Colloid emitters in photostructurable polymer technology: Fabrication and characterization progress report

IEPC-2015-120／ISTS-2015-b-120

Presented at Joint Conference of 30th International Symposium on Space Technology and Science,
34th International Electric Propulsion Conference and 6th Nano-satellite Symposium
Hyogo-Kobe, Japan
July 4–10, 2015

Katharina Huhn* and Torsten Henning†
and Peter J. Klar‡
Institute of Experimental Physics I, Justus Liebig University, 35392 Giessen, Germany

and

Stefan Hengsbach§
Karlsruhe Institute of Technology, 76131 Karlsruhe, Germany

We report on the progress in fabrication and characterization of colloid emitters microfabricated in the photopolymer SU-8. Our experimental setup allows us to perform simultaneously time-dependent measurement of the emission current and optical characterization of the emission process out of the capillary orifice. We analyze and discuss the emission current measurements and based on those estimate the thrust of corresponding capillary arrays. Furthermore, we demonstrate that a 3-D photolithography process can be used to flexibly define the emitter shape. This novel appoach offers additional degrees of freedom in the design of such devices. For example, we plan to use it to reduce ionic liquid wettability of the SU-8 surface by simply optimizing the geometry rather than by including additional coating coating steps in the fabrication process.

Nomenclature

MEMS	= microelectromechanical systems
Emi-BF_4	= 1-ethyl-3-methylimidazolium tetrafluoroborate
PTFE	= polytetrafluoroethylene
CMOS	= complementary metal-oxide-semiconductor
I_{sp}	= specific impulse
T_{peak}	= thrust, peak value
T_{avg}	= thrust, average value over the interval

*PhD candidate, Justus Liebig University, katharina.huhn@physik.uni-giessen.de.
†Senior staff scientist, Justus Liebig University.
‡Professor, Justus Liebig University.
§Graduate engineer (FH), Karlsruhe Institute of Technology.

I. Introduction

Colloid emitters are a promising concept for microthrusters generating high specific impulses since they are expected to provide µN thrust levels with small impulse bits in the µNs range and since their operating principle makes them scale favourably upon miniaturization. In comparison to other thruster concepts suited for this range of tasks, especially plasma-based electrical thrusters, whose losses due to the increasing surface to volume ratio hinder a miniaturization up to the micrometer range, a colloid emitter is an eligible candidate for miniaturization. The extraction voltage scales directly with the nozzle diameter as well as with the distance between capillary orifice and electrode,[1] as a consequence the required extraction voltage can be notably decreased by downscaling the emitter. Since suitable methods and propellants as well as a precise understanding of the occurring electrospray processes exist,[2] the development of this thruster concepts is widely favored. For the fabrication of a miniaturized colloid emitter we use technologies of microelectromechanical systems (MEMS), whose development has sufficiently advanced. Main components like the fuel feed capillary are fabricated by photolithography using the photostructurable polymer SU-8. SU-8 has substantial advantages in comparison to other materials (e.g. silicon) used in MEMS technology. Its high aspect ratio, that is the ratio of height to lateral dimension, its high chemical as well as thermal durability and its electrically insulating property are some of these advantages. Furthermore, SU-8 offers very flexible design options. In particular, by being able to employ 3-D photolithography additional degrees of freedom are gained for shaping the device. No other lithographic method offers such a high design flexibility. Suitable propellants for this kind of device are readily available, i.e. the class of ionic liquids. Ionic liquids are salts with a melting point below 100 °C and a high ionic conductivity which increases with increasing temperature. High ionic conductivity is a prerequisite of the propellant in order to generate high specific impulse in a colloid emitter. The temperature dependence of the ionic conductivity may even be used to control the emitter current by orders of magnitude. In comparison to metals as propellants, no additional heating is required, because many ionic liquids with melting points below room temperature are known. For the use in vacuum the very low vapour pressure is very welcome. Moreover, ionic liquids may be designed to consist of positive as well as negative ions of almost equal mass. Thus, both species can be alternately extracted by applying an alternating extraction voltage, so that a charge up of the emitter and thus the spacecraft may be avoided without employing additional neutralizers.

