2022 Microsystems Annual Research Report



MASSACHUSETTS INSTITUTE OF TECHNOLOGY

# Characterization of 3D-printed, Tunable, Lab-grown Plant Materials

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

Wood has traditionally been viewed as a low-cost, widespread commodity. However, current practices for wood procurement are unsustainable. Wood supply is increasingly strained, and, in many ways, trees are non-ideal to produce wood: they are affected by climate, seasons, and producing a small fraction of wood from the total mass of the tree.

We recently pioneered an approach to generate plant-based materials in vitro without needing to harvest or process whole plants, making possible the high-density production of plant-based materials unaffected by such constraints. In addition, the process is compatible with additive manufacturing. We now report the first physical, mechanical, and microstructural characterization of plant materials generated with Zinnia elegans cell cultures using such methodology. The results show that the properties of the plant materials vary significantly with adjustments to hormone levels present in growth medium. In addition, the data show that the use of bioprinting and casting enables the production of net-shape objects in forms and scales that do not arise naturally in whole plants (Figure 1). Further work could entail the development of processes for other plant species and/ or producing other biopolymers.



▲ Figure 1: (a) Stem samples were halved lengthwise (cut 1) and dried before fixing in wax and sectioning with a microtome along cut 2. (b) Cross-section of a halved and dried Zinnia stem. (c) Cross-section of a grown material sample sliced along the shortest dimension, greatly surpassing the size of the stem, even though they have the same age. Scale bar equals 500 micrometers. From A. Beckwith et al., Materials Today (2022).

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## 3-D Printed Quadrupole Mass Filters for CubeSat Mass Spectrometry

A. Diaz, L. F. Velásquez-García Sponsorship: MIT Portugal

Mass spectrometry is the gold standard for quantitative chemical analysis. Mass spectrometers employ mass filters that generate electromagnetic fields to sort out in vacuum the ionized constituents of a sample based on their mass-to-charge ratio, making it possible to determine the chemical composition of the sample. However, mass spectrometers are typically large, heavy, and power hungry, restricting their deployability into in-situ, portable, and hand-held scenarios, e.g., CubeSats. Miniaturization of electronics and mass spectrometry hardware has made possible the implementation of compact instruments. Nonetheless, instrument miniaturization has been attained at the expense of great loss in performance, caused in part by fabricating unideal electrode shapes and losing assembly resolution via post-assembly. Via additive manufacturing, it is possible to create monolithically and more precisely electrode shapes, avoiding some or most of the key assembly steps in a traditional mass filter, potentially resulting in hardware that performs better.

In this project we are developing compact, monolithically 3D-printed RF quadrupole mass filters that operate in the MHz range. Figure 1 shows an early-stage prototype of this filter. Moreover, the work includes developing compact, precision electronics for running the quadrupole and reading the current transmitted by the mass filter (Figure 2). We will also explore ideas for improving the performance of the mass filter, e.g., operating the devices in the second stability region.



Figure 1: 3-D printed prototype of a quadrupole with conductive electrodes and non-conductive supporting structure.

▶ Figure 2: Full bridge class D amplifier setup to maximize power efficiency with a 2.65-MHz driving frequency.



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# Compact, Monolithic, Additively Manufactured Quadrupole Mass Filters

C. Eckhoff, L. F. Velásquez-Garcia Sponsorship: Empiriko Corporation

Mass spectrometry (MS) is the gold standard for identifying matter. Whether quantitative precision is needed to study absolute amounts of target molecules or qualitative resolving power is needed to discriminate isotopes down to a single neutron of difference, MS is often the tool of choice in the biotech and medical fields. However, the emergent focus of medical device industries on point-of-care (POC) testing has not brought with it a POC MS device satisfying the requirements of physicians and clinical regulatory agencies.

Our group seeks to improve POC MS systems by creating novel micro-electromechanical system (MEMS ) mass filters, using design techniques permitted exclusively by additive manufacturing. This includes arbitrary electrode shapes, precision electrode alignment, and the efficient use of device space. Our manufacturing approach is to use a digital light processing of glass-ceramic resin to produce monolithically fabricated, pre-aligned hyperbolic electrodes (Figure 1). By integrating parts that are usually separate, monolithic designs reduce the need for fastening, alignment, and mounting hardware. This not only reduces costs but increases assembly precision, improving quadrupole resolution. Furthermore, the hyperbolic geometry of the quadrupole electrode rods eliminates field harmonics that are present on common, commercial circular rods. Plating is used to metallize the electrodes (Figure 2), resulting in thermally stable, electrically conductive electrodes.

