

## Ultra-Fast Pulsed High-Current Cold Cathodes with Temporal and Spatial Emission Uniformity

Authors: M. E. Swanwick, L. F. Velásquez-García

Field emission arrays (FEAs) are an attractive alternative to mainstream thermionic cathodes, which are power hungry and require high vacuum and high temperature to operate. Field emission of electrons consists of the following two processes: 1) transmission of electrons (tunneling) through the potential barrier that holds electrons within the material (workfunction  $\varphi$ ) when the barrier is deformed by the application of a high electrostatic field and 2) supply of electrons from the bulk of the material to the emitting surface. Either the transmission process or the supply process could be the limiting step that determines the emission current of the field emitter (FE). Control of the transmission process. Due to the exponential dependence on the field factor and, hence, the tip radius, emission currents are extremely sensitive to tip radii variation; unfortunately, nanometer-sized tip radii in FEAs have a distribution with long tails that causes severe FEA underutilization. A better approach for achieving uniform emission from nanosharp FEAs is control of the supply of electrons to the emitting surface. In a metal, the supply of electrons is very high, making the control of the supply challenging. However, in a semiconductor, where the local doping level and the local potential determine the concentration of electrons, it is possible to configure the emitter such that either the supply process determines the emission current.

We have designed and fabricated FEAs where each field emitter is individually ballasted using a vertical ungated field effect transistor (FET) made from a high aspect ratio (40:1) n-type silicon pillar. Each emitter has a proximal extractor gate that is self-aligned for maximum electron transmission to the anode (collector). Our modeling suggests that these cathodes can emit as much as 30 A.cm<sup>-2</sup> uniformly with no degradation of the emitters due to Joule heating; also, these cathodes can be switched at microsecond-level speeds. The design process flow, mask set and pillar arrays have been completed (Figure 1) with the self-aligned extractor gate to be completed by the spring of 2013. An ultra-high vacuum chamber has been designed and built to test the devices (Figure 2). The chamber can test full 150mm wafers with four high voltage probes at 10<sup>-10</sup> torr pressure.



Figure 1: Large array of high aspect ratio pillars with 1 onm radius tips with 5  $\mu$ m hexagonal packing for individually ballasted field emission arrays.



Figure 2: CAD drawing of the UHV chamber design that is capable of testing 150 mm wafer with four high voltage probes at 10-10 torr.

# Photoactuated Ultrafast Silicon Nanostructured Electron Sources for Coherent X-ray Generation

Authors: M. E. Swanwick, P. D. Keathley, R. G. Hobbs, Y. Yang, W. Graves, K. K. Berggren, F. X. Kärtner, L. F. Velásquez-García Sponsorship: DARPA, DARPA

A collaboration of RLE and MTL investigators is creating the scientific and engineering knowledge for a compact coherent Xray source for phase contrast medical imaging based on inverse Compton scattering of relativistic electron bunches. The X-ray system requires a low emittance electron source that can be switched at timescales of tens of femtoseconds or faster; the focus of our work has been the design, fabrication and characterization of massive arrays of a nanostructured high aspect-ratio silicon (Si) structures to implement low-emittance and high-brightness cathodes that can be triggered very fast using laser pulses to produce spatially uniform electron bunches. Si nanostructure arrays with highly uniform sub-10 nm tip radii have been fabricated via a combined optical lithography and diffusion limited oxidation technique. The fabrication process allows nanometer-level control over the dimensions of the electron emitter structures. Figure 1 shows an array of Si tips with 1.25  $\mu$ m hexagonal pitch have an average radius of curvature of 6.2 nm and standard deviation of 1.1 nm (n=29); when the radius of curvature is changed to 21.6nm, the standard deviation remains approximately the same, i.e., 1.25 nm (n=69).

The tips are illuminated at a grazing incidence of roughly 84 degrees with a 1 kHz titanium sapphire laser (800 nm wavelength) with a pulse duration of 35 fs; the high electric field of the laser pulse is amplified by the silicon tips so the electrons can quantum tunnel from the tips into the vacuum. Experimental results using a time of flight spectrometer show electron beamlet array emission with 3-photon absorption. Work is ongoing to optimize the tip geometry for both low emittance and high current. We are also designing and building a new vacuum chamber to test the devices (Figure 2). The chamber will pump down to  $10^{-7}$  torr in ~15min with an anode bias up to 1100V.



