Flexible Microchannel Development and Fabrication for GC and LC

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Introduction

In capillary analytical techniques there has long been a realization that microchannel devices would offer many advantages over tubing based systems but there have been a number of challengers in design and fabrication techniques.

Microchannel devices with precise control of shapes have been produced in silicon, polymers and glasses. Although precision even nanoscale components can be produced, they each have limitations around difficulty of making good connections, temperature limitations and pressure restrictions that limits their application to chromatography techniques.

The issues we were conscious of and were part of our development objectives were:

1. Precise and reliable channel fabrication at micron tolerances
2. Low volume and robust connection system
3. Deactivated sample flow path
4. High temperature capability
5. High pressure capability.

Fabrication Process

A raft of techniques were developed for constructing a series of stainless steel layers with the channels and other features precisely formed into the stainless steel using either laser or chemical milling techniques. Good dimensional control is achieved in the channel and hole sizes down to the order of 25 microns are possible. When the layers are bonded together they must be precisely aligned to achieve correct register of the different layers and through holes from one layer to the next. The multilayer capability permits very complex flow systems to be fabricated as well as long columns up to 1 metre being feasible. Columns long enough for capillary GC columns is not yet practical or probably desirable but LC columns are feasible. Figure 1 shows the construction of a wafer based LC column.

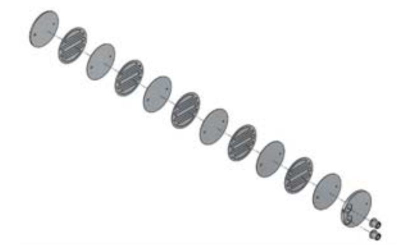

Figure 1. Multilayer wafer column. Column length 1 meter demonstrating the construction of the wafers.

Size of Features that can be Produced

The smallest channels that have been produced to date that have maintained their integrity and channel surface finish are 25 µm X 50 µm. The largest dimensions that have been worked with are in the order of 800 µm X 500 µm features.

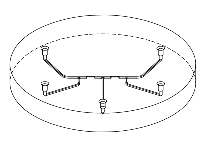

Figure 2. The configuration of a bonded wafer with 5 external connection ports and the internal layout of channels including some restrictors.

Connection System

There are two configurations of wafers developed to enable connecting flow lines to external components in a GC or LC system.

- The wafer itself is a simple disk that is installed in a holder to which the external tubing connections are made. The holder can be mounted onto the wall of the GC oven. This configuration is intended where different configuration wafers can be quickly and easily swapped for different applications. Also, if the wafer becomes damaged with use a new wafer can be installed quickly. See Figure 3 a) and b).
- The connections with the external tubing and columns etc, are made directly where threaded bosses are incorporated into the wafer. See Figure 4.

a) b)

Figure 3. Wafer holder

Figure 4. Connections incorporated onto wafer

Conventional graphite or vespel ferrules cannot be used with the system as the dimensional stability is not adequate and there is also a risk of particles from the graphite or vespel ferrule contaminating the channels in the wafer. SilTite™ metal ferrules must be used for this application. A further development of the SilTite™ metal ferrules is the FingerTight version which was designed to prevent damage to the wafer due to excessive tightening with a spanner. The SilTite™ FingerTite metal ferrule relies on a thin cross section in the area that collapses down onto the fused silica tubing.

A special tool requiring only finger force is used to tighten the ferrules. Finger force is sufficient to deform the metal ferrule onto the fused silica tubing for a reliable seal adequate for MS applications and it does not need retightening after temperature cycling.

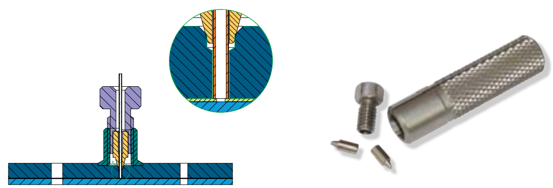

Figure 5. Fused silica to wafer connection system using SilTite™ FingerTite metal ferrule.

Multidimensional GC Applications

The first application of the wafer devices has been in multidimensional GC techniques (see Figure 6).

