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MICROSYSTEMS TECHNOLOGY LABORATORIES
MASSACHUSETTS INSTITUTE OF TECHNOLOGY

Carbon Nanotubes Based-field Emitters by 3-D Printing

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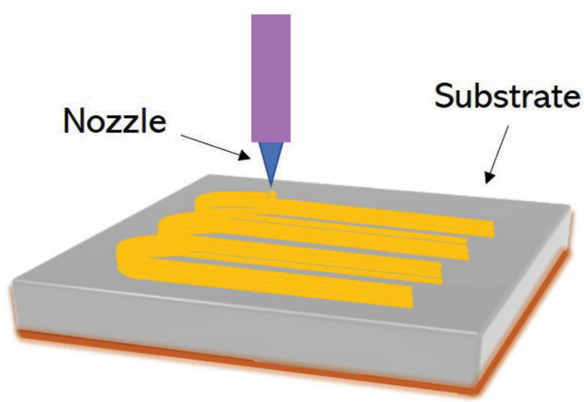
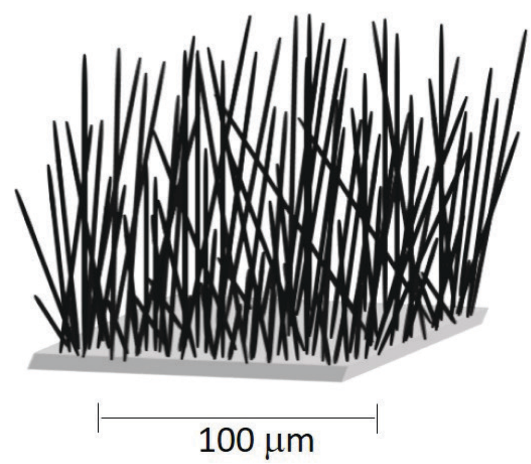
Sponsorship: MIT-Tecnológico de Monterrey Nanotechnology Program

In field emission, electrons are ejected from a solid surface via quantum tunneling due to the presence of a high local electrostatic field. Compared to state-of-the-art thermionic electron sources, field emission cathodes consume significantly less power, are faster to switch, and could operate at higher pressure. Field emission cathodes have a wide range of applications such as X-ray sources, flat-panel displays, and electron microscopy.

Several materials, e.g., Si, ZnO, and graphene, have been explored as field emission sources; however, carbon nanotubes (CNTs) are very promising to implement field emission cathodes due to their high aspect ratio, high electrical conductivity, excellent mechanical, and chemical stability, and high current emission density. Reported approaches for fabricating CNT field emitters include screen printing and direct growth of nanostructures (e.g., plasma-enhanced chemical vapor deposition) where a static stencil, i.e., mask, is involved to produce patterned structures in specific locations. These masks increase the time and cost needed to iterate the pattern, affecting the prototype optimization of the cathode. Ink direct writing (IDW), i.e., the creation of imprints by extrusion of liquid suspensions through a small nozzle, has emerged as an attractive maskless patterning technique that can accommodate a great variety of materials to create freeform imprints at low-cost (Figure 1). An imprint with CNTs protruding from the surface of the imprint (Figure 2), strongly adhered to the substrate can achieve stably high-current emission when an electric field is applied. We are currently working on the design and optimization of the formulation of a CNT-based ink, to eventually demonstrate low-cost field emission sources.



▲ Figure 1: Example of a pattern 3-D printing by means of ink direct writing using a paste made of a filler dispersed into an organic matrix.



▲ Figure 2: Example of an array of porous structure of carbon nanotubes bristling adhered to the substrate showing the tip of CNTs are exposed.