II. Experimental work and results

A. State of the art

The feasibility of fabricating a colloid emitter device, as shown in Fig. 1, consisting of a capillary and a spacer layer made of SU-8 as well as a nickel electrode on top of the spacer layer, was demonstrated by us earlier.[3] At IEPC 2013 we presented first optical observations during extraction tests using a simplified device with an external tip electrode (see Fig. 2).[4] The extension of the corresponding experimental setup by implementing beam current measurements is presented in the following. The simultaneous characterization of the emitter by optical observation and emission current measurement was the aim when designing the setup. In the future it will allow us to study the formation of the so called Taylor cone, the onset of spray emission from the very tip of the cone and also the emitted spray, droplets or particles. These optical observations can then be compared with data of the measured beam current. For the beam current measurements, presented in this paper, 1-ethyl-3-methylimidazolium tetrafluoroborate (Emi-BF_4) was used. Apart from offering a high ionic conductivity, Emi-BF_4 is also liquid over a wide temperature range including room temperature.

B. Time-dependent measurements of the emission current

1. Sample preparation

For an easier understanding of the emission behavior a simplified colloid emitter device was fabricated. It consists of a quadratic (edge length 5 mm), 80 µm thick single layer of SU-8 which contains only one capillary with a diameter of 50 µm in its center. The surface of the SU-8 layer is sputter coated with a thin layer of PTFE in order to optimize the wetting behavior between the liquid propellant (Emi-BF_4) and the surface surrounding the capillary (see Fig. 2) .[4] This coating leads to an increased contact angle between Emi-BF_4 and the surface which is favorable for extraction as it avoids surface wetting, which may cause short circuits.

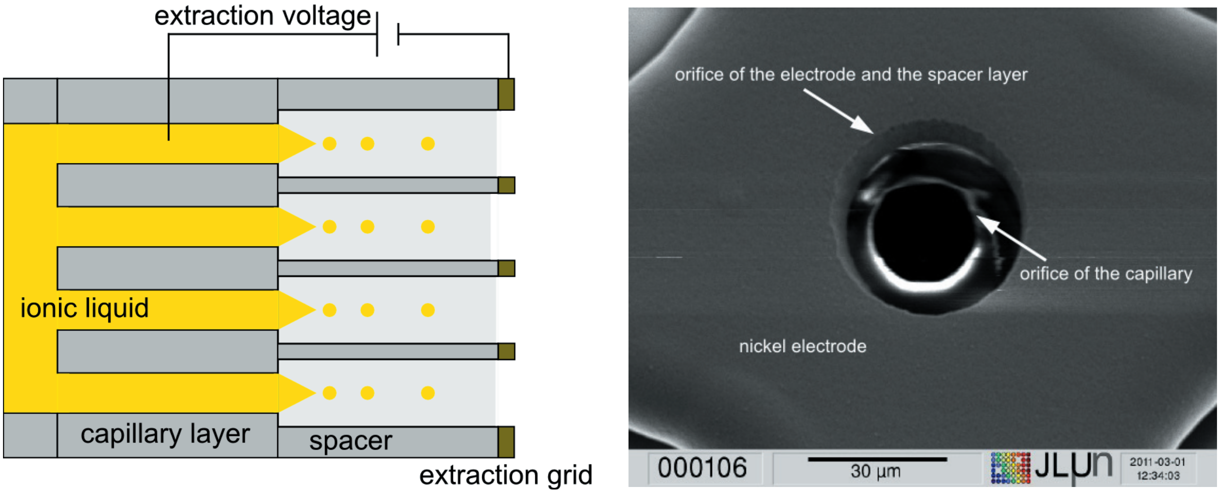

Figure 1. Left: Scheme of the colloid emitter with an integrated electrode and right: SEM-image taken in top view of a colloid emitter sample with an integrated nickel electrode.

