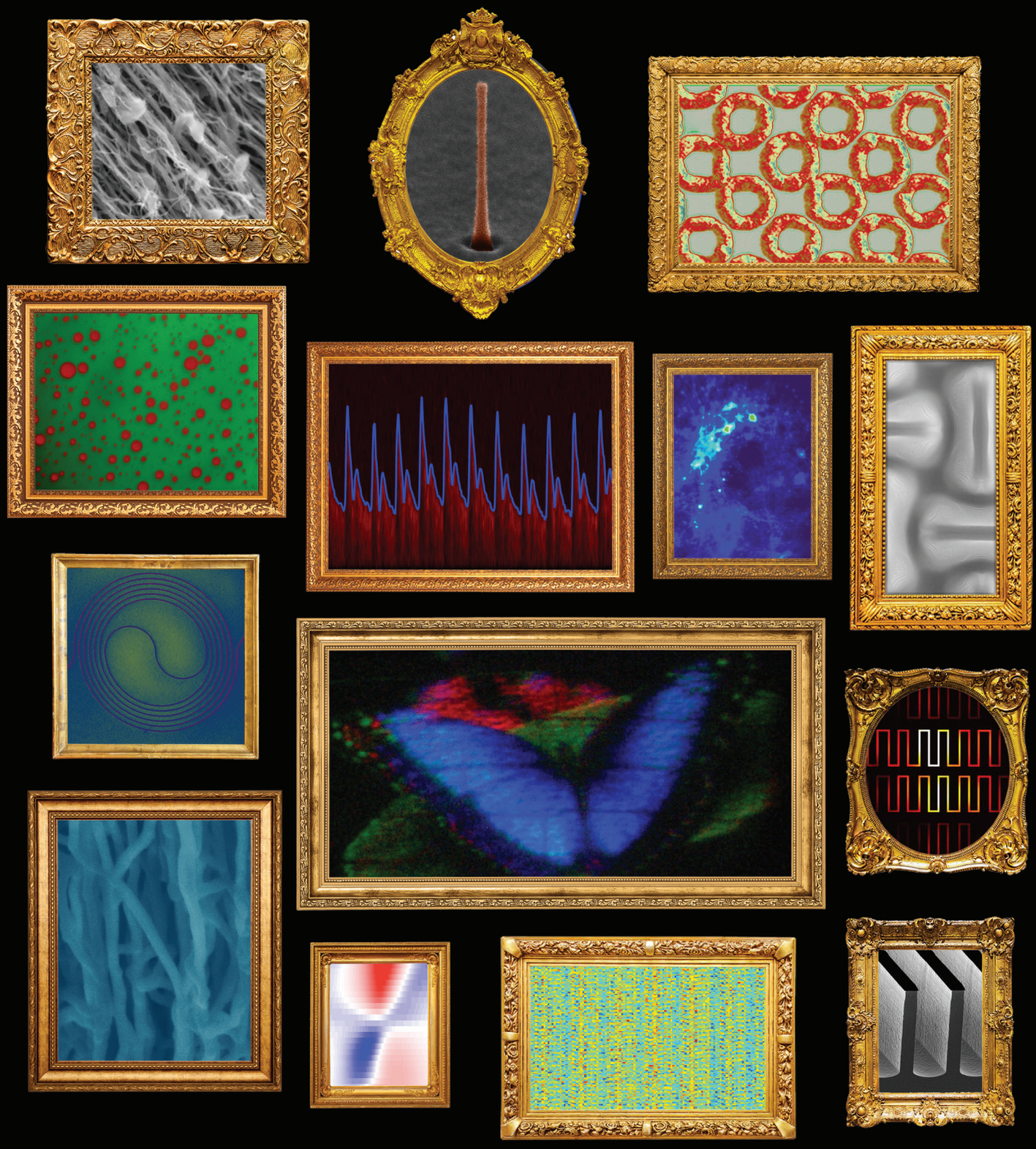


ANNUAL RESEARCH REPORT 2016

MICROSYSTEMS TECHNOLOGY LABORATORIES

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

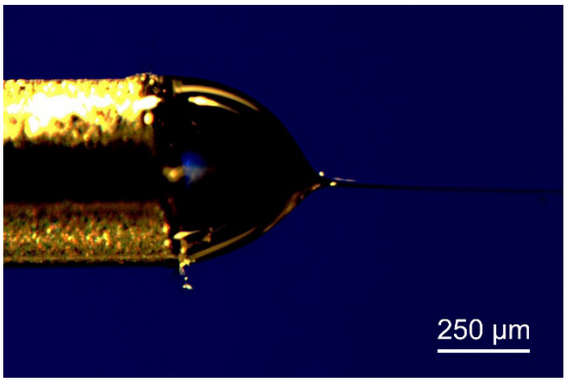
Wearable Energy Harvesters Based on Aligned Mats of Electrospun Piezoelectric Nanofibers

D. Olvera-Trejo, L. F. Velásquez-García
Sponsorship: U.S. Army

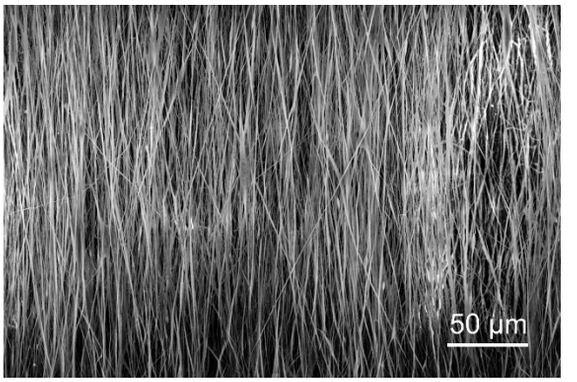
Battery recharging and replacement are still challenging after several decades of developing energy sources for portable and wireless devices. For this reason, new power sources have become essential for current and future stand-alone devices. Energy harvesters are an attractive alternative for supplying power in these systems.

We are developing wearable energy harvesters based on electrospun piezoelectric nanofibers as transducing elements. The proposed harvesting device consists of a set of flexible interdigitated electrodes on a flexible substrate; the electrodes are coated with aligned piezoelectric nanofibers. Each time the substrate is stretched or bent, the piezoelectric nanofibers produce voltage and charge that can be used to feed low-power devices. Our energy harvesters could be integrated into garments, allowing people to carry less weight and volume in batteries, which is particularly advantageous on long journeys and when located far from the electrical grid.