Common terminology in multidimensional GC refers to restrictors in the system but it is more useful to think of them as fluidic logic gates or flow directors. An orifice in the flow path ensures a high gas velocity, high enough so that mixing or diffusion of gas against the flow cannot occur. The ideal fluidic gate has no significant restriction and next to no pressure drop across it. The fabrication of microchannels allows the fabrication of perfect fluidic gates that may only be 0.5 mm long. Figure 7 shows channels fabricated into wafers with an orifice section at 50 micron and 500 micron long. These are the dimensions we have found to be the most effective in GC column switching.

Figure 6. The simplest application of the wafers is for complex connection systems requiring perfect geometry and zero dead volume. Shown are a 4 way splitter, a 3 way splitter and GC multidimensional Deans switch.

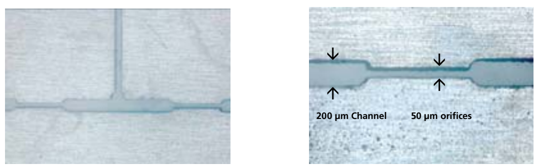

Figure 7. Machined channel orifices in stainless steel layer

Channel Inertness and Peak shape (dead volume)

Stainless steel surfaces are far too active to be used in GC sample flowpaths and there are also some application areas in LC where it is problematic. Even lengths as short as 1 mm have been observed to destroy the inertness integrity of a GC system. The stainless steel must be effectively deactivated.

New surface treatment techniques have been developed for coating and deactivating the stainless steel channels. Figure 8 shows the performance of the system with and without the microchannel wafer installed. The test applied is a standard inertness and efficiency set of test probes, chromatographed under isothermal conditions with sufficient residence time and low enough concentration of the components to induce tailing of the n-Decylamine even on the most inert of chromatography columns and systems.

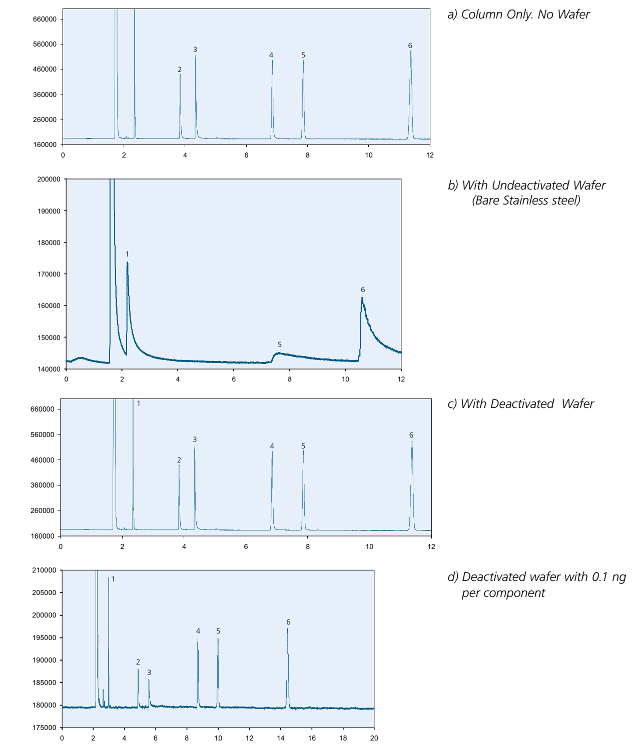

Figure 8.
Column: BPX5 30 m x 0.25 mm ID x 0.25 µm
Column Temp.: 140 °C FID Temp.: 380 °C Injector Temp. 240 °C
Sample: 1 ng on-column of test mix (1) n-Decane (2) 4-chlorophenol (3) n-Decylamine (4) Undecanol (5) Biphenyl (6) Pentadecane

Pressure Capability - Wafer and connection tested successfully at 50,000 psi
Thermal Lag - None detected at 20 °C/min program rate
Maximum Temperature - Defined by the fused silica tubing - 400 °C

Conclusion

The construction of the stainless steel microchannel wafers permits many different channel configurations including different cross section channels and may be readily customized with precisely defined channel dimensions that can be varied from 800 microns down to 25 microns on the same device. The fabrication technique is amenable to multiple layers permitting complex and long flow paths.

With correctly deactivated channels the microchannel wafers have inertness similar to deactivated fused silica and the precise channel geometries and dimensions enable no detectable peak distortion. In addition to GC applications the wafers have also been used in LC applications where the high pressure rating in excess of 50,000psi permits their use in UHPLC applications. The fabrication of these devices is a capability that will change the way we design many components in GC and LC.

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