Reversible Concentric Ring Microfluidic Interconnects

by

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ABSTRACT

A reversible, Chip-to-Chip microfluidic interconnect was designed for use in high temperature, high pressure applications such as chemical microreactor systems. The interconnect uses two sets of concentric, interlocking rings with trapezoidal cross sections to form a fluid seal without the use of gaskets or sealants. Results from analytical and finite element models indicate good sealing capability. Macro scale devices show that the interconnects function as designed and seal up to 80 psi. Micro scale devices have been fabricated, engage as designed and are much stronger than anticipated, however pressure tests have not yet been conducted.

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Chapter 1: Introduction

The benefits of micro scale biological and chemical applications are well understood. Micro Electrical Mechanical Systems (MEMS) use small sample and reagent volumes, reducing cost and waste, increasing reaction yield and reducing the danger of spilled or leaking reactants. They have high mass and heat transfer rates, high surface to volume ratios and operate in the laminar flow regime. They are more efficient, using less space and power.¹ MEMS devices can also be inexpensive, disposable and modular.

However, MEMS devices are also very small and very fragile. They require electrical and fluidic connections to bring power and materials to the chip and a super-structure to prevent them from being damaged in day to day use. Packaging is “one of the most costly and least developed aspects of a micro system”² and this is especially true for microfluidic packaging and the development of interconnects and micro scale fluidic seals. “Interconnections and feedthroughs are often ignored when a packaging technology is developed for MEMS, but usually end up being one of the most important aspects of the package because they are either very large, or cause device failures”³ negating some of the benefits of working on the micro scale.

Although a number of sealing methods have been proposed, none have been sufficiently robust and elegant to emerge as the defacto industry standard. Many rely on brute force or large quantities of sealants, such as epoxy, to create the fluid seals, risking damaging the device during assembly or contaminating the device during operation. To address some of these issues, a static, reversible, high temperature, micro machined, microfluidic interconnect is proposed.

1.1 Failure Modes of Fluid Seals

Fluid seals are a physical barrier placed in a potential leakage path to prevent fluid from exiting the system at a mechanical boundary or interface. There is no such thing as a perfect seal; every seal has some leakage rate. The goal of seal design is to minimize this leakage rate. In general, there are three ways that fluid seals can leak: they can flow around the barrier if the leakage path has not been completely blocked, they can flow through the barrier by diffusion, or they can remove the barrier either by mechanical force or chemical erosion.
1.1.1 Leakage Path

There is also no such thing as a perfect surface. Every surface has some roughness such as scratches, dents and wear marks. When two imperfect surfaces are pressed together, fluid can flow through the interstitial spaces created where material from the first surface does not fill an indentation in the other. Many sealing methods focus on finding some way to fill these gaps and prevent the fluid from leaving the system.

1.1.2 Chemical Diffusion

No matter how well the interstitial spaces are sealed, fluids (especially gases) can still diffuse through some seals. For example, while helium cannot diffuse through stainless steels, the diffusivity of helium through Pyrex glass is very high ($2 \times 10^{-8}$ cm$^2$/s). Seals and sealing methods must employ materials that are impermeable to the fluids used in the system.

1.1.3 Chemical Corrosion

Corrosion of the seal material by strong chemicals can effectively remove the physical barrier and allow fluid to flow out. Seals and sealing methods must employ materials that are chemically resistant and compatible with the fluids in the system.

1.2 Choosing a Sealing Method

Although the sealing method is determined by the application, all fluid seals share a basic set of functional requirements. Each seal must function for a specified temperature range, pressure range and maximum acceptable leakage rate. The seal should minimize dead space and pressure drops. It should be robust, chemically inert, thermally isolated (if necessary), and offer minimal disruption to the overall design of the fluid system. While these requirements are applicable to all fluid seals, the issues below are particularly applicable for micro scale devices.