The piezoelectric nanofibers of our energy harvester are made of poly(vinylidene difluoride), i.e., PVDF, using the electrospinning technique. In electrospinning, a solution rich in long-chain polymers that is subject to a high electrostatic field ejects a jet that is thinned to a submicron diameter due to the interaction of the electric field and surface tension effects on the fiber (Figure 1). Highly aligned fiber deposition on the interdigitated electrodes of the energy harvester is necessary to achieve high efficiency. With this goal in mind, we developed a custom rotating collector system that allows control of the alignment and diameter of the deposited nanofibers. The collected fibers tend to be more aligned and exhibit smaller fiber diameters when the collector drum rotates at thousands of revolutions per minute (Figure 2). Current work focuses on controlling the morphology of the PVDF fibers and nanofiber mats, as well as on testing nanofiber harvester prototypes using a custom apparatus and benchtop electronics.



▲ Figure 1: Electrospinning emitter in operation. A fine jet of liquid is electrohydrodynamically ejected from the emitter tip due to the high electric fields present there; the jet is stretched into a nanofiber due to the surface tension and electrostatic forces it experiments while traveling to the collector electrode.



▲ Figure 2: SEM micrograph of aligned PVDF nanofibers deposited on a rotating drum collector.

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Fabrication of Core-Shell Microparticles Using 3-D Printed Microfluidics

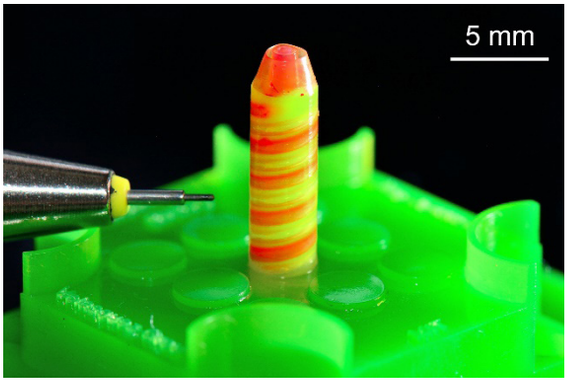
D. Olvera-Trejo, L. F. Velásquez-García
Sponsorship: Tecnológico de Monterrey/MIT Nanotechnology Program