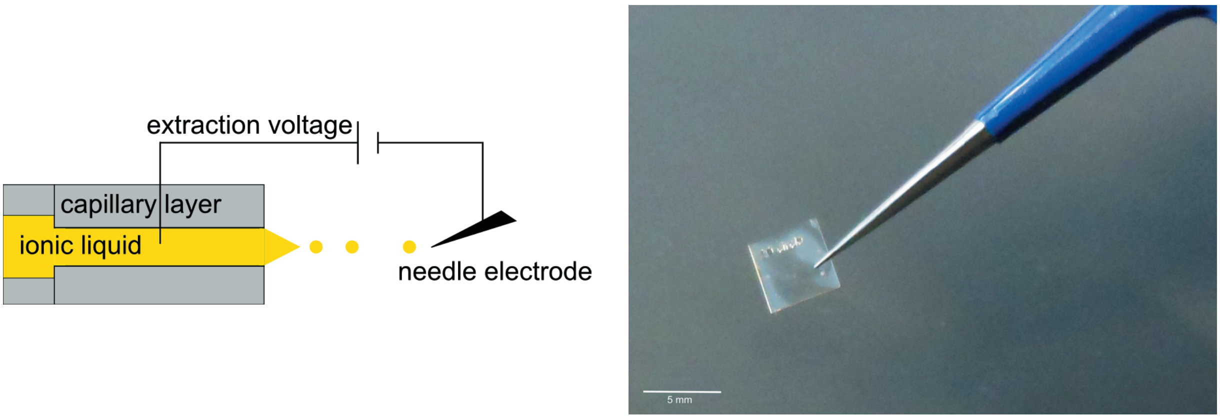

Figure 2. Left: Scheme of the colloid emitter device with an external tip electrode and right: Photograph of a sample fabricated according to the simplified design for the use with an external electrode.

2. *Experimental setup*

A drawing of the setup, as used for the measurements, is shown in Fig. 3. For the implementation of the emission current measurements the simplified colloid emitter device was mounted on the cut plane of a tank (black cylinder in Fig. 3). This device is built into a holder basically consisting of three manually operationable linear stages offering the possibility to align the capillary exactly with respect to the external needle, mounted below this holder (see Fig. 3). For the optical observation of the electrospray behavior by a telescope equipped with a digital camera two additional stages for alignment, suitable for the use in vacuum, are necessary: a linear stage using a piezo stick-slip drive for exact positioning and a rotation stage to observe the Taylor cone and the emitted spray from different angles. The emitter device is connected to the negative high voltage source F.u.G. HCN 7e 3500, which is manually controlled during the measurements. For the measurement of the beam current a collector electrode, a glass plate coated with a transparent and conducting indium tin oxide layer or a copper plate, is mounted in the path of the jet. The current is measured by recording the voltage drop across a 1 MΩ electrical resistance. The electrical circuit includes also amplifiers and frequency filters. The results are displayed directly on an oscilloscope. For the preparation of the measurements the tank is filled with a few milliliters of Emi-BF_4 before the vacuum tank is evacuated down to 10^{-6} mbar. The possibility to observe the extracted spray optically has been retained. For this purpose, an objective (magnification 20 times) in a telescope setup is connected to a digital camera with a 6.6 megapixel CMOS sensor. The depicted setup is designed such that also samples with an integrated

