High-Aspect-Ratio CNT Micronozzles for Electrospray Ionization

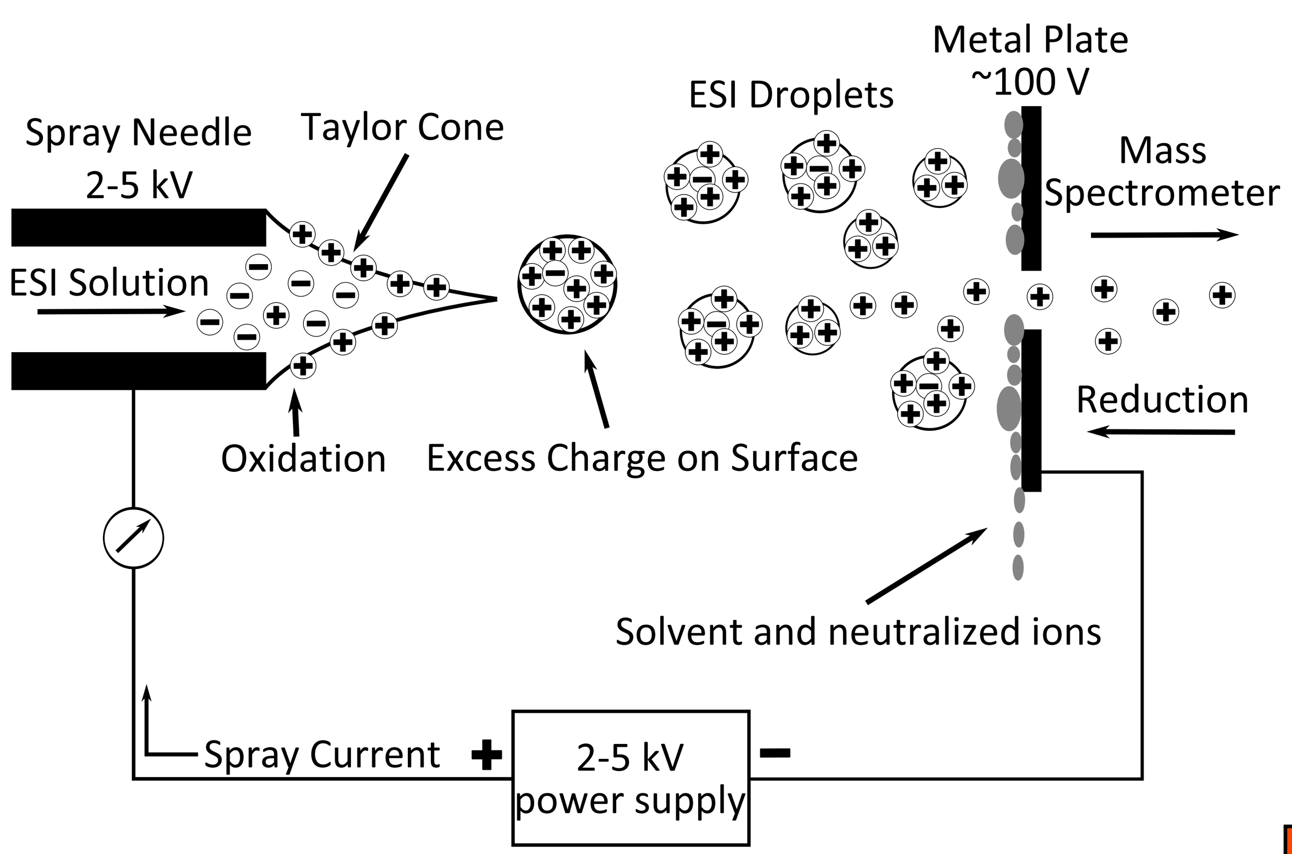
Adam Konneker, Nick Morrill, Stephanie Getty, Yun Zheng, David Allred, Robert Davis, Richard Vanfleet

