

Fabrication of a Double-Emitter Structure for Higher Current Density of Ionic Liquid Electro spray Thrusters

By Shujun GUO,^{1,2)} Masayoshi NAGAO,²⁾ Katsuhisa MURAKAMI,²⁾
Hiromasa MURATA,²⁾ and Yoshinori TAKAO³⁾

¹⁾ Department of Mechanical Engineering, Materials Science, and Ocean Engineering, Yokohama National University, Yokohama, Japan

²⁾ Device Technology Research Institute, National Institute of Advanced Industrial Science and Technology, Tsukuba, Japan

³⁾ Division of Systems Research, Yokohama National University, Yokohama, Japan

(Received August 31st, 2023)

In this study, we have fabricated ionic liquid electro spray thrusters to prevent ionic liquid leakage and provide high current density. A double-emitter structure with a needle protruding from the capillary emitter, where the ionic liquid was intended to be held by the needle, was proposed. The fabrication process employs a fabrication technique of a field emitter array, and a newly designed process to reduce the distance between emitters was used to improve emitter density. As a result of the fabrication, the needle was successfully formed in the emitter electrode, and it was found that emitter density could be improved by changing the deposition conditions of multiple SiO₂ layers.

Key Words: Electric Propulsion, Electro spray, Double-Emitter, Ionic Liquids

1. Introduction

In recent years, the number of launches of nanosatellites has been increasing.¹⁾ These nanosatellites can be developed at a low cost and in a short time. Using nanosatellites makes satellite constellations easier in terms of cost. Satellite constellations must have a propulsion system to control the satellite's orbit and attitude. However, the size of satellites less than 10 kg, which has been the focus of much development, is severely restricted. Therefore, the size of the propulsion system must be reduced. For example, a typical ion thruster using xenon as a propellant requires a high-pressure gas tank and a regulator. Although there are some research activities that use iodine and water as propellants to eliminate the high-pressure gas system,²⁻⁴⁾ ion thrusters are still challenging to install in a satellite weighing less than 10 kg. In this study, we focused on electro spray thrusters that use an ionic liquid as a propellant instead of ion thrusters.

Figure 1 shows a schematic diagram of an electro spray thruster. Electro spray thrusters consist of two electrodes, an emitter and an extractor. First, ionic liquid propellant is supplied to the emitter, and then a voltage is applied between the emitter and the extractor, which causes an electric field to concentrate at the emitter tip.⁵⁾ This causes ions to be released from the ionic liquid to generate thrust.

Electro spray thrusters use the ionic liquid as the propellant, eliminating the need for a high-pressure gas system and dramatically reducing the volume of the thruster compared to ion thrusters. This reduction is because the ionic liquid has an extremely low vapor pressure and can exist as a liquid in space.⁶⁾ However, electro spray thrusters have only about one-tenth the thrust density of ion thrusters. It is necessary to increase the thrust density to produce high thrust with a small propulsion.

In our previous studies, an emitter array with ultra-high

emitter density was fabricated using the field emitter array (FEA) fabrication technique.^{7,8)} The FEA is a field emission electron source that emits electrons by generating a strong electric field on the emitter tips. The emitter arrays fabricated in the previous study were spaced 5 μm apart compared to the 456 μm spacing of S-iEPS developed at MIT,⁵⁾ which is about 10,000 times higher in number density. This high-density emitter array resulted in a current density of 43 mA/cm², an increase of two orders of magnitude higher than that of S-iEPS.^{2,7)} On the other hand, leakage of ionic liquid onto the surface of the emitter array occurred after the ion emission experiment.

As a countermeasure, we fabricated a double-emitter structure with a needle inside the emitter electrode.⁹⁾ While this structure was expected to suppress the leakage of ionic liquid, the emitter density was reduced due to the fabrication process. The challenge is to improve the emitter density because a decrease in emitter density also decreases the emission current density. In this study, we have devised a method to solve the issue mentioned above and verified the fabrication results.