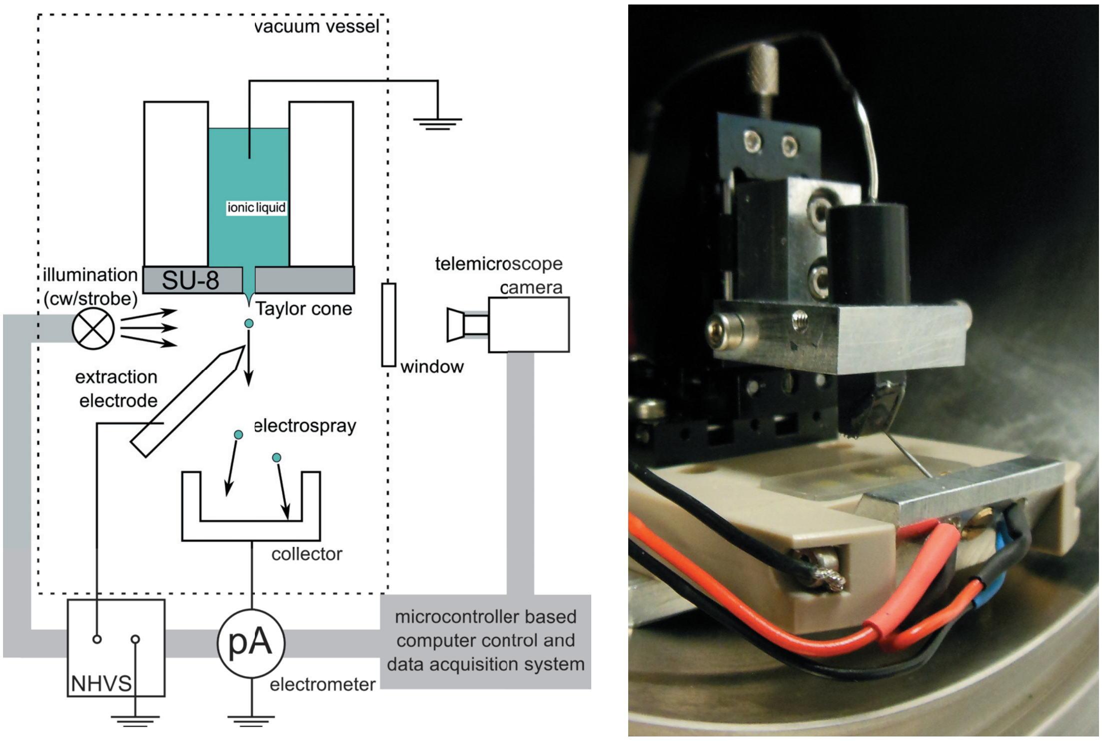

Figure 3. Left: Scheme of the extraction test setup for miniaturized colloid emitters and right: Photograph of the experimental setup with mounted sample.

electrode can be characterized, which is especially important for investigating the influence of varying the geometry of the capillary shape and opening.

3. Results of the emission current measurements

For the analysis described here a sample of the simplified design was used. The diameter of the capillary located at the center of the sample is about 50 µm. The surface of the about 80 µm thick SU-8 layer is sputter coated with a thin layer of PTFE. The distance between capillary layer surface and needle electrode is about 1 mm. The negative voltage is applied to the needle, so that the extracted ions are positive ones.

The left graphs of Fig. 4 show two screenshots of the oscilloscope taken during the current measurements. These are two typical examples to depict the two different spray modi. The top left graph ist a typical result of the peak mode occuring between 2300 V and 2900 V. Current peaks appear in periodic intervals, which decrease with increasing voltage. After exceeding a voltage value of 2900 V the peaks are getting much smaller and appear non-periodically (see lower left graph of Fig. 4).

First observations of a current ocurring were made when the voltage applied between reservoir and needle exceeded a value of about 2300 V. It should be mentioned that the current manifested itself as a sequence of sharp peaks. The effect of increasing the applied voltage on the intensity of the current peaks is shown in the right lower graph of Fig. 4. The highest value was reached at low applied voltages with a maximum value of 18 nA measured at 2450 V. Passing 2450 V the maximum pulse height of the current peaks decreases continuously reaching a minimum pulse height of 11.3 nA at 2800 V. Interesting observations were made when the voltage applied was higher than 2900 V. Many small peaks at frequent intervals suddenly appeared. The duration of the current peaks became smaller with increasing applied voltage (right top graph of Fig. 4). Even more pronounced is the effect on the interval between pulses. There is a practically linear relationship between the increasing applied voltage and the decreasing pulse interval (right center graph of Fig. 4).

