

Investigation of the emission behavior of miniaturized SU-8 based colloid emitters

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We present a concept for sub-micronewton electric thrusters based on miniaturized colloid emitters, made using the photostructurable epoxy polymer SU-8, and employing the ionic liquid EMI-BF₄ as propellant. Typical diameters of the capillaries from whose orifices the propellant is to be extracted are on the order of 40 micrometers. EMI-BF₄ shows a high wetting (low contact angle) on SU-8, which may prevent the formation of a Taylor cone and may also lead to failure of the device due to electrolysis of the electrodes. The surface wettability could be lowered significantly by either sputter coating the SU-8 with PTFE, or by spin coating the SU-8 with a suspension of silicon dioxide nanoparticles in PDMS. From an SU-8 capillary layer so hydrophobised, positive species from an ionic liquid were successfully extracted, as verified by visual observation.

I. Introduction

A need for sub-micronewton thrusters may arise from a number of possible future space missions. For example, earth observation or sky telescope satellites might require a more precise attitude control than the one that can be achieved with today's thrusters. Micronewton thrusters might also be required for highly precise orbit control in deep space formation flight missions à la LISA. Both applications would likely also incur the necessity of sub-micronewtonsecond impulse bits. Finally, the evolving field of picosatellites might profit from smaller thrusters, which would most likely imply sub-micronewton thrust levels, for both orbit and attitude control.

Electric thrusters have the advantage of fast switching with no moving parts, which introduces less mechanical noise on the satellite and offers the possibility to generate sub-micronewtonsecond impulse bits, over their main competitor, cold gas thrusters. It seems to be a prevailing opinion in the micropropulsion community that chemical thrusters are not likely candidates for the applications mentioned above, although the miniaturization of devices in which chemical reactions take place has made appreciable progress in areas like lab-on-a-chip or microprocess engineering.¹

Micronewton thrust levels will require a degree of precision of mechanical dimensions in the fabrication process of the thrusters that can best be met by microfabrication techniques now prevalent in the field of microsystem technology, namely in the manufacture of microelectromechanical systems (MEMS). This will automatically imply a miniaturization of the devices compared to their present-day predecessors, which are almost universally made by conventional mechanical machining, albeit with high precision. Miniaturization will allow a reduction of the overall size of thrusters, which is especially favorable in picosatellite applications, and it opens the possibility of introducing redundancies in the propulsion system that are not affordable with today's thrusters. Thus, the lifetime requirements for an individual microthruster might be considerably

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relaxed compared to today's situation, where, in the best case, a very small number of redundant thrusters are allowed by volume and mass restraints, and where the failure of this very small number of thrusters causes the whole mission to fail.

Not all concepts for electric thrusters are equally favorable for miniaturization. Most prominently, all concepts based on the generation of a plasma are less than ideal candidates for downscaling, since the surface-to-volume ratio increases with decreasing characteristic linear dimension, and the formation of plasma sheath layers at all surfaces makes the generation of a stable plasma rather difficult. A concept that is favorably downscaled is the field emission and acceleration of charged particles in an electrostatic field (field emission electric propulsion, FEEP). The first candidates for FEEP were liquid metal sources, employing for example cesium, and the construction and qualification of FEEP thrusters has made considerable progress. An issue currently under investigation is the possible contamination of the spacecraft by metal recondensing on the surface, especially on windows that consequently lose transparency. Another potential technical obstacle is due to the fact that the liquid metal has to be kept liquid at all times. Therefore, a shutdown and later restart of liquid metal FEEP thrusters is hardly an option, if for example power has to be saved during certain phases of a mission.

Ionic liquids, however, can be designed to be liquid over the temperature range expected on a satellite. From a chemical point of view, ionic liquids are molten salts that can have almost arbitrarily complex anions and cations. Both anions and cations can in principle be extracted, and since both can have a high mass, the electric propulsion is not limited to the emission of positively charged particles, as in the case of plasma based thrusters. This potentially eliminates the need for additional neutralizers, at the expense of requiring both negative and positive high voltage (NHV, PHV) properly conditioned for optimal particle extraction and acceleration. If both the cations and the anions comprising the ionic liquid are complex, a thruster concept will have to include the emission of ions of both positive and negative charges, since otherwise the ionic liquid would charge up and change its composition by electrolysis. Only in special cases, a replenishment of the ions emitted from a suitably designed chemical reaction in the ionic liquid supply system may be possible.