Additive manufacturing, mass filter design, and post-print metallization all pose challenges of their own; when these processes are combined, even greater challenges exist. Our research currently focuses on optimizing each of these processes while considering the needs and effects of the other processes. For instance, we have designed a working quadrupole filter that prints well in ceramic while also being easy to metallize. Proof-of-concept data is being collected for the early prototypes, which will guide future refinement and miniaturization of the quadrupole design.



▲ Figure 1: CAD model of singular, monolithic quadrupole mass filter.



▲ Figure 2: Close-up photo of metallized, 3D-printed electrodes.

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# Langmuir Probes via Rapid Prototyping for CubeSat and Laboratory Plasma Diagnostics

Z. Bigelow, L. F. Velásquez-García Sponsorship: MIT Portugal

Langmuir probes (LPs) are widely considered to be the most versatile in-situ plasma sensors due to the simplicity of their design, small cross section, low maintenance requirements, and compatibility with a very broad range of plasma conditions. When operated with compact, low-power electronics, LPs can be installed onboard CubeSats to characterize the space environment surrounding the spacecraft. Moreover, miniaturized LPs can be used to make local plasma measurements. Basic LPs have a single electrode that collects a current while sweeping a bias voltage; from the current–voltage characteristic, plasma parameters such as the electron temperature and number density can be extracted. LPs can also be operated in groups to improve the measurements: for example, double LPs are less sensitive to plasma fluctuations as they are energized by a floating bias voltage. Also, triple LPs conduct faster measurements because they do not need to sweep any voltages during measurement. However, in a multi-probe LP, care must be taken to spatially spread out the probes to avoid cross-talking.

This project focuses on designing, fabricating, and characterizing compact single, double, and triple LPs with integrated, custom electronics (Figure 1) for CubeSat and laboratory plasma applications. The probes will be manufactured via rapid prototyping, exploring the limits of the technology to implement sensors compatible with the coldest, densest plasmas possible, as well as the ionosphere. The circuitry is being designed to enable low-power, autonomous operation. To fabricate the probes, we plan to use metal 3D printing (Figure 2), laser cutting, and plating for creating the electrically conductive parts of the probes and ceramic 3D printing for the dielectric parts of the probe and housing of the electronics. Current research focuses on completing the hardware design, which will be followed by device/driving electronics manufacture and characterization.



▲ Figure 1: Integrated circuit design for triple LP.



▲ Figure 2: CAD of a 3D-printed, single LP electrode.

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# Compact, Digitally Manufactured Retarding Potential Analyzers Enabled for CubeSat and Laboratory Plasma Diagnostics

J. Izquierdo-Reyes, Z. Bigelow, N. K. Lubinsky, L. F. Velásquez-García Sponsorship: MIT Portugal, MIT-Tecnologico de Monterrey Nanotechnology Program

Retarding potential analyzers (RPAs) are multi-gridded sensors that determine a plasma's ion energy distribution (Figure 1). In this project, we are developing novel, digitally manufactured RPAs for CubeSat and laboratory plasma diagnostics. Unlike most RPAs reported in the literature, our devices enforce aperture alignment across the grid stack, maximizing ion transmission. The core of the device is a set of laser-cut electrodes assembled into a 3-D printed Vitrolite® (a glass ceramic) housing (Figure 2). Characterization of the Vitrolite® printing process shows an in-plane, per axis manufacturing accuracy of 60  $\mu$ m for the as-printed (green) parts and a ~5% shrinkage for the parts annealed at 900 °C; higher annealing temperatures cause significant distortion of the printed part due to material reflow, which is problematic for an engineering application. Also, the assembly misalignment between the grids and the housing significantly worsens when using a housing annealed at 900 °C. Characterization of the devices via simulations and experiments is consistent with expected performance and the literature. Plasmas with a Debye length as small as 50  $\mu$ m have been successfully characterized using the reported sensors, matching the performance of state-of-the-art RPAs manufactured via semiconductor micro-fabrication.





Figure 2: A partially assembled retarding potential analyzer.

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## FFF 3D-Printed Inductors for PowerMEMS

J. Cañada, L. F. Velásquez-García Sponsorship: Empiriko Corporation

Magnetic actuators are among the most essential building blocks of electromechanical systems. In this project, we are exploring ways to manufacture electromagnetic actuators with commodity 3D printers. The 3D printing of electromagnetic actuators can enable fast and inexpensive prototyping and customization in fields like PowerMEMS and robotics, including soft robotics.

Fused filament fabrication (FFF) is a 3D printing method in which a thermoplastic-based material is extruded through a nozzle and deposited layer by layer to construct solid parts. FFF printers equipped with multiple nozzles allow the simultaneous use of several materials, which facilitates the monolithic fabrication of multi-material parts. By using such printers, we intend to monolithically print electromagnetic actuators consisting of permanent magnets, electrically conductive inductors, and rigid or flexible frames.