Figure 1: A SEM micrography of highly uniform nanosharp tips with 1.25  $\mu m$  hexagonal pitch and 12.4 nm average diameter. Upper Right – Close up of a single tip.



Figure 2: CAD drawing of new vacuum chamber to measure current from the photocathodes using a 35 fs 800nm beam at 1kHz at 84 degree angle.

## **Development of Nanostructured Optical Field Emitter Arrays**

Authors: Y. Yang, R. G. Hobbs, V. R. Manfrinato, M. E. Swanwick, W. P. Putnam, P. D. Keathley, F. X. Kaertner, L. F. Velasquez-Garcia, K. K. Berggren

Sponsorship: DARPA

We are interested in the application of arrays of electron field emitters, which can be achieved from a variety of materials, for the preparation of compact and coherent X-ray sources via inverse Compton scattering. Field emission of electrons is commonly achieved by applying a static electric field or optical illumination to sharp metal tips. Sub-wavelength nanostructures can provide geometry-dependent electric field enhancement for both methods. For applications in coherent Xray sources, the field emitter arrays should be able to emit short electron pulses, typically on the femtosecond timescale, which is difficult using conventional electrical circuits. Therefore, optical triggering, whereby a femtosecond laser is used to stimulate electron emission, has been considered.

We have simulated optical fields around various field emitter structures using COMSOL finite element software. Among them, we are particularly interested in the "bullet" structure illustrated in Figure 1. Conical tip structures are widely used to achieve both electrostatic and optical field enhancement in field emitters; however, uniform conical structures pose significant challenges in nanoscale fabrication due to their tapered geometry. Arrays of metallic "bullet" structures, as shown in Figure 1, may be fabricated with a high areal density via positive-tone electron beam lithography with a PMMA resist.

We have designed a fabrication process for the preparation of arrays of optical-field emitters, based on the metallic "bullet" structure shown in Figure 2 (a). The thin  $SiO_2$  layer shown in Figure 2 (a) is used to prevent electron emission from the bulk silicon substrate by acting as an electrically insulating barrier. The thickness of the  $SiO_2$  layer has yet to be optimized. As a proof of concept, we have fabricated an array of Au nanoparticle emitters with an aspect-ratio of 1 (see Figure 2 (b)). Further optimization of the fabrication process is currently underway.



Figure 1: Gold bullet optical-field emitter. (a) Emitter geometry; (b) 2-D electric field amplitude map in a plane of the emitter axis. Radius of the cylinder and hemispherical tip is 5 nm. Cylinder length is 30 nm. The illumination source has a wavelength of 810 nm. Gold ( =-24.9+1.57i) and water (=1.77) are chosen as tip and dielectric materials, respectively. The optical triggering field propagates in x-direction (from left to right) and is polarized along the y-axis (in-plane and along the tip axis). Propagation (k) and polarization (E) directions are shown as arrows. The field enhancement displayed in (b) is normalized with respect to the incident optical field and has a peak enhancement factor ~3.7 at the apex of the "bullet".



Figure 2: (a) Fabrication process of the gold optical-field emitters. The process starts from a Si wafer. A thin SiO<sub>2</sub> layer is grown, preventing electron emission from bulk Si. Electron beam lithography and etching produces a patterned oxide layer. Au deposition and PMMA lift-off allows creation of Au "bullet" arrays. The aspect-ratio of the emitters depends on the Au deposition time. The Si substrate can be electrically grounded to serve as an electron reservoir, or connected to a DC bias to perform electron emission induced by a combination of electrostatic field and optical field stimulation. (b) SEM image of a Au dot array. An oxide layer was not deposited on this sample and the gold dots (aspect-ratio of 1) are deposited directly on the Si wafer. The diameter of each gold dot is 10 nm and the dot pitch is 30 nm. A total of 100 Au dots are packed within an area of less than 0.1 µm2.

1. D. Temple, "Recent progress in field emitter array development for high performance applications," Materials Science and Engineering: R: Reports, vol. 24, pp. 185-293, Jan. 1999.

