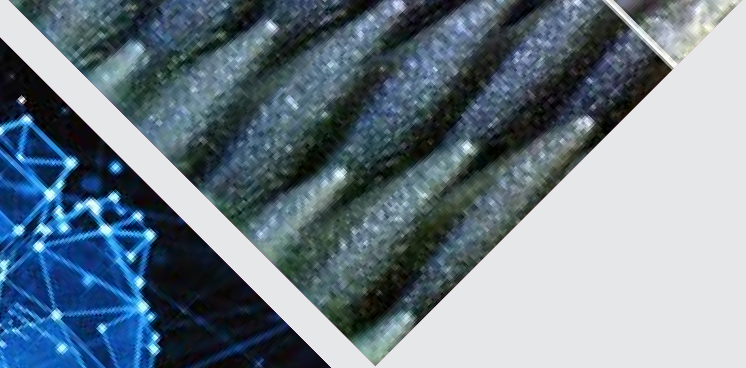
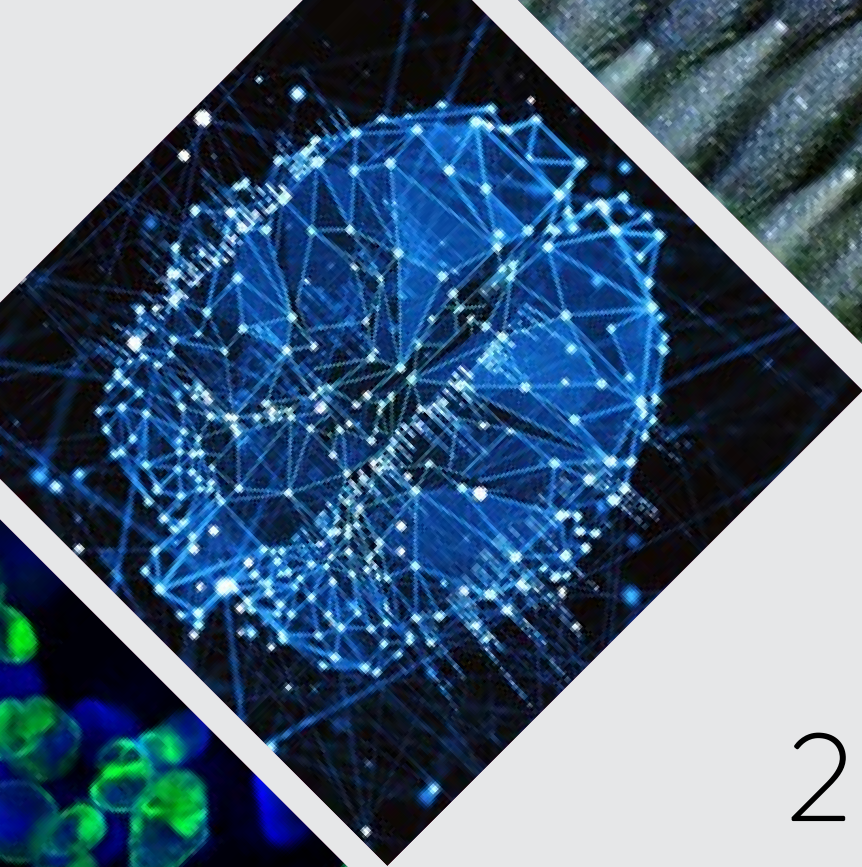


2020 Annual Research Report

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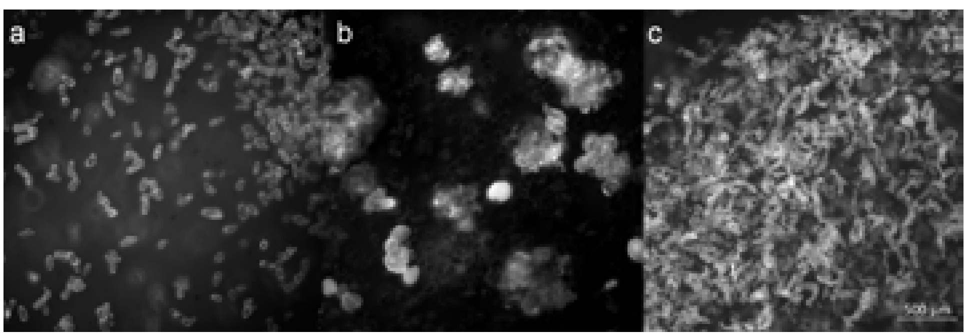
Tuning Plant Cell Culture Parameters for Improved Model Physiologies