The 3D printing of magnets has already been demonstrated in previous work, and we are currently

working on printing inductors using a copper nanoparticle-doped thermoplastic. The resistivity of this material is three orders of magnitude above that of copper, which raises the challenge of generating useful magnetic fields with only moderately conductive material. A spiral printed with this material is shown in Figure 1.

Figure 2 shows an estimate of the magnetic fields that an inductor built up of stacked spirals like the one shown in Figure 1 can generate. This estimation was computed under two restrictions: (i) the current through the inductor can never exceed the maximum current recommended by the material manufacturer, and (ii) the voltage applied across the inductor cannot exceed 200 V. The results indicate that the maximum achievable magnetic field is on the order of a few Gauss. Upcoming work will explore ways to boost these magnetic fields by adding magnetic cores to the printed inductors.



Maximum achievable magnetic field (G)

6

30

▲ Figure 1: Spiral printed using copper-doped thermoplastic.

▲ Figure 2: Estimate of maximum achievable magnetic field as a function of inductor size. Outer radius translates to the number of loops in a spiral; length translates to the number of stacked spirals.

#### FURTHER READING

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- A. P. Taylor and L. F. Velásquez-García, "Miniaturized Diaphragm Vacuum Pump by Multi-material Additive Manufacturing," J. Microelectromech. Sys. vol. 26, no. 6, pp. 1316-1326, Dec. 2017.

# Nanowire-Coated Emitter Electrospray Ionizer Coupled to Digital Microfluidics for Liquid Analysis

A. Kachkine, L. F. Velásquez-García Sponsorship: Empiriko Corporation

Liquid sample processing for mass spectrometry involves the extraction of a target analyte, addition of solvents, and ionization. Solid-state, programmable digital microfluidics have emerged as versatile platforms for sample manipulation, while ionization can be efficiently conducted via electrospray, i.e., the emission of charged molecules from a liquid subjected to a high electric field. Our group previously developed nanowire-coated emitters, which have pure ion emission and are thus of interest to mass spectrometry due to potential noise reduction. We present the first integration of high-efficiency nanowire-coated emitters and digital microfluidics: a single device for sample manipulation and ambient electrospray (Figure 1).

Device fabrication takes ~7 hours and costs ~\$5 in materials, with preparation of the digital microfluidic components taking under 10 minutes at a cost of ~\$1 with no cleanroom processing. The emitter is made out of steel via binder jetting, and then undergoes electropolishing, seed layer deposition, and zinc oxide nanowire growth in a hydrothermal bath. Digital microfluidic components comprise commerciallyfabricated printed circuit boards, laser cut plastics, drop-cast Teflon layers, and a polymeric casing made via digital light processing.

Sample droplets are moved through the device and onto the emitter via a paper conduit. Under comparatively low emitter extraction voltages (2-2.5 kV) our device has two-fold greater electrospray currents than state-of-the-art methods used with digital microfluidics (Figure 2). The electrospray dynamics are time-variant: an initial unstable, shortduration Taylor cone is followed by a secondary, stable electrospray without a visible plume, lasting about 20 seconds while using 5µL of solvent. Ongoing work focuses on characterizing devices interfaced with a mass spectrometer, improving device reliability, and fully characterizing electrospray dynamics. Images are from the first further reading publication below.



 Figure 1: (a) Fabricated device with receptor used for externally actuating device functions and (b) labeled exploded view of device, showing key internal components.

◄ Figure 2: (b) Electrospray of isopropanol from nanowire-coated emitter (device casing removed) and (b) plot of average current versus voltage for nanowire-coated emitter (circles), paper emitter (triangles), and coated blade (squares) with isopropanol as solvent.

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## Internally Fed, Additively Manufactured Electrospray Thrusters for CubeSats

H. Kim, L. F. Velásquez-García Sponsorship: MIT Portugal

Electrospray engines have one or more emitters that electrohydrodynamically eject high-speed charged particles from liquids to produce thrust. Stable electrospray emission, which can be used in a propulsion application, exists for a limited range of electric fields and flow rates. Whether the thruster emits ions or droplets is determined by the flow rate of the propellant. On the one hand, if the flow rate is below a certain threshold value, the engine emits ions, exerting a high-specific impulse, low force to the spacecraft. On the other hand, if the flow rate is above the threshold value, the engine emits droplets, creating a larger thrust with lower specific impulse. In principle, both kinds of emission are useful for propelling a spacecraft.

