition metal-dichalcogenides (TMDCs), have attracted tremendous attention because of their intriguing material properties. Nanomechanical resonators based on 2D materials have been investigated, demonstrating great potential towards applications such as transducers and signal processing devices<sup>[1,2]</sup>. Bismuth oxyiodide (BiOI) is an emerging three-element 2D layered material<sup>[3]</sup>. Here, we report experiment demonstration of the first BiOI resonators, and its unusual temperature coefficient of frequency (TCf).

## Methods

In this work, BiOI nanomechanical resonators are fabricated using a typical dry-transfer method<sup>[4]</sup>, with BiOI flake connected to the electrode patterned by evaporation. We characterize the resonant response using a scheme that incorporates electrical driving and optical detection (Fig. 1a). Vibrations in the BiOI resonator are electrostatically excited through the back gate and interferometrically detected with a 633 nm laser focused on the suspended flake. For all measurements, the resonator sample is mounted in a temperature-controlled sample stage under high vacuum ( $10^{-4}$  mbar).

## Results

We first characterize the thermomechanical resonance without external drive (Fig. 1b), and a clear resonance peak is observed in the noise spectrum, with the resonance frequency  $f_{res} = 3.86$  MHz. We further explore driven response in BiOI resonators using electrical excitations with  $V_g^{DC} = 1$  V, and investigate the TCf in these devices.

At first (294-302K), frequency  $f_{res}$  rapidly decreases as temperature increase. However, when the temperature is increased above 302 K,  $f_{res}$  rapidly increases with temperature (Fig. 2). By using the room temperature (294 K) data as reference (Fig. 3b), we calculate a TCf of -0.365%/K for the device below 302 K, and +0.412%/K above 302 K. Fig. 3a and 3c show the measured resonances of a BiOI resonator and optical images at 294 K and 324 K, respectively. We will present more details about the observed TCf inversion and its physical origin in the full presentation.

## Summary

We have studied TCf in BiOI nanomechanical resonators, for electrically driven resonances with temperature from 294 to 324 K. This device shows very high TCf values, and a TCf inversion is observed around 302 K. Our results suggest that BiOI nanomechanical resonators can be explored for temperature sensitive resonant sensors.

## Reference

- [1] Science, 315, p. 490, 2007.
- [2] ACS Nano, 7, p. 6086, 2013.
- [3] Appl. Catal. B-Environ., 262, p. 118262, 2020.
- [4] 2D Mater., 1, p. 011002, 2014.

## MN-Contributed On Demand-25 Gate-Switchable Bistable Nanomechanical Resonators Based on Two-dimensional Molybdenum Sulfide, Chenyin Jiao, B. Xu, J. Zhu, F. Xiao, Y. Liang, J. Chen, S. Pei, J. Xia, Z. Wang, University of Electronic Science and Technology of China

Nanomechanical resonators based on 2D materials have been widely investigated, demonstrating great potential in applications such as sensors and signal processing devices<sup>[1]</sup>. While typically the frequency can be continuously tuned by applying a gate voltage, it is challenging to realize a resonator that can switch between two distinct states, both of which can be maintained without any external voltage<sup>[2][3]</sup>.

Here we demonstrate a bistable nanomechanical resonator based on two-dimensional molybdenum sulfide (MoS<sub>2</sub>), which has two stable resonant states that can be switched using gate voltage. We use electrostatic force to laterally pull a nearby structure in order to switch the tension in the 2D resonator, and the frequency change can be maintained without any external voltage.

## Methods

The MoS<sub>2</sub> nanomechanical resonator is fabricated by mechanically exfoliating MoS<sub>2</sub> crystal onto Si/SiO<sub>2</sub> substrates with pre-patterned cavities and electrodes<sup>[4]</sup>. We measure the resonant response using a customized scheme that incorporates electrical driving and optical detection (Fig. 1). We also characterize the tension in both the suspended part and on-substrate part of the MoS<sub>2</sub> flake to monitor its tension before and after gate switching (Fig. 2).

Vibrations in the MoS<sub>2</sub> resonator is electrostatically excited through the back gate and interferometrically detected with a 633 nm laser focused on the suspended flake. For all measurements, the resonator sample is mounted in a sample stage under high vacuum ( $10^{-3}$  mbar).