Coaxial electrospraying is an electrohydrodynamic process that creates core-shell microparticles by atomization of a coaxial electrified jet composed of two immiscible liquids. Coaxial electrospraying has several advantages over other microencapsulation technologies including higher encapsulation efficiency and more uniform size distribution. Coaxial electrosprayed compound microparticles can be used in exciting applications such as feedstock microencapsulation, controlled drug release, and self-healing composites.

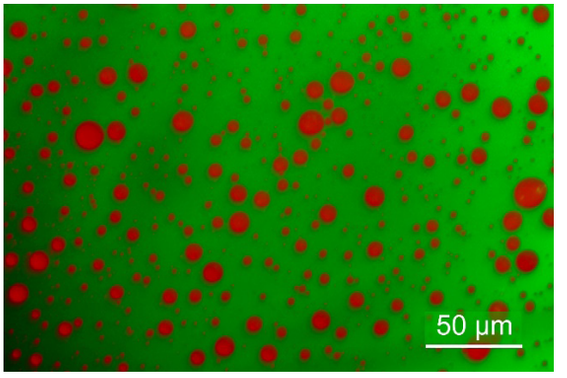
Unlike traditional, i.e., uniaxial, electrospraying that has been investigated for over 100 years and of which many MEMS implementations exist, coaxial electrospraying was first described in 2002 and no microfabricated coaxial electrospray source had been reported due to the inherent three-dimensionality and complexity of its hydraulic system.

Stereolithography (SLA) is a layer-by-layer additive manufacturing process that creates solid objects via photopolymerization of a resin using ultraviolet light. Additive manufacturing started as a visualization tool for mesoscaled objects, but recent developments in the resolution and capabilities of 3-D printing suggest that these manufacturing processes could address the complexity, three-dimensionality, and material requirements of many microsystems. In particular, high-resolution SLA can be used to manufacture freeform microfluidics at a small fraction of the cost per device, infrastructure cost, and fabrication time of a typical silicon-based microfluidic system.

We developed SLA 3-D printed coaxial electrospray sources with one or two emitters that are fed by two helical channels (Figure 1). Each emitter spout is designed to produce a coaxial flow and to enhance the electric field on the liquid meniscus. Using these devices, we produced uniform core-shell microparticles using deionized water as the inner liquid and sesame oil as the outer liquid (Figure 2). The size of the droplets can be modulated by controlling the flow rates fed to the emitters. Electrical characterization of the devices demonstrates that the emitters operate uniformly. Current research efforts focus on demonstrating massively multiplexed sources with uniform array operation.



▲ Figure 1: 3-D printed coaxial electrospray device next to a 0.3-mm-diameter mechanical pencil; the different colors of the liquids supplied to the device evidence the helical channels that feed the emitter nozzle.



▲ Figure 2: Optical image from a fluorescent microscope of core-shell droplets (core is water dyed with Rhodamine B, shell is sesame oil) immersed in water dyed with fluorescein. The compound particles were produced by one of our 3-D printed coaxial electrospray sources. The oil shell covering each red-colored core of the droplets prevents their mixing with green-colored water.

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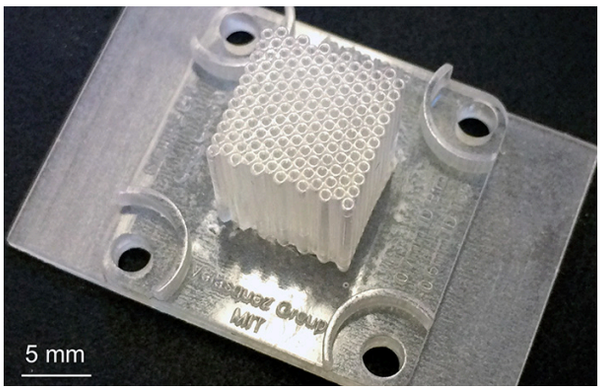
3-D Printed Massively Multiplexed Electrospray Sources