1.2.1 Macro to Micro vs. Micro to Micro

*Will the connect be from the macro scale to the micro scale or from the micro scale to the micro scale?*

All microfluidic devices need to have fluids delivered into and out of the chip. The fluids are transported from a macro scale fluid reservoir to the micro scale chip. At the interface between the tubing carrying the fluid and the surface of the chip, a fluid connection needs to be made and a fluid seal created. This will be designated a “macro to micro scale connection.” Examples of these types of connections make up most of the microfluidic packaging literature and will be discussed further in Chapter 2.
In MEMS devices, inlet tubes are either directly connected to the device substrate or they interface with an intermediate device that is directly connected to the substrate. We will not distinguish between seals created to prevent leakage at the surface of the substrate or seals created to prevent leakage at the tube connection as they all serve the purpose of preventing leakage in the system.

However, the integration of multiple microfluidic components on one chip can be very difficult and expensive and a great deal of research has been focused on the creation of microfluidic systems and microfluidic breadboards to allow micro scale devices to be stacked together or linked to allow more complicated interactions to take place. These types of systems involve creating fluid interfaces between two chips. Instead of transporting fluid from the macro scale world to a chip, a micro scale volume of fluid is being transported from one chip interface to the other. This is designated a “micro to micro scale connection.” Examples of these connections are often found in conjunction with multiple chip microfluidic systems and will be discussed in Chapter 3.

1.2.2 Permanent vs. Demountable Seals

Will the seal at any interface will need to be broken at any time, and if so, how often?

Permanent seals tend to be stronger and more reliable, can offer hermetic sealing (zero leakage and protection against diffusion) and better temperature and chemical resistance. However, they do not allow the seal to be broken and the device(s) to be taken apart easily, if at all.

If the seals need to be dismantled for cleaning, repairs or to swap out one component in a microfluidic system for another and then easily reassembled, a reversible or “demountable” seal must be used. Demountable seals are more vulnerable to fatigue and creep than permanent seals.

The leakage rate and pull-out force are also strong functions of the type of seal. Every type of seal that is not hermetic will have some leakage rate. The flexibility of a demountable seal must be weighed against the maximum allowable leakage rate in the system. If very hazardous materials are being used in the device, a very small or zero leakage rate may be required leading to the choice of a permanent seal. However, a less hazardous system may be able to tolerate a much larger margin for error. In general, the lower the leakage rate, the more expensive the seal.

Pull-out force is the amount of force that is required to break a seal or to pull a fluid tube out of its connection. In general, permanent seals tend to be stronger and have much higher pull-out forces than demountable seals which makes this type of seal attractive for applications where the device is subject to rough handling or conditions. However, for systems where components will be replaced regularly (like microfluidic systems that use disposable chips for medical tests), it is desirable for the pull-out for to be small enough that none of the components or technicians are damaged during the separation process but not so small that the components can separate accidentally.
1.2.3 Disposable vs. Reusable

*Will the microfluidic chip be reusable or disposable?*

Permanent seals tend to be more expensive in terms of skills and resources required to make them and thus are typically not cost effective for disposable systems. In disposable systems, the major driving factors are the cost of the chip (and thus the connection) and the speed and ease with which the seal can be made and broken.

Reusable systems, however, can have equally good reasons for needing permanent and demountable seals. For example, the interface for a disposable chip may need the strength and stability of a permanent seal, however it still requires a set of demountable interfaces where it connects to a disposable part of the system. Dead volume becomes a major issue in reusable systems because any fluid trapped inside the device after use can contaminate future samples. Microfluidic devices can be difficult to clean and minimizing the frequency and extent of cleaning is important. Large dead volumes also increase the operating cost of the system and can impede performance.

1.2.4 Interconnect Size and Geometry

*What are the shape and size requirements of the interconnect?*

Microfluidic devices use extremely small quantities of fluid compared to their macro scale counterparts and need relatively low pressure gradients to drive the flow. If the interconnect is significantly larger than the average flow passage, the pressure drop associated with the transition from the macro to the micro scale can be the largest drop in the system and may lead to reduced device performance. It is often easier and cheaper to create a relatively large interconnect with a large pressure drop and large dead volume, but the decreased performance may not be acceptable.

