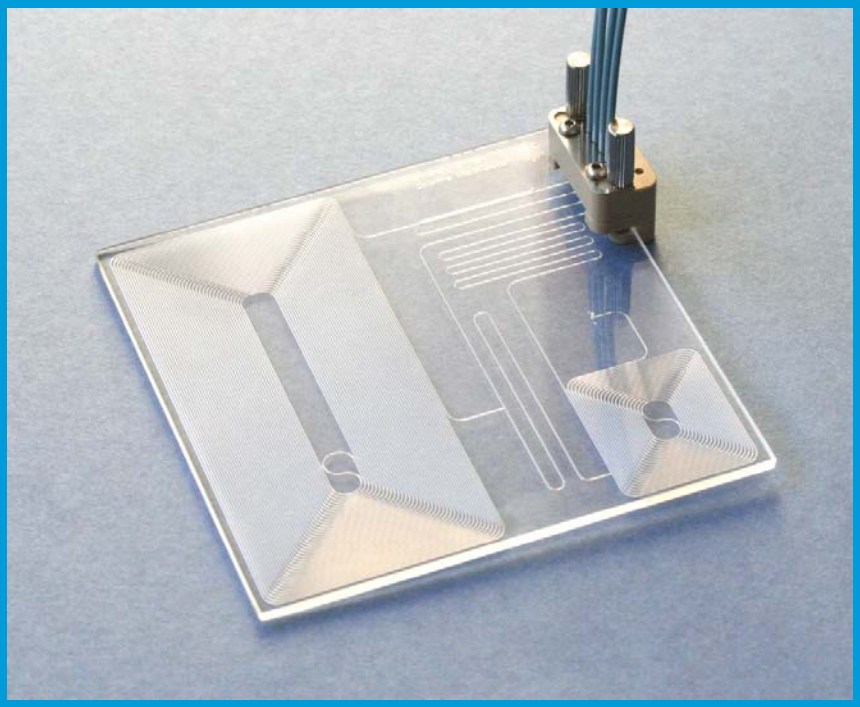
Miniaturisation of Gas Chromatography Equipment for Environmental Testing

The microfabrication of a glass Gas Chromatography chip



dolomite

About Gas Chromatography (GC)

Gas Chromatography (GC) is a highly sensitive chemical analysis technique with a broad range of applications. Existing commercial GC systems are generally quite bulky and fragile. A key part of a GC system is the GC column. In operation, samples are passed through the GC column, where they separate out into chemical components. The separated components then pass through a detector, and a chromatogram is produced which is used to identify the various chemical components. A typical GC column consists of a capillary wound onto a spindle which is heated in a turbulent fan oven. The GC column is normally coated with a 'stationary phase' to aid separation.

For environmental testing, in particular atmospheric monitoring, there is a requirement for portable, robust, low power gas chromatography systems. Microfluidics enables miniaturisation of the gas chromatography column and low power methods for column heating.

Microfabrication of a glass Gas Chromatography chip

The fabrication of the Dolomite GC chip involved wet (isotropic) etching of two glass wafers. This resulted in open channels in each layer with semicircular cross section. Holes were then drilled in one of the wafers for fluid access. The wafers were then diffusion bonded together (without the use of adhesives). Following the diffusion bonding process, the thickness of the base glass layer was reduced to 300µm, to improve the rate of heat transfer to the channels. Figure 1 below shows the chip with a Dolomite Mitos edge connector for making a connection to PEEK fluid pipes.

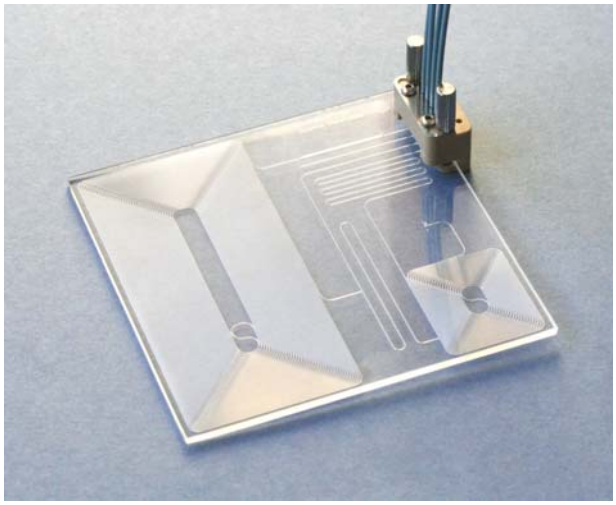


Figure 1 - 100mm x 100mm glass Gas Chromatography chip with Mitos fluid connector

The chip design included an injection zone along with 7.5 meter and 1.4 meter long channels with 320µm internal diameter. These channels replace the wound capillary that is used for existing GC columns. Dolomite successfully overcame the challenge in the photolithography and etching processes, which required the chip footprint area of 100mm x 100mm area to be manufactured without any defects between channels.