The electric field strength needed to activate the electrospray emitters is proportional to the square root of the emitter diameter; therefore, electrospray thrusters benefit from miniaturization, as scaled-down emitters turn-on with less voltage. Miniaturization of electrospray engines has traditionally been accomplished using semiconductor fabrication. However, this manufacturing approach is expensive and time-consuming. Therefore, fabrication approaches that can create complex hardware at a low cost and manufacturing time would greatly help lower the cost of space hardware including CubeSats.

In this project, we are exploring additive manufacturing via digital light processing to create CubeSat electrospray thrusters. We are currently focusing on developing electrospray engines that emit droplets. Before testing multiplexed designs, we are first investigating a single-emitter design to check the essential parameters for electrospray (Figure 1). The high electric field acting on the electrospray emitters is affected by the bias voltage between the propellant and the electrode that extracts droplets, as well as the separation distance between the emitter and the extractor electrode. Therefore, the design needs to be optimized to minimize the start-up voltage. In addition, the upper limit for the flow rate to sustain stable droplet emission needs to be characterized.



 Figure 1: The Taylor cone generated on the additively manufactured emitter.

- D. Olvera-Trejo and L. F. Velásquez-García, "Additively Manufactured MEMS Multiplexed Coaxial Electrospray Sources for High-Throughput, Uniform Generation of Core-Shell Microparticles," Lab on a Chip, vol. 16, no. 21, pp. 4121-4132, Oct. 2016.
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# Soft Material Design of Additively Manufactured Actuators to Control Stress Distribution

H. J. Lee, L. F. Velásquez-García Sponsorship: Empiriko Corporation

Rigid materials are ubiquitous in engineering because their behavior is easy to predict and can support a great amount of weight. However, they are not suitable in unpredictable environments and can be dangerous when used in systems that involve human interaction. Soft and flexible materials, on the other hand, are compliant and can deform to match the shape of any object that comes into contact; this allows the structure to distribute stress and enable safer interaction with the environment. As a result, soft materials are now used in a wide range of applications including flexible electronics, soft robotics, and biological applications. While there are many methods to manufacture structures with soft materials, recent development in additive manufacturing allows fabricating complex designs with geometrical features unmatched by traditional manufacturing methods. Therefore, the structures can be designed to improve many properties for structural or thermal applications.

In this project we are designing and fabricating soft actuator structures to improve the fatigue properties under cyclic compression. Under compression, soft materials tend to fold themselves, which leads to a significant amount of stress concentration. While this might not be a problem for a small number of compressions, the fatigue life becomes an issue when the structure is applied to actual products that require a reasonably long lifespan. In addition to the material properties, structures fabricated through additive manufacturing have anisotropic behavior, where failure is most likely to occur between layers. Figure 1 shows the stress concentration of a flexible ring with a design that distributes the stress to a wider area, reducing the maximum stress within the structure. These designs can easily be fabricated with additive manufacturing, as shown in Figure 2. The sample shown is fabricated with Ninjaflex (a commercial thermoplastic polyurethane printable material) using an extrusion 3D printer.



▲ Figure 1: Solidworks was used to simulate the compression of a hyperelastic material. The structure is designed to distribute stress concentration to improve the fatigue property.



▲ Figure 2: Image shows the structure fabricated through additive manufacturing.

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## Improving Reliability and Performance of Microhydraulic Electrowetting Actuators

J. Kedzierski, I. Liu, L. Racz, L. F. Velásquez-García Sponsorship: MIT Lincoln Laboratory

Electrowetting is the phenomenon of altering the surface tension and wetting properties of liquids by applying electric fields. Applying an external voltage to a liquid droplet changes the droplet's shape and contact angle to solid surfaces, thus affecting the attraction and friction between droplets and solid surfaces. Electrowetting can reversibly transport and shape fluids; thus these effects have been used in multiple applications for manipulating small amounts of liquids, such as adjustable fluid optical lenses and droplet display arrays.

Microhydraulic actuators can be made by assembling a rotor and a stator separated by liquid droplets. Applying external electric fields to the droplets modifies the wetting and surface properties, thus actuating the droplets. Microhydraulic actuators offer several benefits over conventional motors. Microhydraulic actuators are lighter, flexible, and scalable to small dimensions. In contrast, conventional motors that rely on wire coils are often limited by coil size and high resistance when scaled down. The small scale of microhydraulic actuators leads to high power density, enabling effective conversion of electrical to mechanical power in compact spaces.

