

Micro Ion-Optical Systems Technology [MIST] for Mass Spectrometry Using PCBMEMS

David Fries, Stan Ivanov, Heather Broadbent
Marine Sciences

University of South Florida
St. Petersburg Florida USA

Email: dfries@marine.usf.edu, sivanov@marine.usf.edu

Ross Willoughby, Ed Sheehan
R&D

Chem-Space Associates
Pittsburgh Pennsylvania USA

Email: ross@lcms.com, sheehan@lcms.com

Abstract— Within the traditional mass spec instrumentation field there is ongoing interest in new atmospheric ion source designs for more effective and versatile ion generation. The objective of the present study is to apply Organic MEMS microfabrication technologies to generation of atmospheric pressure ion optical devices. We have devised novel materials, processes, and designs for micro ion optical systems for control of ions within sources, and across apertures and conductance arrays. PCBMEMS using LCP have been used in the construction of the devices. Vacuum compatibility of the polymeric material has been found to be similar to glass in performance characteristics. Processes for shaping the polymer dielectric for fluid flow control and the metallization for electrical field control have been devised. Different geometries, both tubular and planar, combined with electrical field shaping circuitry and fluidic flow control networks are part of the effort.

I. INTRODUCTION

The generation of ions and charged particles at atmospheric pressure is accomplished by a variety of macroscopic design and fabrication means. A general characteristic of all atmospheric sources is the dispersive nature of the ions once produced. [1] Needle sources and aerosol techniques disperse ions radially from the tip axis in high electric fields and in the radial flow of gases emanating from tubes and nebulizers, respectively. The radial cross-section of many dispersive sources can be as large as 5 or 10 centimeters in diameter. As a consequence of a wide variety of dispersive processes, efficient sampling of ions from atmospheric pressure sources to small cross-sectional targets or through small cross-sectional apertures and tubes, (typically in the hundreds of microns), into a targeted small region in space becomes quite problematic.

Our goal is to increase the collection and transmission efficiency of ions and/or charged particles through an aperture or tube into a vacuum system, by creating a precisely controlled cross-sectional area and flow of a beam of ions and/or charged particles. Micropatterned and laminated atmospheric ion optical devices offer an alternative to traditional ion optical devices by enabling control of both fluidic and electrostatic pathways to locally control field, flow, heat transfer, and ultimately ion collection efficiency.

Within the traditional mass spec instrumentation field there is ongoing interest in new atmospheric ion source designs for more effective and versatile ion generation. Similarly, we are interested in developing new atmospheric-source based fieldable mass specs beyond the membrane based underwater

mass specs we had developed in the past. [2,3] while designing for maximum ion throughput sensitivity.

Prior work has been accomplished exploring ion optical elements for atmospheric ion sources [4,5] to facilitate and control the transport of ions through the interface region between atmospheric sources and the vacuum region of a mass analyzer. This can be an effective vacuum miniaturization strategy for field portable mass spectrometric instrumentation. The ion optical elements are used to increase the focusing of ions from the atmospheric ion generators into the very small differential pumping apertures at the interface of the vacuum system reducing the gas load and decreasing the need for power hungry high throughput vacuum pumping solutions. Current mass spectrometers have high vacuum throughput systems that need to accommodate the high rate of atmospheric molecules across the aperture that separates the atmospheric source from the vacuum environment of the mass analyzer.

Since greater ion collection efficiency can permit the reduction of the size of the aperture leading into the vacuum chamber, exponential reduction in vacuum throughput will occur. Consequently, there is a need to create controlled, efficient ion transport from atmospheric pressure to vacuum pressure to enable continuous sampling portable mass spectrometer systems.

Recent modeling developments in source and optics design at atmospheric pressure show that maximum control of ion motion may be accomplished by very precise control of geometric shapes and orientations of electrode elements and fluidic pathways. These source features require spatial tolerances down to the 5-10 um range. Microsystems technologies are ideal for integration of these devices The objective of the present study is to apply alternative (non-silicon-based) microfabrication technologies to generation of atmospheric pressure ion optical devices. We have devised novel materials, processes, and designs for micro ion optical systems for control of ions within sources, and across apertures and conductance arrays. Patterned microstructures and geometries for flow and electrostatic field shaping are achieved using PCB-MEMS, also known as Organic MEMS. PCB-MEMS is the next stage of evolution beyond simply providing electrical interconnection and mechanical support. It is the combined insertion of mechanical, fluidic, optical and electronic components into the low cost PCB landscape with high feature resolution and potentially over a large area.