FURTHER READING

- A. P. Taylor and L. F. Velásquez-García, "Electrospray-printed Nanostructured Graphene Oxide Gas Sensors," *Nanotechnology,* vol. 26, no. 50, pp. 505301:1-8, Dec. 2015.
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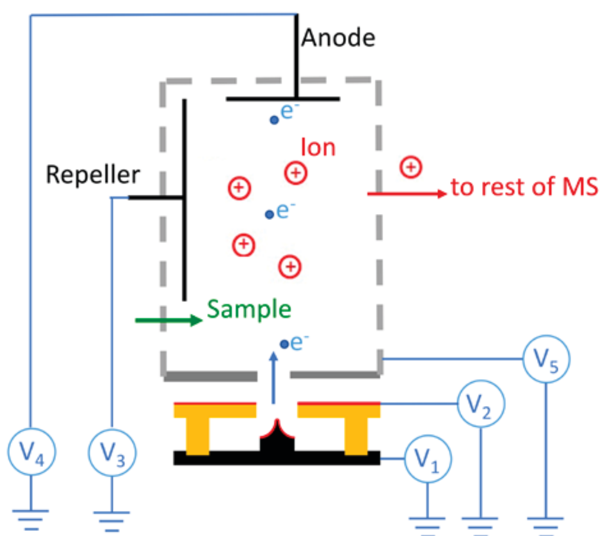
Electron Impact Gas Ionizer with 3-D Printed Housing and NEMS Si Field Emission Cathode for Compact Mass Spectrometry

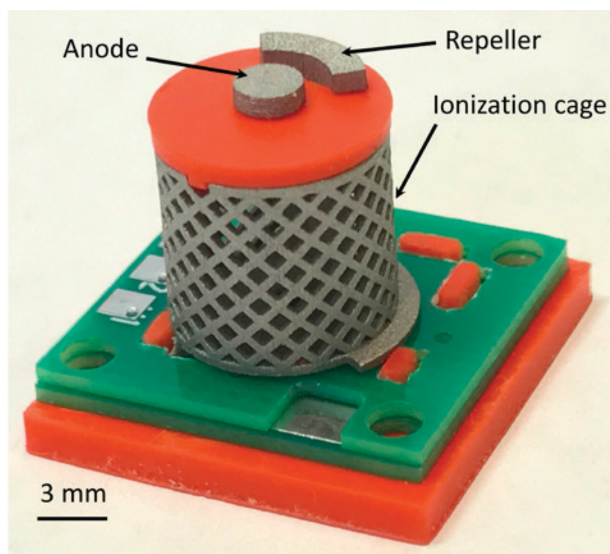
C. Yang, L. F. Velásquez-García
Sponsorship: IARPA

Mass spectrometry is widely used to quantitatively determine the composition of samples. However, the bulky size and high-power consumption of conventional mass spectrometry instruments limit their portability and deployability. One of the key components of a mass spectrometer (MS) is the ionizer. State-of-the-art electron impact gas ionizers use a stream of electrons produced by a thermionic cathode to create ions by fragmentation. Field emission cathodes, based on quantum tunneling of electrons triggered by high electrostatic fields, are a better alternative for portable mass spectrometry of gases compared to mainstream thermionic cathodes because they consume significantly less power, are faster to switch, and could operate at higher pressure.

In this project, we are developing a compact electron impact gas ionizer based on a cleanroom-microfabricated cathode and a 3-D printed ionization housing (Figure 1). The cathode is an array of nano-sharp silicon field emitters with proximal, self-aligned extractor gate, while the ionization housing is composed of an ionization region surrounded by an ionization cage, an anode electrode, a repeller electrode, and a dielectric structure that holds together the electrodes. To produce ions (i) a high enough bias voltage is applied between the extractor gate and the silicon tips, shooting electrons into the ionization region, (ii) the anode electrode attracts the emitted electrons, forcing them to interact with the neutral gas molecules within the ionization region, (iii) the bias voltage of the ionization cage maximizes the ionization yield of the interaction between the electrons and the neutral gas molecules, and (iv) the repeller electrode pushes ions out of the ionization cage. Figure 2 shows an assembled ionizer. Current work is focused on characterization of the field emission cathode and gas ionizer at various conditions.



▲ Figure 1: Schematic of electron impact gas ionizer with field emission cathode. The ions produced by the device are fed to the rest of the MS.



▲ Figure 2: Picture of implemented gas ionizer. The field emitter array chip is mounted between two printed circuit boards.