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

In vitro plant culture models provide valuable insights into factors governing plant growth and development. Improved understanding of genetic and biochemical pathways in plants has facilitated advancements in a variety of industries —from guiding the development of more robust crops, to enabling increased biofuel yields by tuning biomass genetics. Despite the utility of plant culture models, translation of cellular findings to the plant-scale is hindered in current culture systems. These limitations are, in part, because culture systems fail to recapitulate physical aspects of the natural cellular environment. This work investigates the role of extra-cellular mechanical and chemical influences such as scaffold stiffness, hormone concentrations, media pH, and cell density on cell development and growth patterns. Early results indicate that tuning of biomechanical and biochemical cues leads to cell growth which deviates from typical culture morphologies and better resembles natural plant tissue structures.

New analytical methods and measurement metrics were developed to monitor cell enlargement, elongation, and differentiation in response to altered culture conditions. Through factorial design of experiments, optimal conditions for maintenance of long-term cell viability or elevated differentiation rates have been identified. Maps of cell response over a range of extracellular conditions allows for tuning of plant cell models to allow for the exhibition of desired physiological compositions. With the aid of these new data maps, plant tissues which are traditionally difficult to access or study in real-time can be better replicated for study in the laboratory setting.



▲ Figure 1: Zinnia elegans cells (a) shortly after isolation from leaves; cells exposed to varied growth conditions may grow into patterns of (b) bulbous, cell aggregates, or (c) uncoordinated, elongated cells.

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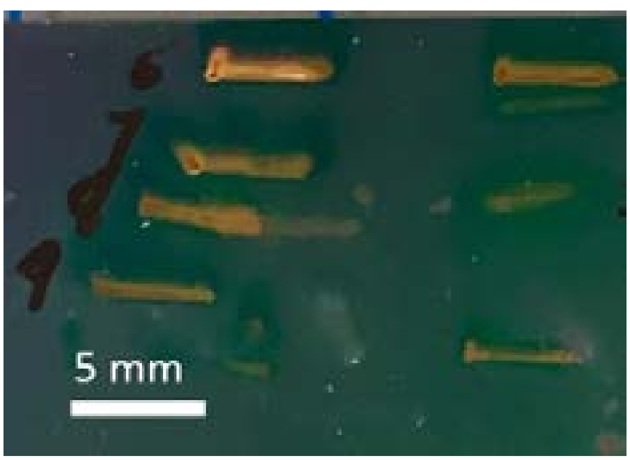
Increasing the Yield of Atmospheric Pressure Microsputtering for Fabrication of Agile Electronics

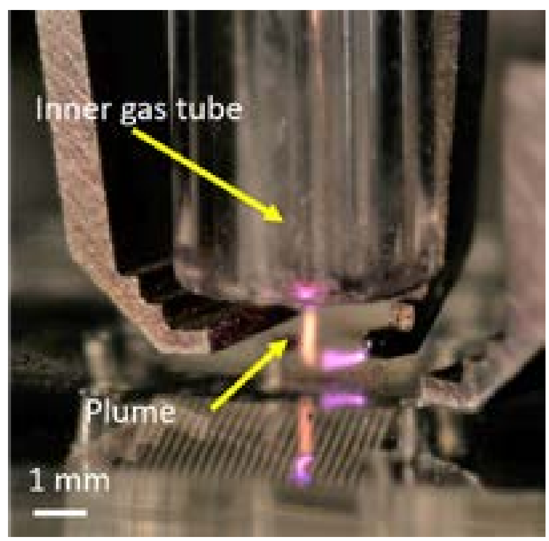
Y. Kornbluth, R. Matthews, L. Parameswaran, L. M. Racz, L. F. Velásquez-García
Sponsorship: U.S. Air Force