- 2. O. J. F. Martin and C. Girard, "Controlling and tuning strong optical field gradients at a local probe microscope tip apex," Applied Physics Letters, vol. 70, pp. 705-707, Feb. 1997.
- 3. L. Novotny, R. X. Bian, and X. S. Xie, "Theory of nanometric optical tweezers," Physical Review Letters, vol. 79, pp. 645-648, July 1997.

## Flush-mounted MEMS Langmuir Probe Arrays for HF-S Band Plasma-sensing

Authors: E. . F. C. Chimamkpam, A. I. Akinwande, L. F. Velásquez-García Sponsorship: NASA

Arrays of MEMS Langmuir probes that are flush-mountable (Figure 1) can serve as a sensorial skin on a spacecraft for fine spatial and temporal resolution of plasma phenomena. The technology can also provide diagnostics for other applications such as tokamaks and nanosatellite scientific payloads <sup>[1]</sup>. The benefits are innumerable for deeper understanding of plasma physics, which is in great need of these microprobes <sup>[2]</sup>. For instance, multiplexed microprobes that are flush-mounted on all the faces of a 3-D "tip" can allow for simultaneous capture of a detailed "whole picture" of plasma behavior in different axes at a given timescale. In addition, two or more different sensory configurations, e.g., single-, double-, triple-probe methods, etc., can be adapted into the same flat die, profiting at the same time from their individual data acquisition strengths. Protruded probes cannot offer these advantages. Another area of deployment is in the observation of electron phase-space holes, self-consistent nonlinear plasma structures that are formed from strong current- or beam-driven turbulence and found in magnetic reconnection regions, which are magnetic field topology modifiers responsible for the explosive release of magnetic energy in magnetospheric storms, solar flares, and laboratory plasmas <sup>[3]</sup>. Fast micro-Langmuir probes that work at high frequencies are indispensable for studying these plasma fluctuations. We developed a system of flush-mounted MEMS Langmuir probes and apparatus with fast timescale; i.e., shorter time compared to the timescale of reconnection events in the Versatile Toroidal Facility at MIT (Figure 2); and wide bandwidth extending across regions of magnetosphere-photosphere, i.e., considering both electron and ion plasma frequencies associated with these regions.



Figure 1: A 10-mm-wide die with total of four individual MEMS Langmuir probes flushmounted on a probe shaft.



Figure 2: Versatile Toroidal Facility at MIT. Magnetic reconnection experiments are conducted in the device.

1. E. S. Field, "Batch-fabricated planar arrays of MEMS Langmuir probes for spacecraft reentry plasma diagnostics and nanosatellite scientific payloads," Master's thesis, Massachusetts Institute of Technology, Cambridge, 2011. [↔]

2. P. Pribyl, W. Gekelman, M. Nakamoto, E. Lawrence, F. Chiang, J. Stillman, J. Judy, N. Katz, P. Kintner, and P. Niknejadi, "Debye size microprobes for electric field measurements in laboratory plasmas," Review of Scientific Instruments, vol. 77, no. 073504, pp. 1-8, July 2006. [4]

3. W. Fox, M. Porkolab, J. Egedal, N. Katz, and A. Le, "Observations of electron phase-space holes driven during magnetic reconnection in a laboratory plasma," *Physics of Plasmas*, vol. 19, no. 032118, pp. 1-12, Mar. 2012. [ $\ominus$ ]

## Silicon Field Emitter Arrays for Chip-scale Vacuum Pumping

Authors: A. A. Fomani, A. I. Akinwande, L. F. Velásquez-García Sponsorship: DARPA