1.2.5 Choosing Materials

*What is the maximum operating temperature and pressure of the system? What are the chemical and biological compatibility requirements?*

The physical properties and conditions of the microfluidic system will determine the materials used in the fluidic connection. All materials have a maximum operating temperature, above which they begin to soften or flow and the fluid devices should be operated well below the maximum operating temperature of the materials used if possible. The material properties will determine how strong (and compressible) the sealing components are and the coefficient of friction between the sealing surfaces. At high pressures, the force on the sealing components may be enough to force the sealing members apart or to overcome the frictional forces keeping them together. Finally, every material reacts with its environment under certain conditions. It is important to ensure that
Choosing Components

the fluids in the system will not corrode or damage the fluid seals and that the materials in the fluid seals will not harm or affect the fluids in the system. For this reason, chemical and biological compatibility are important when choosing materials.

Plastics tend to be inexpensive, have relatively low operating temperatures, moderate to good mechanical strength, poor to moderate chemical resistance and very good biological compatibility. Metals tend to more expensive, have relatively high operating temperatures, good to excellent mechanical strength, moderate to good chemical resistance and moderate biological compatibility. Glass, silicon and ceramics can be very expensive to use in manufacturing, tend to have high operating temperatures, good mechanical strength, excellent chemical resistance and good biological compatibility. They are, however, brittle materials and devices made with them can be very fragile.

1.2.6 Choosing Components

Finally, one must choose whether to use commercially available components to create microfluidic interconnections and seals or whether to design and fabricate custom connections in the substrate of the microfluidic chip. Commercially available components tend to be inexpensive, reliable and easy to use. They are especially good for making macro to micro scale connections and can be combined with custom components if necessary. However, most commercial products are still made on the macro or meso scale (because manufacturing is still easier and cheaper on the macro scale) and few or none exist to make micro to micro scale connections. The MEMS market also is still in its infancy and relatively few products are available for use.

In general, commercially available products are used whenever possible and custom interfaces are designed only when necessary.

References


Chapter 2: Prior Art: Single Chip-to-Chip-to-World Fluid Interconnects

According to Roth, there are five primary classifications of sealing techniques: “permanent seals (welded and brazed metal joints, glass-to-glass, glass-to-metal, and ceramic-to-metal seals), demountable seals (wax and resins, ground, liquid and gasket seals), electrical lead-throughs, seals for motion transmission and seals for transfer of materials (cut-offs, valves, vacuum locks).” Electrical interconnects are often made using mechanisms separate from those used in the fluid connections. And microfluidic interconnects rarely have the need to serve as a means of motion transmission or valves. Examples of these sealing types are rarely (if ever) found in the microfluidic literature. For this reason we will focus only on permanent and demountable seals.

The goal of this section is to introduce the definition, principles, benefits and disadvantages of various permanent and demountable seal types and to discuss examples of these seals found in the microfluidic interconnect literature. This chapter will focus on devices for single Chip-to-World (macro to micro scale) fluidic connections. Arrays of Chip-to-WorldChip-to-Chip (macro to micro scale) and Chip-to-Chip (micro to micro scale) fluid connections for use with microfluidic systems and breadboards will be discussed in the next chapter.

2.1 Permanent Seals

2.1.1 Welds and Welding

Welds are created by locally heating two metal components until they melt and then allowing the metal to join as a single component as it cools. Proper welds are very strong, can create hermetic seals and can stand temperatures up to the melting point of the metals involved.

For MEMS applications, stainless steel is the material of choice when welding because of its high melting point (~ 1400 °C), superior mechanical strength and excellent resistance to chemical corrosion (the notable exceptions being very strong concentrations of sulfate (SO₄²⁻), very low pH, and very high concentrations of chlorides and chlorine)². The weld bond is very strong and when used in conjunction with a stainless steel housing, can greatly reduce the probability of breaking the fragile MEMS devices or snapping off the fluid connections.
However, welding also has some disadvantages. The strength of the weld and the leakage rate are determined by the quality of the weld and requires great skill to reliably reliably. Also, welding technology has traditionally been used on the macro scale and welding, like many other macro scale manufacturing techniques, become much more difficult as size decreases. Finally, microfabrication of stainless steel is more difficult than silicon. All of these factors combine to make stainless steel devices significantly more expensive than their silicon counterparts.