Additive manufacturing (AM) promises new, flexible production; however, while AM excels at creating structural parts, it cannot make functional objects well, e.g. multi-material structures such as electronic components and circuits. Sputtering, which removes material from a target atom-by-atom by using a plasma, is used in IC fabrication finely layered, multi-material fabrication. By miniaturizing the dimensions of the plasma reactor down to sub-millimeter scale, the sputterer can operate at atmospheric pressure, obviating the need for a vacuum. However, at atmospheric pressure, collisions with gas molecules scatter most of the sputtered material, preventing it from reaching the substrate.

We develop plasma microsputterer technology that allows for high-resolution, high-quality deposition of arbitrary patterns, without any templates, pre-, or post-processing; recent results with a gold target include creating imprints with electrical conductivity within an order of magnitude to that of bulk metal. We explore two methods to minimize sputtered material scattering and to increase the deposition rate (yield). The first method minimizes the gap between the sputtering target and the substrate (Fig. 1): the sputtering target is placed 150 μm above the substrate. Dielectric barriers confine the plasma, forcing the plasma to connect the target wire and anode without damaging the substrate. This approach yields 0.2 nm/s (40 pg/s)—twice previous results. However, significant substrate heating occurs, which is incompatible with temperature-sensitive substrates. The second method harnesses convection to drive the sputtered material towards the substrate (Fig. 2). We surround the microsputter target (100 μm diameter) with a strong jet of air (100 m/s, 0.5 mm thick coaxial flow) to force air molecules to transport the sputtered material. This method greatly increases the yield (1 nm/s, 20 ng/s)—30% of the sputtered material reaches the substrate. Current work focuses on further increasing the deposition rate by increasing the rate at which atoms are sputtered.



▲Figure 1: A set of lines printed on a substrate using a gold microsputterer with a small gap (150 μm) between the target and the substrate.



▲Figure 2: Setup with coaxial gas plume; the gas plume (orange) carries ionized air and sputtered gold down to the substrate. We cannot detect any meaningful substrate heating. The purple parts of the plasma are due to high argon and gold content in those areas.

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3D-Printed, Miniature, Multi-material, Valve-less, Magnetically Actuated Liquid Pumps

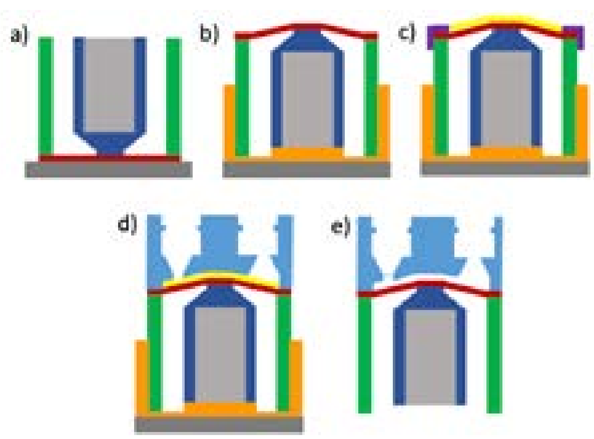
A. P. Taylor, L. F. Velásquez–García
Sponsorship: Edwards Vacuum

Miniaturized pumps can be used to supply precise flow rates of liquid in compact systems. Numerous micro-fabricated positive displacement pumps for liquids with chamber volumes that are cycled using valves have been proposed. Pumps made via standard (i.e., cleanroom) micro-fabrication typically cannot deliver large flow rates without integrating hydraulic amplification or operating at high frequency due to their small pump chambers.

3D-Printing has recently been explored as a processing arena for microsystems; in particular, researchers have reported 3D printed pumps for liquids and gases with performance on par with or better than counterparts made with standard microfabrication.