## Results

We first measure the resonant response of the device with 0 gate voltage, and find its resonance frequency  $f_{res} = 6.33$  MHz. We then gradually increase the gate voltage from 0 to 29 V (Fig. 3a), and then back from 29 V to 0 V (Fig. 3b). It can be clearly seen that the evolution of resonant frequency with gate voltage does not follow the previous trajectory, and upon returning to 0 gate voltage the  $f_{res}$  has increased from 6.33 MHz to 7.65 MHz, clearly illustrating the two distinct resonant states.

We further show that the 0 gate voltage resonance frequency can be switched back and forth through gate voltage sweeps, and that the bistability is due to the lateral tensioning through interaction with a nearby larger membrane, which we will present with additional details at the symposium.

## Summary

We have demonstrated gate-switchable MoS<sub>2</sub> nanomechanical resonators with two distinct and stable resonant states, which can be controlled by gate-induced tension in the structure. Our results show that such devices hold promises for reconfigurable resonators that can be easily switched using voltage.

## Reference

- [1] Sci. Rep., 4, 3919, 2014.
- [2] Nat. Commun., 5, 5158, 2014.
- [3] Sci. Adv., 4, eaao6653, 2018.
- [4] Nano Lett., 16, 5394, 2016.

## MN-Contributed On Demand-28 Thermally Released Spring-Loaded Platform for Capsule Based Drug Delivery and Sensing, Joshua Levy, J. Stine, L. Beardslee, R. Ghodssi, University of Maryland, College Park

The human gastrointestinal tract (GIT) has emerged in recent years as a medium for probing integral biological phenomena within the body,

# On Demand available October 25-November 30, 2021

elucidating many underlying mechanisms of chronic diseases. To better understand these interactions, targeted treatment and monitoring within the GIT has been performed using endoscopic probes and capsule-based platforms. There is much research available demonstrating actuation mechanisms for targeted drug delivery and sensing applications utilizing an embedded balloon or spring to insert a microneedle type structure into the subepithelial space. These devices frequently rely on pH changes in the intestinal tract to trigger actuation, lacking control and limiting the system to passively triggered actuation. Therefore, the development of release mechanisms that can be triggered rapidly and on-demand would enable more precise and targeted therapeutic delivery and sensing.

Here we report the design and fabrication of a novel thermally released 3D-printed spring (Fig. S1) and microfabricated heater as a platform for drug delivery and sensing within epithelial tissue. The spring (H=8 mm and D=3 mm) was fabricated with a high resolution (30  $\mu\text{m}$ ) Digital Light Processing 3D printer using a 40:60 mixture of high- and low-modulus Monocure resin to achieve the desired spring flexibility and strength. To fix the spring in a compressed state a low-melting point (60 C) polymer, polycaprolactone, was applied to the base of the spring and bonded to a 4 mm pillar extending down from the spring tip. The 3 mm diameter resistive microheater was comprised of a Cr/Au (20nm/100 nm) coil patterned on a flexible polyimide substrate through a laser cut mask using electron beam evaporation. The heater design maintains a resistance of 50 Ohm, such that the current through the resistor is compliant with the maximum allowable current from a 3-V 2L76 coin cell battery (60 mA).

The mechanical properties of the spring were characterized using a mechanical tester, observing a spring constant of 13.6 mN/mm (Fig. S2). This force constant is sufficient to insert a microneedle-based drug delivery or sensing system into the epithelial gut layer, as we have previously demonstrated a microneedle tissue insertion force of 0.6 mN. To express the triggered release of the compressed spring, a current of 60 mA was applied to the microheater and successful release of the spring mechanism was achieved within 30 s. Further integration of the spring-heater platform into an ingestible capsule provides potential for targeted drug delivery and sensing within the GIT.

