

ANNUAL RESEARCH REPORT 2019

MICROSYSTEMS TECHNOLOGY LABORATORIES
MASSACHUSETTS INSTITUTE OF TECHNOLOGY

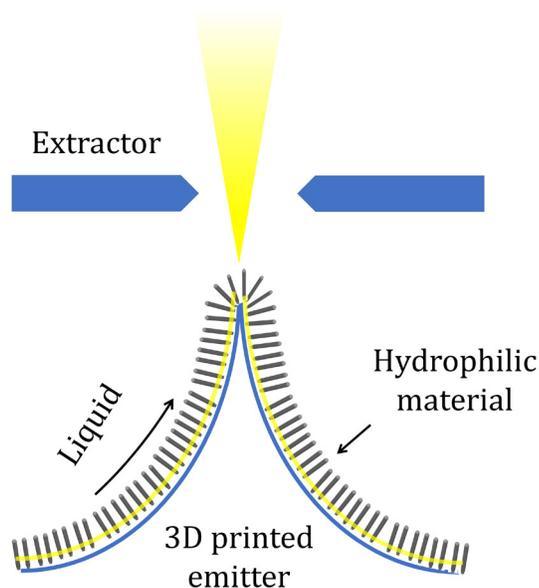
Additively Manufactured Externally-fed Electro spray Sources

D. V. Melo-Máximo, S. Lin, and L. F. Velásquez-García
Sponsorship: MIT-Tecnologico de Monterrey Nanotechnology Program

Additive manufacturing (AM) is a layer-by-layer fabrication technique that creates solid objects by putting material where needed, instead of removing material from stock. Recent advances in AM have made possible the implementation of microsystems that surpass the performance of state-of-the-art counterparts made in a clean room, as well as the demonstration of devices that are challenging or unfeasible to create using standard microfabrication—particularly in the area of microfluidics. In addition, AM is inherently compatible with implementing, with great precision, hierarchical structures with features spanning orders of magnitude in size to accomplish multiple tasks efficiently.

In this project, we are exploring AM to develop, at a low-cost, massively multiplexed externally-fed electrohydrodynamic liquid ionizers (Figure 1) for a wide range of applications such as mass spectrometry,

nanosatellite propulsion, species transport, and agile manufacturing. These devices are mesoscaled arrays of high-aspect-ratio, hundreds-of-microns tall, micron-sharp tips that are conformally covered with a nanostructured layer that transports and regulates the flow of liquid from the reservoir to the emission sites. Manufacturing issues such as inter-process compatibility and tip array uniformity need to be addressed to implement devices that operate efficiently successfully. Current work focuses on exploring and optimizing various manufacturing techniques to monolithically create the electro spray source out of different structures made of different materials; future work includes assessment of device performance, e.g., emission characteristics and uniformity.



▲ Figure 1: Schematic of an externally-fed emitter

FURTHER READING

- D. I. Wimpenny, P. M. Pandey, and L. J. Kumar, "Advances in 3D Printing & Additive Manufacturing Technologies," Springer, 2017.
- B. Gassend, L. F. Velásquez-García, A. Ibitayo Akinwande, and M. Martínez-Sánchez, "A Microfabricated Planar Electro spray Array Ionic Liquid Ion Source with Integrated Extractor," *J. of Microelectromechanical Systems*, vol 18, Issue: 3, pp. 679–694, 2009.
- F. A. Hill, E. V. Heubel, P. J. Ponce de Leon, and L. F. Velásquez-García, "High-throughput Ionic Liquid Ion Sources using Arrays of Microfabricated Electro spray Emitters with Integrated Extractor Grid and Carbon Nanotube Flow Control Structures," *J. of Microelectromechanical Systems*, vol. 23, no. 5, pp. 1237–1248, Oct. 2014.

Additive Manufacturing of Microfluidics via Extrusion of Metal Clay

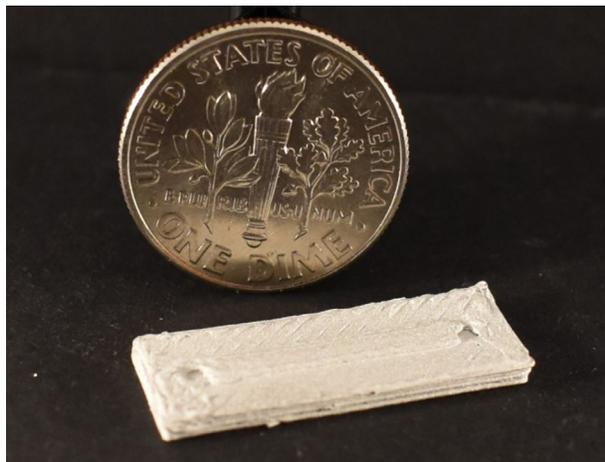
E. Segura-Cardenas, L. F. Velásquez-García
Sponsorship: MIT-Tecnologico de Monterrey Nanotechnology Program

Most microfluidics uses closed microchannels to efficiently accomplish tasks such as species mixing, heat transfer, and particle sorting by increasing the surface-to-volume ratio of the fluid(s) involved in the process. However, the current manufacturing techniques for microfluidics present disadvantages such as high-cost, long production time, no device customization, elaborated design iteration, restriction in the kinds of structures that can be made, and low fabrication yield.

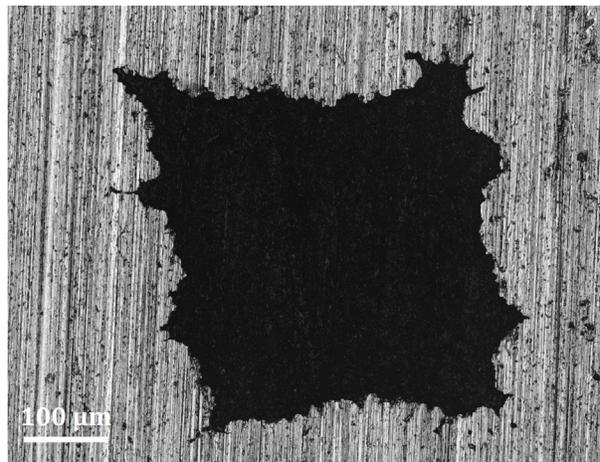
Recent research results demonstrate that additive manufacturing can readily address the shortcomings outlined, often yielding devices that surpass the state of the art or for which traditional microfabrication creates no counterpart. However, most 3D-printed microfluidics are made of polymeric feedstock, which is not compatible with high-pressure and/or high-temperature applications. Mainstream 3D printing methods for metal include lost-wax micro molding, inkjet binder, and direct metal laser sintering; these

processes are unideal to produce monolithic closed-channel microfluidics because they either require internal dummy structures or create internal voids filled in with unprocessed printable material, both of which are challenging to remove from the printed part.