Building on earlier work on printed magnetically actuated liquid pumps, we 3D-printed multi-material, magnetically driven, valve-less miniature liquid pumps. We used the fused filament fabrication (FFF) method: a thermoplastic filament is extruded from a hot nozzle to create, layer by layer, a solid object. The body of the pump is printed in Nylon 12, while the actuation magnet is printed in Nylon 12 containing NdFeB micro-particles. The devices are driven by a non-contact rotating magnet and employ valve-less diffusers to greatly simplify operation.

Our low-cost, leak-tight, miniature devices are microfabricated using 150- and 225-µm layers with a multi-step, multi-material printing process (Figure 1) that monolithically creates all key features with <13-µm in-plane misalignment. Each pump has a frame, a 225-µm-thick membrane connected to a piston with an embedded magnet, a chamber, two diffusers, and two fluidic connectors (Figure 2). Fabrication of the pump requires under 75 minutes and costs less than \$3.89. Finite element analysis of the actuator predicts a maximum stress of 15.7 MPa @ 100 µm deflection, i.e., below the fatigue limit of Nylon 12 for infinite life (i.e., 19 MPa). Water flow rate up to 1.68 ml/min at an actuation frequency of 204 Hz was measured.



▲Figure 1: Process flow to FFF-print liquid pump: a) print pump's membrane (red), piston (blue), frame (green), and magnet (gray). b) While membrane is warm, deform it using a mandrel (orange). c) Place shadow mask (purple) on membrane and apply PVAc film (yellow); d) Remove mask and print chamber ceiling, valves and fittings; e) Dissolve PVAc film.

▲Figure 2: Clockwise from top left: CAD model cross-section of pump; side view of printed pump; top view of printed pump; bottom view of printed pump showing the printed magnet. The scale bar applies to all printed objects.

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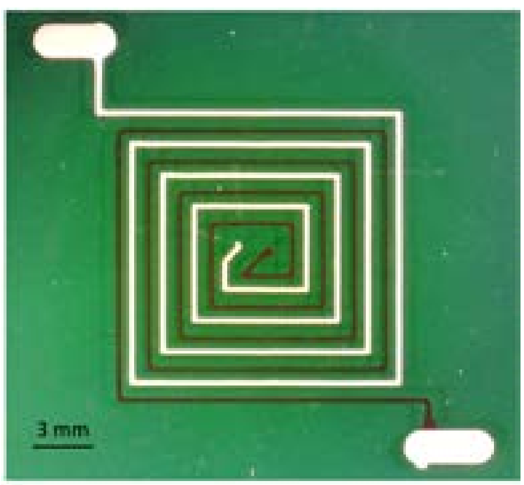
In-plane Gated Field Emission Electron Sources via Multi-material Extrusion

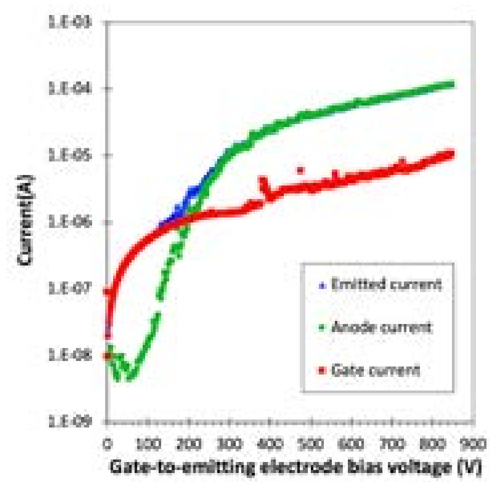
I. A. Perales-Martínez, L. F. Velásquez-García
Sponsorship: MIT-Tecnologico de Monterrey Nanotechnology Program

Field emission is the quantum tunneling of electrons to vacuum due to local high electrostatic fields; such high fields can be generated at a moderate voltage using nanosharp, high-aspect-ratio tips. Compared to thermionic counterparts, field emission cathodes consume less energy, respond faster, and can operate in poorer vacuum, making them attractive in compact applications such as nanosatellite electric propulsion, portable mass spectrometry, and handheld X-ray generation. A wide variety of materials has been explored as field emitters; the research in field emission electron sources has focused on carbon nanotubes (CNTs) due to their nanosized tip diameter, high aspect-ratio, high electrical conductivity, and excellent chemical stability. However, most manufacturing methods for CNT field emission electron sources have associated large cost, long processing time, need of static masks for defining in specific locations the nanostructured emitting material and/or the electrode(s), and large gate interception (or the need for advanced assembly methods to attain high transmission).