Colloid thrusters have been under development at various institutions over the recent past and into the present. These efforts have largely been based on the well-known microfabrication techniques available for both monocrystalline and polycrystalline silicon, among which crystallographic wet etching and high aspect ratio deep etching in plasmas are the most notable.^{2,3} Silicon, however, is intrinsically electrically conductive, albeit at a level that it can be considered an insulator for small voltages. At the high voltages required for electric propulsion with high specific impulses, however, the insulating properties of silicon are insufficient, and insulating layers have to be incorporated into the thruster design.

The photostructurable epoxy polymer SU-8 allows the fabrication of microstructures with aspect ratios not inferior to those achieved in silicon deep reactive ion etching. The use of SU-8 as a photoresist is hindered by its high chemical and mechanical resistance, making its selective removal against functional layers in additive or subtractive pattern transfer processes very difficult. This disadvantage can be turned into an advantage by using SU-8 itself as the material for the functional layers, a concept that has been demonstrated in the fabrication of lab-on-a-chip devices.⁴

Quasi three dimensional structures can be made from SU-8 by a lithographic process involving multiple coating and exposure steps, followed by a single development step. This process flow dictates, since SU-8 is a negative tone resist (that is, irradiated portions become insoluble in developer and remain, non-irradiated portions are dissolved during the development step), that each layer further away from the substrate must be supported by all SU-8 layers closer to the substrate than the layer considered. This limitation, however, can be overcome by extending the fabrication process flow to include sacrificial layer steps, a technique well established in the manufacture of MEMS. Also, recent developments in the field of direct writing three-dimensional photolithography, systems for which are now commercially available, promise a degree of freedom in the design of SU-8 structures that will most likely exceed the possibilities of silicon based technologies. In addition, although qualification data are yet to be collected, it can be stated with a good certainty that SU-8 is electrically insulating and will very likely withstand the electric fields in electric thrusters. One should keep in mind that all SU-8 formulations considered are strictly commercial off-the-shelf compounds, and that no optimization for the needs of space technology has been considered to date.

We have, for the reasons outlined above, proposed to fabricate colloid thrusters employing an ionic liquid as propellant⁵ and started experimental work, the progress of which we report on in the following.

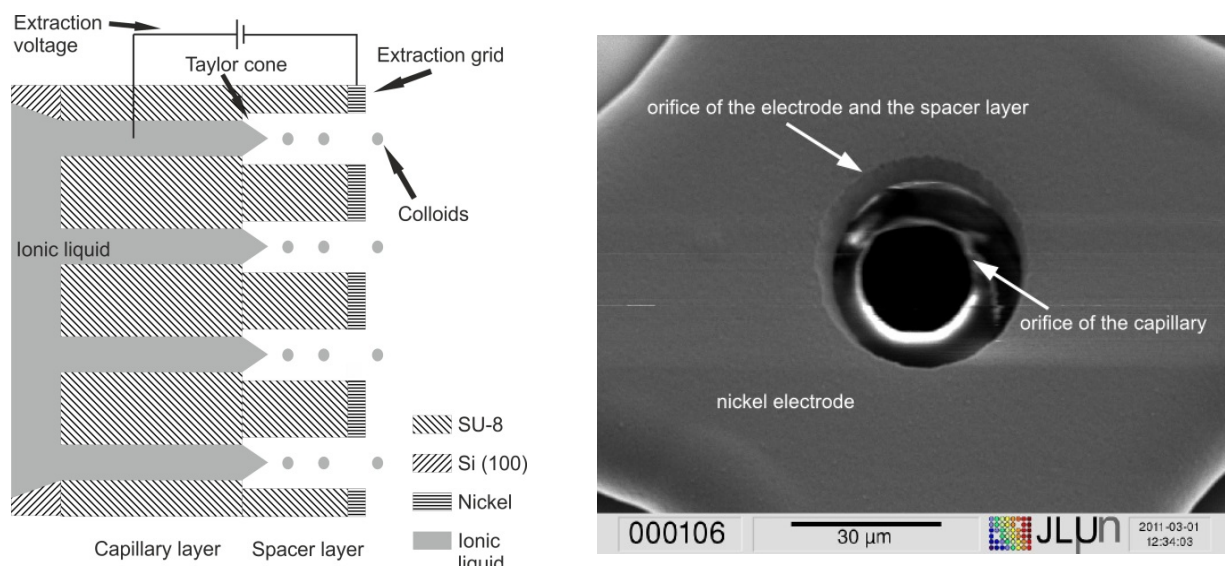


Figure 1. Scheme of a colloid emitter with an integrated extraction grid top electrode (left); SEM image of a sample fabricated according to this design (top view, right).

