

Characterisation of electrospray microemitters fabricated by planar and 3D photolithography

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Electrospray microemitters were fabricated in photostructurable polymers using both conventional planar photolithography and three dimensional microlithography (two photon lithography). The fabrication process of volcano shaped emitters by 3D microlithography is under development, especially for structures made entirely from the photostructurable epoxy polymer SU-8. The most important parameters are identified and results of parameter screening are presented. Pending the availability of samples made entirely by 3D microlithography, emitter arrays fabricated in a mix-and-match approach with 3D microlithography on a substrate pre-patterned by planar photolithography were characterised electrically both in DC measurements and by a time-of-flight method. From the ionic liquid EMI-BF₄, negatively charged droplets with diameters between 0.15 μm and 0.4 μm were extracted. The active propellant feed was identified as a critical part of the characterisation setup.

Abbreviations and acronyms

EMI-BF ₄	1-ethyl-3-methylimidazolium tetrafluoroborate (an ionic liquid)
MEMS	micro electromechanical systems
PEEK	polyether ether ketone (a polymer resistant to high temperatures)
PTFE	polytetrafluoroethylene (a polymer known for its non-stick properties)
PIC	pressure indicator and controller
SU-8	(an epoxy-based negative tone photoresist)
ToF	time-of-flight

I. Introduction

Micropropulsion in the wider sense refers to all propulsion elements providing thrust at the millinewton or sub-millinewton range and is a rather wide field with a large number of potential applications, be it in providing propulsion to very small satellites such as CubeSats^{1,2} or to satellites requiring very high precision, small impulse-bit thrust, as in LISA-like missions.³

Micropropulsion in the narrower sense has the objective of creating very small thrusters (microthrusters) and to assemble as many of these microthrusters as needed into an array that delivers the desired thrust range for the intended application. This concept, known as “scaling-up by numbering-up”, is illustrated in Fig. 1. The individual microthrusters will have a footprint on the order of $100 \times 100 \mu\text{m}^2$, which means that they will have to be manufactured with methods derived from the technology of micro electromechanical systems (MEMS), which in turn have their roots in the technology of microelectronics miniaturisation.

One promising candidate operating principle for microemitters is electrospray (also known as colloid emitters). The propellant in this case is an ionic liquid, from which both positively and negatively charged droplets or, ideally, single ions can be extracted (and have to be extracted), eliminating the need for a

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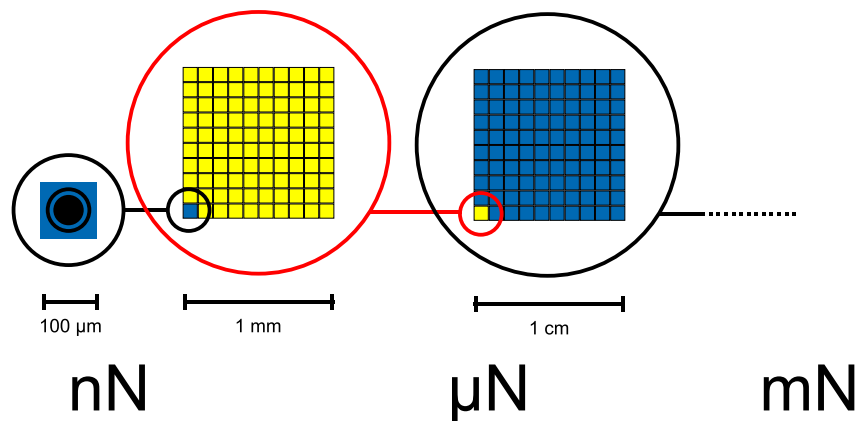


Figure 1. Illustration of the principle of “scaling-up by numbering-up”, creating thrusters of variable thrust range by assembling identical microemitters to arrays of different sizes.

separate neutraliser. For the fabrication of miniaturised electrospray emitters, namely internally wetted emitters which we shall concentrate on in the following, there are basically two technology pathways.

The silicon technology route has been taken successfully in the United States and, in the framework of the MicroThrust consortium, in Europe as well.⁴

An alternative pathway is the photopolymer route, where the critical parts of the electrospray are made by photolithographic methods in suitable polymers (photostructurable polymers or photopolymers). The photopolymer SU-8, an epoxy resin, appears to be a good candidate material due to its stability under harsh conditions. Using planar photolithography as in MEMS technology, we have made emitters from SU-8 and shown that problems inherent to planar technology emitters such as surface wetting can be mitigated by a suitable surface treatment.⁵ Parallel to these developments, the technology of 3D microlithography has matured to market readiness. The feasibility of creating (“writing”) microemitter structures with 3D microlithography has been shown earlier.⁶ With the introduction of a 3D microlithography system to our laboratory in 2019, we can now intensify the research on microemitter fabrication by 3D microlithography, notably in SU-8, which is a resist that has not been optimised for the application in 3D microlithography. The first results obtained using our Nanoscribe PPGT 3D microlithography system (from Nanoscribe, Karlsruhe) are presented in the following.