In this project, we are developing low-cost field emission cathodes via multi-material extrusion. The devices are flat plates with two concentric imprints (Figure 1): an imprint made of CNTs (emitting electrode), symmetrically surrounded on both sides by an imprint made of Ag microparticles (extractor gate). Unlike the great majority of field emission cathodes reported that have an out-of-plane gate electrode, our devices have an in-plane gate that significantly reduces the cost and manufacturing complexity of the device and also facilitates high gate transmission. Our devices can emit electrons in vacuum with as little as 62 V applied between the CNT imprint and the Ag imprint and achieve over 97% gate transmission (Fig. 2). Current work focuses on increasing the imprint density to attain larger current density emission and on developing ballasting structures for attaining large and uniform array emission.



▲Figure 1: Optical top view of a square-loop field emission source with 700-μm pitch between adjacent traces



▲Figure 2: Emitted, gate, and collected currents vs. bias voltage for the device shown in Figure 1.

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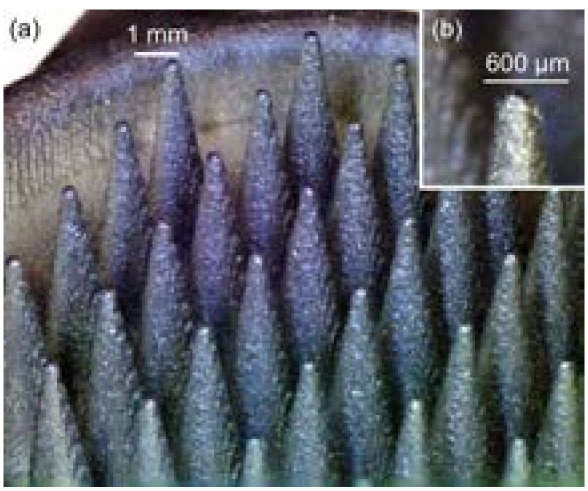
Additively Manufactured, Miniature Electrohydrodynamic Gas Pumps

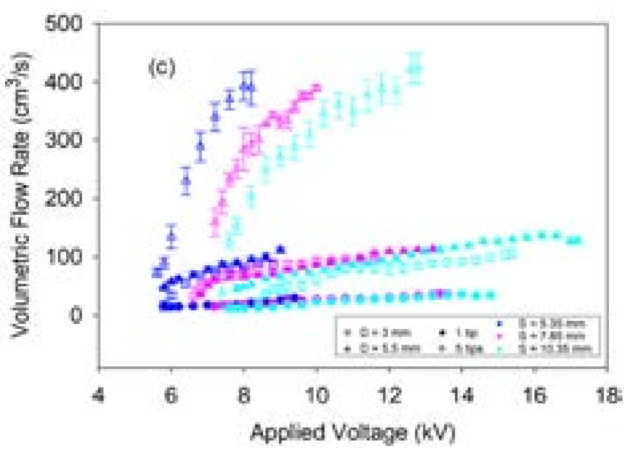
Z. Sun, L. F. Velásquez-García
Sponsorship: MIT-Skoltech Next Generation Program

A corona discharge is a self-sustained physical phenomenon induced around the sharper electrode of a diode due to sharply nonuniform electric fields within the interelectrode space. Ion propagation across such a space is accompanied by collisions with neutral particles, resulting in bulk fluid movement known as ionic wind. In contrast to traditional counterparts, ionic wind pumps have no moving parts, respond faster, and produce significantly less noise, drawing great interest in applications such as air propulsion and electronics cooling. Currently, ionic wind pump technology is far from practical in applications that require large flow velocity, flow rate, and power efficiency; another concern is the stability of the pump, given that ion accumulation in the interelectrode space can cause an electric short during sustained operation. Researchers have proposed using active electrodes with a plurality of field enhancers arranged in parallel (multiplexing) to maximize throughput; however, the reported multi-needle devices are serially assembled, and their performance is inferior to that of single-needle counterparts.