This project is based on prior work at Lincoln Laboratory regarding rotational microhydraulic actuators. The rotor moves relative to the stator by applying external electric fields to the droplets, modifying the wetting and surface properties with electrowetting. The main goal is to improve reliability—currently, the main challenges include lowering friction, stabilizing

rotation, and extending operation time. The project will include experiments to determine the root causes of unreliable operations and incorporate corresponding design changes. The resulting microhydraulic actuators will combine reliable operations with high power density, small size, and precise motion. Thus, the project has vast potential in biomedical and robotics applications.



▲ Figure 1: Operating principle of microhydraulic actuators. Water droplets are shifted along by applying sequential electrical phases. From Kedzierski et al., Science Robotics 3, no. 22, eaat5643, 2018.

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## **3D-Printed Reflectron for Compact Mass Spectrometry**

N. Lubinsky, L. F. Velásquez-García Sponsorship: Empiriko Corporation

Accurate measurement of blood plasma constituents in the medical clinical setting is incredibly important for drug assays and monitoring of biomarkers. However, the traditional gold standard is a triple quadrupole, an investment requiring a large amount of power, space, and a \$10 million budget. Other applications for low false-positive, compact mass spectrometry also face the same economical burdens. Our research group aims to mitigate these with the additive manufacture methods available to 3D printing mass spectrometers for high precision and miniaturization.

We are currently exploring a two-piece reflectron mass spectrometer—a device that measures the timeof-flight (TOF) of ions as they are reflected by the internal electromagnetic fields. The design geometry of a hyperbola (Figure 1) surrounded by a cone creates a potential distribution that is quadratic with distance. Thus, the ions exhibit a repelling force that is independent of the initial energy of the ion; therefore, we can observe a TOF solely dependent on the specific mass-per-charge of the ions.

Methods of operation employed by this reflectron involve the utilization of an entrance gate electrode, with an aperture for ions. Once potential drops to o V on the gate, ions flow into the reflectron, ultimately reflecting towards the entrance, to a nearby anode. Ions colliding will draw current; however, the current intensity will vary in time depending on the TOF of the ions. The delay of the gate switching off to the time we register current intensity is completely dependent on the specific charge of the ion, and mass spectrometry can be performed. In our approach, the electrodes are 3D-printed in glass-ceramic via digital light processing (DLP). Post-print, we selectively plate them, forming conductive surfaces. Current research efforts focus on optimizing the hardware and characterizing such hardware in vacuum.



▲ Figure 1: Hyperbolic electrode 3D printed in glass-ceramic via DLP and coated in nickel.

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# Fully 3D-Printed Electronics via Multi-Material Microsputtering

Y. S. Kornbluth, L. Parameswaran, R. Mathews, L. M. Racz, L. F. Velásquez-García Sponsorship: Kansas City National Security Center

Integrated circuits (ICs) are made in multibillion-dollar foundries, involving extreme processing conditions. To attain low per-chip cost, many identical IC chips are batch processed. Lower volume electronics are manufactured at a low cost using printed circuit boards, where premade components, often made in a foundry, are soldered onto a dielectric plate with an arrangement of thin film conductive traces and a set of drilled vias. However, currently, there is no cost-effective approach to make a small batch of ICs, conduct chip-tochip customization, or rework ICs.

In this project, we are harnessing microsplasma sputtering (i.e., the sputtering of materials at atmospheric pressure using reactors with characteristic length below millimeters) to develop a manufacturing platform for agile manufacturing of ICs. We recently reported the first fully additively

manufactured capacitors as a proof-of-concept demonstration of such multi-material microplasma sputtering manufacturing platform. This is also the first demonstration of a cleanroom-quality, multimaterial electrical device produced entirely through additive manufacturing. The conductive films are created by sputtering gold in air, attaining nearbulk electrical conductivity. The dielectric films are created by sputtering aluminum in a gas blend of argon and air, resulting in alumina films (Figure 1). The frequency response of the capacitor is described by the universal dielectric response typically found in heterogenous dielectrics and suggests the presence of condensed water in the pores of the alumina film. Future work could entail extending the platform to other transducing materials, e.g., semiconductors.



▲ Figure 1: (a) Photograph of a fully microsputtered capacitor composed of two perpendicular gold lines separated by a thin alumina film. (b) SScanning electron Emicroscope (SEM)M cross-section of microplasma-printed capacitor; the gold films are 50 nm thick, while the alumina film is 35 nm thick. From Y. Kornbluth et al., Advanced Materials Technologies (2022).

<sup>•</sup> Y. Kornbluth, R. H. Mathews, L. Parameswaran, L. Racz, and L. F. Velásquez-García, "Fully 3D-printed, Ultrathin Capacitors via Multi-material Microsputtering," Advanced Materials Technologies, to be published, 2022.

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