II. Experimental work and results

A. 3D photolithography of electrospray emitters

1. Principle of two photon lithography

The principle of two photon lithography is illustrated in Fig. 2. A substrate is coated with a photosensitive polymer (photoresist). When the photochemical reaction in the resist is triggered, the resist becomes either more easily soluble in a developer solution and the exposed portions of the resist are finally removed (positive tonality of the resist), or a cross-linking occurs which renders the exposed areas less soluble than the unexposed areas, resulting in the exposed portions of the resist remaining after the development step (negative tonality of the resist). 3D microlithography mostly uses negative resists, and SU-8 is an example of a negative resist.

A near-infrared beam from a femtosecond laser, whose photon energy is too low to initiate the photochemical reaction, is focussed through microscope optics (in the case of the Nanoscribe PPGT, the microscope is actually inverted). In a very small volume element around the focus of the laser beam, the intensity is so high that two-photon processes occur, and the energy of two photons is sufficient to start the photochemical reaction. This volume element (voxel) is elongated along the direction of the beam (the z axis). Its diameter in the x - y plane is less than 500 nm and the extension in z direction generally on the order of 2 μm or less, the exact values depending on several parameters, especially on the laser power. The smaller the power, the smaller the extension of the voxel in all directions.

The voxel is scanned through the photoresist in three dimensions, always starting from the surface of the substrate. The first generation of Nanoscribe instrumentation on which the first demonstrator structures for 3D electrospray microemitters were made⁶ featured only piezo motors that moved the stage with the sample through the fixed laser beam (piezo scan). The next generation, to which the PPGT used here belongs, additionally features a fast scanning of the laser beam in the horizontal plane

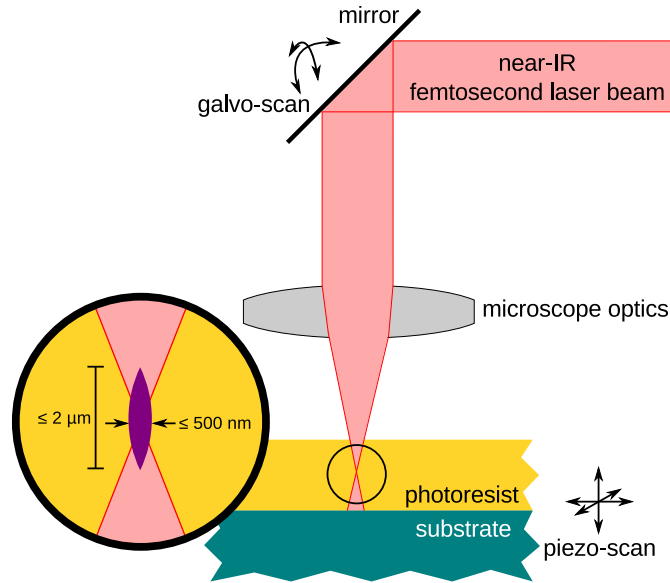


Figure 2. Principle of two photon lithography

by small mirrors in the beam pathway (galvo scan). The writing speed for typical (volcano shaped) emitters is enhanced by almost two orders of magnitude by applying galvo scan instead of piezo scan.

Two photon lithography can be carried out in a number of sample configurations, as summarised in Fig. 3. The highest resolution is achieved in one of the two immersion configurations, with either the objective immersed in immersion oil at the back side of the (obviously transparent) sample and a puddle of resist deposited on the front side, or with the objective immersed in a puddle of resist on the sample's front side. The air gap or non-immersion configuration is marketed by Nanoscribe as the “maskless 2D lithography” configuration, but as will be shown in the following, this configuration is very well suited for the microfabrication of 3D microstructures as long as sub-micrometre details are not required. In this configuration, the substrate is pre-coated with photoresist and mounted face-down in the PPGT device. The obvious disadvantage of this configuration is that the additional interface between air gap and resist surface will degrade the quality, namely the positioning accuracy and the resolution, of the lithography. This disadvantage is, however, more then outweighed by the advantage of having a wider selection of resists available. In particular, this configuration allows the use of SU-8 resist, while the immersion configurations generally are limited to specifically designed resists for 3D lithography, such as IP-Dip or IP-S. Additionally, spincoating of an entire wafer at once and subsequent dicing of that wafer allows much better control of the resist thickness than coating smaller dies, not least since the areas affected by resist thickness inhomogeneities (edge beads) along the edges of the wafer can be generously discarded after dicing.