Development of miniature vacuum pumps that can be integrated with electronic or MEMS components is necessary for producing advanced equipment such as portable analytical instruments <sup>[1]</sup> and high performance sensors <sup>[2]</sup>. The proposed approach graphically illustrated in Figure 1 is based on electron impact ionization (EEI) or field ionization (FI) of the gas molecules using nano-scale sharp silicon tips. The ionized gas molecules are then evacuated from the chamber using a strong electric field to accelerate the ions and implant them permanently into a getter medium made of Ti or Al. In the EEI mode of the operation, a positive voltage is applied between the gate and the emitter to extract electrons that are used to ionize the background gas. In the FI regime, the Si sharp tips are biased at a positive voltage with respect to the gate to extract electrons from the outer shell of the gas molecules in a quantum tunneling process. The former process occurs at electric fields in the range of  $3-6 \times 10^7$  V/cm while the later process initiates at electric fields above  $10^8$  V/cm<sup>[3]</sup>. Despite the larger required voltage, the operation in the FI regime is mandatory since the back-streaming of the positive ions during EEI mode of operation will damage the field emitter (FE) tips at mTorr-pressure range. Although state-of-the-art field emitters have been reported [4] [5] [6], the focus of this work is to improve the reliability of the FE or FI devices for extended operation times and large currents necessary for pumping application. Since these devices demand application of large voltages between the gate and the tip of the FE/FI, wear or breakdown of the insulating dielectric is a major issue. Finite element modeling (shown in Figure 2) has been conducted to optimize the design of the device for pumping application. A new fabrication process is also being developed for high-yield fabrication of an array with more than 300K Si FEs/FIs.



Figure 1: Evacuation of gas molecules using (a) electron impact ionization and (b) field ionization mechanisms.





1. Z. Ouyang and R. G. Cooks, "Miniature mass spectrometers," Annual Review of Analytical Chemistry, vol. 2, pp. 187-214, Feb. 2009. [4]

- 2. M. Esashi, "Wafer level packaging of MEMS," Journal of Micromechanics and Microengineering, vol. 18, no. 7, pp. 073001:1-13, May 2008. [↔]
- 3. R. Gomer, Field Emissions and Field Ionization, New York: Springer-Verlag, Dec. 1992.  $[\leftrightarrow]$
- 4. M. Ding, G. Sha, A. I. Akinwande, "Silicon field emission arrays with atomically sharp tips: turn-on voltage and the effect of tip radius distribution," IEEE Transaction on Electron Devices, vol. 49, no. 12, pp. 2333-2342, Dec. 2002. [+]
- 5. L. F. Velásquez-García, S. A. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform high-current cathodes using massive arrays of Si field emitters individually controlled by vertical Si ungated FETs part 1: Device design and simulation," *IEEE Transactions on Electron Devices*, vol. 58, no. 6, pp. 1775-1782, June 2011. []
- 6. L. F. Velásquez-García, S. A. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform high-current cathodes using massive arrays of Si field emitters individually controlled by vertical Si ungated FETs part 2: Device fabrication and characterization," *IEEE Transactions on Electron Devices*, vol. 58, no. 6, pp. 1783-1791, June 2011. []

## Measuring Ion Energy Distribution Using Batch-microfabricated RPAs

Authors: E. V. Heubel, A. I. Akinwande, L. F. Velásquez-García Sponsorship: NASA

The need to measure particle energies arises in many applications, from calibrating electron sources for electron guns in precision microscopes to determining the efficiency of space-based ion beam thrusters. Retarding potential analyzers (RPAs) are capable of filtering particles based on their energy and have been used as early as the late 1950s and early 1960s for such purposes <sup>[1]</sup>. However, these devices maintain limited application due to stringent dimensional constraints driven by plasma Debye length. Cold dense plasmas require minute apertures and tight spacing tolerances between biasing grids that are difficult to enforce using conventional means. We suggest microelectromechanical system (MEMS) batch-fabrication techniques in order to achieve unprecedented alignment accuracy of successive electrodes while incorporating the necessary micron-scale features. Assembly to a precision of a few tens of microns has been demonstrated with a hybrid RPA (see Figure 1a) <sup>[2]</sup>. Figure 1b shows the fully MEMS-fabricated sensor inspired by in-plane assembly of high-voltage devices, which will have tolerances on the order of 1µm <sup>[3]</sup>.

Augmenting the optical transparency of RPAs provides a more direct path for particles to the collector plate. Signal strength is thus improved as the effective collection area is increased. Preliminary results and comparisons between MEMS-fabricated electrodes and conventional stainless steel mesh have revealed an ameliorated signal quality. Figure 2 shows a greater than two-fold improvement in peak signal strength with the micromachined grids over the conventional RPA<sup>[2]</sup>. Currents captured by the various grids and simulations suggest the possibility of ion beam focusing and interception of ions prior to reaching the collector. Alteration of the internal dynamics of the sensor provides a cleaner signal that may lead to a better interpretation of the measurements than with models that incorporated the stochastic behavior of charged species through randomly oriented electrode apertures.