II. Experimental work and results, and technological issues

A. State of the art

The feasibility of generating fuel feed capillary feedthroughs in an SU-8 layer and the integration with a spacer layer and an extraction electrode support layer, all made in SU-8, were demonstrated by us at IEPC 2011.⁵ Figure 1 shows a schematic drawing of such a structure, illustrating the intended use as a thruster, and a scanning electron micrograph of such an integrated device, in which the extraction electrode was deposited by electroplating on a start and seed layer deposited onto the SU-8 by evaporation coating.

All tests reported in the following were conducted using the ionic liquid 1-ethyl-3-methylimidazolium tetrafluoroborate (EMI-BF₄), whose structure is shown in Fig. 2. The most important properties of EMI-BF₄ are summarized in Table 1.

B. Experimental setup for extraction tests on integrated devices

To characterize the extraction performance of such integrated devices, an experimental setup was built whose core component is a vacuum chamber (cf. Fig. 3) with a fuel supply feedthrough for the ionic liquid leading to one side of the SU-8 structure mounted inside and an optical window on the opposite side allowing the observation through a microscope with a camera.

It was found that it was indeed possible to pull the ionic liquid into the orifice of the fuel feed capillary and beyond. A critical issue was found to be the wetting of the surfaces surrounding the orifice by the

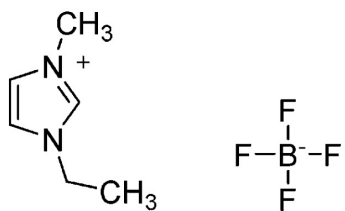


Figure 2. Structure⁶ of the ionic liquid EMI-BF₄

Table 1. Physical properties^{7,8} of EMI-BF₄

Physical properties	Values
Viscosity η at 298 K	37 mPa·s
Density ρ at 298 K	1.28 g·cm ⁻³
Glass transition temperature T_g	181 K
Melting point T_c	286 K
Surface tension γ at 298 K	544 μN·cm ⁻¹
Conductivity at 298 K	1.36 S·cm ⁻¹
Decomposition temperature T_d	720 K

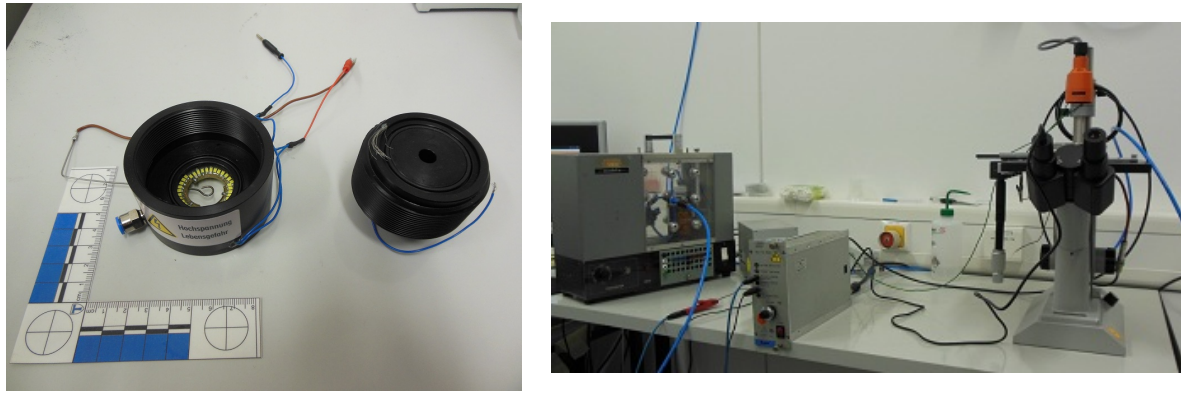


Figure 3. Photographs of the vacuum test chamber used for testing integrated devices (left image) and of the complete experimental setup (right image), comprising (from left to right in the right hand image) a protective atmosphere chamber needed for handling the ionic liquid, a high voltage supply, and an inverted microscope fitted with a camera.