2.1.1.1 Examples of Welded Seals Commercially Available for MEMS devices

A good example of welded seals in commercially available microreactor technology comes from the Institut fur Mikrotechnik Mainz. The Catalyst Testing Micro Reactor (CTMR) uses welding for the fluid inlets and outlet as well as to combine the individual elements of the devices. Figure 2.1 shows one individual reaction plate (left) and 50 reaction plates and fluid interconnects welded together (right). The CTMR was tested up to 5 bar (500 kPa) and 500°C.

Figure 2-1: IMM Catalyst Testing Micro Reactor

2.1.1.2 Examples of Welded Seals in MEMS Research

Two examples of welded seals in MEMS Research both involve the attachment of commercially available fluid interconnects to a stainless steel housing. The first example is from the Forschungszentrum Karlsruhe, where a microreaction system composed of micromixers, microreactors and micro heat exchangers was created. "The reactor is assembled from individual stainless steel plates...diffusion bonded together, resulting in an essentially monolithic stainless steel assembly whose channels are vacuum tight and can also handle high pressures. A stainless steel housing with standard tube connections is welded onto the assembly to complete the reactor." Figure 2.2 (left) shows the reactor assembly in the lower left hand corner and the fully assembled device on the right hand side. In this case, each connection is about twice the size of the entire microreactor.
The second example is from Karlsruhe and Universitat Erlangen involving a microreactor build for rapid, periodic temperature changes. Again the reactor was built up from microstructured stainless steel foils. “All parts of the device are stacked and then connected either by diffusion bonding or electron beam welding. The reaction passage and the cooling passage are welded to fittings with small stainless steel adapters.” See figure 2.2 (right). Both devices operated at temperatures below 300°C.

**Figure 2-2: Welded Research Micro Reactors**

### 2.1.2 Brazed (Soldered) Seals

Brazing or soldering is the process of using a metal with a relatively low melting point to join two other metals. Lead solder is the most common material to use because it is inexpensive, easily obtainable and has a low melting point. For MEMS applications, gold is also very common material to use for brazing because of its low melting point and good biocompatibility.

Braised joints are relatively easy to make and seal very well because the metal will flow between the mating surfaces and fill in all available gaps. They can withstand high pressures, although the mechanical strength of the brazed bond is lower than a welded bond. And brazed joints, unlike welds, can be disassembled if necessary although they cannot be strictly considered demountable.

The primary disadvantages of brazed joints include a relatively low maximum operational temperature (just below the melting point of the solder), insufficient chemical compatibility and resistance, and a potentially more complicated microfabrication process to deposit metals that the solder will adhere to. It is also important to cool the soldered device very slowly to prevent cracking from mismatched thermal expansion coefficients.

#### 2.1.2.1 Examples of Soldered Seals in MEMS Research

Enikov and Boyd demonstrated the use of soldered gold on chemical sensors for both fluidic and electrical connections. To create the fluid seal, windows were opening in the tin layer of the device and 8 µm of gold were electroplated on the surface of the bottom
Adhesive Encapsulation

After fabrication was complete, the bottom chip was paired with a silicon wafer capped with a pyrex wafer that defined the fluid channel and heated to 372° C to create a gold-silicon eutectic bond. The electrical connections used gold-tin eutectic bonds. The device can be seen in figure 2.3. In testing, the strength of the fluid seal exceeded the strength of the silicon.

Figure 2-3: Schematic of Device with Soldered Fluid Seals

2.1.3 Adhesive Encapsulation

Working with metals for welding and brazing can be difficult, expensive and dangerous because of the very high temperatures required to make the metal flow. The use of adhesives like PDMS and epoxy can be used as “brazing” materials to completely encapsulate the fluidic connection. Adhesives are workable at room temperature and are relatively inexpensive and easy to obtain, which makes them both attractive and widely used.

The biggest disadvantage is that adhesives are more susceptible to harsh chemicals than metals which makes adhesives unacceptable for many microreactors and micro reaction systems. They also have lower maximum operating temperatures than welding and brazing, and are more likely to contaminate the MEMS device.