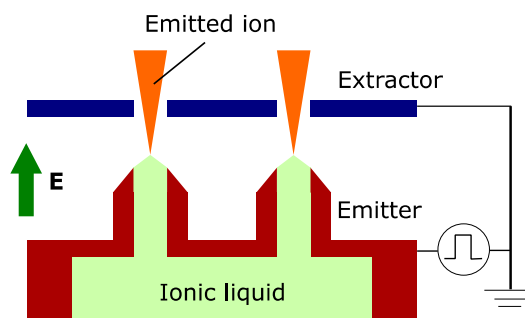


Fig. 1. Schematic of the electro spray thruster.

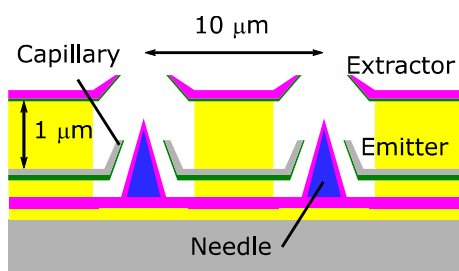


Fig. 2. Schematic of the double-emitter structure.

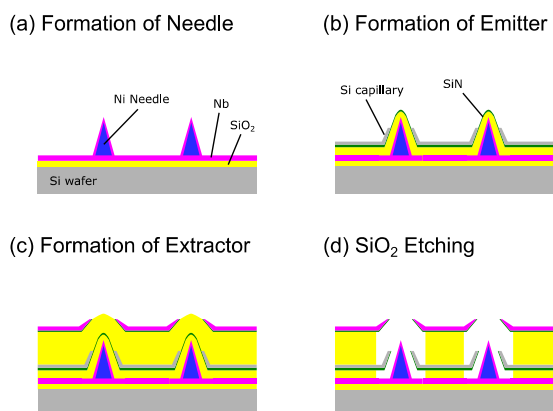


Fig. 3. Fabrication procedure of a double-emitter structure.

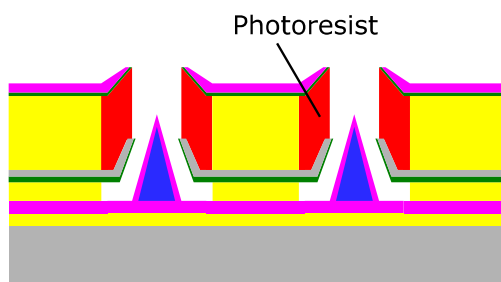


Fig. 4. Schematic of the photoresist filling method.

2. Fabrication of the Double-Emitter Structure using Photoresist Protection

Figure 2 shows a cross-sectional schematic of the double-emitter structure. In this study, the backside Si deep etching for ionic liquid supply was omitted to focus on the double-emitter structure.

In the fabrication of the structure in Fig. 2, the emitter spacing was set to 10 μm due to the difficulty in fabrication. The surface of the emitter electrode is modified to be hydrophobic by plasma treatment with CHF_3 ,⁷⁾ and the needle of Nb is hydrophilic to make the emitter electrode less wettable and reduce the risk of ionic liquid leakage. The needle protruding from the emitter electrode is expected to facilitate the concentration of the electric field at the tip of the emitter electrode, thereby reducing the extraction voltage and increasing the current density.¹⁰⁾ In this structure, the base and height of the needle of Ni covered with Nb are approximately 0.7 and 1.4 μm , respectively.

2.1. Fabrication process

Figure 3 shows the fabrication process of the emitter array.

(a) Formation of needle: A SiO_2 film was deposited on a Si wafer by plasma enhanced chemical vapor deposition (PE-CVD), and Nb was deposited by sputtering. Ni for the needle was deposited by an electron beam (EB) evaporator onto the hole pattern fabricated by photolithography from the top surface of the wafer. The deposited Ni formed a needle shape, and the photoresist was removed. Nb was deposited on top of Ni by sputtering.