FURTHER READING

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- L.-Y. Chen, L. F. Velásquez-García, X. Wang, K. Cheung, K. Teo, and A. I. Akinwande, "A Micro Ionizer for Portable Mass Spectrometers using Double-gated Isolated Vertically Aligned Carbon Nanofiber Arrays," *IEEE Transactions on Electron Devices*, vol. 58, no. 7, pp. 2149-2158, July 2011.
- L. F. Velásquez-García, B. Gassend, and A. I. Akinwande, "CNT-based MEMS Ionizers for Portable Mass Spectrometry Applications," *J. Microelectromech. Syst.*, vol. 19, no. 3, pp. 484-493, June 2010.

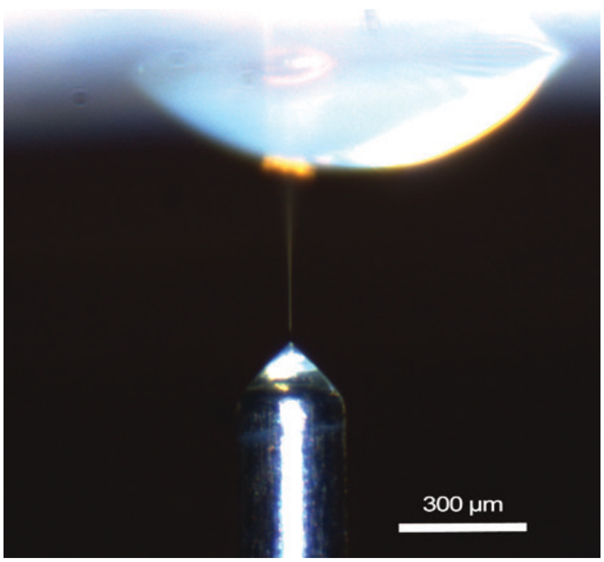
Printed Piezoelectric Thin Films via Electrohydrodynamic Deposition

B. García-Farrera, L. F. Velásquez-García
Sponsorship: MIT-Tecnológico de Monterrey Nanotechnology Program

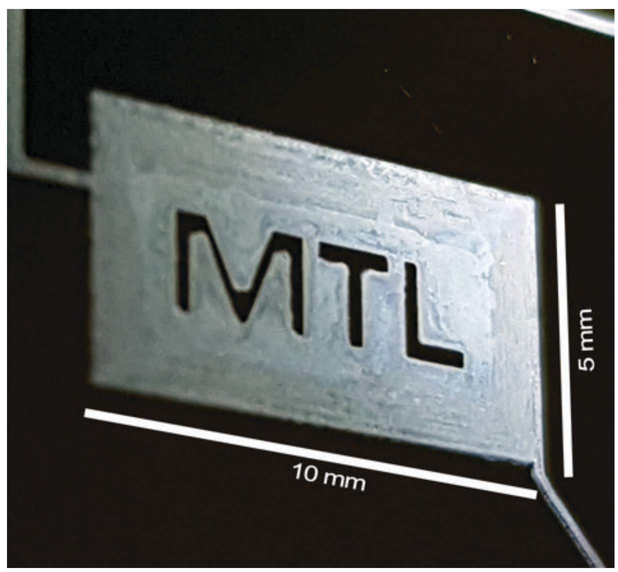
Piezoelectric components have found applications in a variety of fields including energy harvesting, biological and chemical sensing, and telecommunications. The creation of piezoelectric thin films has made possible the implementation of exciting devices that operate at higher frequency (a consequence of the reduction of the thickness of the piezoelectric material) including highly sensitive gravimetric biosensors and acousto-fluidic actuators. However, traditional manufacturing methods for piezoelectrics require a high vacuum, show low deposition rates, involve expensive and complex equipment, and require additional microfabrication processes to achieve the required geometries via patterning and lithography.

Electrohydrodynamic deposition harnesses the electrospray phenomenon to create ultrathin imprints from liquid feedstock (Figure 1). When the electrospray emitter operates in the cone-jet mode, stable jetting of the liquid feedstock allows for the direct writing of structures, thus, eliminating the need for steps for material removal, e.g., mask transfer and etching (Figure 2). In addition, electrohydrodynamic deposition can operate at room temperature without the need for a vacuum and can be scaled-up via electrospray emitter multiplexing.