II. MIST

Micro Ion-Optical Systems Technology [MIST] is the convergence of *fluidic-electrostatic-mechanical* functions into an active or passive system for ion manipulation and control using controlled, shaped electric fields and controlled fluidics. The system may be a system in package (SIP) or eventually system on a chip (SOC). Active systems may dynamically control fields and flow and contain moving mechanical components. Passive systems have no moving parts. We chose passive systems as our initial direction since they are easier to produce cost effectively. Advantages of PCBMEMS MIST include adaptive sampling of ions, laminated 3D systems, feedback control of ion transmission, integration of electronics and large area designs are possible

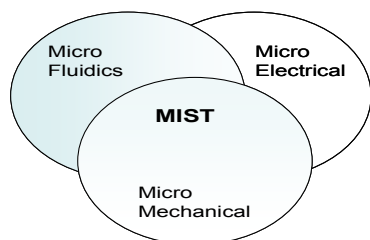


Fig. 1. Micro Ion-Optical Systems Technology [MIST] is the overlap regions of *fluidic-electrostatic-mechanical* functionality into an active or passive system for ion manipulation and control using controlled, shaped electric fields and controlled fluidics

In making Micro Ion-Optical Systems Technology [MIST] one could try to employ standard silicon MEMS technology but silicon has limits in area patterning, the ease of creating 3D systems and is not a favorable material for high voltage applications.

We chose to use polymer dielectrics and metal thin films, a proven combination for high field strength applications in our PCBMEMS process flow. Our particular choice for the dielectric is Liquid Crystal Polymer. The laminates we have designed and fabricated are comprised of liquid crystal polymers with photodefinable bond materials and various electroformed metals. The choice of PCBMEMS was driven by the requirement of a low investment process, a material capable of high levels of electronic systems integration, while providing a vacuum compatibility competitive to glass.

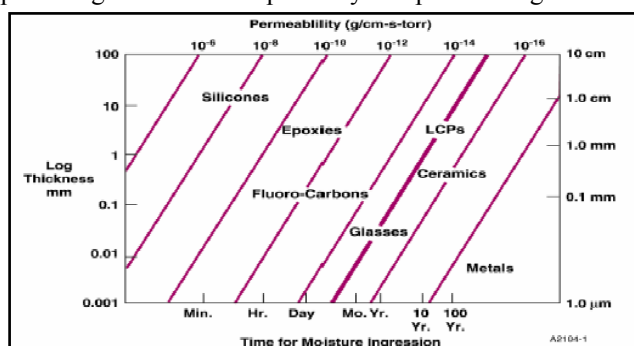


Fig. 2. Biaxially oriented LCP films prevent water transmission and enables good vacuum and environmental performance.

III. ION FOCUSING

These ion optical elements made via PCBMEMS construction are used to increase the focusing of ions from the atmospheric ion generators into the very small differential pumping apertures at the interface of a vacuum system. Initial work has demonstrated an increase in ion transport into the mass spectrometer by a factor of three to ten. Fig 3 shows simulated and experimental results of ion focusing for an electro spray source. Other divergent ion sources will yield similar ion trajectories and performance.