**MN-Contributed On Demand-31 Sidewall Nanochannel Fabrication Using Membrane Projection Lithography and Metal Assisted Chemical Etching, Tong Dang,** University of Pennsylvania; **R. Chaudhary,** ETH Zurich, Switzerland; **N. Xie,** University of Washington; **G. Kim, G. Watson,** University of Pennsylvania

Horizontally enclosed nanochannels have a wide variety of microfluidic applications ranging from biochemical sensing to electrochemical energy conversion and transport [1]. While top-down fabrication of horizontal nanochannels has been reported in materials such as silicon dioxide and polymers, less research has been focused on silicon based horizontal nanochannels. In this work, we report an approach consisting of membrane projection lithography (MPL) [2] and metal assisted chemical etching (MacEtch) [3] to fabricate nanochannels with tunable sizes and shapes that run parallel and beneath the surface of an Si substrate [4]. 2  $\mu\text{m}$  deep Si trenches are first patterned using a direct laser writer and etched using deep reactive ion etching (DRIE) to form smooth sidewalls. Au disks are then deposited onto the sidewalls using MPL, where Au is evaporated at a 45 degree angle through an electron beam lithography (EBL) patterned surface membrane. The Au disks then serve as a catalyst in the MacEtch process to form the nanochannels, in which the silicon directly underneath the Au disks is removed. The fabricated nanochannels are characterized using scanning electron microscopy (SEM), providing insights into effectiveness of the fabrication approach.

This work focuses specifically on the following aspects for enhanced reliability and reproducibility of the approach: improving the smoothness of the DRIE fabricated sidewalls; optimizing the planarization process preceding the patterning of the MPL layer; characterizing the lithography-created trenches and patterns to avoid any pattern size bias leading to misalignment and identifying the direction of the Au disk movement through the Si sidewall.

## References

- [1]Contento, Nicholas M., Sean P. Branagan, and Paul W. Bohn. "Electrolysis in nanochannels for in situ reagent generation in confined geometries." *Lab on a Chip* 11.21 (2011): 3634-3641.
- [2]Burckel, D. Bruce, et al. "Micrometer-scale cubic unit cell 3D metamaterial layers." *Advanced Materials* 22.44 (2010): 5053-5057.

- [3]Huang, Zhipeng, et al. "Metal-assisted chemical etching of silicon: a review." *Advanced materials* 23.2 (2011): 285-308.

- [4]Chaudhary, Rimjhim, et al. "Sidewall channel fabrication using membrane projection lithography and metal assisted chemical etching." *J. Vac. Sci. & Technol.B.* 37.6 (2019): 061813.

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**MN-Contributed On Demand-34 On-Demand Reconfigurable Transmission Grating for Neutron and X-ray Interferometry, Sarah M. Robinson,** National Institute of Standards and Technology (NIST)/University of Maryland, College Park; **R. Murphy, K. Weigandt, D. Hussey, N. Klimov,** National Institute of Standards and Technology (NIST)

We report on the development of a real-time reconfigurable transmission grating for neutron and x-ray interferometry. Such dynamic grating can be used as source of a quasi-coherence for phase imaging using incoherent sources of radiation such as neutron or x-ray beams. Our DynAmic ReconfigURable Source grating (DARIUS) is a silicon-based microfluidic device, which allows on-demand modulation of neutron and/or x-ray intensity in one dimension by adjustment of transmission grating period ranging from 20  $\mu\text{m}$  to 20,000  $\mu\text{m}$ . The main component of the DARIUS device is 51.2  $\times$  51.2 mm<sup>2</sup> transmission grating consisted of 2,560 microfluidic diffraction channels etched in silicon with 10  $\mu\text{m}$  x 100  $\mu\text{m}$  (width  $\times$  depth) cross-section and 20  $\mu\text{m}$  pitch. Transmission grating period tunability is realized by dynamically changing the channels' opacity to neutrons and/or x-rays. Opacity of each of 2,560 diffraction channels can be switched independently between non-transparent and transparent state by filling/drainage of the channels with neutrons/x-ray absorbing fluid. In this work, we will provide details on the fabricating the first prototype device, DARIUS-1, consisting of 128 independently controlled channels. We will also discuss our method of channel's infilling with opaque fluid and overall device performance.