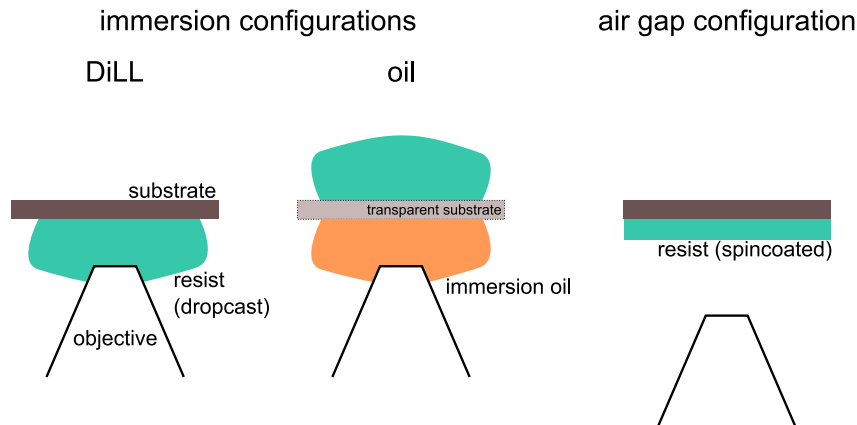


Figure 3. Two photon lithography configurations that the Nanoscribe PPGT offers. The immersion configurations are needed in order to obtain the highest resolutions, but the air gap configuration was chosen in this work since it allows the use of pre-coated substrates.

2. Writing parameters for SU-8 resist

All samples were made on silicon substrates. Two inch silicon wafers, single side polished, with a nominal thickness around 280 μm , were coated with SU-8 50 resist purchased from Microresist Technology, Berlin. After a single bake of at least 5 minutes duration on a 180 °C hotplate, 2 mL of SU-8 50 were dispensed in the centre of the wafer with a syringe. The rotation speed was ramped from 0 to 500 rpm within 5 s in order to spread the resist evenly over the wafer, held at 500 rpm for 8 s, then ramped within 2 s to the final rotation speed of 1000 rpm. Holding at the final rotation speed for 30 s resulted in a homogeneous resist coating. The softbake (pre-exposure bake) was carried out in two steps (one minute at 65 °C, ten minutes at 95 °C) in order to minimise thermal stress. The resist thickness of the samples ready for exposure was found to vary slightly with the exact amount of resist dispensed and lay between 160 and 190 μm . One die of each wafer was selected for a test of the Nanoscribe interface finding parameters.

The laser energy, measured as percentage of the full laser power available, is a very critical parameter for writing in SU-8. When this laser power is too high, the heat generated around the focus cannot be dissipated sufficiently, and this results in the formation of small bubbles that in most cases will burst and destroy the resist coating and hence render the sample useless. We found 50 percent to be a good value for our SU-8 50 resist. The writing speed, on the other hand, was found to have practically no impact on the bubble formation problem, so there was no reason to deviate from the 50 000 $\mu\text{m s}^{-1}$ scan speed that the Nanoscribe PPGT allows.

3. Design parameters

There are essentially two ways of generating the CAD data that go into the lithography system. One option is to draw a structure using almost any CAD system, export that structure as an STL file and import that STL file in the NanoWrite software that comes with the Nanoscribe system. The conversion dialogue will then query a (large) number of parameters including the following:

slicing is the height (z) increment between subsequently written layers,

hatching is the lateral (x or y) increment between subsequently written lines in the same layer,

stitching determines how patterns that are too large to be written without moving the stage are to be composed of smaller patterns,

scaffolding determines to which extent massive structures are not to be written in their entirety, but only along their surfaces and internal support structures, with the rest of the resist remaining unexposed. After development, that remaining unexposed resist can be exposed in a flood exposure (of course, this only makes sense for negative resist).

An alternative pathway to drawing and importing a structure is to write a programme in the Nanoscribe GWL language. One can either use a high level programming language and make that output low-level GWL code, or one can use the structures (especially loops) that GWL in itself offers for scripting. This approach, which we have chosen for the fabrication process development described in the following, gives the user full command over the writing process and easily allows a systematic screening of parameter ranges, for example by arranging patterns in an array and varying one parameter with each array index.

Figure 4 and shows the result of such a parameter screening for tubes with a square cross section. Both images in that figure very well illustrate the high aspect ratios that can be achieved in SU-8 resist. A wall thickness of a few micrometres is sufficient for a wall height exceeding a hundred micrometres.

4. Mix-and-match with 2D vs. integral 3D photolithography

The geometry of the emitters is tapered around the orifice in order to prevent wetting of the surface by the ionic liquid. These “volcano” emitters need to be placed on an SU-8 baseplate in order to be handled, especially, to be glued onto the propellant tank. Such a baseplate should measure at least seven by seven millimetres and its thickness should be at least 30 μm to allow safe handling. In the past, we have therefore adopted a mix-and-match approach to sample fabrication. First, a membrane of SU-8 with the desired thickness of at least 30 μm was patterned with a fluidic via pattern on a silicon substrate by planar photolithography, using a broadband ultraviolet light mask aligner (Suss MA56) and a photomask procured from a mask shop (Rose Fotomasken, Bergisch Gladbach). The SU-8 on the silicon substrate was then dropcast with Nanoscribe 3D specialty resist and patterned, with the volcanoes aligned to the fluidic vias in the SU-8. This mix-and-match approach speeds up the process substantially, but it has its disadvantages. Using SU-8 in the second mix-and-match stage is difficult due to the small difference in

