A Study of Miniature Cylindrical Ion Trap Mass Spectrometers as a Basis for Micro-fabricated Underwater Mass Spectrometer Sensors.

F.H.W. van Amerom, A. Chaudhary, M. Telrande and R.T. Short,
Center for Ocean Technology, University of South Florida, St. Petersburg, FL

Introduction

It is of major importance to continuously monitor marine waters to protect living organisms from hazardous chemicals and to aid in understanding ecological systems [1]. Miniature Cylindrical Ion Trap (CIT) Mass Spectrometers are ideal candidates for in-situ underwater mass spectrometry sensors allowing for longer deployments and portability due to the reduced power consumption and the smaller weight and size. In addition, CIT Mass Spectrometers are capable of ms^n [2], which is particularly useful in analysis of complex mixtures of chemicals. Several methods of building small CIT Mass Spectrometers are investigated.

Experiments

A set-up was built (fig. 1) to test miniature CIT Mass Spectrometers. The set-up allows ramping of a sinusoidal waveform to 2200 V_{op} at 1.5-6 MHz in 15-65 ms. Analytes are ionized using a rasterable electron gun. Scanning of the electron beam over an array of traps is thus possible.

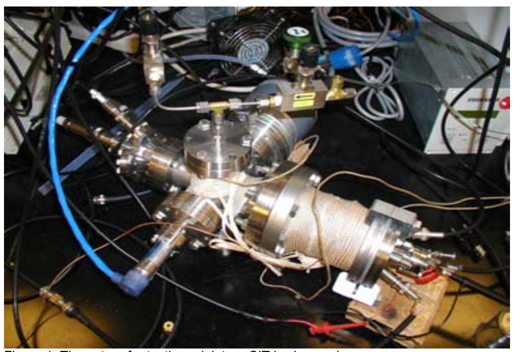

Figure 1. The set-up for testing miniature CIT is shown above.

The vacuum housing set-up includes an inlet for buffer gas and analytes, a turbo pump capable of reaching pressures as low as 10^{-8} Torr and a flange with electric feedthroughs. A CIT can be mounted on the flange with the electrical feedthroughs.

Results

To test the set-up, a 2.00 mm radius CIT, with endplates having a 1.00 mm radius aperture, and 0.64 mm end gap spacing (r_0=2 mm, z_0=2.31), was mounted in a holder on the vacuum flange inside the vacuum chamber. Helium gas and perfluoro-tributyl-amine (PFTBA) were introduced at $1\bullet10^{-4}$ Torr and $1\bullet10^{-6}$ Torr. An air-coil was attached on one side to the cylinder of the trap and on the other side to a broadband power amplifier. This formed a series resonant circuit resonating at 1.760 MHz and gave a high voltage sine wave on the cylinder of the trap. The sine wave was held constant at 85 V_{op} for 15 ms to trap ions during the ionization of the PFTBA. This was followed by ramping the RF voltage from 85 V_{op} to 500 V_{op} in 45 ms. Ions ejected from the CIT during ramping of the RF voltage were detected as a function of time. The resulting time resolved mass spectrum was stored on a Le Croy digital oscilloscope.

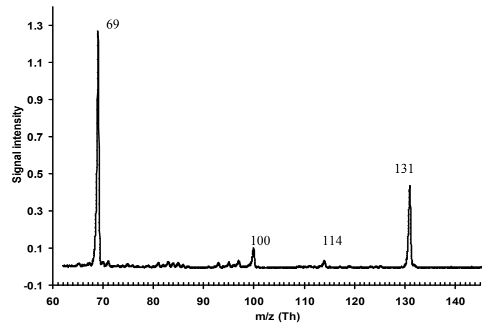

Figure 2. A mass spectrum of PFTBA at 10^{-6} Torr and Helium buffer gas at 10^{-4} Torr, measured with a 2 mm radius miniature CIT. Axial modulation at 1/3 of f_0 is applied to improve the resolution.

Miniaturization

Several techniques, such as steel etching and silicon etching, along with various materials, for example, green tapes, bulk ceramics and steel cylinders, were examined for construction of small CIT's. Green tapes are soft sheets, which are easily machined and can be layered, laminated, punched and drilled. The laminated Green-tape CIT structures turn into ceramics after firing in an oven at 850°C.

Figure 3. On the left, holes are drilled in the laminated green tape before firing at 850°C. Lamination of green tape sheets allows for various thicknesses. On the right, electro-less nickel is applied to the laminated and fired green tape structures.

Figure 4. On the left, a CIT is fabricated by compressing green tapes in a die. After firing at 850°C, the CIT is first plated with electro-less nickel and after that with electro-less gold. Shown on the right is the stainless steel die.

A die, see figure 4, was used to compress the green tapes. More complex structures are allowed by this technique.

Several 4 x 4 arrays of miniature CIT's were micro-fabricated and are to be tested (see figure 5). The endplates were fabricated out of thick Si wafers using Deep Reactive Ion Etching (DRIE). Ring electrode arrays were created from a 1600-microns thick wafer using ultrasonic drilling.

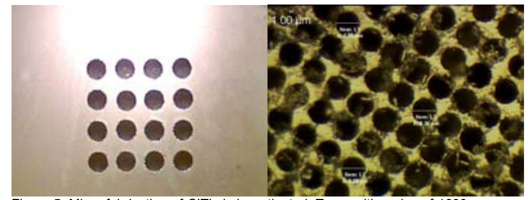

Figure 5. Micro fabrication of CIT's is investigated. Traps with a size of 1600 µm were drilled using an ultrasonic disc driller. Shown on the right is an endplate mesh with a hole size of 5 µm made from Si with deep reactive ion etching (DRIE).

The Si wafer components were plated with electro-less Ni and electro-less gold to create conducting surfaces and bonded together using CYTOP, a cyclized perfluoro polymer, as the insulating layer and glue at the same time.

Conclusions

Work is in progress to improve the techniques and quality of the CIT's. Green tapes are very easy to use and a large variety of shapes can be produced with a die. One of the drawbacks of miniaturization of Mass Spectrometers is that storage capacity is diminished and mass resolution can degrade, both of which tend to decrease their value for in-field analysis. Storage capacity can be maintained by careful construction and limiting the mass range for specific analysis.

References

[1] "Underwater Mass Spectrometers for in situ Chemical Analysis of the Hydrosphere", R. T. Short, D. P. Fries, M. L. Kerr and C. E. Lembke, J. Am. Soc. Mass Spectrom., 2001, 12, p 676-682.

[2] "Miniature cylindrical ion trap mass spectrometer", G. E. Patterson, A. J. Guymon, L. S. Riter, M. Everly, J. Griep-Raming, B. C. Laughlin, Z. Ouyang and R. G. Cooks, Anal. Chem., 2002, 74, p 6145-6153.

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