L. F. Velásquez-García
Sponsorship: U.S. Army

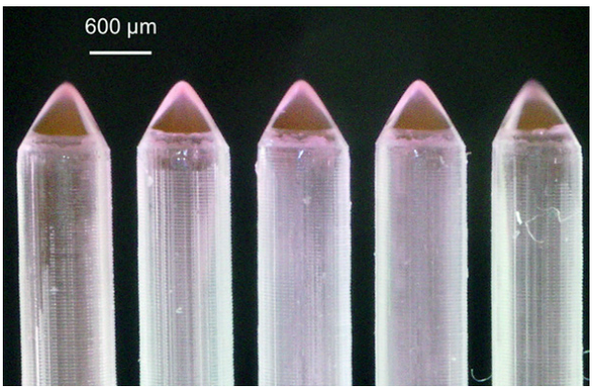
Electrospray is a electrohydrodynamic phenomenon that produces from a meniscus a stream of micro/nanoparticles that, depending on the properties of the liquid and the process conditions, can be droplets, ions, or fibers. The low spread in size and specific charge of the emitted particles makes the use of electrospray attractive in applications such as combustors, maskless micro/nanomanufacturing, and nanosatellite propulsion. However, the throughput of an electrospray emitter is very low, limiting the applicability of single-emitter electrospray sources to a few practical cases, e.g., mass spectrometry of biomolecules.

An approach to increase the throughput of an electrospray source without increasing the size variation of the emission is implementing arrays of electrospray emitters that operate in parallel. Miniaturization of the electrospray emitters results in less power consumption and lower onset voltage; in addition, using micro-fabrication, monolithic arrays of miniaturized emitters with large array size and emitter density can be made. Researchers have demonstrated a variety of MEMS multiplexed electrospray sources that operate uniformly. Although these devices work satisfactorily, they present a number of issues: (i) the device architecture is often a compromise between what should be made based on the modeling and what can be made given the limitations of traditional microfabrication, sacrificing device performance; (ii) a change in any of the in-plane features of the design requires the redesign and fabrication of one or more lithography masks while causing added costs and time delays; (iii) these devices are fairly expensive because they are made in a multi-million semiconductor-grade cleanroom with advanced tools that are operated by highly trained staff, which restricts their application to high-end applications and research.

We recently demonstrated the first 3-D printed multiplexed electrospray sources in the literature (Figure 1). The devices were fabricated with stereolithography and have associated two orders of magnitude less fabrication cost per device, fabrication time, and manufacturing infrastructure cost compared to a silicon MEMS multiplexed electrospray source. The 3-D printed devices include features not easily attainable with other microfabrication methods, e.g., tapered channels and threaded holes. Through the optimization of the fabrication process, arrays with as many as 236 internally fed electrospray emitters (236 emitters in 1 cm^2) were made, i.e., a twofold increase in emitter density and a sixfold increase in array size compared with the best reported values from multiplexed, internally fed, electrospray sources made of polymer. The characterization of devices with a different array size suggests a uniform emitter operation (Figure 2).



▲ Figure 1: A 3-D printed planar array of 143 tapered, internally fed electrospray emitters in 1 cm^2 (143 emitters/cm^2, hexagonal packing) The emitters are fed by 12 mm long tapered internal channels with 400 µm diameter at the emitter spouts.



▲ Figure 2: External row of 5 emitters part of a 49-emitter planar array (70 emitters/cm^2, square packing). The scalloping on the exterior of the emitters, due to the layer-by-layer manufacturing, is visible.

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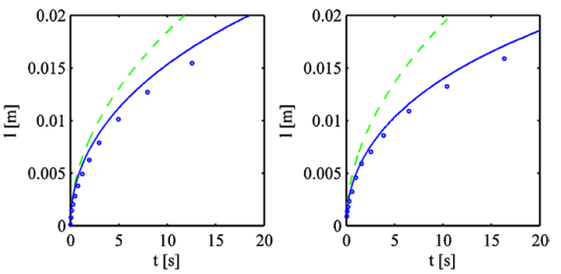
Optimization of Capillary Flow through Open Microstructured Arrays