This project uses metal additive manufacturing and electropolishing to create miniature, multi-needle ionic wind pumps. Our devices are needle-ring corona diodes composed of a monolithic inkjet binder-printed active electrode (Figure 1), made in stainless steel 316L, with a plurality of sharp, conical needles and a thin plate copper counter-electrode, with electrochemically etched apertures aligned to the needle array. Five-needle ionic wind pumps eject air at 2.9 m/s and at a volumetric flow rate of 343 cm3/s, three times larger than the flow rate of a single-tip device with comparable efficiency (Figure 2). Current work systematically studies the relevant parameters to optimize the design of the electrohydrodynamic pump.



▲Figure 1: Field view (a) and tip close-up (b) of an as-printed 32-tip active electrode; the structure is binder inject-printed in stainless steel 316L.



▲Figure 2: Volumetric flow rate vs. bias voltage for 1-needle and 5-needle ionic wind pumps with different counter-electrode aperture D and inter-electrode separation S.

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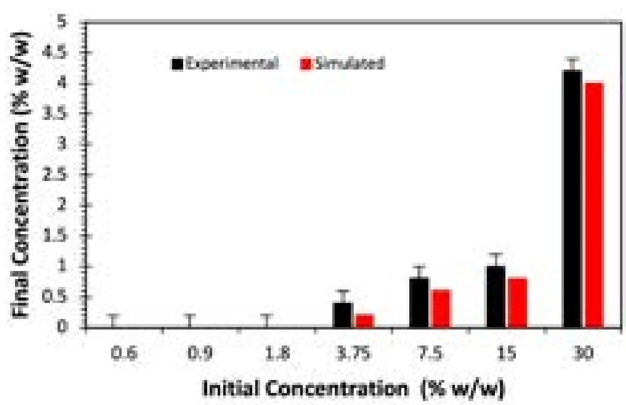
3D-Printed Silver Catalytic Microreactors for Efficient Decomposition of Hydrogen Peroxide

E. Segura-Cardenas, L. F. Velásquez-García
Sponsorship: MIT-Tecnologico de Monterrey Nanotechnology Program

Microreactors increase the surface-to-volume ratio of their reactants and by-products, resulting in faster, more efficient reactions and better heat transfer than in their non-miniaturized counterparts, leading to higher throughput per unit of reactor active volume and to better selectivity in the species produced by the reactor. The great majority of microreactors are made of polydimethylsiloxane (PDMS)—a material that cannot operate at elevated pressures or temperatures. Other reported microreactors are made in silicon, ceramics, or metals; although these materials are compatible with high-pressure and high-temperature operation, they have associated a very high production cost because they are made in a semiconductor cleanroom or with specialized, low-throughput tooling, e.g., electro discharge machining.

Hydrogen peroxide (H_2O_2), a water-soluble oxidant, spontaneously decomposes in the presence of heat or a catalyst. Applications of a H_2O_2 catalytic reactor include monopropellant rocket propulsion, steam generators, and pumping; miniaturized versions of such catalytic reactors are of great interest to PowerMEMS. Here, we developed a novel additive manufacturing technique based on silver clay extrusion to create high-pressure compatible and high-temperature compatible, monolithic microfluidics; silver is also a very efficient and effective catalyst for the decomposition of H_2O_2. Our microreactors are composed of a water-tight microchannel connected to the exterior via two fluidic ports (Figure 1). The experimental performance of the microreactor as a catalytic decomposer of H_2O_2 matches well our reduced-order modeling estimates (Figure 2), attaining a decomposition efficiency of 87% for a flow rate of 5 μL/min of H_2O_2 with an initial concentration of 30% w/w. Current research focuses on exploring other applications, e.g., heat exchangers.

▲Figure 1: Apparatus for experimental characterization of 3D-printed silver catalytic microfluidics using H_2O_2 (top); 3D-printed, monolithic silver microfluidic with a 350-μm-wide, 2.5-cm-long, and 350-μm-tall microchannel and inlet and outlet fluidic ports (bottom).