(b) Formation of emitter: SiO_2 was deposited by PE-CVD, and SiN was deposited on top of SiO_2 by sputtering as an etch stop layer, which will be described later. Then, Si was deposited by sputtering. Next, the Si layer was opened by photolithography and the etch-back technique,¹¹⁾ and then the photoresist was removed.

(c) Formation of extractor: SiO_2 was deposited by PE-CVD. SiN and Nb were deposited on top of SiO_2 by sputtering. The Nb and SiN layers were opened sequentially using the etch-back technique, and then the photoresist was removed.¹¹⁾

(d) SiO_2 etching: In this process, buffered hydrofluoric acid (BHF) is used to form ionic liquid channels around the needle. The etching of the SiO_2 layer with BHF is isotropic, so the SiO_2 layer between the extractor and emitter is laterally removed as the etching proceeds. As a result, the lateral distance per emitter becomes larger, making it difficult to increase emitter density. Therefore, as shown in Fig. 4, we attempted to suppress etching in the lateral direction by filling the space between the electrodes with a photoresist. This method can reduce the distance between neighboring emitters, which is expected to increase the emitter density in the future. Specifically, the BHF etching is divided into two stages. In the first stage, an etch stop layer is provided to stop the BHF etching, and then a photoresist is applied to fill the space between electrodes. When the entire surface is exposed after filling with the photoresist, the photoresist under the overhanging extractor electrode is not exposed because the extractor electrode serves as a mask. In contrast, the photoresist in other areas is exposed and can be removed by the developer, making it possible to selectively fill the space under the electrode with photoresist.

2.2. Fabrication results

Figure 5 shows an SEM image of the emitter array after process (d). It can be confirmed that emitter arrays are formed with a pitch of 10 μm . The extractor, emitter, and needle can be seen in the view.

Figure 6 shows a cross-sectional SEM image of the same wafer as Fig. 5 after process (d). The needle in the emitter electrode was successfully protruded from the Si capillary electrode. However, the capillary of the emitter electrode, as shown in Fig. 2, was almost lost. This loss is because the etching rate of the Si layer was faster than expected in etching process (b) in Fig. 3, and it is expected to be solved by shortening the etching time. In addition, the SiO_2 in the upper layer of the emitter electrode was etched laterally about 1 μm more than the lower layer. This lateral etching is thought to be because the photoresist could not completely fill the space between the emitter and extractor electrode shown in Fig. 7.

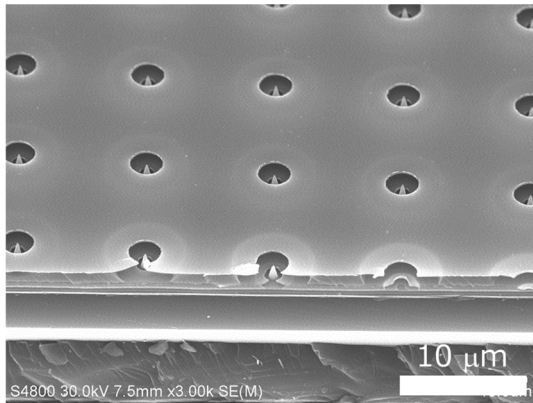


Fig. 5. SEM image of the fabricated emitter array.

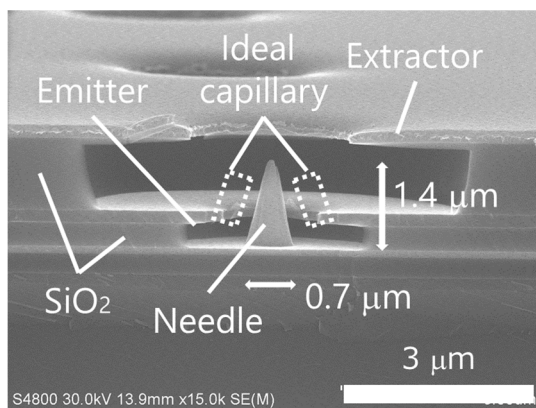


Fig. 6. Cross-sectional SEM image of the fabricated emitter array.