In this project, we are exploring the use of extrusion of metal clay to implement closed-channel microfluidics; the technique is arguably similar to fused filament fabrication and can readily create voids without spurious infill or post-processing required. Via the extrusion of metal clay, leak-tight metal microchannel with monolithic, working ports have been created (Figure 1). A cross-section of the microfluidic shows an unclogged microchannel, evidencing the feasibility of the technique to create closed channels with hydraulic diameters of relevance to microfluidics (Figure 2). Current work focuses on exploring the design space of the technology and demonstrating an application of relevance.



▲ Figure 1: Extruded monolithic microfluidic with 350- μm wide and 350- μm tall microchannel and inlet and outlet ports.



▲ Figure 2: Cross section of closed microchannel.

FURTHER READING

- R. Frykholm, Y. Takeda, B. G. Andersson, and R. Carlström, "Solid State Sintered 3-D Printing Component by using Inkjet (Binder) Method," *J. of the Japan Society of Powder and Powder Metallurgy*, vol. 63, no. 7, pp. 421–426, 2016.
- Z. Sun, G. Vladimirov, E. Nikolaev, and L. F. Velásquez-García, "Exploration of Metal 3-D Printing for the Microfabrication of Freeform, Finely Featured, Mesoscaled Structures," *J. of Microelectromechanical Systems*, vol. 27, no. 6, pp. 1171 - 1185, Dec. 2018.
- S. Marre, J. Baek, J. Park, M. G. Bawendi, and K. F. Jensen, "High-Pressure/High-Temperature Microreactors for Nanostructure Synthesis," *JALA: J. of the Association for Laboratory Automation*, vol. 14, no. 6, pp. 367–373, Dec. 2009.

3D-Printed, Low-cost, Miniature Liquid Pump

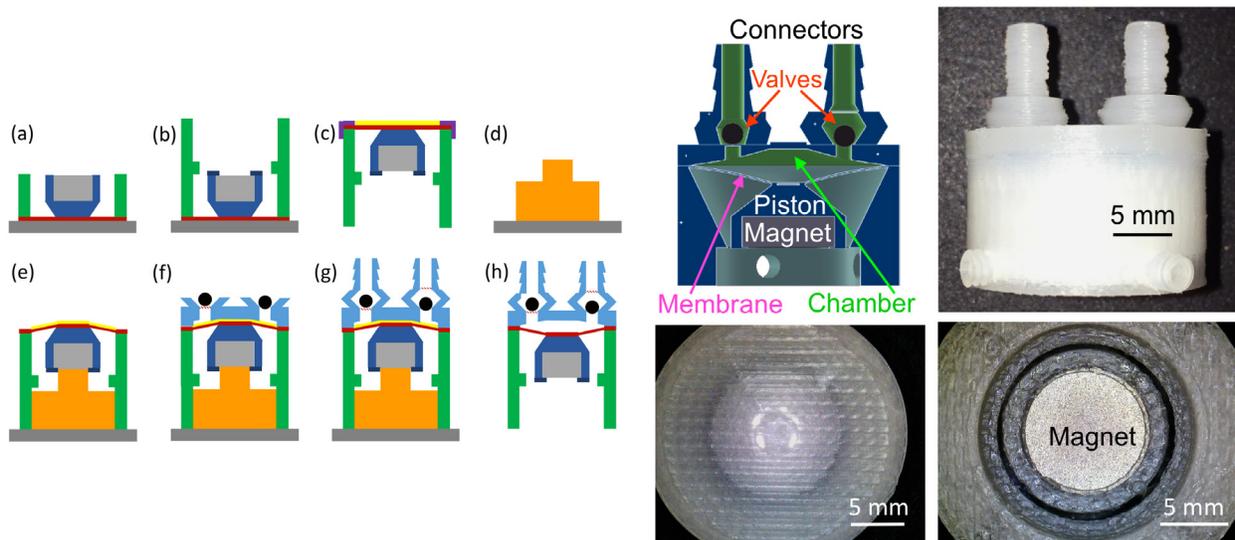
A. P. Taylor, L. F. Velásquez-García
Sponsorship: Edwards Vacuum

Many compact systems use pumps to precisely set flow rates of liquid or, in general, to manipulate small liquid volumes for effective mass transport, cooling, or momentum transfer. Numerous microfabricated positive displacement pumps for liquids with chamber volumes that are cycled using valves have been proposed. Pumps made via standard (i.e., cleanroom) microfabrication typically cannot deliver large flow rates without integrating hydraulic amplification or operating at high frequency due to their small pump chambers.

Additive manufacturing, i.e., the layer-by-layer fabrication of objects using as template a computer-aided design model, has recently been explored as a processing arena for microsystems. In particular, researchers have reported 3-D printed pumps for liquids and gases with performance on par or better than counterparts made with standard microfabrication. Building upon earlier work on printed MEMS magnetic actuators, we recently developed miniature liquid pumps printed in pure nylon 12 via fused filament

fabrication (FFF) whereby a thermoplastic filament is extruded from a hot nozzle to create a solid object layer by layer.

Our low-cost, leak-tight, miniature devices are microfabricated using 150- to 300- μm layers with a multi-step printing process (Figure 1) that monolithically creates all key features with $<13\text{-}\mu\text{m}$ in-plane misalignment. Each pump has a rigid frame, a 21-mm-diameter, 150- μm -thick membrane connected at its center to a piston with an embedded magnet, chamber, passive ball valves, and two barbed fluidic connectors (Figure 2). Pump fabrication under 2 hours and costs less than \$4.65 are achieved. Finite element analysis of the actuator predicts a maximum stress of 18.7 MPa @ 2-mm deflection, about the fatigue limit of nylon 12 (i.e., 19 MPa). A maximum water flow rate of 1.37 ml/min at 15.1 Hz actuation frequency is calculated, comparable to reported values of miniature liquid pumps with up to 200X higher actuation frequency.



▲ Figure 1: Half is printed (a)-(b) with membrane (red) and inserted magnet (grey). Partial pump is prepared, (c)-(e); second half printed on top, (f)-(h) with chamber (light blue), valve grids (red hatch), inserted balls (black).