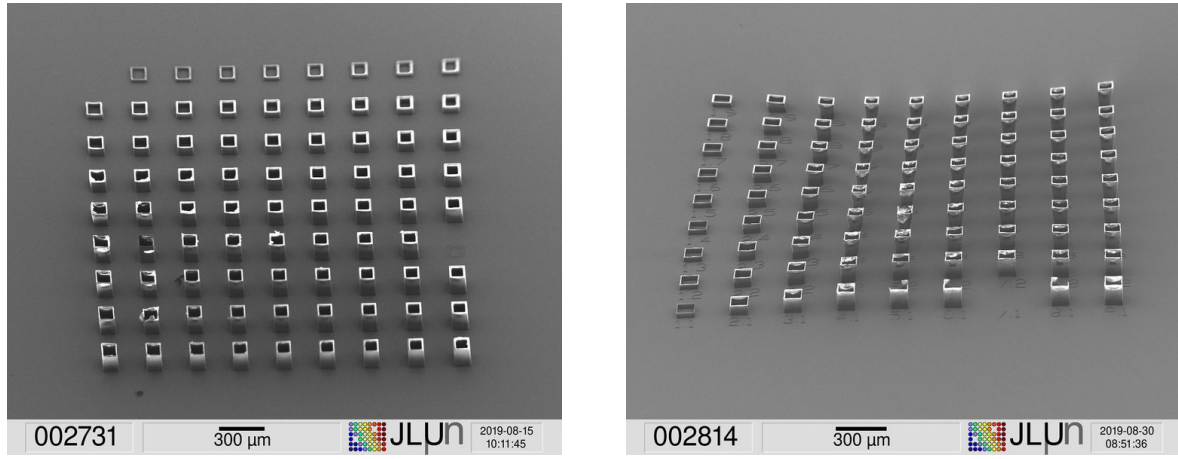


Figure 4. Systematic parameter variation for tubes with square cross sections in SU-8. The nominal tube height was varied from 20 μm to 180 μm (from top to bottom in the left image, from left to right in the right image) and the wall thickness was varied from 3 μm to 12 μm from left to right in the left image and from 2 μm to 6 μm from front to back in the right image. The tube height levels off as the nominal tube height exceeds the actual resist thickness of approximately 160 μm .

diffraction index between exposed (and developed) and unexposed SU-8. In addition, there is a danger of surface contamination between the two stages that will endanger the proper adhesion of the resist in the second stage. It is therefore clearly preferable to write the whole emitter structure as needed in the experiment in a single step. This in turn means that one will have to write the surface of the base plate and an array of supporting walls, while leaving the vast part of the resist inside the baseplate unexposed ("shell writing" and scaffolding). The base plate will then be cross-linked by a subsequent flood exposure.

Figure 5 shows the result of a shell writing test. A wall thickness of 2 μm is obviously insufficient for columns of even moderate heights, but as soon as the wall and cover thickness reaches and exceeds 3 μm , the columns are indeed very stable. One can expect from these results that when a large area is to be stitched together from such columns, a support wall interval of 100 μm with a wall width of 3 μm should provide enough mechanical stability. Tests for writing such large areas are now underway.

The volcano emitters shown in Figures 6 and 7 were written in the bulk (as opposed to shell writing). It is obvious that the Nanoscribe PPGT facilitates the fabrication of high quality volcano emitters. The process of optimising the design with respect to speed is presently going on. The algorithmic approach to defining the structures (as opposed to CAD drawing and importing) allows to have both the critical structures (capillaries and orifices) written with parameters optimised for quality, and at the same time,