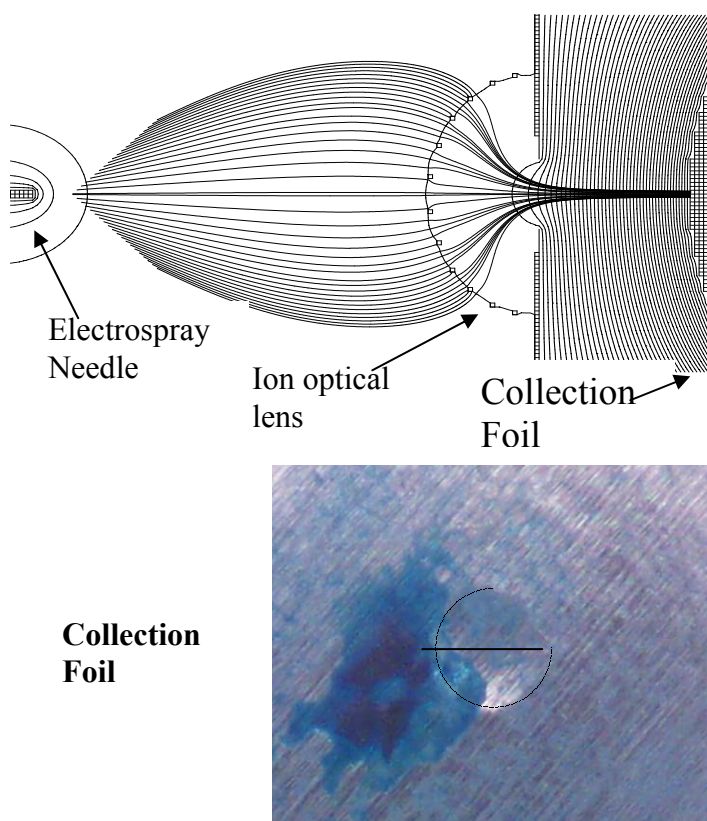


Fig 3. Upper: SIMION simulation of ion trajectories from electro spray dye experiments at atmospheric pressure with a hemispherical shaped HTE. The simulation shows the approximate geometry of the apparatus with needle voltage at 5+5 kV, HTE at 0 V, and the collector surface covered with aluminum foil at -12 kV. Lower: Photomicroscopy of the collection of blue dye through a lens optimized for compression. The resulting area compression ratio is greater than 5000. The dye was collected for 16 hours. Note the indentation of the foil [outlined in black] where the sampling aperture exists. The aperture diameter indicated by the dotted black line was 500 μm.

Using Ion Selective Aperture Arrays as our targeted design (Fig. 4), we tried creating our multilayer microholes arrays using: 1. conventional mechanical drilling 2.laser drilling, 3.photoimageable dielectric etch and deposition, 4. combinations of photoimaging, etch, deposition (i.e PCBMEMS). We have found the PCBMEMS process to be

the most versatile in making heterogeneous organic microsystems. An enabling aspect of the LCP PCBMEMS is that the LCP etching provides a sacrificial layer for isolating the conductor *and* the conductor provides a sacrificial layer for isolating or suspending the dielectric. This process is similar in versatility as the silicon/SiO₂ etch systems in standard MEMS.

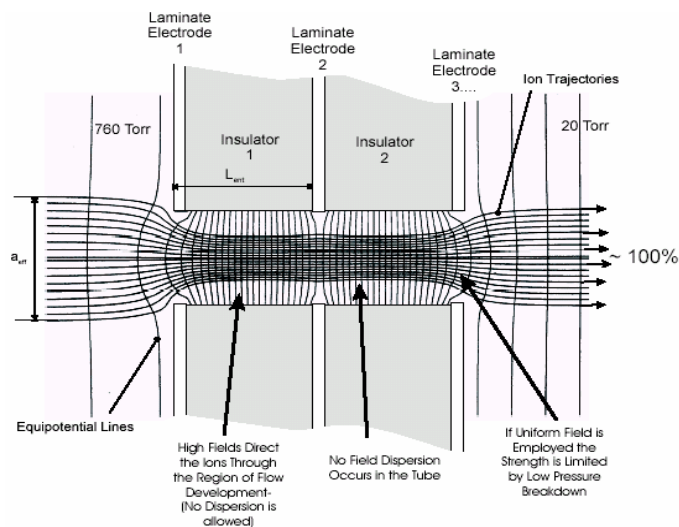


Fig. 4. Schematic diagram of a laminated aperture that ensures that the ions are presented with a uniform field as they traverse the entire length of the conductance pathway. The lack of dispersive fields at the entrance and within the tube maintains the axial trajectories of ions across the entire flow development region. Simulations generated from SIMION. The scale of the aperture is on the order of 50-100 μm .

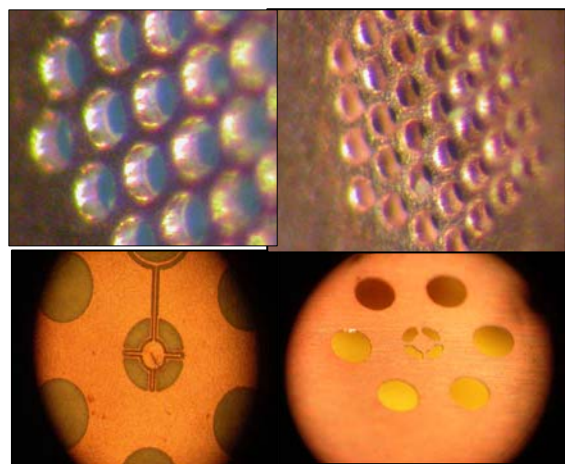


Fig. 5. Process results for array patterning the LCP polymer dielectric for fluid flow control and the Cu metallization for electrical field control. The holes in the array are 70 μm in diameter and form the tube flow condition. The holes are equivalent to nonplated vias. The upper planar array photo set is of one design, the lower set is of a second design. Both designs have been verified for ion transmission,