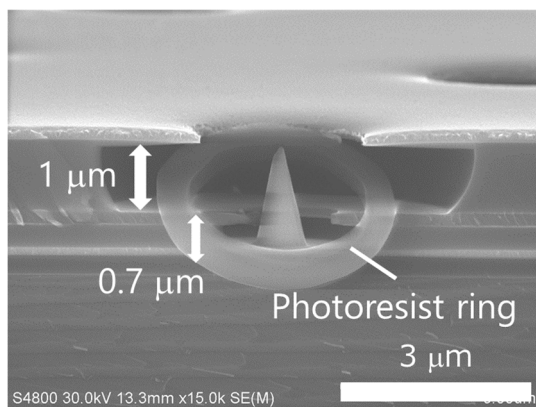


Fig. 7. Cross-sectional SEM image of the fabricated emitter array with photoresist ring.

Figure 7 is a cross-sectional SEM image of process (d) in Fig. 3. The photoresist ring, which was intended to suppress etching in the lateral direction, can be seen. The size of the photoresist ring and the etching area of the upper SiO₂ layer must be identical in order that SiO₂ is not etched by BHF. However, the etched region of the upper layer (1 μm in Fig. 7) is wider than the photoresist ring (0.7 μm in Fig. 7). The difference in size results in a gap between the electrodes and the photoresist. This result is probably due to a gap between the photoresist and the extractor and emitter electrodes, where

BHF penetrated through the gap. One of the reasons for the gap is that the photoresist was baked to solidify the applied photoresist. At this time, the solvent contained in the photoresist evaporated, and the volume of the photoresist was thus reduced. The gap was created where the photoresist was in contact with the electrode. The gaps that were created differ from the results of the methods used in previous FEA studies.¹²⁾ The reason for this is that the SiO₂ layer thickness in this process is about 1 μm, which is about twice as thick as the 500 nm thickness in previous FEA studies. Therefore, it is necessary to devise another method to improve emitter density.

3. Revised Fabrication of the Double-Emitter Structure

As mentioned in the previous section, process modifications are necessary to improve emitter density. We changed the method of filling the photoresist, but could not avoid the formation of gaps after baking. Furthermore, Fig. 6 reveals that Nb may be etched by BHF. In particular, Fig. 4 shows that if the Nb covering the needle is etched, the Ni inside of Nb cannot be protected from BHF. Therefore, the Nb in the emitter electrode was changed to Si, and the Nb covering the needle was changed to Cr and Si. The surface of the emitter electrode was also modified to be hydrophobic by plasma treatment with CHF₃.⁷⁾ The outermost cover of the needle is Si, and Si forms a natural oxide film, which is also hydrophilic. In addition, a change in SiO₂ deposition conditions was introduced as a process to improve emitter density as described below.

SiO₂ in the double emitter structure is deposited by PE-CVD, and the etching rate of BHF varies with each deposition temperature. The etching rate of BHF decreases as the SiO₂ is deposited at higher temperatures. In this fabrication, the upper and lower SiO₂ layers were deposited at 200 and 100°C, respectively. As a result, the etching rate of the upper layer is about half that of the lower layer. Therefore, it is thought that the upper layer can suppress lateral spreading during etching with BHF.

3.1. Fabrication process

Figure 8 shows the fabrication process of the emitter array.

(a) Formation of needle: Cr was deposited by sputtering. Ni for the needle was deposited by the EB evaporator onto the hole pattern fabricated by photolithography from the top surface of the wafer. The deposited Ni formed a needle shape, and the photoresist was removed. Cr and Si were deposited on top of Ni by sputtering.

(b) Formation of ionic liquid supply holes: After photolithography, Si and Cr near the needle were etched to form the supply hole.

(c) Formation of emitter electrodes: SiO₂ was deposited at 100°C followed by SiN and Si as capillary emitter electrodes. SiO₂ was removed in a later step to form the ionic liquid channel.