▲ Figure 2: Clockwise from upper left: cross section of pump model; side view of printed pump; bottom view of printed pump with embedded SmCo magnet; top view of pump membrane, prior to printing second half.

FURTHER READING

- A. P. Taylor and L. F. Velásquez-García, "Miniaturized Diaphragm Vacuum Pump by Multi-material Additive Manufacturing," *J. Microelectromech. Syst.*, vol. 26, no. 16, pp. 1316-1327, Dec. 2017.
- A. P. Taylor and L. F. Velásquez-García, "High-temperature Compatible, Monolithic, 3D-Printed Magnetic Actuators," *J. Phys. Conf. Ser.*, vol. 1052, 012046 (4 pp), Jul. 2018.
- A. P. Taylor and L. F. Velásquez-García, "Low-cost, Monolithically 3D-Printed, Miniature High-flow Rate Liquid Pump," *PowerMEMS 2018 Conference Proceedings*, Dec. 2018.

3D-Printed Microfluidics to Evaluate Immunotherapy Efficacy

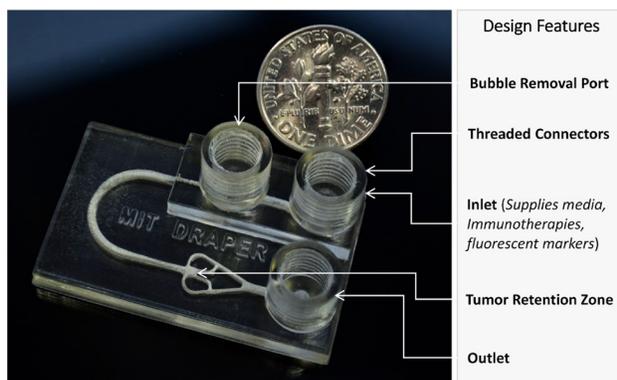
A. L. Beckwith, J. T. Borenstein, L. F. Velásquez-García
Sponsorship: Draper

Microfluidic devices are conceptually an ideal platform for the provision of personalized medical evaluations as they require small analyte volumes and facilitate rapid and sensitive investigations. However, 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.

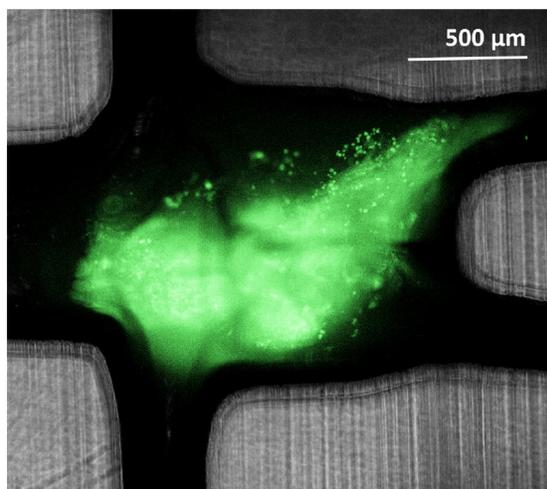
We have developed an entirely 3D-printed microfluidic platform that enables modeling of interactions between tumors and immune cells, providing a microenvironment for testing the efficacy of immunotherapy treatment. The monolithic platform allows for real-time analysis of interactions between a resected 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 vs. traditionally-fabricated microfluidic systems. The 3D-printed device sustains viability of biopsied tissue fragments under dynamic perfusion for at least 72 hours while enabling simultaneous administration of drug treatments, illustrating a useful tool for drug development and precision medicine for immunotherapy. Confocal microscopy of the tumor tissue and resident lymphocytes in the presence of fluorescent tracers provides real-time monitoring of tumor response to various immunotherapy. The platform and accompanying analysis methods distinguish between a positive immune response and a lack of tissue response in the presence of immunotherapeutic agents.

This platform introduces novel methodologies in modeling and analyzing tumor response to improve prediction of patient-specific immunotherapy efficacy. To the best of our knowledge, this is the first report of human tumor fragments cultured in a dynamic perfusion system capable of testing the effect of circulating immune checkpoint inhibitors on resident tumor-infiltrating lymphocytes.



▲ Figure 1: An optical picture of a 3D-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.

FURTHER READING

- A. L. Beckwith, J. T. Borenstein, and L. F. Velásquez-García, "Monolithic, 3D-Printed Microfluidic Platform for Recapitulation of Dynamic Tumor Microenvironments," *Journal of Microelectromechanical Systems*, vol. 27, No. 6, pp. 1009-01022, Dec. 2018.
- A. L. Beckwith, L. F. Velásquez-García, and J. T. Borenstein, "Microfluidic Model for Evaluation of Immune Checkpoint Inhibitors in Human Tumors," *Advanced Healthcare Materials*, 2019.

Electrohydrodynamic Printing of Ceramic Piezoelectric Films for High-frequency Applications

B. García-Farrera, L. F. Velásquez-García

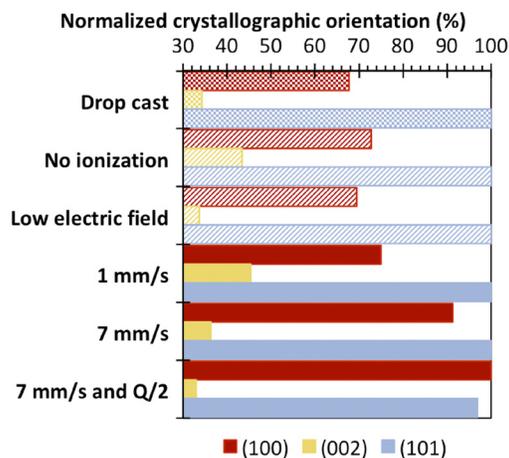
Sponsorship: MIT-Tecnológico de Monterrey Nanotechnology Program

The high operating frequencies that ceramic piezoelectric ultra-thin films attain have made possible exciting applications such as energy harvesting, telecommunications' filters, high sensitivity biosensors, and acoustofluidic devices; however, the inherently high cost and complexity of current manufacturing methods limit, in general, their widespread use. Additive manufacturing (AM), which has proven successful in creating complex devices and components of relevance to micro and nanosystems, could overcome these disadvantages; nevertheless, AM of piezoelectrics has been achieved only with polymer-based materials—unsuitable for said applications.