Figure 2: Comparison of ion energy distributions obtained from a 10V ion source with our hybrid sensor showing a more pronounced peak using microfabricated grids over conventional stainless steel mesh  $[\mathfrak{A}]$ .

Figure 1: Exploded views of a) hybrid RPA with stainless steel housing and b) fully micromachined RPA  $^{[2]}$ .

1. W. C. Knudsen, "Evaluation and demonstration of the use of retarding potential analyzers for measuring several ionospheric quantities," *Journal of Geophysical Research*, vol. 71, no. 19, pp. 4669–4678, Oct. 1966. [↔] 2. E. V. Heubel, A. I. Akinwande, and L. F. Velásquez-García, "MEMS-enabled retarding potential analyzers for hypersonic in-flight plasma diagnostics," in *Proceedings of the 15th Solid-State Sensors, Actuators, and* 

Microsystems Workshop, Hilton Head Is., SC, June 2012, pp. 324–237. [↔] [↔] [↔]

3. 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," *Journal of Microelectromechanical Systems*, vol. 18, no. 2, pp. 332–346, Apr. 2009. [ $\leftrightarrow$ ]

### Cathode for X-ray Generation with Arrays of Individually Addressable Field Emitters Controlled by Vertical Ungated FETs

Authors: F. A. Hill, L. F. Velásquez-García Sponsorship: DARPA

This work focuses on the design and fabrication of a cathode for a portable x-ray source. The cathode is made of an array of individually addressable electron guns, each containing double-gated field emitters. Compared to thermionic cathodes, field emission arrays operate at lower vacuum and lower temperatures, use less power and are more portable. The electron beam from each gun is extracted by a proximal gate and collimated using a distal gate before it hits an anode in a micron-sized spot that generates Bremsstrahlung x-rays. The architecture of the cathode is shown in Figure 1. Each field emitter is fabricated on top of a vertical ungated field-effect transistor (FET)<sup>[1][2]</sup> that acts as a current source due to the velocity saturation of electrons in silicon when the voltage across the FET is above a saturation voltage. Current source-like behavior provides spatial and temporal uniformity of the output current across the emitter array; it also protects against emitter burnout and current surges. Individual addressability is achieved by fabricating the structure on SOI wafers to create electrically isolate strips of silicon. The extractor and focus gates are monolithically integrated with the cathode chip. They are patterned in strips that are orthogonal to the silicon strips, so that a single electron gun can be turned on at once. Each vertical ungated FET is a  $25-\mu$ m-tall column with a 0.5- $\mu$ m diameter, and emitter tip radius is in the range of 20 nm. The saturation current and saturation voltage of the silicon columns are plotted as a function of doping density in Figure 2. Wafer doping of 10-20  $\Omega$  cm provides a saturation current of 0.5  $\mu$ A and an output impedance of  $2\times10^9 \Omega$ . With 100 emitters per chip, the total output current per chip is 50  $\mu$ A, corresponding to a current density of 139  $\mu$ A/cm<sup>2</sup>.



Figure 1: Architecture of the cathode chip layout. The chip contains a 10 by 10 array of electrode guns, each containing field emitters.



Figure 2: FET saturation current vs. doping (top) and FET saturation voltage vs. doping (bottom).

1. L. F. Velásquez-García, S. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform high-current cathodes using massive arrays of Si field emitters individually controlled by vertical Si ungated FETs—Part 1: Device fabrication and characterization," *IEEE Transactions in Electron Devices*, vol. 58, pp. 1783-1791, June 2011. [ $\leftrightarrow$ ]

2. L. F. Velásquez-García, S. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform high-current cathodes using massive arrays of Si field emitters individually controlled by vertical Si ungated FETs—Part 2: Device design and simulation," *IEEE Transactions in Electron Devices*, vol. 58, pp. 1775-1782, June 2011. [↔]

# Batch-Microfabricated Electrospray Arrays with Integrated Electrode Stack for Ionic Liquids

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

Electrospray is a process to ionize electrically conductive liquids that relies on strong electric fields; charged particles are emitted from sharp tips that serve as field enhancers to increase the electrostatic pressure on the surface of the liquid, overcome the effects of surface tension, and facilitate the localization of emission sites. Ions can be emitted from the liquid surface if the liquid is highly conductive and the emitter flowrate is low. Previous research demonstrated successful operation of massive arrays of monolithic batch-microfabricated planar electrospray arrays with an integrated extractor electrode using ionic liquids EMI-BF<sub>4</sub> and EMI-Im <sup>[1]</sup> <sup>[2]</sup> – liquids of great importance for efficient nanosatellite propulsion. The current work aims to build upon the previous electrospray array designs by increasing the density of the emitter tips, increasing the output current by custom-engineering suitable nanofluidic structures for flow control, and improving the ion optics to gain control of the plume divergence and exit velocity.