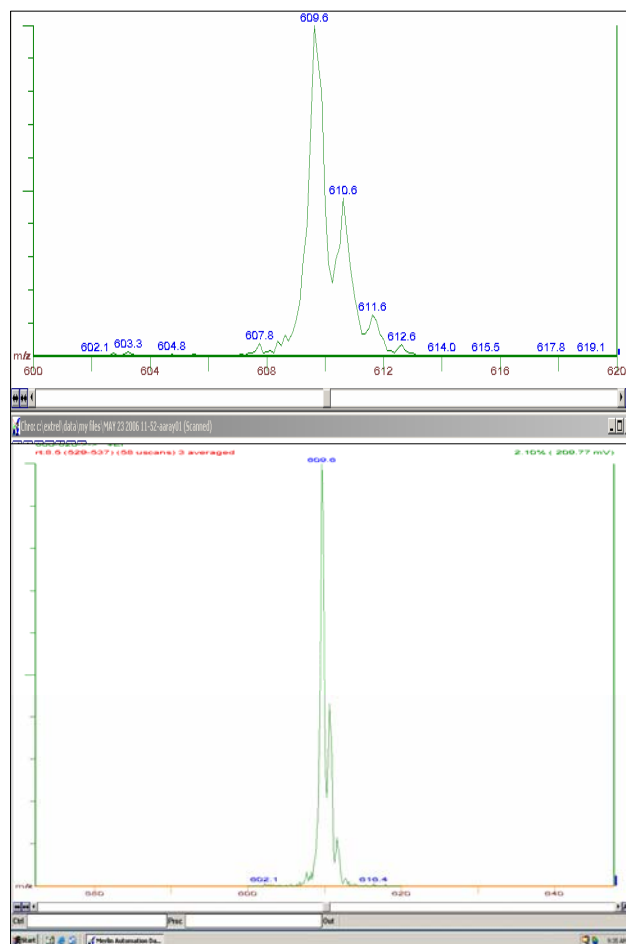


Fig. 6. Mass spectrum of reserpine electrosprayed and focused through the multilayer aperture array shown in Fig 5

IV. CONCLUSIONS

The laminates designed and fabricated are based on liquid crystal polymers (LCP) with photodefinable bonding materials and various electroformed metals. The choice of PCBMEMS was driven by the requirement of a low investment process, a material capable of high levels of electronic systems integration, while providing a vacuum compatibility competitive to glass. Liquid Crystalline Polymer (LCP) based components have been developed for numerous atmospheric sensing applications and designs for atmospheric ion control and control of conductance across interfaces has been completed. PCBMEMS fabrication techniques have been used in the construction of these devices. Vacuum compatibility of the polymeric material has been found to be similar to glass in performance characteristics.

Liquid Crystalline Polymer (LCP) based designs and components have been created for atmospheric ion transport and control of conductance across an interface. Different geometries, planar and laminate, have been fabricated. Initial processing results indicate that this route to miniaturization and

rapid fabrication is an attractive path to designing affordable, accessible integrated ion optical systems and provides optics designers advantages over standard MEMS processing. The use of this technology may ultimately lead to more compact mass spectrometers and analyzers capable of being used in the field that can directly sample ions from ion sources operating at atmosphere.

ACKNOWLEDGEMENT

This work was supported by the Navy (Grant number N00014-03-1-0480) and NAVSEA Crane ICIT. The authors are grateful to George Steimle for his tremendous support on process developments contributions on completing this project.

REFERENCES

- [1] [Hutchens, T. W, Yip T. T. 1993. New desorption strategies for the mass spectrometric analysis of macromolecules. *Rapid Commun. Mass Spectrom.* 7:576-580.]
- [2] Fries DP, Short R, Toler, S, Lembke, C, Kerr M and Byrne R. Mass spectrometry in the hydrosphere. *49th Proceedings of the American Society for Mass Spectrometry*. Long Beach, California, 2000.
- [3] Fries DP, Short RT, Langebrake, LL, Patten JT, Kerr ML, Kibelka G, Burwell DC and Jalbert JC. In water field analytical chemistry: mass spectrometers, robots, numerical models for wide area chemical plume localization. *Field Anal. Chem. Tech. Journal* 5: 121-130.D. 2001
- [4] Sheehan EW, Willoughby RC and Fries DP. Atmospheric Pressure Focusing. *52nd Proceedings American Society for Mass Spectrometry*,. Nashville, TN. May 23-27 2004.
- [5] Willoughby RC, Sheehan EW and Fries DP. Transmission of Ions Through Conductance Pathways from Atmospheric Pressure. *52nd Proceedings American Society for Mass Spectrometry*. Nashville, TN. May 23-27 2004.