Taking into account that the size of the extracted portions of the propellant and their charge state, thus their charge to mass ratio, are unknown, the thrust and specific impulse can only be vaguely estimated. Based on an applied voltage of 2600 V and assuming drops with a single positive charge and a diameter of 100 nm a single capillary yields a thrust peak value of $T_{\text{peak}} = 3\,\mu\text{N}$ and average value over the interval of

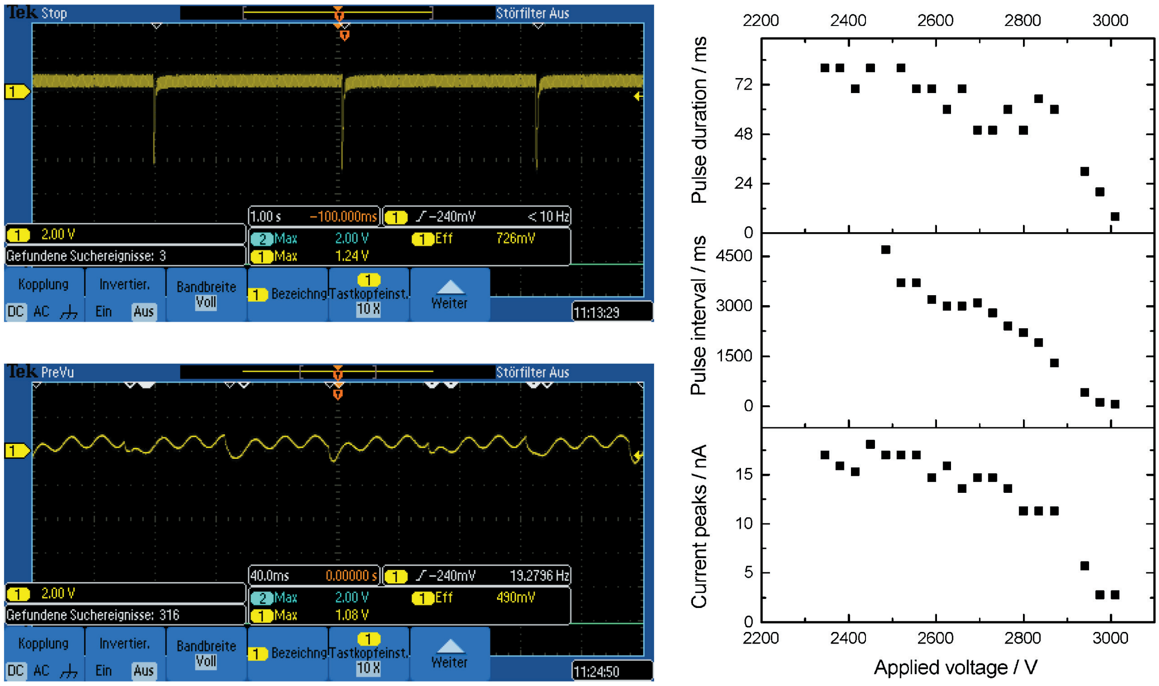

Figure 4. Left: Screenshots of the oscilloscope taken during the current measurements at an applied voltage of 2695 V (top) and 3010 V (bottom). Right: Analysis of the current measurement data: pulse duration (top), pulse interval (center) and strength of the current peaks (below) against applied voltage.

$T_{avg} = 0.85$ nN. Consequently the I_{sp} results in 3.6 s. Assuming that the extracted portions of the propellant are ions with a single positive charge the calculated T_{peak} is 1.5 nN and the corresponding average value is $T_{avg} = 43$ pN only, while the I_{sp} is 7000 s. Assuming that it is feasible to define 2500 emitters per square centimeter and that these can be used simultaneously, the total thrust is about in $T_{avg} = 2.125$ µN per square centimeter if singly positively charged droplets with a diameter of 100 nm are extracted, while under the condition that only positive ions are extracted the total thrust per square centimeter is $T_{avg} = 107.5$ nN.

C. Variation of the geometry of the capillary orifice

At IEPC 2013[4] we discussed the importance of reducing the ionic liquid wettability of SU-8. A wetting of the capillary surface leads to an undefined liquid surface, which makes the colloid emitter difficult to operate with a good reproducibility. Using the contact angle measurements, we found, that the contact angle can be increased by a sputter coated layer of PTFE, which means that the wetting behavior is minimized. As the wetting is minimized, this layer of PTFE also avoids a crawling of the liquid up to the electrode, which generally leads to a short circuit. An alternative approach to prevent the wetting is to employ optimized geometries of capillary environment.

