Investigation of Microscale Electromechanical Technologies for Continuous Monitoring

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Final Report
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Efforts are under way worldwide to integrate electronic and mechanical components on solid state devices to perform the functions of traditional sensors at the micro-scale. For example, chemical sensors have been envisioned that involve pumping air or liquid samples at small volumetric flow rates over long periods of time for continuous monitoring of environments. The mechanical components on these devices are fabricated into monolithic silicon or other solid state media. These types of devices, if proven successfully, would offer new capabilities for remote monitoring at very small size, weight, and power consumption. When coupled with micro-scale monolithic communications components, the technical option could exist for extremely small remote monitoring devices.

Heretofore, little effort has been invested at the Laboratory in examining specific design problems that could capitalize on the emerging component technologies under development at both large and small computer and electronics firms worldwide. Our program involved conceptual designs and selected prototype manufacturing of three different types of micro-scale sensors that could be applied to problems in the nonproliferation mission areas.

Ruggedized Gas Chromatography Column

This project was designed to address some of the technical issues associated with taking miniature open-tubular gas chromatography columns into the field in a small, portable package. Principal design goals include high separation efficiency, column ruggedness, low manufacturing cost, and low thermal mass and the consequent requirement for heater power supply capacity. Applications include remote monitoring of chemical effluents in unattended ground sensors, portable mass spectrometer instruments, and stationary portal installations.

Most open-tubular gas chromatograph columns used today are made of flexible, fused silica thin wall tubing, with greater chemical compatibility than the original stainless steel designs. Drawbacks to quartz that our design seeks to overcome include size, fragility, cost, and heater power required.

Miniature gas chromatographs previously manufactured using silicon technology have suffered from less than optimum mass transfer characteristics and consequent low performance because of the inherent rectangular or square cross section associated with the technique. To overcome this limitation, there are some people trying to isotropically etch silicon to produce more rounded micro machined capillaries. Issues remain with this approach.

We devised a process that overcomes the silicon manufacturing limitations and simultaneously leads to enhanced height equivalent theoretical plates/meter as a natural byproduct. The design employs a flat stainless steel plate with a chemically milled channel, a process that produces circular cross section channels with rough surface finishes, both desirable gas chromatograph performance features.

The gas chromatography devices were made using a 304L stainless steel substrate, with approximate dimensions of 100 mm x 100 mm, 3 mm thick. Using the chemical

milling technique, we machined a long (27 m) serpentine channel into the surface, 0.1 mm deep, on 0.2 mm centers, leaving 0.1 mm lands between channels. This technique involves designing a photoresistive coating that locally protects the surface, applying it to the steel, and chemically etching the unprotected regions. Other designs and manufacturing techniques were also completed. We built eight different prototypes using combinations of laser machining, single point turning, and chemical milling techniques. Of these total, we selected the most promising combination of high performance and ultimate low production cost approaches and fabricated two assemblies to test in a standard gas chromatograph oven.

Tests were limited to basic mass flow analysis runs using the bare column with no stationary phase. We used standard GC test materials, including methylene chloride and methane, to determine the fundamental flow properties of the column including average linear velocity. In our 13 m test column, we measured 390 mm/s average linear velocity with atmospheric pressure inlet and high vacuum outlet, close to the desired performance. More substantive tests of the GC performance were to have been conducted in the (canceled) subsequent year of the program.

Optrode

The objective of this segment of the LDRD project is to demonstrate the feasibility of micro machining basic elements of optrode on a silicon wafer. An optrode is a chemical sensor using an optical fiber. It utilizes an effect in which a reagent (e.g., pyridine), when exposed to certain chemical (such as certain chlorinated compounds), develops an intense red color. A typical optrode comprises an UV (or blue) light source for excitation, a porous tube filled with the reagent, a pump to flow the reagent, wavelength filters, and a detector. The excitation light (UV) and the signal light (red) are transmitted through optical fibers. The size of the whole system is substantial, typically over 50 cm in length and 15 cm in diameter.

Under this project, the porous tube was replaced by a V-groove channel micro machined into a U-shape configuration on a silicon wafer. Through-holes were prepared at the ends of the U-channel to connect to reagent source and drain. Also prepared were channels for input and output optical fibers. An aluminum block was machined to be provided with two reservoirs for the reagent liquid (one worked as the reagent source and the other as the drain), and a manual pump mechanism was installed to induce forced liquid flow. The V-groove was about 0.1 mm wide. The overall size of the micro machined part was about 2 cm by 4 cm. Liquid flow through the micro machined channel was successfully demonstrated using water as a liquid. Optical fibers were seated in the alignment grooves and light transmission from input fiber through the liquid then to output fiber was demonstrated.

Miniature capillary electrophoresis sensor

Capillary electrophoresis (CE) has been regarded by many in recent years as a major breakthrough in fluid phase separation science. The technique combines the strengths of both high performance liquid chromatography (HPLC) and conventional electrophoresis to yield rapid, precise, automated, and highly efficient analysis of complex chemical mixtures using minimal injected sample volumes (picoliter-nanoliter). Unlike most forms of high performance liquid chromatography that require non-aqueous solvents, CE is capable of operation in aqueous media, making it the ideal choice for trace analysis of inorganic ions, small organic molecules, organic acids, water soluble polymers and biomolecules (proteins, peptides, neurotransmitters, DNA etc.) Samples for analysis can be obtained directly in the fluid phase, or as extracts from solids or condensates.

High CE separation efficiencies result from the use of small separation channels or capillaries, 20-100 microns in diameter. Since the efficiency is independent of channel length, the entire approach is extremely amenable to micro fabrication and miniaturization. Direct application of this technique to rapid trace analysis and real time monitoring of waste water, leachates, and condensates from suspect facilities associated with nuclear proliferation activities should be possible using miniature field deployable, hand held sensors. Additional applications, include sensing and analysis of chemical and biological warfare agents and residues both in battlefield conditions and in facility inspection scenarios.

Recently, advances in CE miniaturization have resulted in the fabrication of entire CE systems including electrokinetic sample injectors on palm sized glass "chips". This type of planarized chip technology is ideal for interfacing with a new type of integrated optic detection system currently under development at LLNL for DOE-NN Advanced Concepts Program. Under LDRD funding, we have developed and tested a micro-fabrication strategy for electrokinetically injected planarized CE systems on advanced ceramic substrates. This is a significant result, since it allows CE system optimization by "fine tuning" of the physical properties of the chip substrate. As a result of the Joule heating accompanying electrophoresis, for example, thermal management is a crucial parameter in determining both efficiency and resolution in CE separations. Thermal conductivity of the CE chip substrate can easily be increased one to two orders of magnitude over conventional fused silica and glass based systems by careful selection of the CE chip substrate material used in microfabrication. New substrate materials also permit optimization of crucial solute/capillary wall interactions via choice of inherent substrate surface charge states. We manufactured two prototype ceramic (Aluminum oxide) capillary electrophoresis chips, designed to be integrated into the optical detection system described above. This device consists of a 15 mm square set of ceramic plates, sandwiched together to form a miniature assembly complete with reservoirs, 1.5 mm diameter optical windows, and two capillary electrophoresis channels 0.1 mm wide and a few millimeters long. Testing was limited to preliminary (successful) leak and hydrodynamics checks. Additional tests will be completed under advanced concepts funding from DOE-NN.