The basic version of the MEMS electrospray array consists of an emitter die and an extractor die (shown in Figure 1), both made of silicon and fabricated using deep reactive ion etching. The two dies are held together using a MEMS high-voltage packaging technology based on microfabricated springs that allows precision packaging of the two components with less than 1% beam interception <sup>[3][4]</sup>. The emitter die contains dense arrays of sharp emitter tips with as many as 1,900 emitters in 1 cm<sup>2</sup>. A voltage applied between the emitter die and the extractor electrode creates the electric field necessary to ionize the ionic liquid (see Figure 2). A nanostructured material transports the liquid from the base of the emitters to the emitter tips. The present research focuses on engineering the nanofluidic structure to attain higher emitter current while maintaining good array emission uniformity and on developing batch microfabricated advanced ion optics to control the electrospray plume.





Figure 2: Schematic of externally fed electrospray emitter tips and the electrodes that create an electric field to produce a beam of ions.

Figure 1: Design of the basic version of the MEMS electrospray array, consisting of an emitter die and an extractor die.

- 1. L. F. Velásquez-García, A. I. Akinwande, and M. Martínez-Sánchez, "A planar array of micro-fabricated electrospray emitters for thruster applications," *Journal of Microelectromechanical Systems*, vol. 15, no. 5, pp. 1272-1280, Oct. 2006. []
- 2. B. Gassend, L. F. Velásquez-García, A. I. Akinwande, and M. Martínez-Sánchez, "A microfabricated planar electrospray array ionic liquid ion source with integrated extractor," Journal of Microelectromechanical Systems, vol. 18, no. 3, pp. 679-694, June 2009. [4]
- 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," *Journal of Microelectromechanical Systems*, vol. 18, no. 2, pp. 332-326, 2009. [↔]
- 4. L. F. Velásquez-García, A. I. Akinwande, and M. Martínez-Sánchez, "Precision hand assembly of MEMS subsystems using DRIE-patterned deflection spring structures: An example of an out-of-plane substrate assembly," *Journal of Microelectromechanical Systems*, vol. 16, no. 3, pp. 598–612, 2007. [↔]

# Externally-fed, Microfabricated Electrospinning Device for Increased Throughput of Polymer Nanofibers

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

Electrospinning is a process in which a membrane-like web of thin fibers can be produced using high electrostatic fields and polar liquids with high viscosity. It is the only known technique that can generate continuous fibers with controlled morphology in the 10-500 nm diameter range and has tremendous versatility as it can create non-woven or well-aligned mats of polymer, ceramic, semiconductor, and/or metallic fibers using the same hardware. Electrospinning is also capable of conformally coating 3D complex shapes with ultrathin layers that have complex multi-layered structure and thickness variation across the surface. In particular, polymer electrospun fibers have been proposed to develop multi-stack functional fiber mats for protective gear, because they show high breathability, elasticity, and filtration efficiency. In addition, electrospun fibers made of the appropriate materials could also be used in flexible electronics (graphene) and in structural reinforcement against mechanical trauma. However, the production of electrospun nanofibers has very low throughput due to the small fiber diameter, which limits their applications to high-end products. In this project we are investigating the development of high-throughput electrospun nanofibers using batch-microfabricated arrays of externally fed electrospinning emitters. Externally-fed emitters are attractive, because they do not require high pressure drops as internally-fed emitters do. Also, they do not clog and can process liquids that bubble.