This project aims to produce piezoelectric thin films suitable for acoustic resonators and actuators via electrospray jetting of nanoparticle-doped liquid feedstock. Initial work revolved around the optimization of the deposition parameters and formulation of the liquid feedstock for the reduction of the printed line's width and thickness, elimination of the "coffee ring" effect, and analysis of the crystallographic orientation of the films. Current work focuses on improving the film's homogeneity, increasing the crystal orientation towards a highly oriented film, and its piezoelectric characterization and application as a sensor.



▲ Figure 1: Electrospray of nanoparticle-doped liquid feedstock. The electric field from the bias voltage between the capillary and the substrate ejects a fine jet of solution from the apex of the Taylor cone.



▲ Figure 2: Negative of the Microsystems Technology Laboratory logo printed with piezoelectric liquid feedstock via electrospray.

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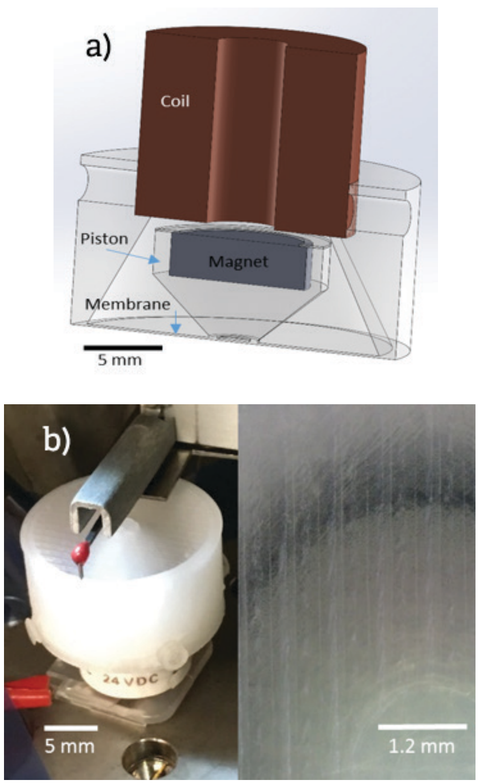
Additive Manufacturing of High-temperature Compatible Magnetic Actuators

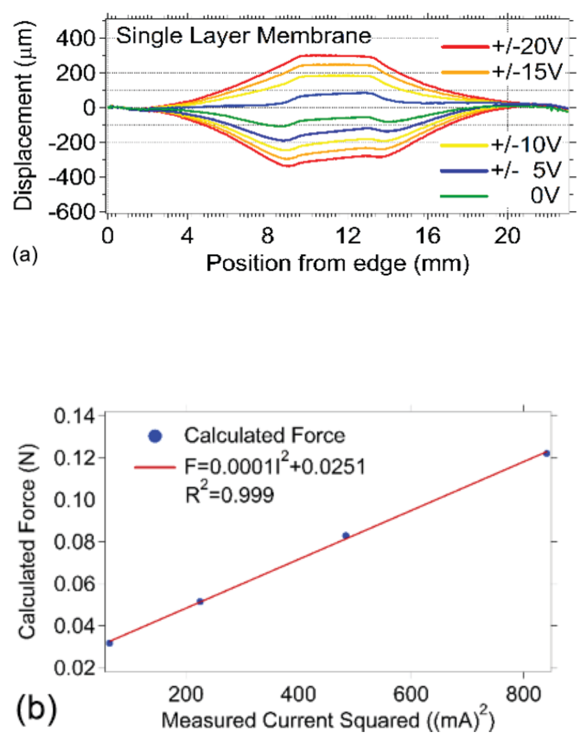
A. P. Taylor, L. F. Velásquez-García
Sponsorship: Edwards Vacuum

Various MEMS devices require large displacement and large force actuation to be efficient, such as miniature pumps. Magnetic actuation delivers large displacement and large force in a compact form factor. Additive manufacturing has recently been explored as a processing toolbox for MEMS; researchers have reported additively manufactured microsystems with performance on par or better than counterparts made with standard microfabrication. In this work, miniature actuators are printed in pure Nylon 12 using the fused filament fabrication method where a thermoplastic filament is extruded from a hot nozzle to create layer by layer a solid object. The actuators have embedded magnets that are not demagnetized by the heated nozzle (@ 250 ˚C) while being sealed in place midstream in the printing process.

