A MEMS-based Gas Chromatograph Front-end for a Miniature Spectrometer

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We have made all the key components of a miniature gas chromatograph: the pre-concentrators, different gas separation columns and detectors. All components have been designed for low temperature die-to-die assembly and connections to conventional GC tubing have also been included. The development is one step forwards to truly portable gas analysis instruments.

The miniaturization of gas chromatographs (GC) has many advantages, amongst which the reduction of power needed to heat the system and the reduction of the carrier gas consumption. We have developed a GC that will function as a front end for a mass spectrometer for space applications. The results of the project can also be used towards the portable gas analysis systems.

The schematic diagram of the GC is shown in Figure 1. The dashed structures in this figure are the MEMS components, the blue lines below a device indicate that it can be cooled, and the red ones, heated. The sample gas is drawn into the cooled pre-concentrator by a vacuum where it is adsorbed. Once sufficient material has been adsorbed, the sample gas and vacuum valves are closed and a carrier gas is led to the pre-concentrator and into the column. The temperature in the pre-concentrator is abruptly increased to obtain a sharp desorption peak of the sample material into the column, where it is separated. The separated sample is then analyzed by a Thermal Conductivity Detector (TCD) that compares its conductivity with that of a reference gas. The reference gas is the carrier gas whose flow has been reduced by a flow restrictor to the same level as the gas that elutes from the long gas column.

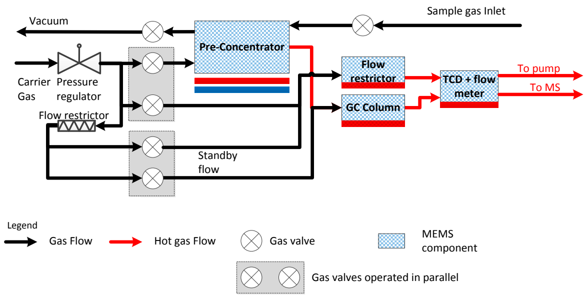


Figure 1: Schematic diagram of a gas chromatograph.

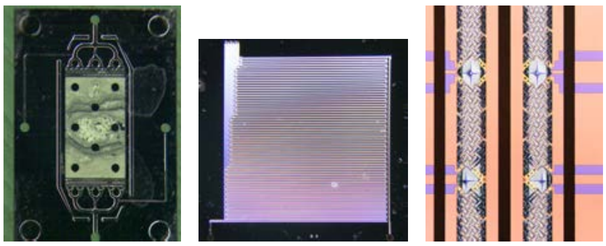
The pre-concentrator is a small cavity made of silicon and glass with metallic feedthroughs. The metallic feedthroughs connect to silicon pillars in the cavity which can be heated resistively to create a sharp desorption peak. The entire pre-concentrator will be mounted on a thermo-electric cooler to lower its temperature during the adsorption phase. The cavity of the pre-concentrator is filled with a commercial Tenax® absorbent.

The column consists of a silicon-glass sandwich, with a long serpentine channel dry etched into the silicon. The dimensions of the channel are 100 µm width, 250 µm depth and up to 4 m length, which is similar to the smallest commercial columns. The cross-section of the channel is rectangular, which presents a challenge for the deposition of the stationary phase. The thickness of stationary phase must be uniform, since thickness variations of the stationary phase result in variations of the time substances are retained. This retention time variation results in peak broadening, making it more difficult to separate different species passing through the column. Recently developed techniques like Atomic Layer Deposition (ALD) and Molecular Vapor Deposition (MVD) are very suitable for the deposition of uniform layers in structures with extreme aspect ratios and have been used to deposit both aluminum oxide and a silane monolayer on the surfaces of column channels.

The TCD consists of a total of ten resistors, suspended over two independent flow channels. This allows the TCD to measure differentially between a reference flow and a separated sample flow. Two resistors in each channel allow the connection of the resistors in a Wheatstone bridge. The three additional resistors in each channel allow the measurement of the flow using a differential heat measurement. Each resistor is defined in a platinum layer sandwiched between two isolating LPCVD silicon nitride layers.

An especially challenging task of the project has been the development of fluidic connections to the external world and between the different devices of the GC. The column can be heated up to 330°C, a temperature that the fluidic connections must be able to handle. At the same time, some of the column coatings cannot withstand temperatures over 250°C, limiting the bonding temperatures. The selected method has been the Au-Sn liquid phase soldering of miniature tubes to specially made gold coated silicon structures. Once this solder has molten at approximately 280°C, it absorbs the gold on the silicon structures, increasing its melting temperature to over 350°C. All silicon structures are gold coated and are connected using thermocompression bonding at temperatures under 250°C. The bonded gold is a noble metal that will not interact with the sample gases and the thermocompression bond yields a hermetically sealed connection between the two wafers.

All GC components have been successfully fabricated and are now being tested for their chemical performances prior to a complete assembly.



(a) (b) (c)

Figure 2: Pictures of the different components of the GC: (a) the pre-concentrator, (b) the column, and (c) the TCD.

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