One way of realizing an optimized geometry is to define a trench between capillary and electrode by planar photolithography, but this is somewhat labor-intensive. The reason is, when employing conventional photolithography, the freedom of design is restricted by the fact that only 2-D layers can be structured, which have to be stacked on top of each other. Fig. 5 shows the layout of a colloid emitter consisting of three layers of SU-8 in order to create a trench between capillary and electrode where the design restrictions described above have been overcome. The SEM image demonstrates that, in principle, a precise alignment of the three layers is possible. However, it is not possible to structure the upper layer in places where the underlying layer has been removed.

Another promising and even better approach is employing novel 3-D photolithography technology which is far more flexible than conventional 2-D photolithography and allows one to fabricate 3-D structures of

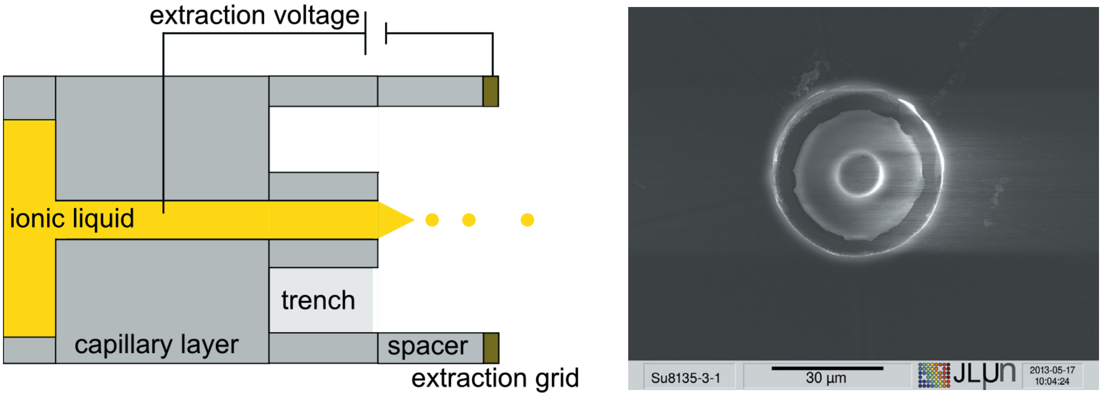

Figure 5. Left: Scheme of the colloid emitter with an integrated electrode and trench between capillary and electrode structured by 2-D photolithography and right: SEM-image taken in top view of a colloid emitter sample structured according to this design.

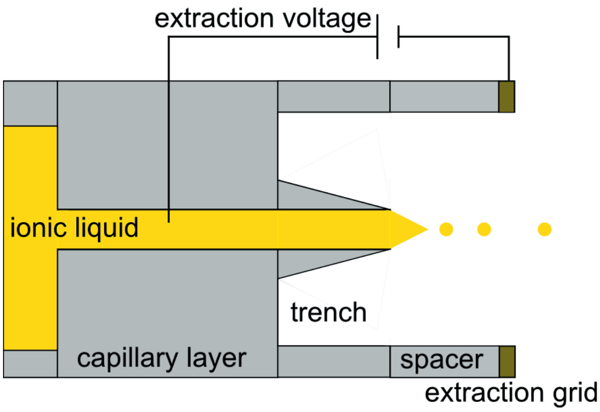

Figure 6. Scheme of the colloid emitter with an integrated electrode and a truncated cone-like capillary orifice fabricated using 3-D photolithography.