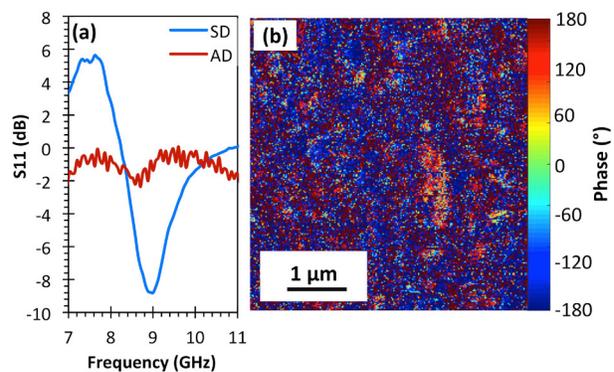
We report the first additively manufactured ceramic ultra-thin piezoelectric films compatible with high-frequency applications using electrohydrodynamic deposition (EHD) at room temperature. The films were made by electro spraying a zinc oxide (ZnO) nanoparticle liquid feedstock, directly writing line imprints as thin as 213 nm and as narrow as 198 μm .

We harness a previously unreported effect to

align the polar axis of the imprint and obtain overall piezoelectricity. As Figure 1a shows, the (100) orientation monotonically increases as the linear density of the deposition is reduced by increasing the raster speed or reducing the feedstock flow rate (Q)—provided two conditions are met: the feedstock is ionized (via EHD), and a small separation between emitter and substrate is used. Notably, the orienting effect directly acts on the direction of the polar axis by means of the rastering direction (Figure 2a), allowing for vibration modes and resonator configurations that were previously unfeasible. The macroscopic piezoelectric behavior is shown through piezoforce response microscopy (PFM) (Figure 2b) and the suitability for high-frequency applications was demonstrated by testing thin-film bulk acoustic resonators (FBAR) on a flexible polymer substrate, where the resonant frequency of ~ 5 GHz was used to calculate the acoustic speed of the films ($\sim 2,000$ m/s), which is close to the transversal wave speed of ZnO.



▲ Figure 1: Main crystallographic orientations for ZnO, normalized as a percentage of the highest peak. Fill patterns: reference (checkerboard), no impact on crystallography (dashes) and successful conditions (solid).



▲ Figure 2: (a) Frequency response of the reflection coefficient (S_{11}) for laterally excited FBARs with films made of traces printed in the same (SD) or alternating (AD) directions. (b) 2D phase mapping of PFM. All films were printed at 3 mm/s.

FURTHER READING

- B. García-Farrera and L. F. Velásquez-García, "Room Temperature Direct Writing of Ultrathin Zinc Oxide Piezoelectric Films via Near-field Electrohydrodynamic Jetting for High-frequency Flexible Electronics," [to be presented] Transducers 2019–EUROSENSORS XXXIII, Germany, Jun. 2019.
- A. P. Taylor and L. F. Velásquez-García, "Electrospray-printed Nanostructured Graphene Oxide Gas Sensors," *Nanotechnology*, vol. 26, pp. 505301:1–8, Nov. 2015.
- A. P. Taylor, C. V. Cuervo, D. P. Arnold, and L. F. Velásquez-García, "Fully 3D-Printed, Monolithic Magnetic Actuators for Low-cost, Compact Systems," *J. of Microelectromechanical Systems*, doi:10.1109/JMEMS.2019.2910215, 2019.

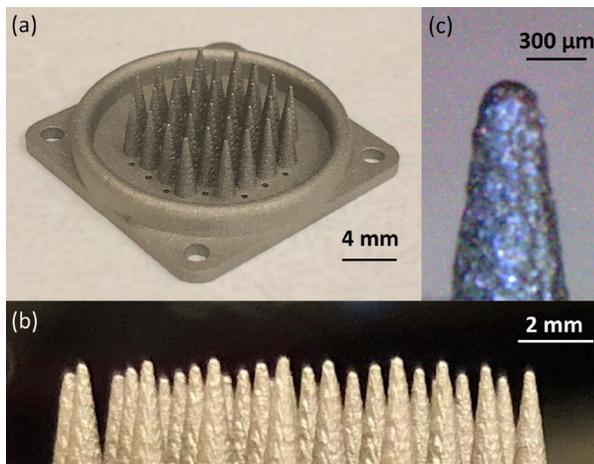
3D-Printed, Monolithic, Multi-tip MEMS Corona Discharge Ionizers

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

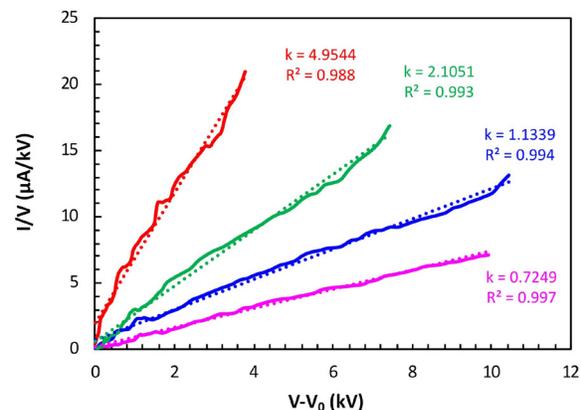
A corona discharge is a high-electric field ionization phenomenon caused by the development of a self-sustained electron avalanche between a sharp electrode (i.e., *corona* electrode) and a blunt electrode; the ions create a plasma region around the corona electrode and in their travel to the opposite electrode transfer momentum to the surrounding fluid. In this project, we are harnessing advanced metal inkjet printing technology to demonstrate massively multiplexed MEMS corona discharge ionizers (Figure 1), with the aim to increase greatly their ionization throughput and optimize their transduction mechanism to be able to implement exciting applications such as no-moving-parts pumps for gases and compact ion mobility spectrometers.