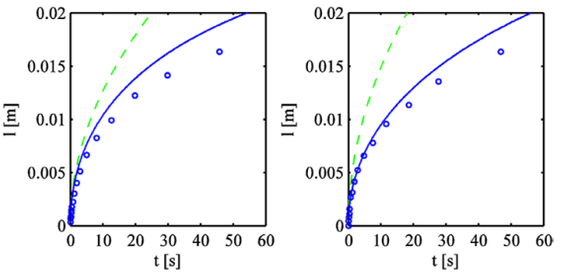
P. Ponce de Leon, L. F. Velásquez-García
Sponsorship: DARPA

Liquid propagation through porous microstructures has received significant attention due to the importance of precisely controlling flow in microfluidic systems. Periodic surface structures, e.g., arrays of open micropillars or open microchannels, sometimes can be used to control the flow in a microsystem, introducing benefits such as direct access to the porous structure, device reusability, and resilience against clogging. In an open fluidic structure, the liquid is not actively pumped, e.g., using an upstream pressure signal; instead, the microstructured surface passively drives the liquid via capillary action. However, the same surfaces driving the flow via surface tension's pull simultaneously impede it by way of viscous resistance. Therefore, optimization of the geometry of the microstructured surface is required to maximize the flow rate it transports.

We developed semi-analytical models that describe the dynamics of capillary flow against gravity in (i) vertical arrays of open microchannels with rectangular cross-section and (ii) arrays of open micropillars with square packing and square cross section. We also extended our analysis to capture the shear-thinning behavior typical of many non-Newtonian fluids. Our models indicate the existence of multiple flow rate maxima with respect to pore size. One maximum, which occurs only in micropillar arrays, arises from the trade-off between capillary pressure and viscous resistance. The two other maxima, which occur for both micropillar and microchannel arrays, are related to meniscus and gravitational effects and only appear at low aspect-ratio (i.e., in channels/gaps between adjacent pillars that are about as wide as they are deep) and high Bond number, respectively. Experimental capillary rise data demonstrate that incorporating first-order gravitational effects and the impact of meniscus curvature improved flow rate predictions relative to models that neglect these factors (Figures 1 and 2; in both figures the working liquid is 1% PEO in 40/60 ethanol/water). Experimental capillary rise data also confirm the existence and location of a flow maximum with respect to the width of an open-microchannel; operating at any of the maxima decreases the sensitivity of flow rate to geometric variation, allowing for more robust microfluidic systems. Finally, we demonstrated electrospray emission from the edge of a microstructured surface as an example of an application of the porosity geometries we investigated in this study; the supply-limited regime of the current-voltage characteristics of these devices are in agreement with the literature on electrospray droplet emission, opening the possibility to implement arrays of externally-fed electrohydrodynamic jetting emitters that can operate continuously while producing droplets or nanofibers using suitable working liquids.



▲ Figure 1: Height of rising liquid front vs. time for various open-microchannel geometries. In each subplot the blue circles are measured data points. The solid blue line shows the predictions of our model, and the dashed green line shows predictions neglecting gravitational and meniscus-permeability effects. Left – microchannels 139 μm deep, 149 μm wide, 101-μm thick walls; right – microchannels 141 μm deep, 211 μm wide, 289-μm thick walls.



▲ Figure 2: Height of rising liquid front vs. time for various open-micropillar array geometries. In each subplot the blue circles are measured data points, the solid blue line shows the predictions from our data, and the dashed green line shows predictions of the model proposed by Xiao *et al*, ***Langmuir,*** 2010. Left – micropillars 140 μm tall, 93 μm wide, 157 μm gap; right – microchannels 143 μm tall, 289 μm wide, 211 μm gap.

