Microfabricated Silicon Nib for Low-Cost, Mutiplexed Micro-Coating

Erik V. Mukerjee
(925) 423-4841
mukerjee2@llnl.gov

Chemical and biological detection systems are key areas of interest at LLNL, directly supporting its mission of nonproliferation and homeland security. Many modalities of sensor platforms are presently being pursued (*e.g*,. BAMS, IMS, GC-MS), including novel microscale detection systems. Current LLNL micro-cantilever-based detection systems have shown promise for both biological and chemical detection and identification. This system couples organic polymer (polyolefin) swelling to mechanical stress causing resistance changes in the sensing element as a function of analyte exposure.

However, the fabrication and integration of these systems involves time-consuming serial processes for coating each micro-cantilever with the chemically sensitive polymer. The current coating set-up consists of a single-machined stainless steel nib (tip of a fountain pen) structure engineered to wick and store fluid into a reservoir until dispensed via contact with a higher surface energy material. Mechanical contact between the nib and cantilevers often causes the cantilevers to break. The fixed large reservoir capacity is governed by macroscale machining and therefore the dispensing volume is controlled by differences in surface energies and not by total reservoir volume.

The effort of this work was to use a modified coating structure similar to the meso-contact printing mechanism currently in use. Creating an in-plane silicon array of micro-coating structures will enable much faster throughput of polymer film selection and trials. Film thickness uniformity across devices is essential for device functionality. The sheer volume of polymers trials/selection necessary for sensor viability and selectivity almost certainly precludes serial coating methods.

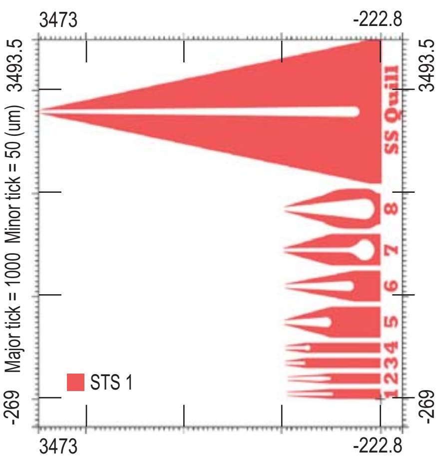

Figure 1. Schematic of nib geometries including the current stainless steel configuration.

Project Goals

The goal of this project is to fabricate a multiplexed polymer micro-coating system. Near term application is specific to the GS chemical agent detection project; however, the application space can be expanded to include chemical/biological assays and microscale organic materials deposition processes.

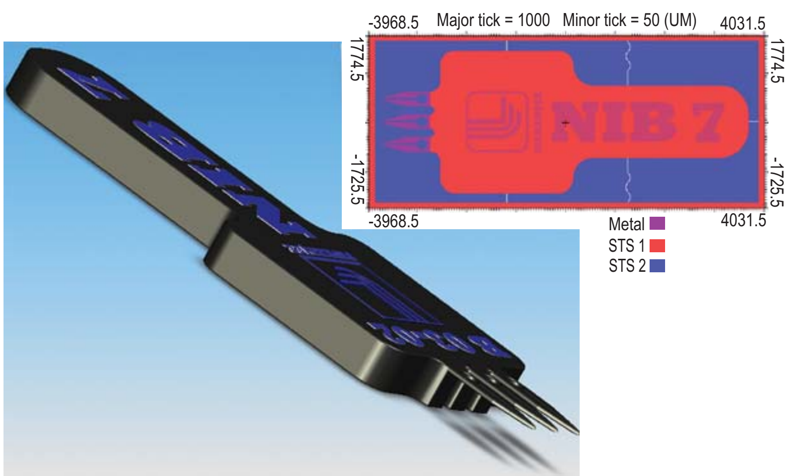

Figure 2. Illustration of final nib structure and mask (insert).

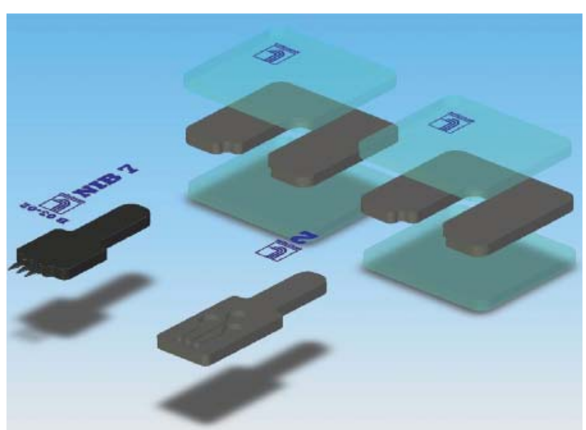

Figure 3. Exploded schematic of nib, reservoir, and universal socket structures.