2.1.3.1 PDMS Encapsulation

An example of irreversible PDMS encapsulation comes from the University of Glasglow. During the construction of the substrate, posts were placed on the device over the inlets of the silicon part of the device before a thin PDMS layer was poured over top. These posts were removed after curing and plastic tubes, syringes and pipettes were inserted in their place. A thicker layer of PDMS was then poured over the surface of the device and cured, permanently sealing the connection to the macro world. In this case, the PDMS served
both as a sealing mechanism as well as a means to make the entire device more robust and less prone to failure. This sealing method has good biocompatibility and very low dead volume, but lower mechanical strength than other methods. No information was given for performance or pressure rating of the seal.

Figure 2-4: Micro array Platform with PDMS Fluid Seals

2.1.3.2 Plastic Encapsulation

Gartner et al. demonstrate the use of injection molding the encapsulate standard fluidic connectors for biomedical applications. In this case, a clinical diagnostic chip was created using hot embossing and assembled with an injection molded cover plate with “the male Luer Lok fittings directly molded onto the plate.” No picture or data on the performance of the seal is available.

Puntambekar and Ahn at the University of Cincinnati proposed fluid interconnects made from flanging thermoplastic tubing (PEEK or teflon). The application of pressure and/or heat causes the tubing to flow and fill a cavity in the microfluidic device, creating a leak tight seal. After the interconnect cools, a second piece of tubing can be press fit into the flanged tubing to connect the microfluidic device to the macro scale world. The interconnects sealed up to a pressure of 30 psi (200 Pa) and had a pull out force between 15 and 40 N depending on fabrication method.
Gonzalez, Collins and Smith at the University of California-Davis demonstrated a tubing interconnect that press fit plastic tubing over a micro fabricated hexagonal silicon tube. The silicon tubes were created by etching two silicon wafers using a KOH solution and then fusion bonding them together. Because the outer diameter of the bonded silicon tube was 700 um and the plastic tubing had an inner diameter of 762 um, “heat was applied to flow the plastic tubing over the silicon tube, sealing the connection.”

The primary advantages of epoxy, as stated above, are that it is inexpensive, easy to obtain, easy to use, relatively strong and creates a relatively reliable seal. It requires no special equipment and in most cases, no modification to the device design and can be used to reinforce other sealing methods.

While epoxy and other adhesives can be removed using trichlorethylene or similar chemicals, for the most part they can be considered permanent seals. However, epoxy, more than any of the other adhesives, causes problems with contamination. During the manufacturing process, low viscosity epoxy can flow through the small spaces that it is
supposed to fill and seal and enter the MEMS device instead of stopping at the end of the interface. The use of epoxy can also be an inelegant method of sealing. Large globs of epoxy increase the footprint of the connection and can also be difficult to assemble if alignment methods were not micromachined into the substrate during fabrication.

The combined advantages and disadvantages of epoxy as a sealing method make it ideal for research applications and undesirable for commercial applications. For this reason, there are no examples of epoxy encapsulation as primary or secondary seals in commercial MEMS devices. For research applications, epoxy encapsulation is the most prevalent sealing method cited in the literature.

2.1.3.3.1 Examples of Epoxy Encapsulation in MEMS Research

A common method for macro-to-micro scale fluidic interconnects is to use commercially available ferrules as an interface between macro scale tubing and the micro scale devices. The ferrule provides strength to the connection as well as an increased surface area for contact with the microfluidic chip and the epoxy wetting surface. De Mas from MIT, Edwards et al. from Louisiana Tech and Saldanha from Louisiana Tech all demonstrated the use of this sealing method in their work.

Jackman et al. at MIT used alignment holes punched into the PDMS layer of a microreactor with integrated MIR infrared spectroscopy to hold inlet and outlet capillary tubes in place until an epoxy layer could create a fluid seal and structural support.12 No information is given for the maximum pressure of the system, but it is assumed that the PDMS, and not the epoxy seal, would be the limiting factor.