ionic liquid. While good wetting of the SU-8 by the ionic liquid is desirable inside the fuel feed capillary to assist or even replace fuel pumping by the action of the capillary force, wetting of the surrounding surfaces prevents the formation of a proper Taylor cone necessary for extraction. In addition, as soon as the ionic liquid reaches the extraction electrode metal, an intense electrochemical reaction occurs that results in the immediate destruction of the extraction electrode and hence of the device.

C. Experimental setup for extraction tests on single layer devices

To address the problem of wetting, experimental investigations were conducted on single SU-8 layer samples comprising only the fuel feed capillary layer, in a simplified experimental setup in which small portions of these layers were supplied with ionic liquid from the back and mounted opposite an external extraction electrode. The diameter of the $80\text{ }\mu\text{m}$ long capillary could vary from 15 to $50\text{ }\mu\text{m}$, while the electrode could consist of a sharp tungsten carbide tip or a copper plate. Details on the different geometries and directions of illumination and observation are shown in Fig. 5. The samples were mounted in air instead of in vacuum, which is slightly detrimental especially in the high voltage regime, but which on the other hand offers a rather high flexibility in the arrangement of the illumination and observation optics since their respective axes can be tilted with respect to another.

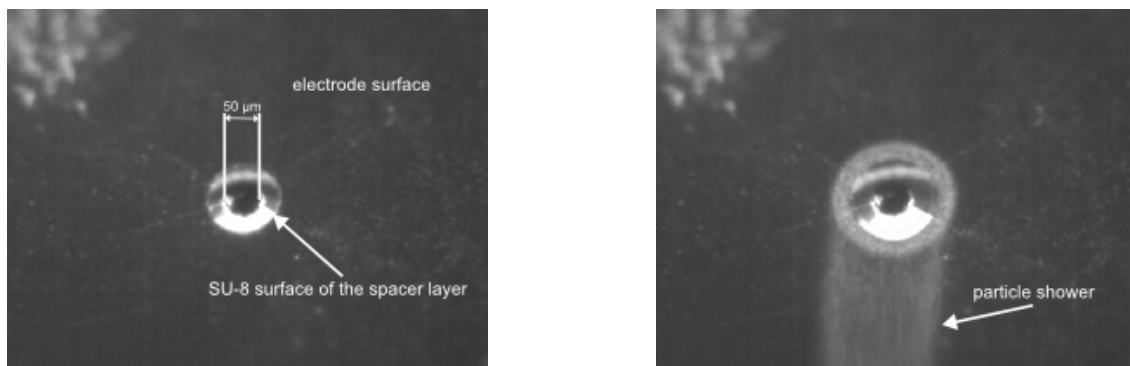


Figure 4. Images taken during an extraction test with an integrated device. Left: ionic liquid has been pulled out of the orifice and has started wetting the surrounding areas, causing an electrochemical reaction resulting in erosion of the extraction electrode material. Right: as the destruction progresses, occasionally particle jets are observed.