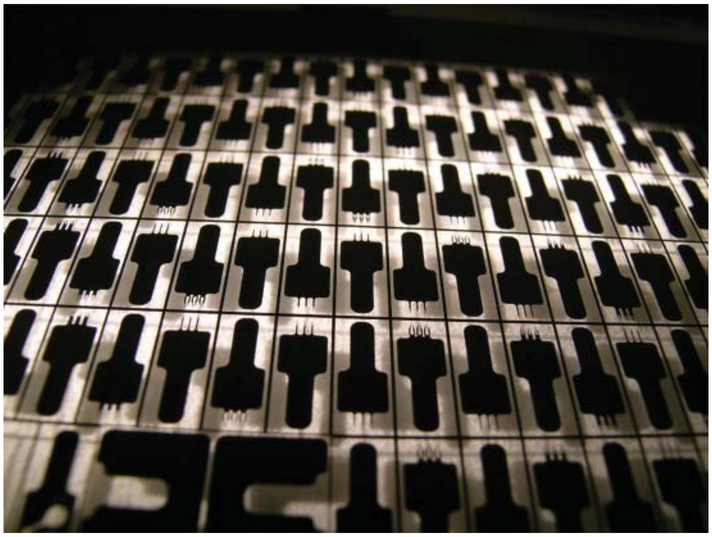

Figure 4. Wafer level view of various nib configurations.

Relevance to LLNL Mission

Work on this device advances several processing techniques to extend LLNL's capabilities and will produce a significant technological impact on the instrumentation community. The resulting technologies will aid in the fabrication of deployable detection systems that are well aligned with the national security mission of LLNL, *e.g.*, biodetection of pathogens and chemical warfare agent detection.

FY2007 Accomplishments and Results

In FY2007 the milestones that were achieved include layout, fabrication, characterization, and testing of several silicon micro-nib structures. An overarching technical challenge was thickness uniformity of the individual final nib elements. Cross-wafer etch rates of the deep reactive ion etcher (DRIE) varied as a function of position on the silicon substrate (1.8 µm/min to 2.4 µm/min, ~25%). This resulted in final device thicknesses ranging between 5µm to 125 µm on a single silicon substrate. Changing localized loading effects (*i.e.*, amount of exposed silicon in a given region) improved etch uniformity to an acceptable level of ~10%.

Each lithographic mask set produced 1) eight nib geometries; 2) three reservoir geometries; 3) universal sockets; and 4) a set of larger demonstration structures (4x larger).

The nib geometries varied in length (0.5 mm to 1mm, and 3.5 mm for stainless steeel); total reservoir volume (4 nl to 25 nl, and 400 nl for stainless steel); and volume to surface area (15:1 to 50:1, and 35:1 for stainless steel). The universal sockets facilitated rapid switching of nib configuration during characterization runs and fit both the nibs and the reservoir structures. The testing assembly consisted of two micromanipulator stages (X-Y cantilever mounted stage, X-Z nib mounted stage) and a stereoscope for visualization of cantilever coating.

Results are as follows:

1. Maximum fracture load
   a. Average nib 1 through 4: 1.3 N
   b. Average nib 5 through 8: 3.8 N
2. Average spot size (0.1% polyolefin/dioxane, nibs 5 through 8 on planer silicon nitride surface): 423 µm +/- 5%
3. Nib thicknesses 20 µm to 30 µm most consistent dispensing
4. Cantilever coating
   a. Dummy cantilevers structures (nibs 6 and 7 most consistent)
   b. Real cantilever sets (nibs 6 and 7 to be tested and run against various analytes)

From these results, optimal nib geometries (nibs 6 and 7) and thicknesses (25 µm+/- 5 µm) were determined for consistent/uniform polyolefin dispensing. This preliminary data suggest that the microfabricated silicon nib arrays can be used as a cost/time effective alternative to the serial stainless steel system currently in use.

Figure 5. Demonstration of 1-to-1 registering of nib elements with micro-cantilevers and size comparison with current stainless steel nib structure.

Related References

1. Baller, M. K., "A Cantilever Array-Based Artificial Nose," *Ultramicroscopy*, **82**, pp. 1-9, 2000.
2. Porter, T. L., "Sensor Based on Piezoresistive Microcantilever Technology," *Sensors and Actuators A-Physical*, **88**, pp. 47-51, 2001.
3. Sepaniak, M. "Microcantilever Transducers: A New Approach to Sensor Technology," *Analytical Chemistry*, **74**, pp. 568A-575A, 2002.
4. Then, D., "A Highly Sensitive Self-Oscillating Cantilever Array for the Quantitative and Qualitative Analysis of Organic Vapor Mixtures," *Sensors and Actuators B-Chemical*, **117**, pp. 1-9, 2006.