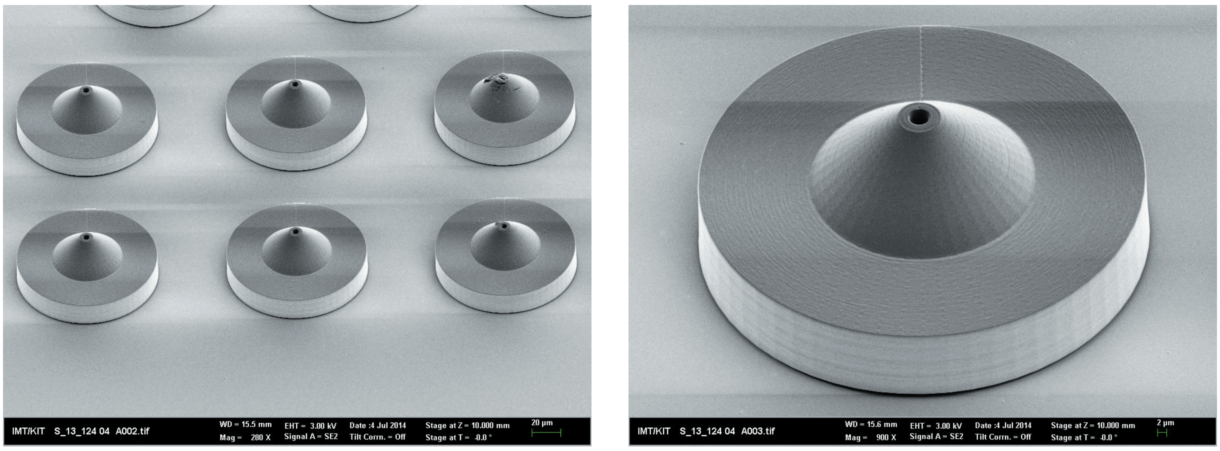

Figure 7. SEM-images of a truncated cone-like capillary orifice fabricated using 3-D photolithography. Left: Overview and right: Closeup view.

almost arbitrary shapes. This method can be used to systematically study the wetting behavior of capillaries with orifices of different shape. In order to maximize the contact angle between the cone shaped liquid surface and the orifice of the capillary, we tried to structure a tapered emitter. The capillary layer as well as a second layer, which serves as a spacer between capillary layer and the extraction electrode, were still structured by common 2-D photolithography. Afterwards a truncated cone like capillary orifice was structured on top using 3-D photolithography (see Fig. 6).

The SEM images in Fig. 7 prove the feasibility of fabricating such conic orifices using the combination of 2-D and 3-D photolithography. The diameter of the capillary is about 2 µm, much smaller than for emitters structured using 2-D photolithography where the smallest capillary diameter obtainable was typically larger than about 10 µm. Thus, the novel 3-D technique also renders the possibility to structure even smaller capillaries and therefore define even more emitters per area unit.

III. Conclusion

We have reported on the progress in characterizing SU-8 based colloid emitters. The time-dependent measurement of the emission current was succesfully introduced. The results of the measurements show two different spray modi: At lower voltages, applied between the liquid propellant Emi-BF_4 and the extraction electrode, the current appears as a periodic sequence of sharp peaks. With increasing applied voltage, the current peaks get smaller and appear at frequent non periodic intervals. We also have reported on the progress in fabricating novel thruster designs using a 3-D photolithography process which offers an alternative approach to optimize the wetting behavior of the capillary surface. The SEM-images presented in this article prove the feasibility of fabricating truncated cone-like capillary orifices.

Acknowledgments

This work was supported in the framework of LOEWE-Schwerpunkt RITSAT and by DLR space administration (grant No 50 RS 1504). Samples prepared by 2D-photolithography were made in the Micro- and Nanofabrication Laboratory at Justus Liebig University Giessen and those by 3D-photolithography were fabricated at KIT.

References

[1]Martinez-Sanchez, M., "16.522 Space Propulsion," http://ocw.mit.edu (Accessed 11 May, 2015), Spring 2004.

[2]Wilm, M. S. and Mann, M., "Electrospray and Taylor-Cone theory, Doles beam of macromolecules at last?" *International Journal of Mass Spectrometry and Ion Processes*, , No. 136, 1994, pp. 167–180.

[3]Piechotka, M., Huhn, K., Henning, T., Feili, D., and Klar, P. J., "Microfabrication of Colloid Emitters based on the Photo Resist SU-8 and Electro Plating," International Electric Propulsion Conference, 2011.

[4]Huhn, K., Piechotka, M., Henning, T., and Klar, P. J., "Investigation of the emission behavior of miniaturized SU-8 based colloid emitters," International Electric Propulsion Conference, 2013.