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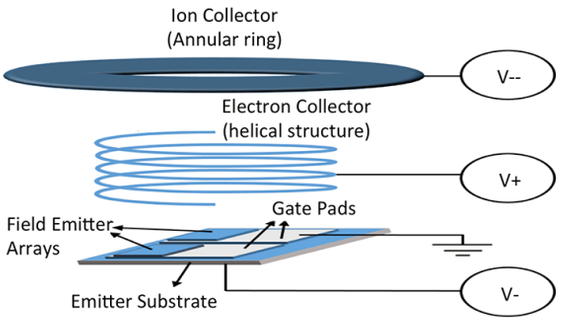
Chip-Scale Electrostatic Vacuum Ion Pump with Nanostructured Field Emission Electron Source

A. Basu, L. F. Velásquez-García
Sponsorship: DARPA

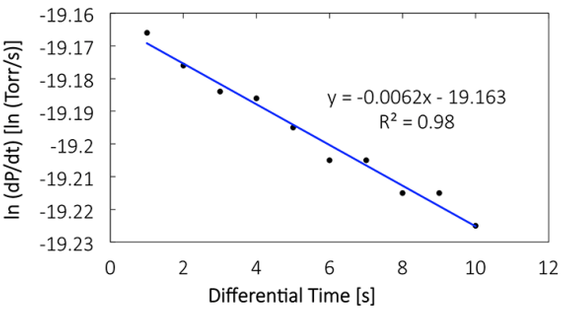
Cold-atom interferometry of alkali atoms can be used in a variety of high-precision sensors and timing devices such as atomic clocks, gyroscopes, accelerometers, magnetometers, and gravimeters. These devices require ultra-high vacuum (UHV, pressure < 10^{-9} Torr) to operate; therefore, chip-scale versions require miniaturized UHV pumps resilient to alkali metal vapors that consume power at levels compatible with device portability. In a macro-sized chamber, UHV-level vacuum can be maintained using a conventional magnetic ion pump, where electrons that swirl around the magnetic lines of a magnet create ions by impact ionization of neutral molecules, which in turn sputter a Ti getter. While scaled-down versions of magnetic ion pumps have been reported, these are incompatible with miniaturized cold-atom interferometry systems because (i) a reduction in the pump size increases the required threshold magnetic field for electron trapping, and (ii) the larger magnetic field associated with a miniaturized ion pump can interfere with the operation of the cold-atom sensor, yielding flawed readings. Non-evaporable getter (NEG) pumps are used in some cold-atom interferometry systems, e.g., commercial chip-scale atomic clocks; however, NEG pumps are unable to pump noble gases such as He and N2 that are present in the chamber, and they inefficiently pump alkali vapors.

We are developing vacuum ion pumps compatible with chip-scale cold-atom interferometry devices. The proposed field emitter array (FEA)-based magnet-free ion pump architecture is shown in Figure 1. In this pump design, a helical electron collector pulls the electrons toward itself, forcing them to first travel beyond the height of the electron collector, to then get pushed back due to the electrostatic mirror effect of the annular-shaped ion collector. Therefore, the trajectory of the electrons is significantly increased compared to a pump design with a parallel-capacitor electrode configuration, augmenting the probability of impact ionization. The FEA consists of arrays nano-sharp silicon tips, each surrounded by a self-aligned gate electrode; we have shown that these FEAs do not degrade in the presence of Rb vapor.

Figure 2 shows the semi-log plot of the minus time derivative of the pressure versus time during pump-down, with the horizontal axis denoting the time since the beginning of each pump-down cycle; in these experiments, the pressure inside the chamber reached values as low as ~7×10^{-7} Torr. Each data point in the plot represents an average of the minus time derivative of the pressure considering all pump-down cycles. The R^2 of the linear fit of the data evidences that our reduced-order model accurately explains the dynamics of the pump. The slope of the linear fit of the data estimates the experimental pumping time constant at about 161 seconds.



▲Figure 1: Schematic of the FEA-based, magnetic-less ion pump architecture.



▲Figure 2: Semi-log plot of the negative of the time derivative of the chamber pressure vs. time. From the slope of the linear fit, τ = 161.2 s.

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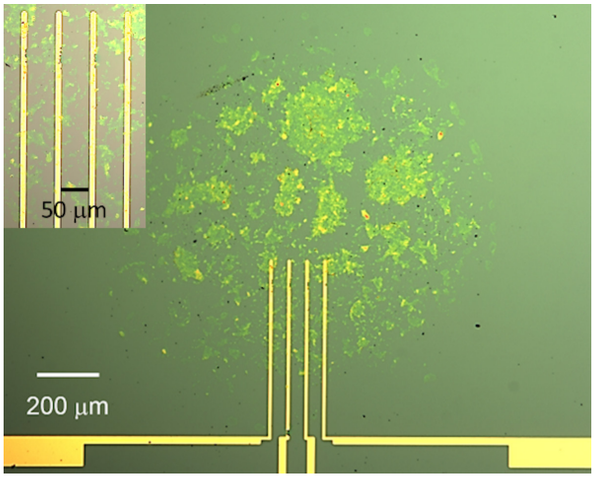
Electrospray-Printed Graphene Oxide Nanostructured Humidity Sensor