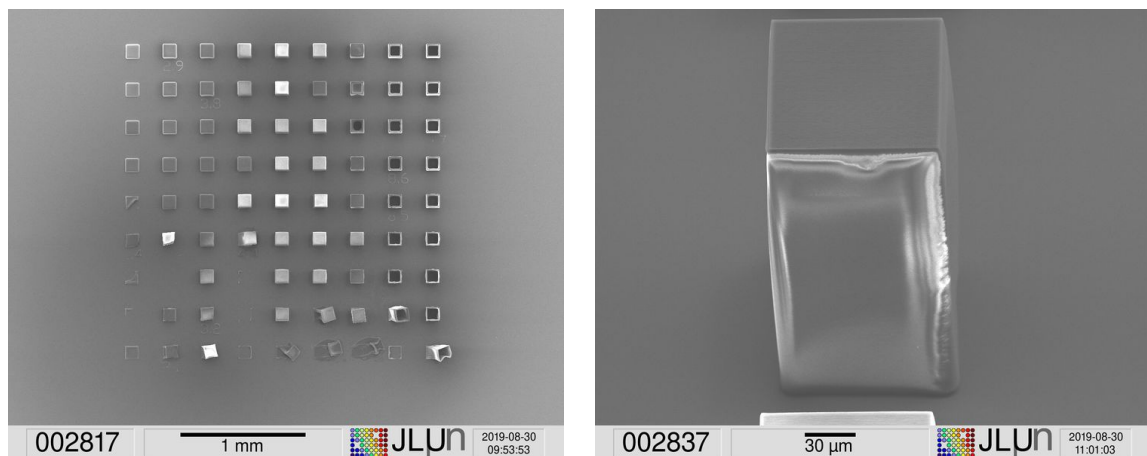


Figure 5. Shell writing of columns with square cross section in SU-8. The nominal column height was varied stepwise from 20 μm to 180 μm from left to right in the left image and the thickness of the column walls and the top cover was varied stepwise from 2 μm to 6 μm from bottom to top in the left image. When the nominal column height exceeds the actual resist thickness around 150 μm , the column covers are missing. The right hand image shows a column seen under a tilt angle of 40°. The resist inside the columns is still unexposed in the examples shown here.

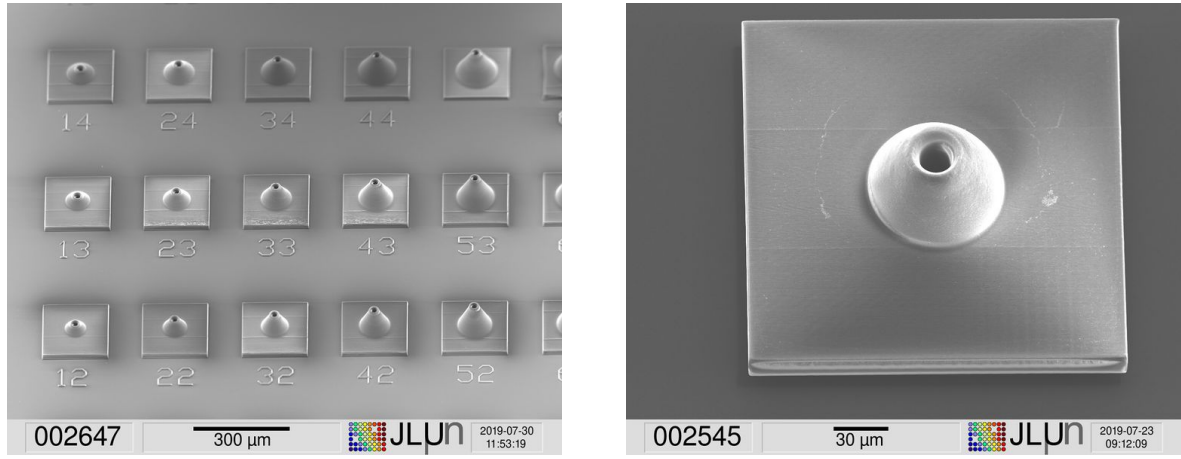


Figure 6. Volcano emitters on SU-8 baseplates written in the bulk in SU-8. All capillaries have an inner diameter of $20\text{ }\mu\text{m}$. The height h of the capillary's orifice above the baseplate surface was varied stepwise from $10\text{ }\mu\text{m}$ to $50\text{ }\mu\text{m}$ from left to right in the left image and the base radius of the volcano was varied stepwise from $\frac{h}{2} + 10\text{ }\mu\text{m}$ to $\frac{h}{2} + 20\text{ }\mu\text{m}$ from front to back in the left image. For the structures in the left image, slicing and hatching was $0.3\text{ }\mu\text{m}$ for the capillary tube and $0.6\text{ }\mu\text{m}$ for the supporting structures including the volcano. The capillary tube was written with a radial hatching of $0.1\text{ }\mu\text{m}$ and an angle increment between straight sections corresponding to an arc of $0.1\text{ }\mu\text{m}$ length. The volcano structure in the right image extends $20\text{ }\mu\text{m}$ above the baseplate surface and has a base radius of $30\text{ }\mu\text{m}$, it was entirely written with slice and hatch of $0.3\text{ }\mu\text{m}$ and with the same radial parameters as for the structures in the left image.