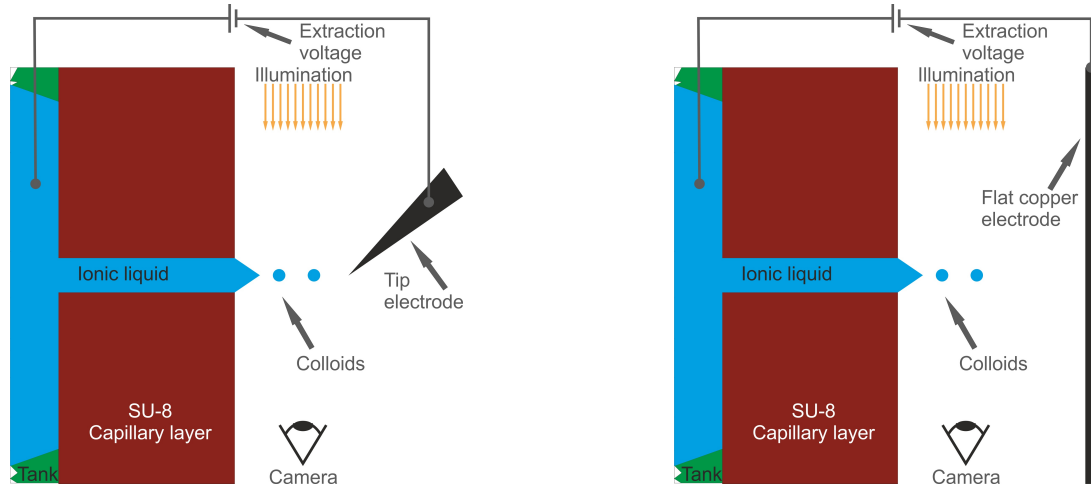


Figure 5. Schematic drawings of the colloid emitter extraction test setups for single layer devices, i.e. with external electrodes (left: tip electrode, right: plate electrode), indicating the respective principal directions of illumination and observation (the angle of observation could be tilted up to 30° against the direction shown in the drawings).

D. Methods of reducing the ionic liquid wettability of SU-8

The contact angle measurement is an important tool to investigate the wetting properties of liquids on surfaces. The contact angle is defined as the angle between the droplet and surface. This angle results from the equilibrium of the three boundary surface tensions. The contact angle of EMI-BF₄ on an SU-8 surface is about 38° . This small contact angle is desired for the inner surface of the capillary, since the capillary force is directly associated to the contact angle. On the surface of the capillary layer a high contact angle can avoid wetting and hence the loss of functionality.

We have measured the contact angle of EMI-BF₄ in combination with several treated surfaces. The measurements were taken with a contact angle measurement device using the sessile drop method. The sample is mounted on a desk while the droplet of the test liquid is falling on the sample. A camera takes a photo of the resting droplet. Finally the contact angle is determined.

A simple attempt of reducing the ionic liquid wettability of SU-8 surfaces by treating them with HMDS (hexamethyldisilazane, IUPAC name bis(trimethylsilyl)amine), commonly used in semiconductor technology to create a nonpolar termination of a silicon surface and thus making the surface hydrophobic, failed. In the case of HMDS, no significant deviation from the contact angle of 38° observed on untreated SU-8 surfaces was seen.

A significant increase of the contact angle to 85° was, however, achieved when the surface was sputter coated with a layer of PTFE (polytetrafluorethylene). A minimum thickness of the PTFE layer was required to achieve this increase in hydrophobicity. The value for this minimum thickness can only be roughly estimated to lie around 500 nm due to equipment instrumentation restraints in the sputter system.

A further increase of the contact angle to 121° was obtained when the SU-8 surface was spin coated with a suspension of silicon dioxide nanoparticles in PDMS (polydimethylsiloxane). The silicon dioxide particles had a diameter of 10 to 30 nm. An amount of 1.5 wt% of these particles and 0.5 wt% PDMS (including 10 wt% curing agent) of PDMS were dissolved in toluene, applied to the surface at 3000 rpm for 20 s and dried at 353.15 K for about one hour on a hot plate.⁹ Figure 6 shows the contact angle measurement results on the untreated SU-8 surface and on the successfully hydrophobised surfaces.

E. Extraction tests with an external tip electrode

The diameter of the 80 μm long capillary is about 40 μm . An about 500 nm thick layer of PTFE is sputter coated onto the SU-8 surface. The extraction electrode used in the extraction test is a tungsten carbide needle electrode mounted in a distance of about 1 mm. The image sequence in Fig. 7 shows the extraction at an angle of 30° . Thus the capillary appears as a dark shadow in the almost transparent SU-8 layer. The liquid crawls up in the capillary while increasing the voltage. At a certain value the liquid is extracted and