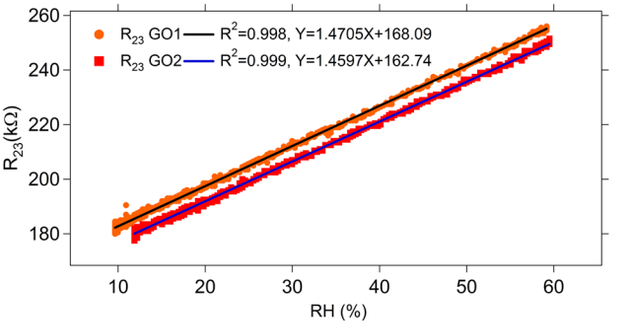
A. P. Taylor, L. F. Velásquez-García
Sponsorship: Edwards Vacuum

Conductometric gas sensors based on the chemoresistive response of semiconducting metal oxide films are widely used due to their simplicity, flexibility in production, and broad applicability to many fields. Typically, the adsorption of a gas molecule on the surface of a metal oxide alters surface electronic properties, causing a change in electrical conductivity. Although many metal oxides could be used for gas sensing, only a few show the appropriate combination of adsorption ability, catalytic activity, sensitivity, and thermodynamic stability. These select metal oxides (e.g., SnO_2, TiO_2, and ZnO), however, are the least active from the catalytic point of view. To alleviate this problem, doping with redox-active noble metal nanoparticles, such as Pt, Au, and Pd, is done to enhance conductivity response. Unfortunately, noble metals are expensive, thereby precluding their use in low-cost applications. An appealing alternative material for reactive gas sensing is graphene oxide (GO) because of its high sensitivity to adsorbed surface species and compatibility with harsh environments.

We developed low-cost conductometric gas sensors t hat use an ultrathin film made of a matrix of GO nanoflakes as a transducing element. The devices were fabricated by lift-off metallization and near-room temperature, atmospheric pressure electrospray printing using a shadow mask. The sensors detect humidity at room temperature without requiring any post-heat treatment, harsh chemical reduction, or doping with metal nanoparticles. The printed GO devices (Figure 1) show a linear relationship between the resistance of the GO sensors and relative humidity in the 10-60% range (Figure 2); considering that they were fabricated with different electrospray printing recipes, the similarity between the linear response of the two devices suggests a common underlying physical sensing mechanism dependent on the intrinsic properties of the material. The power consumption of the printed sensors was estimated at 6 µW or less in the 10-60% relative humidity range.



▲ Figure 1: Optical micrograph of a conductometric GO humidity sensor with a four-point probe electrode configuration and an inset showing a close-up view of the active area (top left corner). From A. P. Taylor and L. F. Velásquez-García, *Nanotechnology*, 2015.



▲ Figure 2: Resistance versus relative humidity for two electrospray-printed GO sensors. From A. P. Taylor and L. F. Velásquez-García, *Nanotechnology*, 2015.

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Micro- and nano-enabled multiplexed scaled-down systems for space, energy, healthcare, manufacturing, and analytical applications. 3-D printed MEMS/NEMS, carbon nanotubes; electrospray, electrospinning, field emission, field ionization, plasmas, X-rays; electrical and chemical nanosatellite propulsion, plasma sensors, portable mass spectrometry, high-voltage, 3D MEMS packaging, x-ray sources, tactile displays and sensors, ultracapacitors.

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SELECTED PUBLICATIONS

D. Olvera Trejo and L. F. Velásquez-García, "SLA 3-D Printed MEMS Coaxial Electrospray Sources For Uniform Generation of Core-Shell Microparticles," *Technical Digest 17th Solid-State Sensor, Actuator and Microsystems Workshop,* Hilton Head Island SC, USA, pp. 176 -179, June 5-9, 2016.

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