Design, Fabrication and Study of Micro-Electrospray Chips

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Abstract. This paper described design, fabrication and study of micro electrospray chips. The micro electrospray chips are fabricated by using micro-electro-mechanical system (MEMS) technology. The micro electrospray techniques can be used in many applications, including high viscous fluid dispenses for 3D rapid prototyping and biological sample analysis in mass spectrometer. In this study, micro electrospray chips with different wedge angels are investigated by using microscopic visualization techniques. Four spray modes are identified at various operation voltages and currents. It is found that the voltage for stable cone-jet mode decrease as wedge angle becomes smaller. On the other hand, it is also found that stable cone-jet mode represent a constant current range in current (I)-voltage(\textPhi) curve. It allows us to easily identify if the micro electrospray chips operate at cone-jet mode.

Introduction

With the shrinking size of chemical and biological analysis tools, the evolution of microfluidic technology is mirroring that of the microelectronics industry in the quest for ‘smaller, faster and cheaper’ devices and systems. There is, hence, a current need for the development of new easy-to-fabricate droplet ejection devices for high viscosity fluid. Microfluidic devices are being particularly developed for these applications. One way to eject high viscosity fluid at the micro system is to use electrospray techniques. Electrospray is of particular interest when working with high viscosity samples. In an electrospray system (Figure1), a solution of the sample fluid is passed through a capillary which is held at high potential. When the electric field is strong enough to overcome the surface tension force, the liquid forms a cone shape, so-called Taylor cone, at the nozzle tip. At the tip of Taylor cone, the effect of the high electric field as the solution emerges is to generate a mist of highly charged droplets which pass down a potential and pressure gradient towards a ground electrode. During that transition, the droplets reduce in size by evaporation of the solvent or by “Coulomb explosion (droplet subdivision resulting from the high charge density).

The electrospray study has been investigated for several decades. In 1971, Zeleny et. al [1] used electrospray in industry for fuel or paint spray. In 1968, Dole et. al. [2] applied electrospray techniques un separation technology successfully separate the large molecules into many small molecules. In their experiments, Faraday Cage is used in their experiments to detect current signal and use the current signal to estimate the small molecular size. Yamashita and Fenn [3] integrated electrospray with mass spectrometer (ESI-MS) technology since the electrospray can separate samples without any damage to the samples. It is also easy to integrate with any other tools in sample analysis. Aleksandriv, M. L. et. al.[4] demonstrated a methods to combine the ESI-MS and liquid chromatography (LC) technology together. This greatly enhances the ESI capability in sample analysis.

Electrospray now has been applied in my commercial instruments. Wilm and Mann [5] set up mathematical
model for capillary electrospray in 1994. In 1997, Karger, and Ramsey published paper for micro-electrospray chips [6,7]. Different from using capillary tube as a nozzle, they use photolithography fabrication techniques to etch a microchannel on a glass substrate and the exit of microchannel is surface modified to become hydrophobic. At the same year, Amish Desai et. al.[8] use MEMS technology to fabricate the micro electrospray nozzle. The nozzle is microfabricated on a silicon substrate. Xuan-Qi Wang et. al.[9] used Parylene-C polymer to fabricate the nozzle in 1999. This nozzle is flexible and will not be deform due to internal residual stress. Patrick Griss et. al.[10] use silicon and silicon nitride to fabricate the roof shooting type multi-array nozzle. The nozzle is fabricated by using deep reactive ion etching (DRIE) and wet etch. In 2003, Séverine Le Gac et. al.[11] use SU-8 to fabricate a pen-like nozzle and investigate the effects of different wedge angle in electrospray performance.

Figure 1: The basic principle of electrospray

**Design, Fabrication and Package of Micro electrospray chips**

Figure 2 shows the sketch of micro electrospray chip with wedge angle α=90°. There is a liquid inlet at the center of the chip. The liquid enters the chip from the inlet and ejects out of the chip from the nozzle tip. Two bonding pads are also shown in the Figure 2. In this study, fused silica glass is used as substrate. There are two parts in a micro electrospray chip. The first part is the electrode chip for high voltage input. The second part is the microchannel chip for liquid flow. These two chips are fusion bonding together and cut into different wedge angles with a dicing saw.