Gray et al. suggest creating two types of microfluidic interconnects by epoxying glass capillary tubes into holes in the substrate that were created using DRIE. The first relies on “matching the inside and outside diameter of the capillaries, thus eliminating any dead space.” The second type of coupler uses a silicon “sleeve around the etched bore” which “acts as a barrier to adhesive and enhances the mechanical integrity of the coupling”13 although it introduces some dead volume. Both couplers were demonstrated to hold at pressures of up to 500 psi (3.4 MPa).
Meng, Wu and Tai have focused on creating self-aligning micromachined fluidic couplers that “serve as a convenient intermediary between external fluidic plumbing and the inlet/outlet ports of a fluidic device”\(^1\). Their design uses a fitting which has either been micromachined or molded from a micromachined mold. A capillary tube is inserted (press fit or molded) into the fitting, forming a macro to micro scale fluid seal. The fitting mimics the geometry of the fluid port on the microfluidic device (usually a KOH pyramidal pit or a DRIE circular hole), making the fittings self-aligning, with low dead volume. The micro to micro scale fluid seal is created using an adhesive material like epoxy. The matched geometry between the fluid ports and the fitting geometries ensures that “only a minimal amount of the adhesive material is wetting by fluid flowing through.” These devices are reported to seal against at least 1200 psi (8.2 MPa).

Pattekar and Kothare introduced a microfluidic interconnect based on “localized high-temperature deformation” of teflon capillary tubes to “obtain in situ temporary sealing.”\(^1\)\(^5\) In this example, a teflon capillary tube is inserted into a round hole in a pyrex wafer that has been bonded to a silicon wafer until it touches the silicon surface. The wafer is then placed on a hot plate and the end of the teflon tube is heated until it deforms into a dome shape. The capillary tube is then pulled up and the deformed end forms a weak fluid seal. Finally, this seal is reinforced by coating the capillary connection in epoxy. The resulting
Adhesive Encapsulation

seal has low dead volume, little risk of epoxy leaking into the microfluidic device and very
good mechanical strength. The connection seals for pressures up to 315 psi (2.2 MPa) and
temperatures up to 275°C.

![Figure 2-10: Thermally Deformed Microfluidic Interconnects](image)

Finally, Tsai and Lin suggest using an integrated sealing polymer layer (Mylar) with
epoxy based fluidic seals. In this case, a capillary tube is punched through a small piece
of Mylar film and the inserted into a fluid port in a microfluidic device, ensuring that the
mylar is in contact with the device surface. The device is then heated to allow the mylar to
soften and create a weak fluid seal. Finally, a layer of epoxy is used to cover the
connection to add mechanical strength. An integrated process was also presented which
involved depositing and patterning mylar on the wafer surface. These fluid connections
have a low dead volume and minimize the risk of epoxy entering the microfluidic device.
They can be used up to a temperature of 180°C and a pressure of 190 kPa without
leakage.

![Figure 2-11: Integrated Mylar Microfluidic Interconnect](image)
2.1.3.4 Double Sided Tape as Adhesive Encapsulation

Most of the innovation in using epoxy encapsulation for the creation of microfluidic seals has been focused on how to align the mating parts and hold them in place during the adhesive application and curing, and methods to prevent the adhesive from entering the microfluidic device. In many cases, this required creating a weak fluid seal before applying the adhesive to the connection.

The use of double sided tape to form micro to micro scale fluidic connections has a number of advantages over epoxy. Double sided tape is cheap and widely available. The clamping force of double sided tape is very strong, so parts won’t move once they have been aligned and pressed together. This eliminates the need for external clamping and leaves more of the surface of the chip free for optical and electrical access. The tape acts as both a gasket and an adhesive seal once it has been cured, preventing leakage in the system. The adhesive on the tape is not a fluid, so it will not act as a source of contamination in the fluid system. And finally, it does not add a significant amount of dead volume inside or increase the packaging size outside of the device.

However, double sided tape does not eliminate the need for a separate alignment method and can only be used in microfluidic systems where the reactants won’t be affected by the materials in the tape and where the tape and its adhesives won’t be dissolved by the reactants.