A 1D electrohydrodynamic coaxial cylinder model was implemented in COMSOL Multiphysics to study the ionization and collision processes in air at atmospheric pressure and room temperature of a 1-tip device, predicting a 400- μm -thick corona region surrounding the corona tip. The onset voltage estimated from the simulation is 5.849 kV, which is close to the theoretical value from Peek's formula of 6.416

kV. In addition, current over voltage (I/V) versus bias voltage minus the onset voltage ($V-V_0$) characteristics were collected for different ionizer array designs while varying the separation between the corona electrode and the collector electrode; the data follow the Townsend current-voltage model (Figure 2). Moreover, the data show that the corona current decreases with increased spacing of the corona electrode-to-collector electrode due to the decrease of the electric field on the tips; however, a smaller separation between the corona electrode and the collector electrode results in larger fluctuations in the corona discharge current. Devices with different numbers of tips tend to generate the same total corona current at the same bias voltage although more tips are set to discharge as the number of tips increases; this increase can be ascribed to the stronger interference between adjacent tips when the tip-to-tip spacing decreases. Current research efforts focus on optimizing the array design to minimize electric field shadowing and sharpening the tips to achieve operation at a lower bias voltage.



▲ Figure 1: Tilted view (a) and near-front view (b) of a binder ink-jet-printed SS 316L planar array with 32 high-aspect-ratio tips; close-up top view of a tip (c).



▲ Figure 2: I/V vs. $(V-V_0)$ characteristics for a 5-emitter ionizer with 7.95-mm (red), 11.12-mm (green), 14.30-mm (blue), and 17.47-mm (magenta) electrode separation.

FURTHER READING

- P. Ferrer and M. P. Tchonang, "Miniaturization of Electrostatic Ion Engines by Ionization and Acceleration Coupling," *J. Phys. D: Appl. Phys.*, vol. 44, no. 33, pp. 33520, Aug. 2011.
- Z. Sun, G. Vladimirov, E. Nikolaev, and L. F. Velásquez-García, "Exploration of Metal 3-D Printing for the Microfabrication of Freeform, Finely Featured, Mesoscaled Structures," *J. of Microelectromechanical Systems*, vol. 27, no. 6, pp. 1171–1185, Dec. 2018.
- Z. Sun and L. F. Velásquez-García, "Miniature, 3D-Printed, Monolithic Arrays of Corona Ionizers," *Technical Digest of the 18th International Conference on Micro and Nanotechnology for Power Generation and Energy Conversion Applications (PowerMEMS 2018)*, pp. 37, 2018.

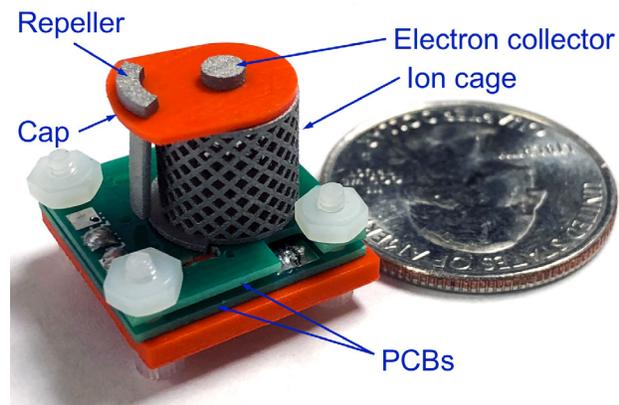
3D-Printed Gas Ionizer with CNT Cathode for Compact Mass Spectrometry

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

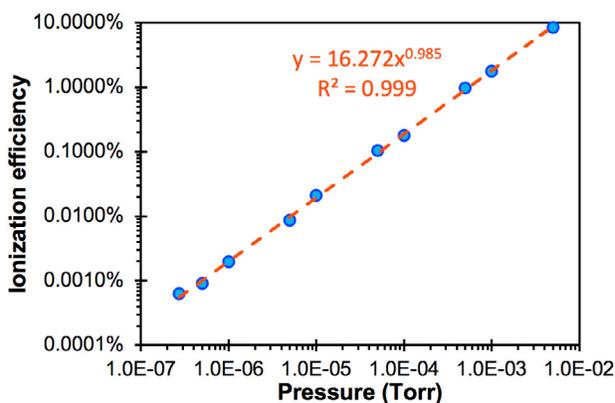
Mass spectrometers are powerful chemical analytical instruments used to quantitatively characterize the composition of unknown samples via ionization and mass-to-charge ratio species sorting. However, mainstream mass spectrometers are large, heavy, power hungry, and expensive, limiting their applicability in real-time and in-situ applications. Gas molecules can be ionized via electron impact ionization (EII), for which a source of electrons, i.e., a cathode, is required. Cold cathodes emit electrons into a vacuum via quantum tunneling due to high surface electric fields that lower and narrow the barrier that traps electrons within the material; typically, high-aspect-ratio, nano-sharp tips are used to produce such fields with moderate bias voltages. Compared to thermionic cathodes, field emission electron sources have faster response and less power consumption. Compared to other field emitters, carbon nanotubes (CNTs) are less affected by back-ion bombardment and chemical degradation. There are numerous reports of gas ionizers with CNT cathodes EII;

however, these devices are microfabricated using clean-room technology and/or use ion-generating structures machined with standard technologies, affecting their cost and size.

In this project, we are harnessing additive manufacturing (AM) to develop novel electron impact ionizers that circumvent these challenges. AM has unique advantages over traditional manufacturing methods including compatibility with creating complex 3D geometries, print customization, and waste reduction. Our design (Figure 1) uses inkjet binder printing of SS 316L to create electrodes to efficiently generate ions and steer charged species, stereolithography of polymer resin to define the dielectric structures that electrically isolate the different electrodes, and an additively manufactured CNT electron source. We have successfully characterized the ionizers at pressures as high as 5 mTorr while achieving ionization efficiencies as high as 8.5% (Figure 2).



▲ Figure 1: Fully additively manufactured electron impact gas ionizer with CNT cathode. Ion cage sets a region with uniform ionization cross section. Electron collector gathers electrons emitted by cathode and released during ionization. Ion repeller pushes ions out of ion cage. The cap interfaces with electrodes to create a 3D structure while providing electrical insulation.



▲ Figure 2: Ion current-to-transmitted electron current ratio versus pressure when field emission electron source of EII is biased at 700. There is linear dependence between pressure and ionization efficiency, in agreement with EII model.

FURTHER READING

- C. Yang and L. F. Velásquez-García, "Low-cost, Additively Manufactured Electron Impact Gas Ionizer with CNT Field Emission Cathode for Compact Mass Spectrometry," *J. of Physics D – Applied Physics*, vol. 52, no. 7, 075301, p. 9, Feb. 2019.
- L. F. Velásquez-García, B. Gassend, and A. I. Akinwande, "CNT-based MEMS Ionizers for Portable Mass Spectrometry Applications," *J. of Microelectromechanical Systems*, vol. 19, no. 3, pp. 484-493, Jun. 2010.
- B. Gassend, L. F. Velásquez-García, and A. I. Akinwande, "Precision In-plane Hand Assembly of Bulk-microfabricated Components for High-voltage MEMS Arrays Applications," *J. of Microelectromechanical Systems*, vol. 18, no. 2, pp. 332-346, Apr. 2009.