We have demonstrated the first miniature, additively manufactured, monolithic magnetic actuators compatible with high temperature (>200 ˚C) operation (Figure 1). The displacement of a 150 µm-thick, single-layer membrane actuator is characterized by various DC coil bias voltages, resulting in a maximum membrane displacement of 302 µm with 20V DC applied to the driving coil; in addition, the magnetic force is proportional to the square of the current drawn by the coil as expected from theory (Figure 2).



▲Figure 1: (a) Cross-section of magnetic actuator with additively manufactured Nylon 12 body, embedded SmCo magnet, and off-the-shelf driving coil. (b) Photograph of a single-layer, 150 µm-thick membrane actuator while being tested (left) and close-up of the membrane showing the striations due to the rastering of the nozzle (right).



▲Figure 2: (a) Membrane displacement vs. radial position from the edge for various DC bias coil voltages for an actuator with a 150 µm-thick single-layer membrane. The membrane sags at 0V due to the weight of the magnet. (b) Magnetic force acting on the SmCo magnet versus the square of the current drawn by the driving coil.

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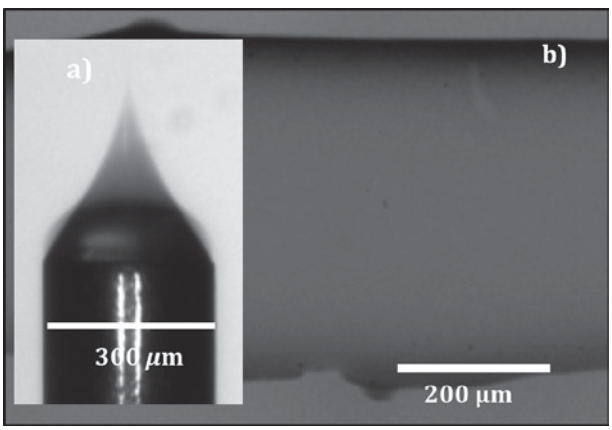
Electrospray-printed Physical Sensor

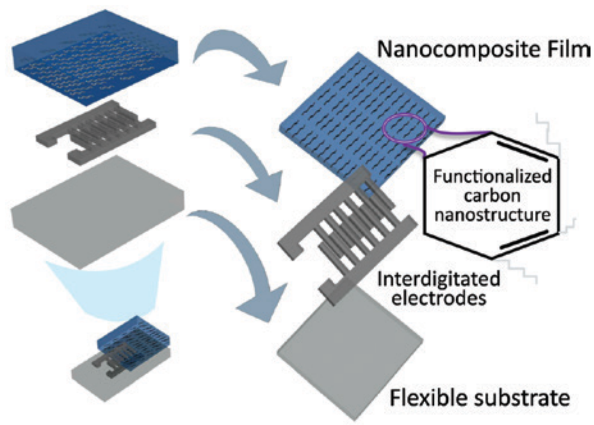
A. O. Sustaita-Narváez, L. F. Velásquez-García
Sponsorship: MIT-Tecnológico de Monterrey Nanotechnology Program

Electrospray deposition (ESD) has recently gained attention as a manufacturing technology to develop novel nanostructured composites to produce low-cost micro- and nano-devices. ESD is also a remarkably versatile printing technique due to its capability to create ultrathin films made from a great variety of liquid feedstock (e.g., suspensions of polymeric, dielectric, metallic particles) that can be doped with organic nanostructures to modulate the physical properties of the imprint. Notably, the resulting nanoreinforced composites might show enhanced transduction, which, in combination with printing on flexible substrates, might be relevant for exciting applications such as wearable biomedical devices.

This project aims to develop an additively manufactured, low-cost, flexible physical sensor based on an ultrathin nanocomposite film doped with functionalized carbon nanostructures. The Taylor cone on an electrospray emitter fed with nanocomposite feedstock is shown in Figure 1a, while an electrospray-deposited imprint on a substrate is shown in Figure 1b. Essentially, this project is divided in *(i)* down-selecting and optimizing the formulation of the liquid feedstock, *(ii)* optimizing the fabrication of the ultrathin (~100 nm) nanostructured composite, and *(iii)* demonstrating a flexible physical sensor with transducing component made of the optimized nanostructured composite (see Figure 2).