An example of the use of double sided tape in microfluidic seals is from Sandia National Laboratories. Galambos et al. developed an Electro-Microfluidic Dual Inline Package (EMDIP) which is intended to combine the standard electronics DIP with microfluidic connections for an integrated MEMS packaging system.17 In this case, holes are drilled through a standard ceramic or plastic DIP to create fluid ports. A silicon or PEEK insert is then used to connect the widely spaced fluid ports in the DIP to the closely spaced fluid ports of a silicon microfluidic device. Double sided tape is used to seal the connection between the silicon chip and the silicone/PEEK insert and between the insert and the top of the EMDIP. The seal was shown to hold up to pressures of 23 atm (2.3 MPa).

![Figure 2-12: EMDIP with Adhesive Tape Microfluidic Interconnect](image-url)
2.1.4 Glass-to-Glass Seals

Pyrex and other forms of glass can also be “welded” together by melting the joint between two glass objects until soft and then allowing the interface to cool again. Glass frits (the equivalent of solder rings for brazing) can also be used to create an interface between two glass surfaces.

Glass-to-glass seals can be used for very high temperature applications (up to the melting point of the glass), have excellent chemical resistance and compatibility and are quasi-hermetic seals, completely preventing fluid leakage but allowing the diffusion of helium gas over long periods of time. They are best for MEMS devices that use glass or pyrex substrates instead of or in addition to silicon.

Glass-to-glass seals are fragile because of the brittle nature of the substrate, the glass frit and the glass capillary tubes used. Rapid cooling can seal stresses in the joint, further decreasing the mechanical strength of the bond. However, the strength of the bond between silicon and pyrex wafers is often weaker than the glass weld and delamination is the most common failure mode for these seals. And the glass bonds are more difficult to create than many other sealing methods.

An example of glass-to-glass sealing in MEMS Research comes from MIT where it was used to study catalytic processes. In this case, Pyrex and Kovar capillary tubing was bonded to a pyrex wafer using melted brazing beads. The Kovar tubing held to 525°C and 50 atm of pressure while the pyrex tubing held to 25 atm. See figure 2.13.

![Figure 2-13: Glass Frit Microfluidic Interconnects](image)

2.2 Semi Permanent and Demountable Seals

Demountable seals are very important for bioMEMS devices where inexpensive microchips can be used to perform tests on fluid samples and then discarded. They are also important for building microchemical systems out of many individual MEMS components. It is more difficult to get a negligible leakage rate with demountable seals but
they are also less expensive to create and make the devices more versatile. Examples of compression seals with and without gaskets and seals created with O-rings from the MEMS literature will be presented.

2.2.1 Compression Seals without Gaskets

Some seals can be created by compressing the surfaces together if one or both materials is sufficiently compliant. In this case, the softer material deforms to fill the gaps between the two surfaces and prevent fluid from flowing in the interface. This works particularly well for soft plastics. Compression seals without gaskets are leak tight but not hermetic. They are reversible with little degradation of seal quality over time as long as the material stays within the perfectly elastic regime and the materials involved are very inexpensive.

The disadvantage of these seals is that the materials involved have relatively low melting points and cannot be used for high temperature reactions. Even small leakage rates may not be acceptable when using potentially hazardous chemicals or biological samples and the chemical resistance of these materials is frequently inadequate.

Kovacs filed a patent (5,890,745) for Stanford University in 1997 whose claims covered a number of instances of press fitting capillary tubes into each other and directly into microfluidic devices. Variations included using tapered sidewalls to guide the press fit tube and using thermal contraction and expansion to aid insertion of the tubes.

2.2.1.1 Examples of Compression Seals without Gaskets Commercially Available for MEMS Devices

An example of compression seals without gaskets comes from Upchurch Scientific. Upchurch has designed an entire line of microfluidic fittings including the NanoPort Assemblies. NanoPort assemblies consist of a port (coned or flat bottom), a gasket to seal the interface between the port and the substrate, a ferrule (flat bottomed only) and a nut (coned or flat bottom). The gasket is pressed into a recess in the bottom of the port and the port is permanently attached to the surface of the microfluidic chip with an adhesive. The fluid tubing is inserted in the ferrule and the nut (the coned nut acts as both the nut and the ferrule) and the nut/tubing assembly is screwed into the port. By screwing the nut into the port, the nut is compressed around the tubing creating a fluid seal without the use of gaskets. The seal is reversible by unscrewing the assembly. NanoPort connections can withstand pressures of 1500 psi (10.3 MPa) and 6.0 in-lbs (0.07 kg-m) of torque.
2.2.1.2 Examples of Reversible Compression Seals without Gaskets in MEMS Research

Gray et al. developed an array of “silicon/plastic coupler[s] with injection-molded press fittings” that allow glass capillary tubes to be inserted and removed at will.\textsuperscript{20} The coupler alone was not leak proof at 60 psi (414 kPa) but was able to withstand a maximum pressure of 60 psi (414 kPa) by adding a silicone gasket between the plastic and the silicon.