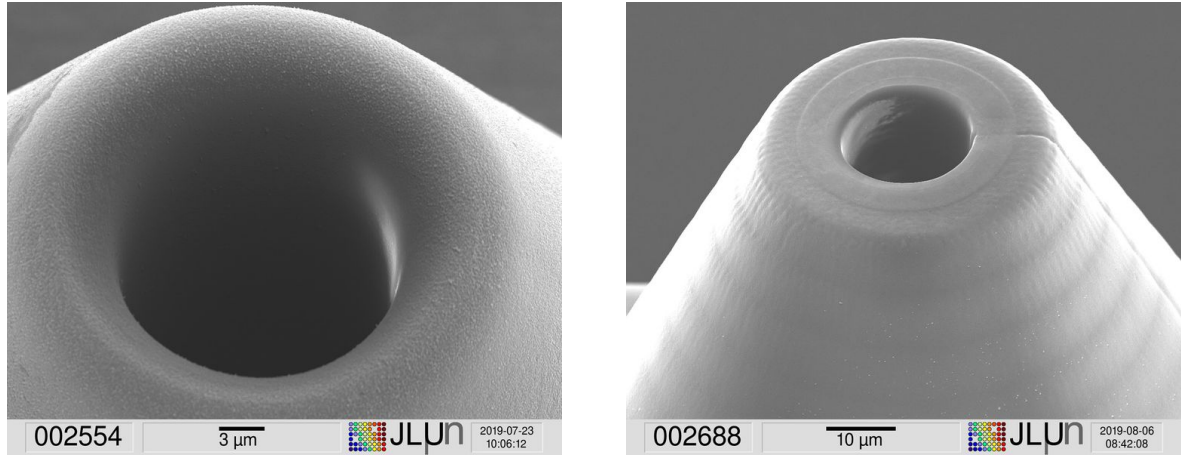


Figure 7. SEM images of volcano emitter capillary orifices demonstrating the high quality of the surfaces. In the right image, the distinction between the capillary tube written with a radial hatching of $0.1\text{ }\mu\text{m}$ and the surrounding volcano taper written with a radial hatching of $0.4\text{ }\mu\text{m}$ is visible. The seam visible at the three o'clock position in the right image indicates the zero degrees angular position where the sequence of lines approximating the circles started and ended, respectively. A slight hint of the voxel structures is visible in the right image written at the relatively coarse slice and hatching of $0.6\text{ }\mu\text{m}$.

to have the supporting volcanoes and especially the baseplate written with parameters optimised for speed, in order to keep overall writing times acceptable.

B. Characterisation of electrospray emitters

1. Electrical (emission) characterisation setup

The measurement setup is shown as a schematic drawing in Fig. 8. The SU-8 membrane with the capillary or array of capillaries is glued to a PEEK tank filled with EMI-BF₄ ionic liquid. An external extraction electrode with an inner diameter of 5 mm is placed at a distance of a few millimetres from the sample. The ionic liquid tank is put either at negative or positive high potential, and the extraction electrode is grounded. Electrospray is collected on a copper plate, and the resulting current is amplified in a circuit, placed inside the vacuum chamber in order to minimise noise. The amplified signal is fed to the outside and measured both with a digital multimeter, averaging the DC signal over a period of approximately one second, and with an oscilloscope, providing time resolution down to the limit given by the 11 kHz cutoff frequency of the amplifier circuitry.

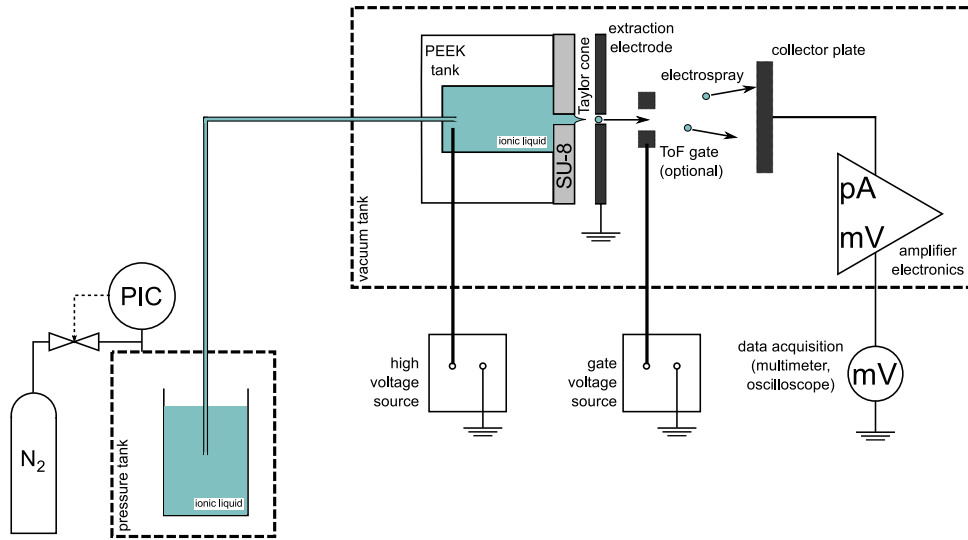


Figure 8. Measurement setup with active propellant feed system and with a shutter gate for time-of-flight (ToF) measurements

A crucial element of the setup is the active propellant feed system, modelled after the system used by Busek.⁷ A reservoir of ionic liquid is kept in a separate vacuum container and the replenishment feed rate is affected by the pressure difference between the propellant reservoir (pressure chamber) and the main vacuum chamber.

2. DC characterisation of mix-and-match microemitters

The device under test for the data presented in the following was an array of 4×4 microemitters. The sample was fabricated by pre-patterning an SU-8 membrane with fluidic vias with a diameter of $50 \mu\text{m}$ each. Cones with a capillary diameter of $10 \mu\text{m}$, a base diameter of $75 \mu\text{m}$ and a taper of approximately 40 degrees were then placed on top of each of the sixteen vias by Nanoscribe 3D microlithography. After development, the substrate wafer was dissolved in a 20 % solution of potassium hydroxide at a temperature of 60°C . Optical microscope inspection indicated that at least fourteen of the sixteen emitters should have been operational. A single external extraction electrode with an inner diameter of 5 mm placed at a distance of 3 mm from the sample surface was used in the following.