In the fabrication of electrode chip part, 300Å Ti and 1200Å Pt are first deposited on the glass substrate by using E-beam evaporator, and patterned by using lift-off technique. The width of electrode is 200µm and the bonding pads size is 2mmx2mm. For the microchannel fabrication, wet chemical etch is used to manufacture microchannel. Photoresist is first applied and patterned on the substrate. The glass chip is immersed in a BOE etchant to etch a microchannel with width 160µm and depth 20µm. Finally, photoresist is stripped and hole is drilled for the liquid inlet. After the completion of the microchannel and electrode chips, two chips are fusion bonding together at 610°C.

Finally, the chip is cut to a specific angle at the tip of nozzle. The wedge angles include 30°, 60°, 90°, 120°, and 150°. Figure 3 shows the micro electrospray chips with different wedge angles. Figure 4 depicts the SEM picture of the micro electrospray chips with wedge angle α=120°. In Figure 4, there is no clear interface between the electrode and the microchannel chips. It indicates the fusion bonding is perfect in the fabrication process. Figure 5 shows the package of micro electrospray chip.
Figure 2  The sketch of micro electrospray chip

Figure 3  The micro electrospray chips with different wedge angles

Figure 4 SEM picture of the micro electrospray chips with wedge angle $\alpha=120^\circ$.

Figure 5 Package of micro electrospray chip

Experimental setup

Figure 6 and Figure 7 show the sketch and photograph of the microscopic visualization experimental setup. The setup includes microscope (NIKON SMZ 1000), color camera from TELGEM, syringe pumps (Cole Parmer 74900 Series), high voltage power supply (YSTC High Voltage Power Supply, 0~30KV), multi-meter (TTI 1705), display, image acquisition, 100K$\Omega$ resistor and light source.

Experimental results and discussion

Micro electrospray chips with five wedge angles, ranging from 30$^\circ$ to 150$^\circ$, are used in the current study. The microchannel is 160$\mu$m in width and 20$\mu$m in depth. The parameter of experiments are listed in Table 1. The experimental results are described in the following sections.
A. Electrospray Modes

According to different operation voltages $\Phi$, the electrospray shows the different liquid shape at the tip of the nozzle. Figure 8 shows the electrospray mode photograph at different voltages. There are dripping mode, pulsating mode, cone-jet mode and multi-jet mode in Figure 8.
B Stable Cone-Jet mode

The operation voltage range for cone-jet mode is defined as the stable operation range $\Delta \Phi$. Figure 9 is electrospray pictures for the wedge angle $\alpha=90$, flow rate $Q=8.33 \times 10^{-11} \text{ m}^3/\text{s}$, fluid is 70% methane mixed with water (case7). The onset voltage is 6.5Kv. As the voltage increases, Taylor cone shrinks. When the voltage exceeds 9.0Kv, multi-jet mode appears.

![Figure 9 Electrospray pictures for wedge angle $\alpha=90^\circ$, and flow rate $Q=33.3 \times 10^{-11} \text{ m}^3/\text{s}$](image)

C. Relation between Current $I$ and operation voltage $V$

In Figure 6, 100K$\Omega$ resistor is used in the setup to measure current. The relation between current $I$ and applying voltage is shown in Figure 10. Figure 10 is the relation between current $I$ and applying voltage for micro electrospray chip with wedge angle 150$^\circ$. The operation condition is the same as case 14 in Table 1.

![Figure 10 Electrospray picture and the current (I) v.s. voltage($\Phi$) curve for micro electrospray chip with wedge angle $\alpha=150^\circ$](image)
Conclusions

As a final remark, this work indicates that the use of the electrospray techniques for dispense liquid application. Four operation modes are observed. They are dripping mode, pulsating mode, cone-jet mode and multi-jet mode. For the cone-jet mode, a constant current in I- Φ curve is identified.

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References