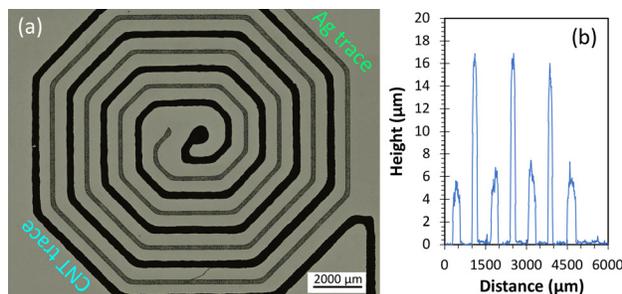
Printed CNT Field Emission Sources with Integrated Extractor Electrode

I. A. Perales-Martínez, L. F. Velásquez-García
Sponsorship: MIT-Tecnológico

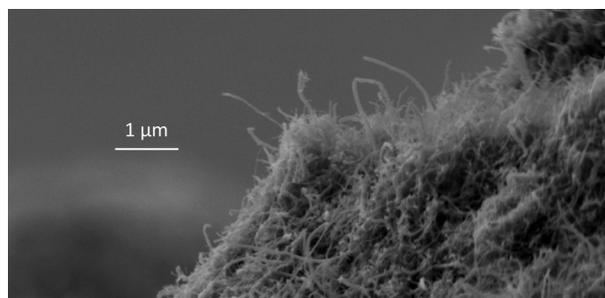
Field emission cathodes are promising electron sources for exciting applications such as flat-panel displays, free-electron lasers, and portable mass spectrometry where fast switching, low-pressure operation, and low power consumption are favored metrics. A field emitter quantum tunnels electrons to a vacuum due to the high electrostatic fields at its surface; this tunneling is typically done at low voltage using a whisker-like structure. Carbon nanotubes (CNTs) are attractive structures to produce electron field emission due to their ultrasharp tip diameter, high aspect ratio, high electrical conductivity, and excellent mechanical and chemical stability. Although CNT-based cold cathodes have been widely reported in the literature, their manufacture could be quite expensive (e.g., devices partially or fully made in a semiconductor cleanroom), or the extractor electrode of the cathode is an external mesh, causing high-beam interception (e.g., in screen-printed devices) or requiring an advanced method of assembly to the emitting component to achieve high transmission.

In this project, we are developing novel field emission sources that are fully additively manufactured

to circumvent the aforementioned challenges. The devices are made via direct ink write (DIW) printing, which is one of the least expensive and most versatile additive manufacturing methods as is capable of monolithic multi-material printing. Compared to screen printing, DIW does not involve static masks to transfer patterns and produces significantly less waste. The fully-printed field emission electron source is composed of two continuous imprints: a spiral trace made of a CNT compound, acting as an emitting electrode, symmetrically surrounded on both sides by a spiral trace made of silver nanoparticles, acting as in-plane extractor electrode (Figure 1). After printing, the CNT spiral receives a mechanical treatment that releases the CNT tips from the bulk of the imprint (Figure 2), enabling field emission from the CNT imprint. Characterization of the printed CNT field emission sources in triode configuration (i.e., using an external anode) shows low turn-on voltage and low interception of the emitted current by the extractor electrode. Current work focuses on design optimization and experimental characterization of the devices.



▲ Figure 1: Fully-printed CNT field emission source (a) and linear scan of structure in radial direction using confocal microscope (b). CNT traces are about 300 μm wide and 5 μm tall; Ag traces are about 200 μm wide and 16 μm tall.



▲ Figure 2: Close-up scanning electron micrograph of surface of CNT trace after mechanical treatment that releases CNTs from bulk is executed. Procedure creates grass-like structure suitable for field emission.

FURTHER READING

- C. Yang and L. F. Velásquez-García, "Low-cost, Additively Manufactured Electron Impact Gas Ionizer with CNT Field Emission Cathode for Compact Mass Spectrometry," *J. of Physics D – Applied Physics*, vol. 52, no. 7, 075301, p. 9, Feb. 2019.
- L. F. Velásquez-García, B. Gassend, and A. I. Akinwande, "CNT-based MEMS Ionizers for Portable Mass Spectrometry Applications," *J. of Microelectromechanical Systems*, vol. 19, no. 3, pp. 484-493, Jun. 2010.
- B. Gassend, L. F. Velásquez-García, and A. I. Akinwande, "Precision in-Plane Hand Assembly of Bulk-microfabricated Components for High voltage MEMS Arrays Applications," *J. of Microelectromechanical Systems*, vol. 18, no. 2, pp. 332-346, Apr. 2009.

Controlling the Nanostructure in Room-temperature-microsputtered Metal

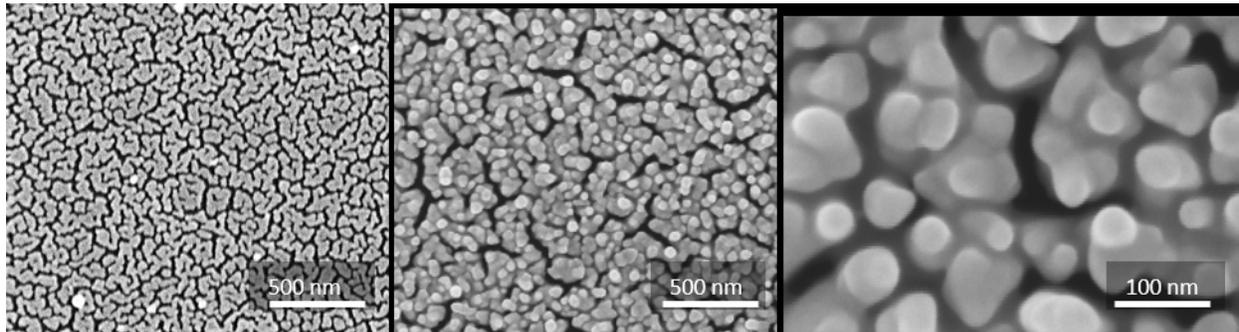
Y. Kornbluth, R. Matthews, L. Parameswaran, L. M. Racz, L. F. Velásquez-García
Sponsorship: MIT Lincoln Laboratory

Sputter deposition involves the ejection of atoms from a target and the atoms' subsequent deposition on a nearby substrate. Because the deposition is done on the atomic level, the nanostructure of the deposit can vary significantly. This variance is of concern, as it can greatly affect material strength and conductivity. Traditional sputtering relies on vacuum and thermal annealing to ensure dense, highly conductive deposits. However, agile manufacturing on temperature-sensitive substrates is not compatible with these two solutions.