The pressure difference between the pressure tank containing the ionic liquid supply and the vacuum

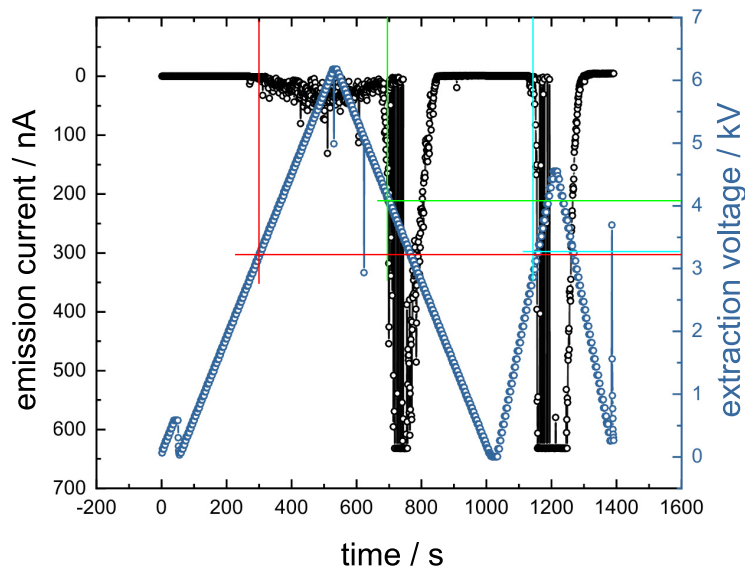


Figure 9. Time traces of extraction voltage and emission current for a 4×4 array of volcano microemitters

Table 1. Properties of the emitted species (negative), derived from time-of-flight measurements

time of flight	27 ms	43 ms	78 ms
specific charge	$4.2 \cdot 10^{-3} \text{ C kg}^{-1}$	$1.7 \cdot 10^{-3} \text{ C kg}^{-1}$	$0.5 \cdot 10^{-3} \text{ C kg}^{-1}$
contribution to thrust	196 μN	77 μN	142 μN
droplet radius	0.19 μm	0.26 μm	0.39 μm

tank, applied via a capillary of length 0.5 m and an inner diameter of $\frac{1}{16}$ inch, actively pushed the propellant into the tank at the emitter's backside. At a pressure difference of 0.15 MPa, the capillaries could be filled by applying high voltage, but it took the maximum voltage value of 6 kV that the high voltage source could deliver. Still, a time lag between application of the high voltage and onset of the extraction current of up to a minute remained for pressure differences below 0.2 MPa, even after the initial filling.

Figure 9 shows time traces of the extraction voltage and of the emission current at a propellant feed pressure difference of 0.22 MPa. Obviously, there is a strongly hysteretic component in the relation between the extraction voltage and the emission current that results in a current peak occurring upon reduction of the extraction voltage from the maximum. This extraction voltage value also marks the threshold for the extraction at a subsequent cycling of the extraction voltage.

It should be noted that no ionic liquid was extracted from the capillaries unless by application of an extraction voltage, regardless of the pressure difference in the range up to 0.28 MPa. The horizontal mounting of the device under test inside the vacuum tank implies that any ionic liquid extracted and not emitted through the extraction electrode would have wetted the surface and that a short circuit would have been created that would have been detected as an overload in the high voltage source.

The conclusion to be drawn from these observations is that the propellant filling and replenishment is a rather complex process. In the range of propellant feed pressure differences applicable, other pressures, probably mostly surface tensions, are non-negligible, so that there is no simple relation between the pressure difference and the mass flow rate of the propellant. The interplay between the parameters governing the propellant replenishment need to be understood better in order to map the operating regime of the microemitters.

3. Time-of-flight characterisation of a single microemitter

As shown in Fig. 8, the setup was equipped with a Bradbury-Nielsen gate that allowed time-of-flight measurements for the characterisation of the emitted species. The device under test for the data shown

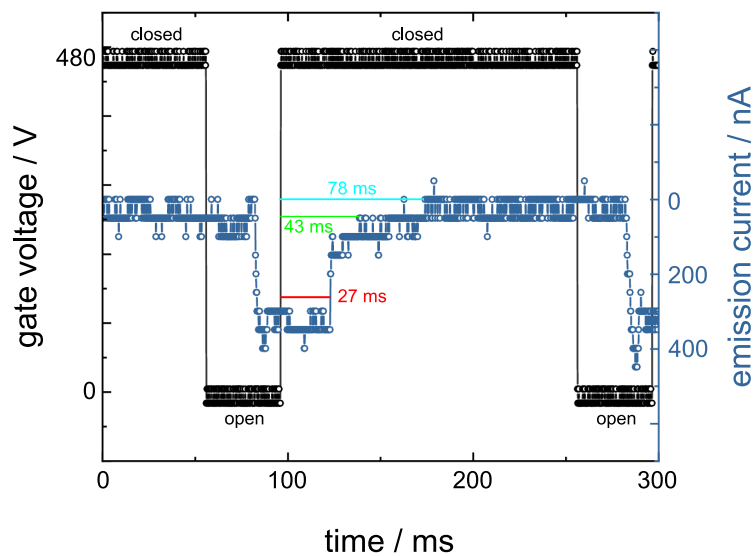


Figure 10. Time-of-flight measurement: traces of emission current and gate voltage for a single microemitter