**MN-Contributed On Demand-37 QCM Study of Tribotronic Control in Ionic Liquids and Nanoparticle Suspensions, Caitlin Seed, B. Acharya, J. Krim,** North Carolina State University

A Quartz Crystal Microbalance (QCM) technique has been employed to tune friction at liquid-solid interfaces with tribotronic methods employing externally applied electric fields in both nanoparticle suspensions and ionic liquid systems. The setup consists of a QCM immersed in liquid containing electrically charged constituents whose sensing electrode faces a nearby counter electrode. An electric field perpendicular to the QCM surface is created when a potential is applied between the two electrodes, which allows the charged constituents in the surrounding liquid to be repositioned. QCM measurements are able to detect differences in friction under various field conditions, and thus detectably tune the friction in both nanoparticle and ionic liquid systems. The versatility and simplicity of QCM friction measurements renders it an ideal tool for the rapidly expanding research area of tribotronics.

## Author Index

### Bold page numbers indicate presenter

— A —

Acharya, B.: MN-Contributed On Demand-37, **4**

— B —

Baca, E.: MN-Contributed On Demand-13, **2**

Ballance, M.: MN-Contributed On Demand-16, **2**

Beardslee, L.: MN-Contributed On Demand-28, **3**

Bingham, A.: MN-Contributed On Demand-16, **2**

— C —

Chaudhary, R.: MN-Contributed On Demand-31, **4**

Chen, J.: MN-Contributed On Demand-25, **3**

— D —

Dang, T.: MN-Contributed On Demand-31, **4**

— F —

Feng, P.: MN-Contributed On Demand-7, **1**

— G —

Ghodssi, R.: MN-Contributed On Demand-28, **3**

Gibson, C.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-16, **2**

— H —

Herrera, S.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-16, **2**

Hirabayashi, M.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-13, **2**

Hollowell, A.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-13, **2**

Hummel, G.: MN-Contributed On Demand-16, **2**

Hussey, D.: MN-Contributed On Demand-34, **4**

— J —

Jiao, C.: MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**

Jordan, M.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-13, **2**; MN-Contributed On Demand-16, **2**

— K —

Kaisar, T.: MN-Contributed On Demand-4, **1**

Kim, G.: MN-Contributed On Demand-31, **4**

Klimov, N.: MN-Contributed On Demand-34, **4**

Krim, J.: MN-Contributed On Demand-37, **4**

— L —

Lee, J.: MN-Contributed On Demand-4, **1**

Lepkowski, S.: MN-Contributed On Demand-10, **2**

Levy, J.: MN-Contributed On Demand-28, **3**

Li, J.: MN-Contributed On Demand-1, **1**

Liang, Y.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**

— M —

McClain, J.: MN-Contributed On Demand-13, **2**

McDow, J.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-13, **2**

Menk, L.: MN-Contributed On Demand-13, **2**

Moore, C.: MN-Contributed On Demand-16, **2**

Murphy, R.: MN-Contributed On Demand-34, **4**

— N —

Nordquist, C.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-16, **2**

— P —

Pei, S.: MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**

— R —

Robinson, S.: MN-Contributed On Demand-34, **4**

Ruyack, A.: MN-Contributed On Demand-10, **2**; MN-Contributed On Demand-16, **2**

— S —

Schiess, A.: MN-Contributed On Demand-16, **2**

Schmitt, R.: MN-Contributed On Demand-13, **2**

Seed, C.: MN-Contributed On Demand-37, **4**

Stine, J.: MN-Contributed On Demand-28, **3**

— W —

Wang, F.: MN-Contributed On Demand-1, **1**

Wang, Z.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**

Watson, G.: MN-Contributed On Demand-31, **4**

Weigandt, K.: MN-Contributed On Demand-34, **4**

Wen, T.: MN-Contributed On Demand-1, **1**

Wu, S.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**

— X —

X.-L. Feng, P.: MN-Contributed On Demand-4, **1**

Xia, J.: MN-Contributed On Demand-25, **3**

Xiao, F.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**

Xie, N.: MN-Contributed On Demand-31, **4**

Xu, B.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-25, **3**

— Y —

Yang, R.: MN-Contributed On Demand-1, **1**

Yousuf, S.: MN-Contributed On Demand-7, **1**

— Z —

Zhang, P.: MN-Contributed On Demand-1, **1**

Zheng, X.: MN-Contributed On Demand-7, **1**

Zhu, J.: MN-Contributed On Demand-1, **1**; MN-Contributed On Demand-19, **2**; MN-Contributed On Demand-22, **3**; MN-Contributed On Demand-25, **3**