An aspect of this project is looking into the physics of wicking to optimize the fluidic micro/nanostructures that control the emitter flow rate. For solids with intrinsic contact angles below some critical value determined by roughness geometry, it becomes energetically favorable for a droplet to completely impregnate the roughness and spread through it <sup>[1]</sup>. This process of hemi-wicking has been described in pillar arrays of varying shapes and sizes <sup>[2][3]</sup>. For externally-fed electrospinning, we must ensure a sufficient and steady flow rate of polymer solution to avoid broken or irregular fibers. We are theoretically and experimentally investigating optimal morphologies of both the micro/nano fluid control structures and the emitter geometry to attain good array emission uniformity.



Figure 1: Fluid diffusion rate as a function of micropillar height and diameter for a fixed pitch of 10  $\mu m.$  At constant height, there exists some intermediate diameter where the flow is maximized.



Figure 2: Photograph of fluid being transported through the micropillar patterned surface. Several different spreading regimes can be achieved by varying micropillar geometry.

1. D. Quéré, "Wetting and roughness," Annu. Rev. Mater. Res., vol. 38, pp. 71-99, April 2008. [↩]

3. C. Ishino, M. Reyssat, E. Reyssat, K. Okumura, and D. Quéré, "Wicking within forests of micropillars," Europhys. Lett., vol. 79, p. 56005, 2007. [+]

<sup>2.</sup> L. Courbin, J. C. Bird, M. Reyssat, and H. A. Stone, "Dynamics of wetting: From inertial spreading to viscous imbibition," Journal of Physics: Condensed Matter, vol. 21, p. 464127, 2009. [+-]

### Electrospray Nanoprinting on Electrospun Nanofiber Mats for Low-cost Biochemical Detection

Authors: M. Overlin, E. V. Heubel, K. Senecal (US Army NSRDEC), P. Marek (US Army NSRDEC), L. F. Velásquez-García Sponsorship: US Army

An electrospray emitter ionizes polar liquids using high electrostatic fields. The electric field produces suction on the free surface (meniscus) of an electrically conductive liquid, and the surface tension of the liquid tends to counteract the effect of the electrostatic suction. If the electric field is larger than a certain threshold, the meniscus snaps into a conic shape called a Taylor cone<sup>[1]</sup> (see Figure 1). A Taylor cone emits charged particles from its apex due to the high electrostatic fields present there; these particles can be ions, droplets, fibers, etc., depending on the working liquid and the emitter flowrate [2]. In particular, electrospray in cone-jet mode<sup>[3]</sup> creates near-monodispersed charged droplets that can be used for many applications including mass spectrometry<sup>[4]</sup>, etching<sup>[5]</sup>, and nanosatellite propulsion<sup>[6]</sup>. In this project we are exploring electrospray in cone-jet mode as a technology to create controlled nanoimprints on electrospun nanofiber mats with liquids such as fluorescent dye and nanoparticles solutions, as an alternative technology to nano-pipetting or ink jet printing. Using a shadow mask, we have shown imprints in close agreement with the dimensions of the mask aperture (see Figure 2). The longterm goal of the project is to investigate the design space of the technology to make low-cost and low false-positive biochemical detectors by exploring the multiplexing and scaling-down limits of cone-jet mode electrospray sources using batch micro- and nanofabrication<sup>[7]</sup>.





Figure 2: Sub-millimeter electrospray imprints on an electrospun fiber mat using a slit.

Figure 1: A Taylor cone at the end of a 300-µm OD capillary. Charged droplets are emitted from the apex of the cone. The working liquid is a fluorescent dye.

- 1. G. I. Taylor, "Disintegration of water drops in an electric field," Proc. Royal Society of London A, 1964, vol. 280, pp. 383-397. [↔]
- 2. J. Fernandez de la Mora, "The fluid dynamics of Taylor cones," Ann. Rev. of Fluid Mec., vol. 39, pp. 217-243, 2007. [↔]
- 3. J. Fernandez de la Mora, "The current emitted by highly conductive Taylor cones," *J. Fluid Mechanics*, vol. 260, pp. 155-184, 1994. [ $\leftrightarrow$ ]
- 4. J. B. Fenn, M. Mann, C. K. Meng, S. F. Wong, and C. M. Whitehouse, "Electrospray ionization for mass spectrometry of large biomolecules," *Science*, vol. 246, no. 4926, pp. 64-71, 1989. [↔] 5. M. Gamero-Castaño and M. Mahadevan, "Sputtering of silicon by a beamlet of electrosprayed nanodroplets," *Appl. Surf. Sci.*, vol. 255, pp. 8556-8561, 2009. [↔]
- 6. L. F. Velásquez-García, A. I. Akinwande, and M. Martinez-Sanchez, "A planar array of micro-fabricated electrospray emitters for thruster applications," J. of Microelectromech. Sys., vol. 15, no. 5, pp. 1272-1280, 2006.