(d) Formation of extractor electrodes: SiO₂ was deposited at 200°C followed by SiN and Nb as emitter electrodes.

(e) Formation of ionic liquid channel: SiO₂ was etched with BHF through the electrode aperture to form the channel.

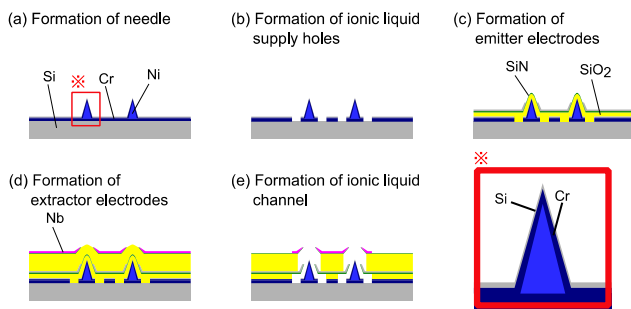


Fig. 8. Fabrication process of the emitter array.

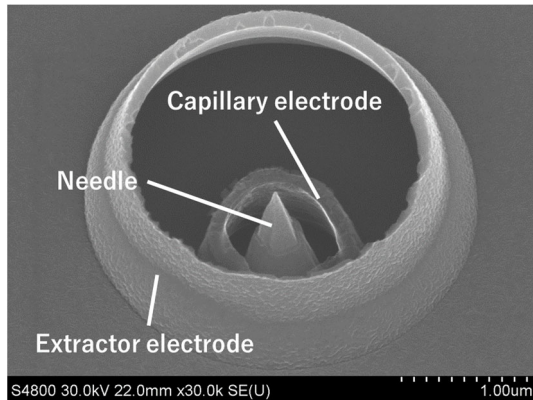


Fig. 9. SEM image of emitter array.

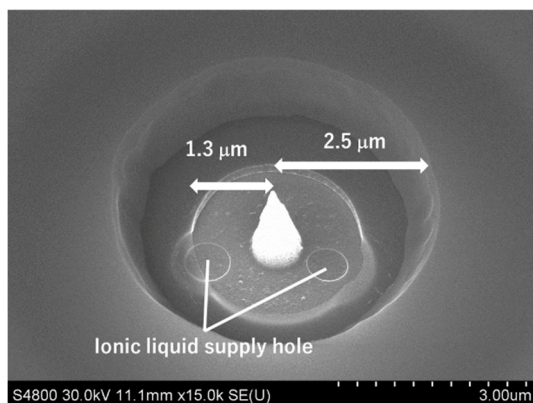


Fig. 10. SEM image of emitter array with capillary removed.

3.2. Fabrication results

Figure 9 shows an SEM image of the emitter array after immersion in BHF. The extractor electrode, emitter electrode, and internal needle can be seen in Fig. 9. From Fig. 9, a needle was successfully formed inside the capillary of the emitter electrode. The electrodes were then removed by reactive ion etching to observe the extent to which the upper SiO₂ layer was etched by BHF and the ionic liquid supply holes. The etched result is shown in Fig. 10. As shown in the figure, the etching amount of SiO₂ in the upper layer and lower layer are about 2.5 µm and 1.3 µm, respectively. The etching amount in the upper layer is reduced from 3.0 µm in Fig. 6. At the same time, the ionic liquid supply holes could be contained within the etching area of the lower layer. Therefore, it is possible to reduce the pitch between emitters from 10 µm to approximately 6 µm. The etching amount of

the upper layer SiO₂ can be narrowed by increasing the deposition temperature of the upper layer SiO₂ from 200°C to higher temperatures, which is left for future work.