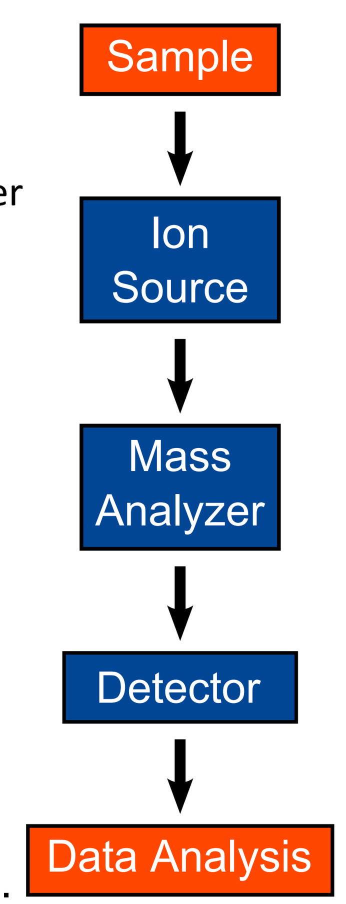
Project Goal

Fabricate micronozzles on-chip for use in a microfluidic electrospray ionization system.

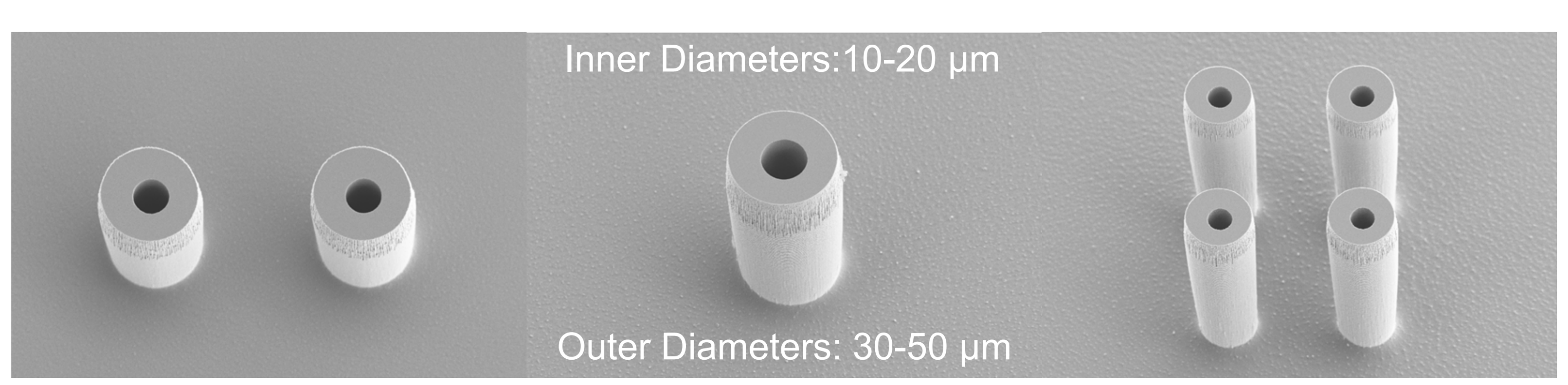
This system will enable the production of a compact mass spectrometer for applications astrobiology such as in-situ analysis of organic molecules.

Electrospray Ionization (ESI)



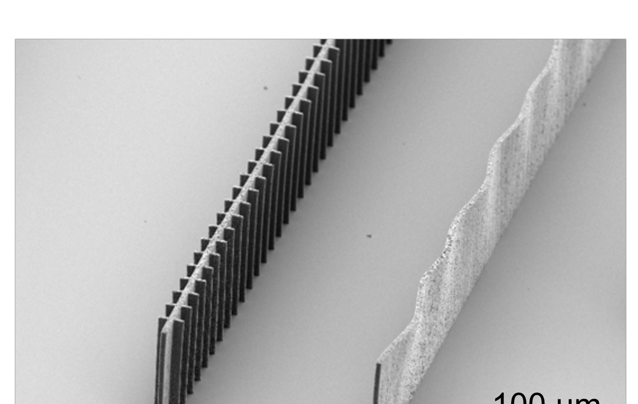


ESI is a technique for ionizing molecules used in mass spectroscopy. It is particularly useful for analyzing large, organic molecules because it does not cause them to fragment as readily as other methods.