To enable high-quality material without heating the material or requiring a vacuum, we performed a statistically-motivated set of experiments to determine what deposition parameters improve the material quality. We developed an empirical model and found that an appropriate electrical bias voltage, applied either to the substrate or to a conductive plate under the substrate, has the greatest impact on the material quality. This is due to the presence of charged nanoparticles, formed by collisions between sputtered

atoms in the dense plasma around the sputter target. The applied electric field attracts positively charged nanoparticles, allowing the nanoparticles to strike the substrate with more energy than their temperature alone would dictate. This extra energy enhances the mobility of the deposited metal, allowing it to form denser, more energetically favorable coatings (Figure 1) without significant substrate heating. With this technique, we have improved the conductivity of the sputter coating to 5x bulk metal ($15 \mu\Omega\text{-cm}$) at room temperature.

Applied electric fields also improve the coating's thickness. In the absence of electric fields, the sputtering process is self-limiting. As the positively charged sputtered material reaches the substrate, charge builds upon the substrate, repelling charged sputtered material and preventing the deposit from thickening. However, biasing the substrate with a negative voltage prevents this charge from accumulating, allowing for thicker ($> 200 \text{ nm}$) films.



▲ Figure 1: Deposit nanostructure without (left) and with (center, right) applied bias voltage (-500 V). The electric field attracts nanoparticles, allowing them to strike the substrate with sufficient energy to rearrange into an energetically favorable, dense nanostructure, resulting in high electrical conductivity of the imprint. The voids in the right-hand side image are due to the limited depth of focus of the electron microscope.

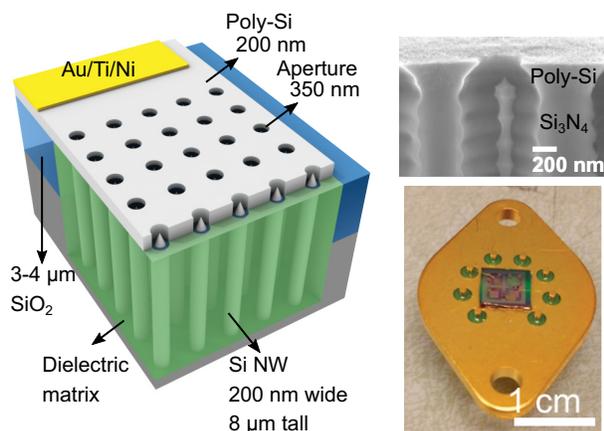
FURTHER READING

- Y. Kornbluth, R. H. Mathews, L. Parameswaran, L. Racz, and L. F. Velásquez-García, "Room-temperature, Atmospheric-pressure Deposition of Dense, Nanostructured Metal Films via Microsputtering," *Nanotechnology*, vol. 30, No. 28, 285602 (10pp), Jul. 2019.
- Y. Kornbluth, R. H. Mathews, L. Parameswaran, L. M. Racz, and L. F. Velásquez-García, "Microsputtering with Integrated Ion-drag Focusing for Additive Manufacturing of Thin, Narrow Conductive Lines," *J. of Physics D – Applied Physics*, vol. 51, no. 16, p. 165603, Apr. 2018.
- R. Messier, A. P. Giri, and R. A. Roy, "Revised Structure Zone Model for Thin Film Physical Structure," *JVST A*, vol. 2, p. 500, 1984.

Gated Silicon Field Ionization Arrays for Compact Neutron Sources

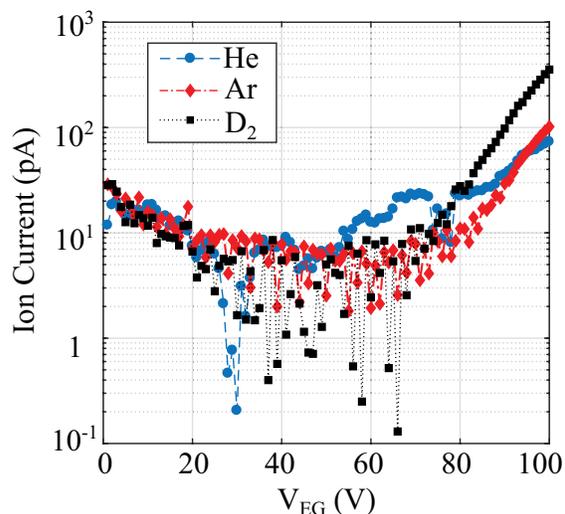
G. Rughoobur, A. I. Akinwande
Sponsorship: Defense Advanced Research Projects Agency

Neutron radiation is widely used in various applications, ranging from the analysis of the composition and structure of materials and cancer therapy to neutron imaging for security. However, most applications require a large neutron flux that is often achieved only in large infrastructures such as nuclear reactors and accelerators. Neutrons are generated by ionizing deuterium (D_2) to produce deuterium ions (D^+) that can be accelerated towards a target loaded with either D or tritium (T). The reaction generates neutrons and isotopes of He, with the D-T reaction producing the higher neutron yield. Classic ion sources require extremely high positive electric fields, on the order of 10^8 volts per centimeter (10 V/nm). Such a field is achievable only in the vicinity of sharp electrodes under a large bias, and consequently, ion sources for neutron generation are bulky.



▲ Figure 1: Schematic of gated field ionization array, with SEM cross-section of a single field ionizer and photograph of a packaged chip with arrays of different sizes for neutron generation

This work explores, as an alternative, highly scalable and compact Si field ionization arrays (FIAs) with a unique device architecture that uses self-aligned gates and a high-aspect-ratio (~40:1) silicon nanowire current limiter to regulate electron flow to each field emitter tip in the array (Figure 1). The tip radius has a log-normal distribution with a mean of 5 nm and a standard deviation of 1.5 nm, while the gate aperture is ~350 nm in diameter and is within 200 nm of the tip. Field factors, β , $> 1 \times 10^6 \text{ cm}^{-1}$ can be achieved with these Si FIAs, implying that gate-emitter voltages of 250-300 V (if not less) can produce D^+ based on the tip field of 25-30 V/nm. In this work, our devices achieve ionization current of up to 5 nA at ~140 V for D_2 at pressures of 10 mTorr. Gases such as He and Ar can also be ionized at voltages (<100 V) with these compact Si FIAs (Figure 2).