▲ Figure 1: a) Taylor cone of nanocomposite solution (needle has 300 μm outer diameter). b) 150X scanning electron microscope image of a line of electrospray-deposited ultrathin nanocomposite film.



▲ Figure 2: Schematic of flexible physical sensor.

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- A. P. Taylor and L. F. Velásquez-García, "Electrospray-printed Nanostructured Graphene Oxide Gas Sensors," *Nanotechnology*, vol. 26, pp. 505301:1-8, 2015.
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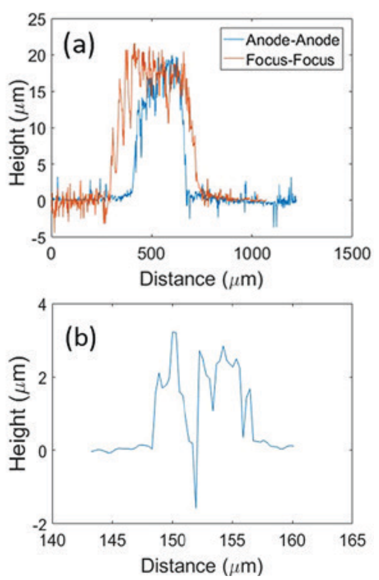
Atmospheric Microplasma Sputter Deposition of Interconnects

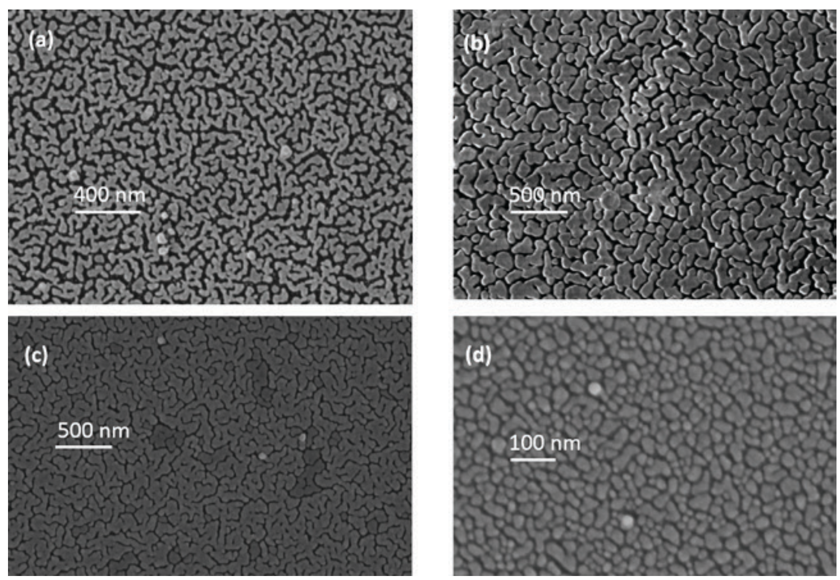
Y. Kornbluth, R. Mathews, L. Parameswaran, L. M. Racz, L. F. Velásquez-García
Sponsorship: MIT Lincoln Laboratory

We have preliminarily developed an apparatus that allows for the continuous, direct writing of interconnect-quality conductive lines. An atmospheric-pressure microplasma obviates the need for a vacuum while allowing for fine resolution imprints. We tested and characterized a novel focusing mechanism in which collisions with the working gas are harnessed to transfer electrostatic force to neutral sputtered atoms. This method compresses the deposit's width in one dimension while expanding its length in the perpendicular dimension. We find that for an ideal set of parameters, the imprint is narrower than the sacrificial sputtering target (i.e., 9 µm wide imprint from a 50 µm diameter target). Other sets of parameters lead to other results, as computer simulation predicted, ranging from an unfocused spot 400 µm in diameter to a narrow line with 20:1 compression in the direction of focus, i.e., width, and 20:1 expansion in length (Figure 1), as compared to the unfocused spot.