Earlier microfluidic GC chips were fabricated in Silicon with a square channel cross-section. These channels suffer from problems with uneven application of the coating. The Dolomite chip has a near circular cross section (Figure 2) with accurate alignment of the two chip layers. This allows the stationary phase to be applied evenly to the inside surface of the channels.

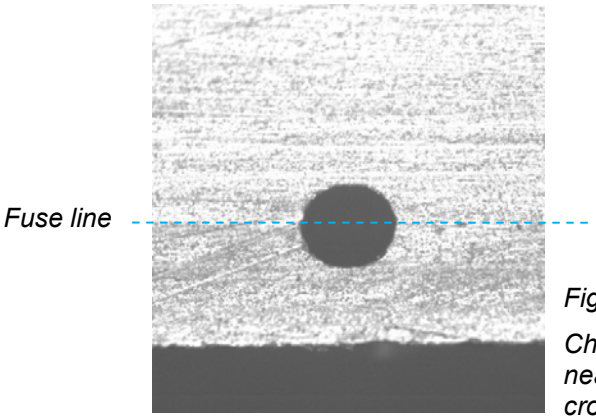


Figure 2

Chip sectioned to show near-circular channel cross section (320µm diameter)

Figure 3 below shows a constriction etched into in the channel. This constriction allowed activated carbon particles to be loaded in the area shown in Figure 4, and retained by the constriction, forming a sample absorption column in the injection zone of the chip.

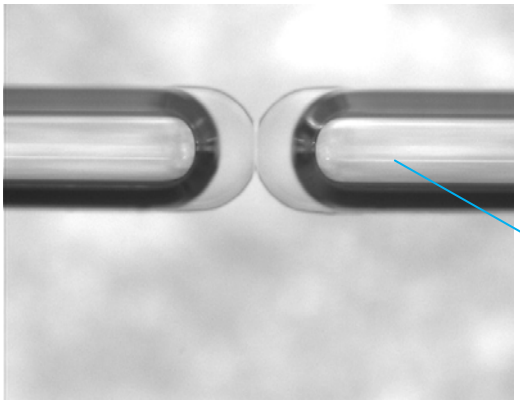


Figure 3 - 320µm diameter channel with 50µm constriction

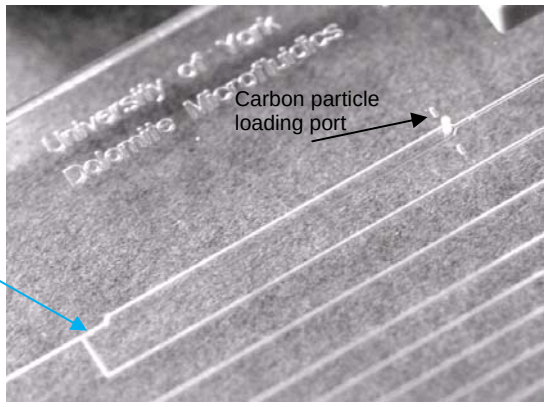


Figure 4 - Sample absorption column area

Gas Chromatography Analysis

Gas Chromatography requires independent heating and cooling of the two on-chip columns and the injection zone. Direct column heating and cooling was achieved using a combination of resistive heaters and Peltier devices. The low thermal conductivity of glass allowed for multiple uniform temperature zones within a single glass chip. Temperature control over the range 10–200°C was achieved. Peak power demand was approximately 25W, two orders of magnitude less than a conventional turbulent fan oven. Figure 5 shows the temperature profile under the 300µm thick chip layer:

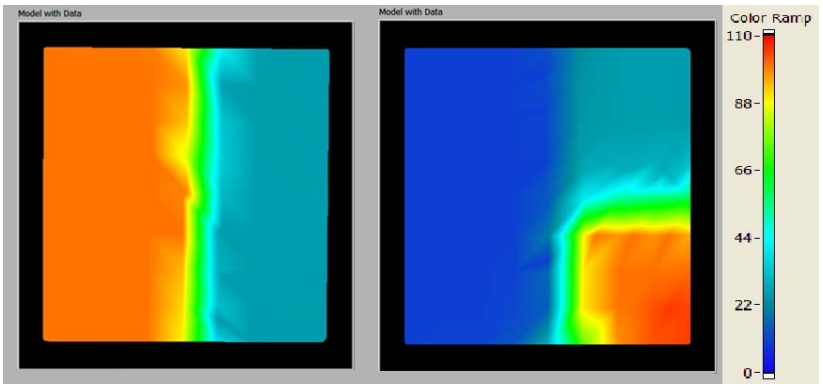


Figure 5 - Measured temperature profile (on left) with the 7.5m column heated to 100°C, all other areas left at ambient. Measured temperature profile (on right) with the 7.5m column cooled to 10ºC whilst holding the temperature of the 1.4m column at 100◦C. The temperature remained uniform (±2°C) over the 7.5m column and the unheated area remained at ambient temperature.

For detection of the separated components a standard FID (Flame Ionization Detector) and a lightweight 100mW PID (Photoionization Detector) were coupled to the column. Column performance was tested with gas mixtures of monoaromatic and monoterpene species at the parts per million (ppm) concentration level. The low power GC-PID device showed good performance for a small set of VOCs (Volatile Organic Compounds) and sub ng detection sensitivity to monoaromatics. Figure 6 shows a chromatogram of the separated components of the gas mixture.

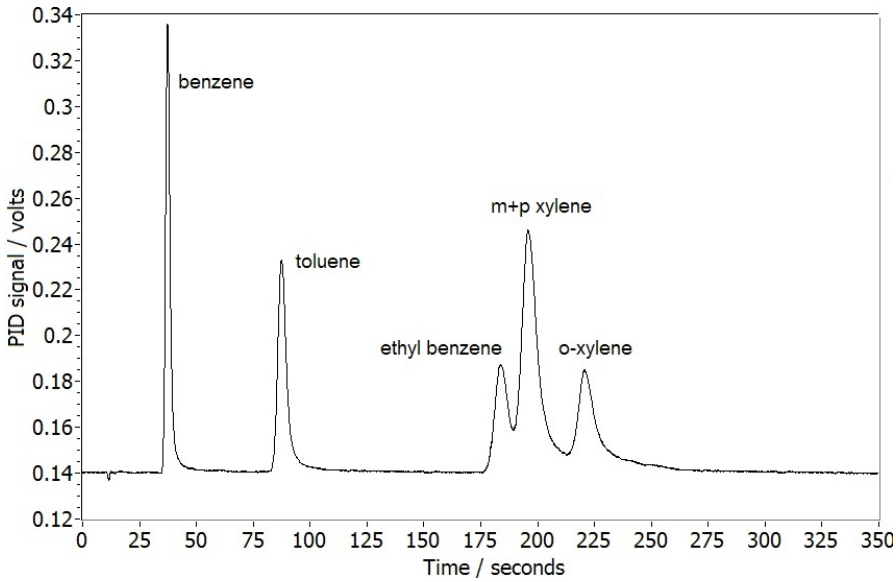


Figure 6 - Chromatogram showing separation of the 10ppm BTEX standard gas mixture. 0.5mL sample loop in combination with the planar glass 7.5m GC column programmed from 10 to 100◦C at 20◦C/min heated using Peltier and resistive elements. Detection is by low power photoionization detector.

Conclusion

The microfluidic glass GC device with a low power photoionization detector offers substantial potential as a field portable GC instrument and is a useful alternative to typically square channelled silicon devices of restricted physical size and higher material cost. The larger glass wafer areas that may be micro-manufactured at reasonable costs (when compared to silicon) offers the potential for planar devices with column dimensions analogous to those used in laboratory GC instruments. In common with other planar GC approaches, the ability to directly cool this device using the Peltier effect may offer substantial advantages for the analysis of very volatile species over typical cryogenic cooling of drawn capillaries in standard GC ovens.

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