7. B. Gassend, L. F. Velásquez-García, A. I. Akinwande, and M. Martinez-Sanchez, "A microfabricated planar electrospray array ionic liquid ion source with integrated extractor," J. of Microelectromech. Sys., vol. 18, no. 3, pp. 679-694, 2009. [↩]

## Luis Fernando Velásquez-García

Title: Principal Research Scientist Department: Microsystems Technology Laboratories URL: multiplexed.mit.edu

#### Collaborators

- A.I. Akinwande, EECS, MIT
- K. Berggren, EECS, MIT
- W. Graves, Nuclear, MIT
- R. Gupta, CIMIT, Mass General
- V. Hruby, Busek Co.
- F. Kaertner, EECS, MIT
- R. Lanza, Nuclear, MIT
- C. Livermore, MechE, NEU
- P. Marek, US Army NSRDEC
- M.A. Schmidt, EECS, MIT
- K. Senecal, US Army NSRDEC
- S. Teller, EECS, MIT

#### Graduate Students

- E. S. Field, Research Assistant, MechE
- S. Guerrera, Research Assistant, EECS (co-supervised with A.I. Akinwande)
- E. V. Heubel, Research Assistant, MechE
- V. Jayanti, Research Assistant, EECS
- D. Jeng, Research Assistant, EECS (co-supervised with A.I. Akinwande)
- P. Ponce de Leon, MechE

### Visiting Student

• Z. Liu, EECS, BU

### Postdoctoral Associates

- A. Fomani (co-supervised with A.I. Akinwande)
- F. Hill
- M. Swanwick

### Visiting Scientist

• E. Chimamkpam

• M. R. Overlin, EECS, MIT

### Support Staff

• C. Collins, Administrative Assistant II

#### **Publications**

S. Guerrera, L. F. Velásquez-García, and A. I. Akinwande, "Scaling of High-Aspect-Ratio Current Limiters for the Individual Ballasting of Large Arrays of Field Emitters," accepted for publication, *IEEE Transactions on Electron Devices*, 2012.

E. V. Heubel, A. I. Akinwande, and L. F. Velásquez-García, "MEMS-Enabled Retarding Potential Analyzers for Hypersonic In-Flight Plasma Diagnostics," *Technical Digest of the 15th Solid-State Sensor, Actuator and Microsystems Workshop*, pp. 324 – 327, Hilton Head Island SC, USA, June 3 – 7, 2012.

P. D. Keathley, W. P. Putnam, A. Sell, S. Guerrera, L. F. Velásquez-García, and F. X. Kaertner, "Laser Induced Annealing Dynamics of Photo-Electron Spectra from Silicon Field Emitter Arrays," *Technical Digest of the Conference on Lasers and Electro-Optics – CLEO*, San Jose, CA, May 6-11, 2012.

S. A. Guerrera, L. F. Velásquez-García, and A. I. Akinwande, "Scaling of High Aspect Ratio Current Limiters for the Individual Ballasting of Large Arrays of Field Emitters," *Technical Digest 24th International Vacuum Nanoelectronics Conference*, Wuppertal, Germany, July 2011.

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, 2011.

L. F. Velásquez-García, S. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform High-Current Cathodes Using Massive Arrays of Si Field Emitters Individually Controlled by Vertical Si Ungated FETs – Part 1: Device Design and Simulation," *IEEE Transactions on Electron Devices*, Vol. 58, No. 6, pp. 1775 – 1782, 2011.

L. F. Velásquez-García, S. Guerrera, Y. Niu, and A. I. Akinwande, "Uniform High-Current Cathodes Using Massive Arrays of Si Field Emitters Individually Controlled by Vertical Si Ungated FETs – Part 2: Device Fabrication and Characterization," *IEEE Transactions on Electron Devices*, Vol. 58, No. 6, pp. 1783 – 1791, 2011.