▲ Figure 2: Ion current measured for different gases (He, Ar, and D_2) at 1 mTorr pressure demonstrating low ionization voltages using 1000 by 1000 Si FIAs.

FURTHER READING

- M. Araghchini, S. A. Guerrero, and A. I. Akinwande, "High Current Density MEMS Deuterium Ionizers," *2016 29th International Vacuum Nanoelectronics Conference*, pp. 1-2, 2016.
- A. Fomani, L. F. Velasquez-Garcia, and A. I. Akinwande, "Low-voltage Field Ionization of Gases up to Torr-level Pressures using Massive Arrays of Self-aligned Gated Nanoscale Tips," *IEEE Trans. Electron Devices*, vol. 61, no. 5, pp. 1520-1528, 2014.
- Bargsten Johnson, P. R. Schwoebel, P. J. Resnick, C. E. Holland, K. L. Hertz, and D. L. Chichester, "Field Ionization Characteristics of an Ion Source Array for Neutron Generators," *J. Appl. Phys.*, vol. 114, no. 17, p. 174906, Nov. 2013.

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.

Rm. 39-415B | 617-253-0730 | lfvelasq@mit.edu

POSTDOCTORAL ASSOCIATE

Chenye Yang, MTL

GRADUATE STUDENTS

Ashley L. Beckwith, MechE
Yosef S. Kornbluth, MechE
Zhumei Sun, MechE

UNDERGRADUATE STUDENT

Sharon Lin, EECS

VISITORS

Brenda García Farrera, Tecnológico de Monterrey
Dulce Viridiana Melo Máximo, Tecnológico de Monterrey
Imperio Anel Perales Martínez, Tecnológico de Monterrey
Emmanuel Segura Cárdenas, Tecnológico de Monterrey
Alan Osiris Sustaita Narváez, Tecnológico de Monterrey
Anthony Taylor, Edwards Vacuum

SUPPORT STAFF

Jami L. Mitchell, Administrative Assistant

SELECTED PUBLICATIONS

Y. Kornbluth, R. H. Mathews, L. Parameswaran, L. Racz, and L. F. Velásquez-García, "Room-temperature, Atmospheric-pressure Deposition of Dense, Nanostructured Metal Films via Microsputtering," *Nanotechnology*, Vol. 30, no. 28, 285602 (10pp), Jul. 2019.

A. P. Taylor, C. Vélez Cuervo, David Arnold, and L. F. Velásquez-García, "Fully 3D-Printed, Monolithic Magnetic Actuators for Low-Cost, Compact Systems," *J. of Microelectromechanical Systems*, vol. 28, no. 3, pp. 481–493, Jun. 2019.

A. L. Beckwith, L. F. Velásquez-García, and J. T. Borenstein, "A 3D-Printed Microfluidic for Patient-specific Assessment of Immunotherapy Efficacy," *Advanced Healthcare Materials*, vol. 8, no. 11, 1900289 (6pp), Jun. 2019.

B. García-Farrera and L. F. Velásquez-García, "Near-room Temperature Direct Writing of Ultrathin Zinc Oxide Piezoelectric Films via Near-field Electrohydrodynamic Jetting for High-frequency Flexible Electronics," [presented] *20th International Conference on Solid-State Sensors, Actuators, and Microsystems (Transducers 2019-Eurosensors XXXIII)*, Berlin, Germany, June 23-27, 2019.

C. Yang and L. F. Velásquez-García, "Low-cost, Additively Manufactured Electron Impact Gas Ionizer with CNT Field Emission Cathode for Compact Mass Spectrometry," *J. of Physics D-Applied Physics*, vol. 52, no. 7, 075301, p. 9, Feb. 2019.

Y. Kornbluth, R. Mathews, L. Parameswaran, L. M. Racz, and L. F. Velásquez-García, "Room-temperature Printing of Micronscale-wide Metal Lines for Microsystems via Atmospheric Microsputtering," *Technical Digest 32nd Conference on Micro Electro Mechanical Systems (MEMS 2019)*, Seoul, South Korea, January 27-31, 2019.

Z. Sun, G. Vladimirov, E. Nikolaev, and L. F. Velásquez-García, "Exploration of Metal 3-D Printing for the Microfabrication of Freeform, Finely Featured, Mesoscaled Structures," *J. of Microelectromechanical Systems*, vol. 27, no. 6, pp. 1171-1185, Dec. 2018.

A. L. Beckwith, J. T. Borenstein, and L. F. Velásquez-García, "Monolithic 3D-Printed Microfluidic Platform for Assessment of Immunotherapy Treatments," *J. of Microelectromechanical Systems*, vol. 27, no. 6, pp. 1009-1022, Dec. 2018.

A. P. Taylor and L. F. Velásquez-García, "Low-cost, Monolithically 3D-Printed, Miniature High-flow Rate Liquid Pump," *Technical Digest of the 18th International Conference on Micro and Nanotechnology for Power Generation and Energy Conversion Applications (PowerMEMS 2018)*, Daytona Beach, FL, December 4-7, 2018.

C. Yang and L. F. Velásquez-García, "Compact, 3D-Printed Electron Impact Ion Source with Microfabricated, Nanosharp Si Field Emitter Array Cathode," *Technical Digest of the 18th International Conference on Micro and Nanotechnology for Power Generation and Energy Conversion Applications (PowerMEMS 2018)*, Daytona Beach, FL, December 4-7, 2018.

Z. Sun and L. F. Velásquez-García, "Miniature, 3D-Printed, Monolithic Arrays of Corona Ionizers," *Technical Digest of the 18th International Conference on Micro and Nanotechnology for Power Generation and Energy Conversion Applications (PowerMEMS 2018)*, Daytona Beach, FL, December 4-7, 2018.

A. Beckwith, L. F. Velásquez-García, and J. Borenstein, "An Additive Manufacturing-based Microfluidic System for Evaluation of Cancer Immunotherapies," *Technical Digest MRS Fall 2018*, Boston, MA, November 25-30, 2018.