▲Figure 2: Final H_2O_2 concentration versus initial concentration using a 3D-printed silver microfluidic prototype. Reduced-order modeling simulations and experimental results agree.

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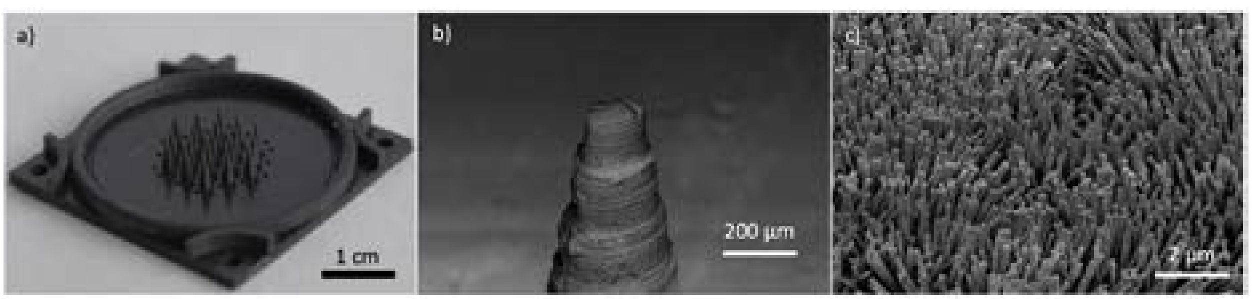
Nanostructured, Additively Manufactured, Miniature Ionic Liquid Ion Sources

D. V. Melo-Máximo, L. F. Velásquez-García
Sponsorship: MIT-Tecnológico de Monterrey Nanotechnology Program

Electrospraying is a high-electric field physical phenomenon that transforms electrically conductive liquids into fine, uniform streams of micro/nanoparticles; the applications of electrospraying include mass spectrometry, nanosatellite propulsion, combustors, and agile manufacturing. Unfortunately, electrospray emitters have very low throughput; consequently, several research groups have investigated, for about two decades, greatly increasing the electrospray source's throughput via emitter multiplexing, using micro- and nanotechnology to attain lower startup voltage and denser emitter arrays. Although successful, the reported implementations harness cleanroom microfabrication, which has an associated high cost that is incompatible with many applications of electrospraying. In this project, we explore the use of additive manufacturing to create, at a very low cost, monolithic arrays of electrospray emitters capable of ion emission.

We have succeeded at demonstrating the first additively manufactured ionic liquid electrospray sources in the literature; our devices produce per-emitter current comparable to that produced by silicon microfabricated counterparts, at a small fraction of their fabrication cost. The devices are diodes composed of an emitting electrode and an extractor electrode: the emitting electrode is a monolithic array of digital light projection (DLP)-printed solid, conical, polymeric needles covered by a conformal layer of hydrothermally grown zinc oxide (ZnO) nanowires as a wicking material (Figure 1), while the extractor electrode is a laser-cut SS 316L plate with an array of apertures that matches the pattern of the array of needles. Characterization of the devices in vacuum using the ionic liquid EMI-BF_4 demonstrates bipolar, uniform array emission of solvated ions—in agreement with the literature on ionic liquid ion sources. Current research efforts focus on increasing the number of emitters per unit of area and on exploring other materials and designs for implementing the devices.



▲ Figure 1: Selected views of an additively manufactured electrospray source with 19 emitters in 1 cm^2 of active area: a) emitting electrode made in DLP-printable resin; b) close-up SEM image of one tip of the array, showing conformal growth of a ZnO nanowire forest; c) close-up SEM of ZnO nanowire forest (the material that transports the ionic liquid from the reservoir to the emission sites at the tips of the emitters), showing that the forest is composed of uniform, 150 nm-diameter nanowires.

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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, ionizers, microfluidics, microplasmas, nanosatellite propulsion, portable mass spectrometry, pumps, sensors, X-ray sources.

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SELECTED PUBLICATIONS

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