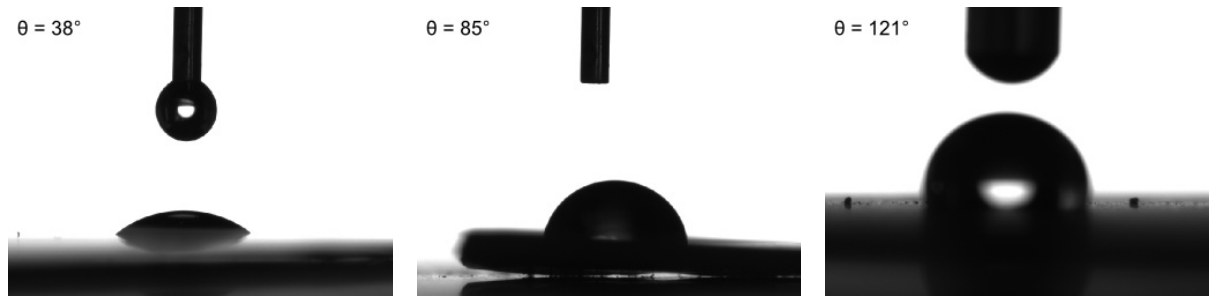


Figure 6. Photographs of the contact angle measurements, showing drops of EMI-BF₄ on untreated SU-8 (left), PTFE (centre), and on SU-8 coated with a suspension of silicon dioxide nanoparticles in PDMS (right).

accelerated to the electrode without wetting the surface of the device.

III. Discussion, remaining technological issues

We have shown the feasibility of extracting positive ions from an ionic liquid using a manually controlled negative high voltage (NHV). This manual control is being replaced by an automatic control that will allow the ionic liquid to be pulled towards the orifice slowly, and that will enable a subsequent increase of the NHV leading to the formation of the Taylor cone and to extraction of ionic liquid positive ions for a (short) limited time and at a well-defined point in time.

The extraction of negative ions may require a different ionic liquid and is not planned for the immediate future, since no fundamental differences in the processes and technology are anticipated.

Ionic liquids are hygroscopic,¹⁰ and we have seen phase separation effects during the extraction tests under atmospheric conditions that we attribute to water in the ionic liquid. Occasionally, we observed arc discharges between the electrode and the ionic liquid. Therefore, all extraction tests will have to be moved to appropriately designed vacuum chambers, allowing the ionic liquids to be kept from atmospheric water during their entire lifetime, and preventing discharges due to ionised air.

The optical imaging of the extracted ionic liquid leaves much room for improvement. A first step will be the proper timing of the moment of extraction, determined from the output of the automated NHV supply, with an LED flash illumination. This should enable at least a rough estimate of the velocity of the extracted ionic liquid, to be calculated from the known duration of the flash. From such data, one can assess the feasibility of further characterising the extracted ionic liquid by high speed videography.

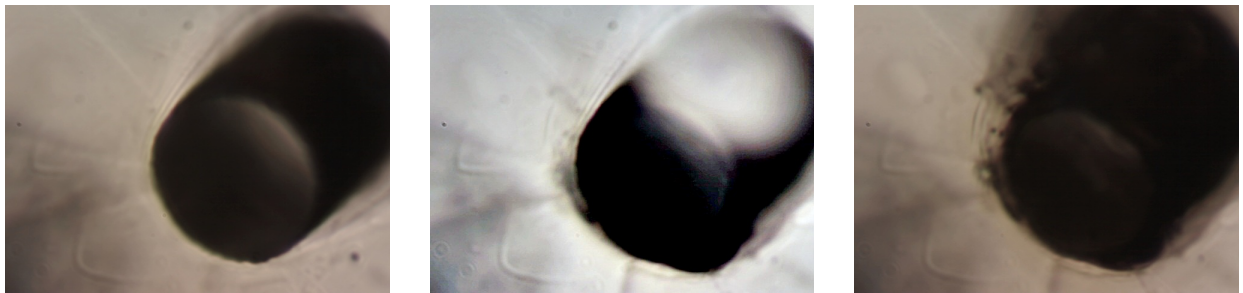


Figure 7. Images from a sequence recorded during an extraction test, taken before (left), during (center), and after (right) the extraction of a small plume. The recording was done with 15 frames per second. The sample is observed at an angle of 30°. Thus the capillary appears as a dark shadow in the almost transparent SU-8 layer. The diameter of the capillary opening is 40 μm . The difference in average image brightness results from the images having been individually contrast enhanced by applying a Retinex filter.

IV. Conclusion

We have shown that it is possible to extract positive species from a colloid emitter made from SU-8. The surface wettability of the SU-8 by ionic liquids can be reduced by sputter coating with PTFE as well as by a surface treatment with silicon dioxide nanoparticles in PDMS, which is essential for a working device with integrated extraction electrodes.

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