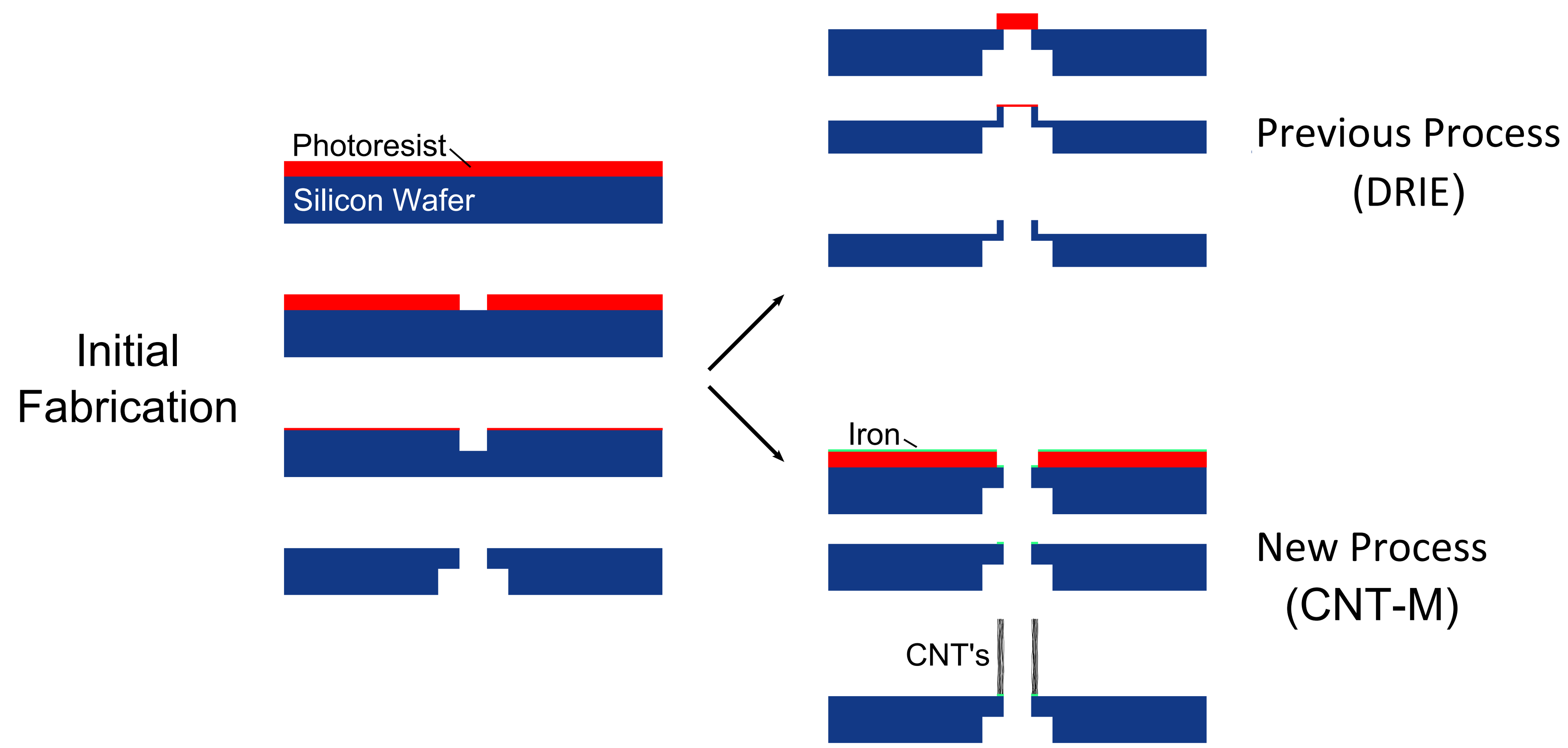


Initial attempts to fabricate silicon micronozzles utilized deep reactive ion etching (DRIE), but the best results only produced nozzles approximately 200 µm tall.

Carbon nanotube templated microfabrication (CNT-M) has been shown to be an effective technique for fabricating high aspect ratio microstructures with very small pore sizes, and this is the primary reason why it was chosen as an alternative to DRIE. CNT-M is still under development however, and it has unique design challenges and restrictions.