The microstructure of the deposit is of particular interest. As is typical of sputterers, the deposit could be smooth (55 nm roughness), and the resistivity can be as low as 1.1 µΩ·m (with no annealing). However, the resistivity greatly depends on the microstructure, which in turn depends on the deposition conditions. It is well known that sputtering at high-pressure results in a grain structure, as the early deposits shadow parts of the bare substrate, keeping sputtered material from fully coating the substrate. Traditionally, vacuum sputtering prevents this problem by allowing the sputtered material to impact the substrate normal to the surface; however, we sputter at atmospheric pressure, and thus, the sputtered material is redirected by random collisions. In our case, we use a combination of directed gas flow and electrostatic forces to prevent this shadowing effect (Figure 2).



▲ Figure 1: Height profiles of (a) partially focused and (b) well-focused deposits. The well-focused deposit is cracked; an artifact of the microscopy extends the crack into the substrate.



▲ Figure 2: SEM micrographs of deposits (a) without gas flow, (b) with gas flow but no electrostatic attraction, (c) with gas flow and electrostatic attraction, and (d) with gas flow and larger electrostatic attraction. Note how the microstructure improves from (a) to (c), and in (d), there is too much attraction for proper deposition.

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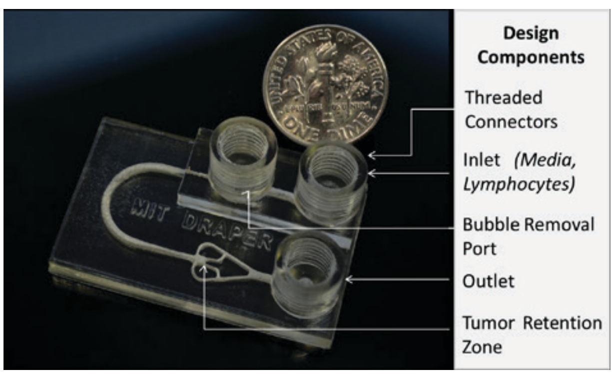
A Printed Microfluidic Device for the Evaluation of Immunotherapy Efficacy

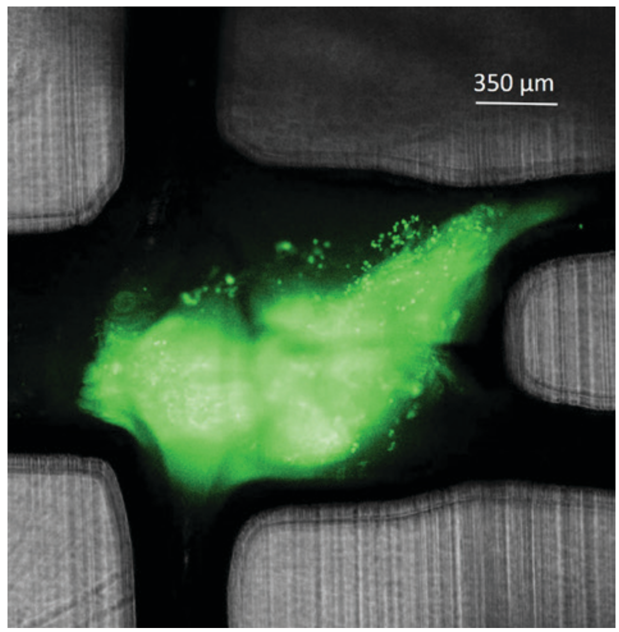
A. L. Beckwith, J. T. Borenstein, L. F. Velásquez-García
Sponsorship: Draper

Inherent challenges in device fabrication have impeded the widespread adoption of microfluidic technologies in the clinical setting. Additive manufacturing could address the constraints associated with traditional microfabrication, enabling greater microfluidic design complexity, fabrication simplification (e.g., removal of alignment and bonding process steps), manufacturing scalability, and rapid and inexpensive design iterations.

We have fabricated an entirely 3-D-printed microfluidic platform enabling the modeling of interactions between tumors and immune cells, providing a microenvironment for testing immunotherapy treatment efficacy. The monolithic platform allows for real-time analysis of interactions between a resected tumor fragment and resident or circulating lymphocytes in the presence of immunotherapy agents. Our high-resolution, non-cytotoxic, transparent device monolithically integrates a variety of microfluidic components into a single chip, greatly simplifying device operation when compared to traditionally-fabricated microfluidic systems. Human tumor fragments can be kept alive within the device. In addition, the tumor fragment within the device can be imaged with single-cell resolution using confocal fluorescence microscopy.