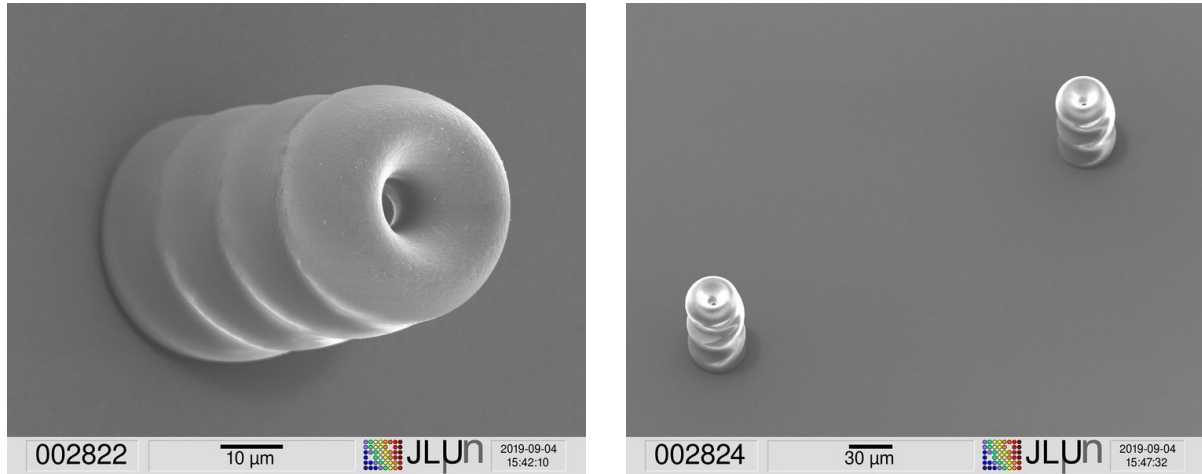


Figure 11. Spiralling capillary tubes, an illustrative example of the freedom in design that 3D microlithography provides

here was a single emitter made by planar photolithography with a capillary diameter of $50\text{ }\mu\text{m}$, surface treated by sputter coating PTFE in order to mitigate wetting of the SU-8 surface by the ionic liquid. The distance from the sample surface to the extraction electrode was 5.9 mm , the flight distance from the extraction electrode to the collector plate was 110.8 mm . The ionic liquid was fed with a pressure difference of 0.15 MPa and extracted by applying a voltage of 2 kV . The Bradbury-Nielsen gate was operated with a square voltage signal of 480 V at $5\text{ cycles per second}$, with an open time of 40 ms and a close time of 160 ms in each cycle. Figure 10 shows a typical example of the emission current as a function of time, superimposed with the gate voltage signal.

The resolution of the emission current is relatively low due to the low absolute value of the emission current of this single emitter. Still, one can derive from the measurements that the majority of current was carried by three species. Their times of flight, specific charges, contribution to thrust, and droplet radii (the latter two calculated assuming a single charge) are tabulated in Table 1.

Upon variation of the extraction voltage, the composition of the emission varied as well. However, all droplet radii as derived from the respective observations lay in the range between $0.1\text{ }\mu\text{m}$ and $0.4\text{ }\mu\text{m}$ and were thus considerably smaller than the $50\text{ }\mu\text{m}$ radius of the capillary orifice.

A further reduction and possibly even the transition to the ionic regime might be achievable when 3D microlithographically defined samples optimised for very small orifice radii become available. It remains to be seen what implications further reduced diameters in the fluidic path have on the propellant feed process.

III. Conclusion and Outlook

With the introduction of 3D microlithography, electrospray emitter design has gained an unprecedented freedom of design. This applies both to the outer shape of the emitters, especially the geometry of the surface in the critical areas around the capillary orifices, as well as to the design of the fluidic path between the tank and the orifice.

As an example of what is possible in 3D microlithography, Fig. 11 shows spiralling tubes, written in SU-8 using the processes as described above. Such spirals might be integrated inside the volcanoes if it turned out that this would favourably affect the emission properties.

The possibilities of 3D microlithography also re-open the subject of microfabrication of the extraction system, either in the same lithography process as the emitters themselves, or in a separate process with proper microassembly and packaging at a later stage. Feasibility studies in this area will have to indicate the best technology road to be followed.

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