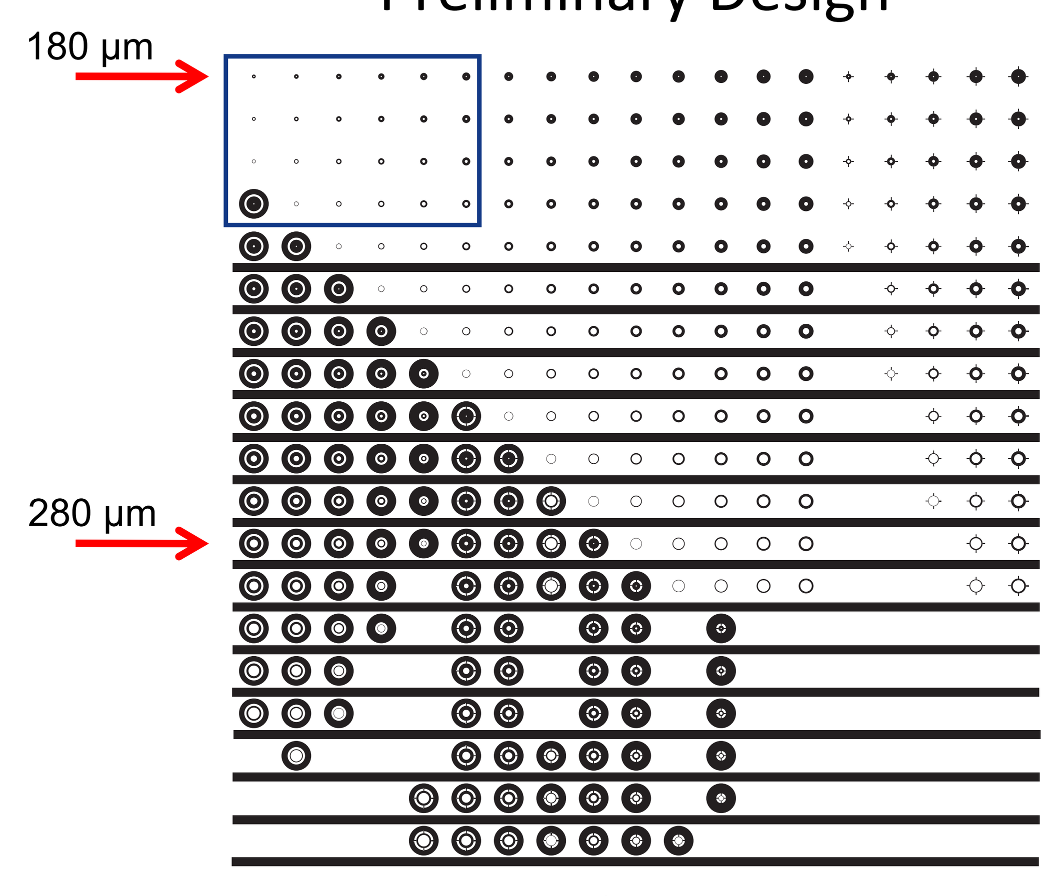


Nozzle Fabrication



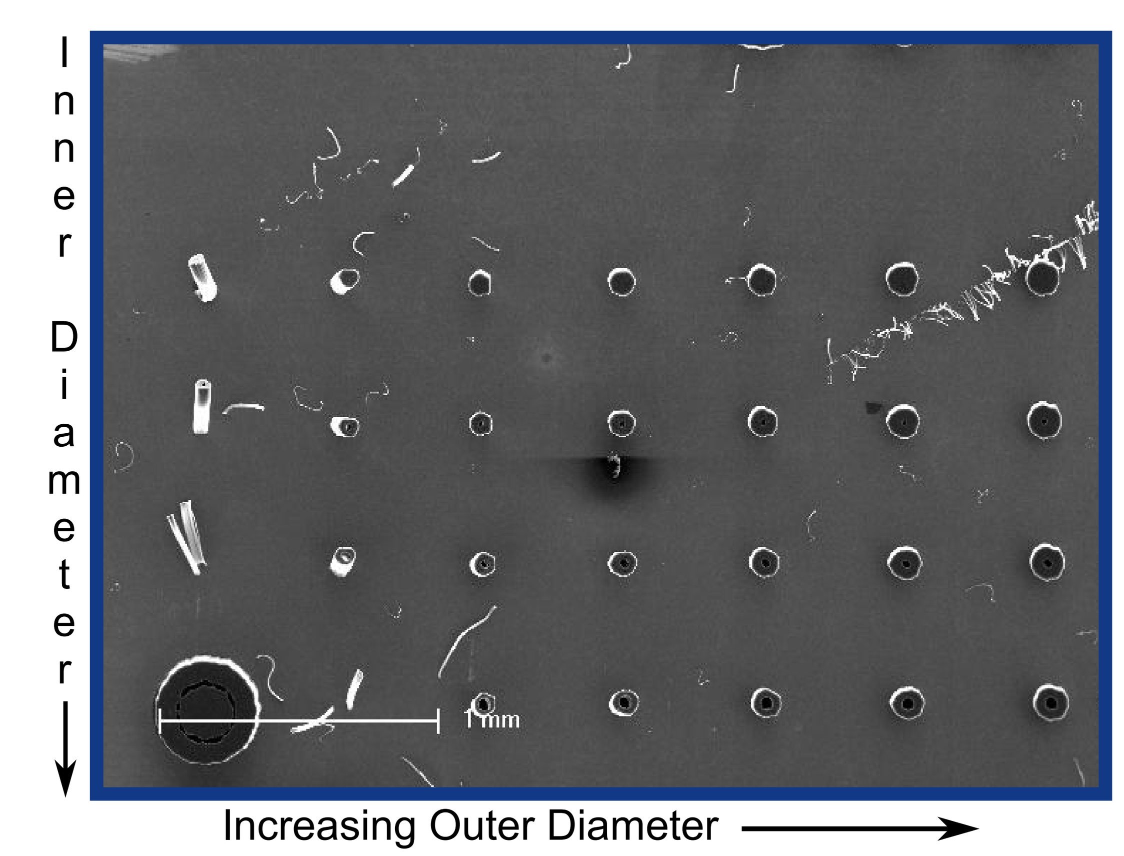
The CNT-M method will use the same initial steps as the DRIE process to pattern and create holes through the silicon substrate. The nozzle fabrication step is much different, however, and requires the deposition of aluminum oxide (30 nm) as a diffusion barrier followed by a thin film of iron (4-10 nm) which serves as a catalyst for CNT growth. The grown CNT nozzle is then coated and filled with another material to create a solid structure. This step was not done in this preliminary study due to the fact that the CNT forest provides the shape of the final striucture.