▲Figure 1: An optical picture of a 3-D-printed, transparent, non-cytotoxic microfluidic platform for analysis of the efficacy of immunotherapy, with features labeled.



▲Figure 2: Overlaid bright-field and fluorescence images enable visualization of device geometries in addition to the stained tumor fragment. Single cells are visible.

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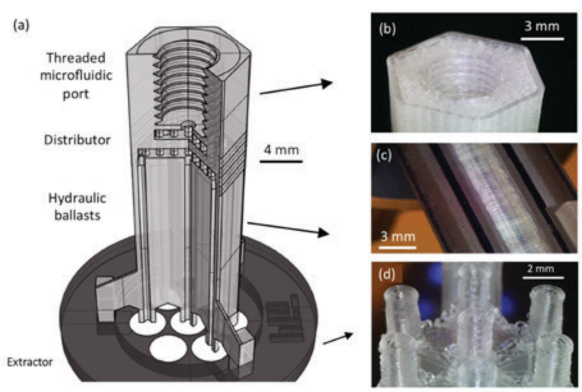
Biocompatible Dielectric-conductive Microsystems Monolithically 3-D Printed via Polymer Extrusion

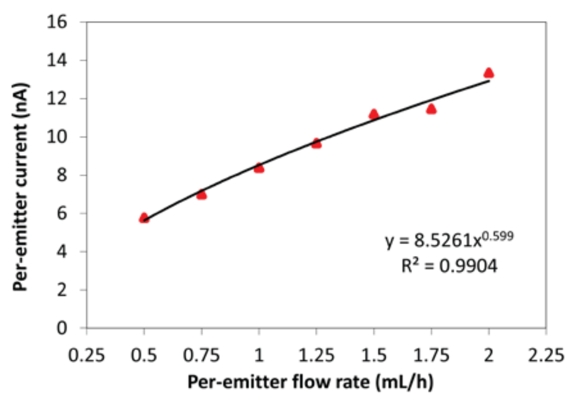
Z. M. Sun, L. F. Velásquez-García

Additive manufacturing (AM), i.e., the layer-by-layer construction of devices using a computer-aided design (CAD) file, has been recently explored as a manufacturing toolbox for MEMS. The demonstration of monolithic multi-material devices in 3-D printed MEMS has the potential to implement better, more complex, and more capable microsystems at a small fraction of the time and cost typically associated with semiconductor cleanroom microfabrication. Fused filament fabrication (FFF) is an AM technique based on extrusion of thermoplastic polymers that is arguably the simplest and cheapest commercial 3-D printing technology available.

Here, we report additively manufactured monolithic microsystems composed of conductive and dielectric layers using an FFF dual extruder 3-D printer. The base material is a biocompatible polymer, polylactic acid (PLA), which can be doped with micro/nanoparticles to become electrically conductive. Characterization of the printing technology demonstrates close resemblance between CAD files and printed objects, generation of watertight microchannels, high-vacuum compatibility, and non-cytotoxicity. A large (~23) piezoresistive gauge factor was measured for a certain graphite-doped conductive PLA, suggesting its utility to implement 3-D printed strain transducers via FFF. Multiplexed electrohydrodynamic liquid ionizers (Figure 1) with integrated extractor electrode and threaded microfluidic port were also demonstrated. The per-emitter current vs. per-emitter flowrate characteristic shows a power dependence with 0.6 coefficient (Figure 2), close to the square-root dependence predicted by de la Mora's law for the cone-jet emission mode.



▲ Figure 1: CAD schematic and selected close-up views of FFF-printed 7-emitter electrospray array with hydraulics made of dielectric PLA and extractor electrode made of graphite-doped conductive PLA.



▲ Figure 2: Per-emitter current vs. flow rate characteristic for a fully-printed 7-emitter array with integrated extractor electrode and microfluidic port.

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Luis Fernando Velásquez-García
Principal Research Scientist
Microsystems Technology Laboratories

Micro- and nano-enabled multiplexed scaled-down systems that exploit high electric field phenomena; powerMEMS; additively manufactured MEMS/NEMS. Actuators, cold cathodes, nanosatellite propulsion, ionizers, microplasmas, portable mass spectrometry, pumps, sensors, X-ray sources.

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