4. Conclusion

In this study, a double-emitter structure was fabricated to prevent ionic liquid leakage and increase the current density of electro spray thrusters. As a result of the fabrication, a needle protruding from the emitter electrode was successfully formed, but an issue arose in establishing a new SiO₂ etching method. Since the etching amount of the upper SiO₂ layer in the lateral direction could not be suppressed, the etching method of the SiO₂ layer needed to be changed. Therefore, we set up a difference in the etching rate by changing the SiO₂ deposition temperature of the upper and lower layers. As a result, we succeeded in narrowing the etching area of the upper layer. In the future, a deep etching from the backside of the emitter array will be performed. The ion emission and current-voltage characteristics of the completed emitter array will also be investigated to demonstrate the current density improvement without ionic liquid leakage.

Acknowledgments

This work was financially supported in part by JSPS KAKENHI Grant Number JP21H01530, JST FOREST Program Grant Number JPMJFR2129, the Canon Foundation, and the Futaba Foundation.

References

- SpaceWorks: 2020 Nano/Microsatellite Market Forecast 10th ed, 2020.
- Rafalskyi, D., Martínez, J. M., Habl, L., Zorzoli Rossi, E., Proynov, P., Boré, A., Baret, T., Poyet, A., Lafleur, T., Dudin, S., and Aanesland, A.: In-Orbit Demonstration of an Iodine Electric Propulsion System. *Nature* **599** (2021), pp. 411–415
- Tsay, M., Frongillo, J., and Hohman, K.: Iodine-Fueled Mini RF Ion Thruster for CubeSat Applications, Joint Conference of 30th International Symposium on Space Technology and Science, 34th International Electric Propulsion Conference, and 6th Nano-satellite Symposium, Hyogo-Kobe, Japan, IEPC-2015-273 /ISTS-2015-b-273, 2015.
- Ataka, Y., Nakagawa, Y., Koizumi, H., and Komurasaki, K.: Improving the Performance of a Water Ion Thruster Using Biased Electrodes. *Acta Astronautica* 2021, 187, 133–140.
- Krejci, D., Mier-Hicks, F., Thomas, R., Haag, T., and Lozano, P.: Emission Characteristics of Passively Fed Electro spray Microthrusters with Propellant Reservoirs, *J. Spacecr. Rockets*, **54** (2017), pp. 447–458.
- Watanabe, M.: Current and Future Scopes of Ionic Liquid studies, *J. Surf. Sci. Jpn.*, **28** (2007), pp. 298–303 (in Japanese).
- Suzuki, K., Nagao, M., Liu, Y., Murakami, K., Khumpuang, S., Hara, S., and Takao, Y.: Fabrication of Nano-Capillary Emitter Arrays for Ionic Liquid Electro spray Thrusters, *Jpn. J. Appl. Phys.*, **60** (2021), pp. SCCF07-1–6.
- Nagao, M. and Yoshida, T.: Fabrication of Gated Nano Electron Source for Vacuum Nanoelectronics, *Microelectron Eng.*, **132** (2015), pp. 14–20.
- Suzuki, K., Nagao, M., Murakami, K., Khumpuang, S., Hara, S., and Takao, Y.: Fabrication of Micro Capillary-Needle-Type Emitter Electrodes for Ultra-High-Density Fabricated Electro spray Thrusters, Proceeding of 63rd Space Sciences and Technology Conference, Tokushima, Japan, JSASS-2019-4801, 2019 (in Japanese).
- Zhang J., Cai G., Shahzad A., Liu X., Wang W.: Ionic liquid

- electrospray behavior in a hybrid emitter electrospray thruster, *Int. J. Heat Mass Transf.*, **175** (2021), pp. 121369–1–10.
- 11) Nagao, M.: Micro-fabricated Field Emitter Arrays ~For New Applications~, *J. Vac. Soc. Jpn.*, **60** (2017), pp. 47–54 (in Japanese).
- 12) Nagao, M., Yoshida, T., Kanemaru, S., Neo, and Y., Mimura, H.: Fabrication of a Field Emitter Array with a Built-in Einzel Lens, *Jpn. J. Appl. Phys.*, **48** (2009), pp. 06FK02.