Preliminary Design



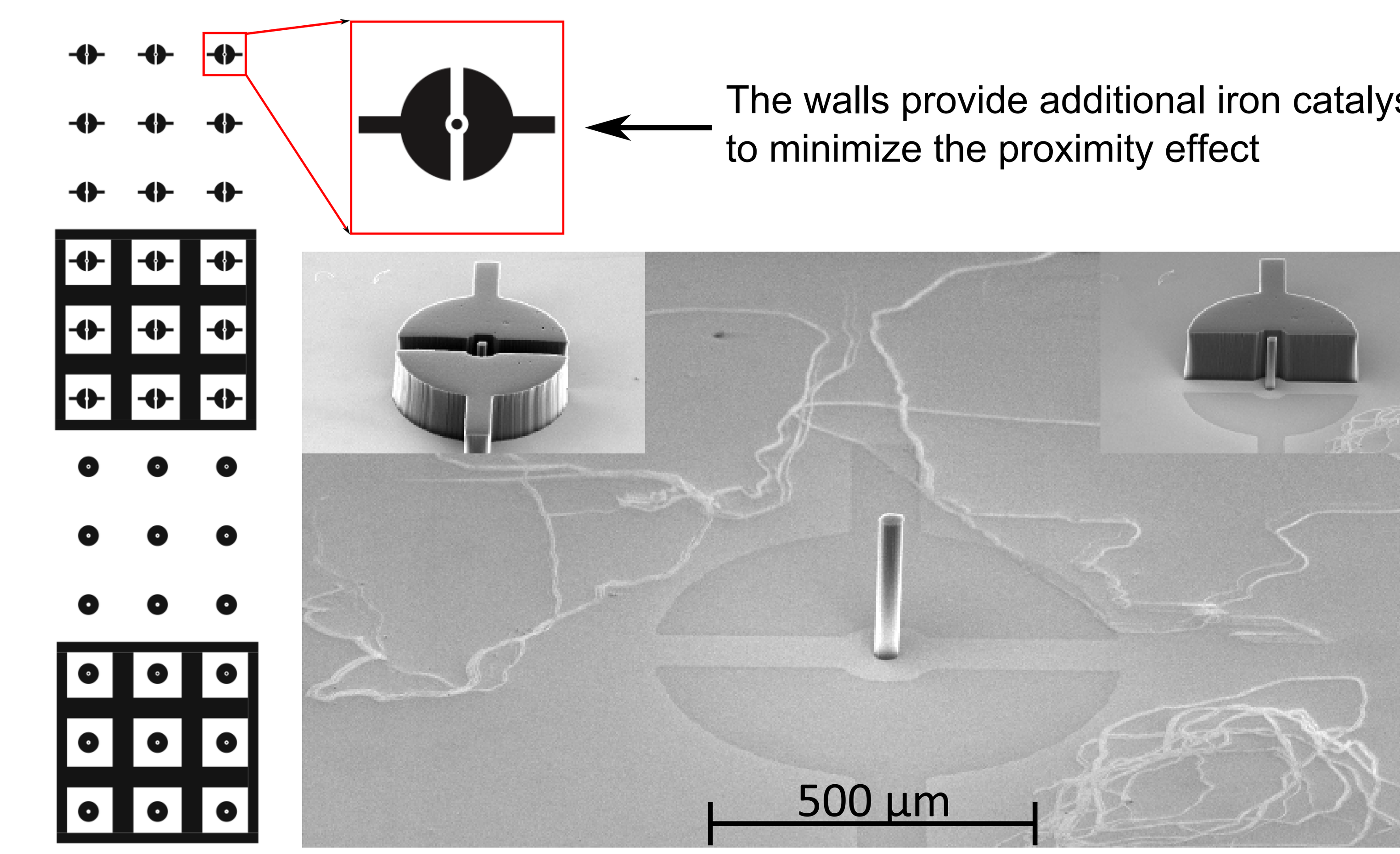
The first test mask contained a wide variety of nozzle shapes with varying inner and outer diameters. The various geometries were designed to explore the feasible pore sizes that could be created using the CNT-M process. This mask was also designed to compare the structural stability of simple cylinders to nozzles with reinforcing elements such as crossbeams.

Proximity Effect



Shown above is an electron micrograph of one of our first test samples, focused on the region with the smallest nozzles. In initial tests using this design, it was observed that the smallest nozzles always grew to a final height much shorter than any feature towards the center of the pattern. This is due to the 'proximity effect' of iron on CNT growth, which causes regions of high iron surface density to grow faster and taller than areas with lower catalyst density.

Refined Design



Outer Diameter: 50 µm Height: 371 µm

In our second test mask, we focused on the smaller diameter nozzles and added sacrificial walls into the design to avoid the limitations of the proximity effect. This tactic produced straighter nozzles and a fourfold increase in maximum height from the initial design.

Summary

Preliminary work has been done on the use of the CNT-M process for the fabrication of high aspect ratio micronozzles. It has been demonstrated that design geometry must be optimized to allow a relatively large amount of iron catalyst around important features. This optimization allows the minimization of the proximity effect of iron catalyst in CNT growth, and allows for the creation of devices with higher aspect ratios. In the future, these CNT nozzles will be filled with other materials to create working nozzles for electrospray ionization.

References and Acknowledgments

We would like to thank Stephanie Getty and Yun Zheng of Goddard Space Flight Center; Robert Davis, Richard Vanfleet, and David Allred of Brigham Young University for their advice and support. We would also like to thank NASA and the Rocky Mountain Space Grant Consortium for funding this project.