Armani, Liu and Aluru demonstrated a device which used a commercially available microfluidic coupler was embedded in PDMS and removed after the curing process was complete.\textsuperscript{21} This left a perfect impression of the coupler in the PDMS and allowed the coupler to then be snapped in and out at will in the final device. No data was given for the pull out force or the maximum pressure for operation.
2.2.2 Compression Seals with Gaskets

In cases where the materials involved are insufficiently elastic to seal without an intermediate layer, gaskets are used. Gaskets are often elastomers that have the same problems with heat and chemical resistance as compression seals without gaskets. Other gasket materials like graphite, woven or solid metals, and asbestos have very high temperature resistance and better/different chemical resistances.

Gaskets are inexpensive and add very little to the cost of the device which makes them attractive for applications that require disposable devices. Gaskets also made excellent secondary seals for applications that desire a backup seal. However, gaskets do have to be replaced on occasion because of damage caused by repeated loading.

The largest disadvantage of using compression seals (especially those that sandwich the microfluidic device between two plates) is that silicon and pyrex wafers tend to be very fragile and often the compressive force of the sealing process is enough to damage or destroy the device. It is difficult to regulate the compressive force when tightening screws and to repeatably obtain the same compressive force. And this packaging system tends to significantly increase the total volume of the system.

Victor et al. was granted a patent (6,319,476) for Perseptive Biosystems, Inc in 2001 whose claims covered a fluid connector consisting of a “housing, a clamping member, a first load support surface and a sealing member” whose sealing member “in its simplest form... is a gasket.” Variations include the surface of the top plate of the housing and compression screws as the “clamping member” and the use of springs and alignment mechanisms.
2.2.2.1 Examples of Compression Seals with Gaskets in MEMS research

An example of compression seals with gaskets in MEMS devices come from the Jensen group at MIT. Losey et al. and Ajmera et al. demonstrate the use of a Kalrez or Viton gasket sandwiched between two plates to form a compression fluid seal with both a multiphase packed-bed reactor and a differential reactor. The assembly seals at pressures up to 10 atm (1 MPa) and can operate at temperatures up 250° C (the maximum operating temperature of the gasket materials).

Haverkamp et al from the Institut fur Mikrotechnik Mainz (IMM) created a micro bubble column gas/liquid reactor which used graphite gaskets as compression seals in the research version. No information was given about the operating pressure or leakage rates for this system. A commercial version of the micro bubble column is now available and uses O-rings for the sealing method. The commercial version allows a maximum leakage of 0.01 mg nitrogen per second per meter of gasket length at 10 bar (1 MPa) and 20 degrees C.
2.2.3 O-ring Seals

An O-ring is a flexible ring of gasket material that operates on the principle of self-help. The pressure difference over the seal causes the O-ring to flatten out, and in many cases, to extrude into and fill any gaps in the seal. O-rings have the same advantages and disadvantages as gasket seals.

2.2.3.1 Examples of Compression Gaskets Commercially Available for MEMS Devices

Lawrence Livermore National Laboratory has created a microfluidic interconnect that allows standard liquid chromatography tubing to be connected to a MEMS device via a module that contains an O-ring. A maximum pressure of 750 psi (5200 kPa) was achieved using this interconnect. The technology is covered by a patent granted to Benett and Krulevitch for the University of California in 2001 (6,273,478) and although this product is not yet commercially available, they are seeking partners to commercialize the technology.
The Institut fur Mikrotechnik Mainz also uses O-rings to create the seal in many of its devices, including the Caterpillar Splot-Recombine Micro Mixer (CPMM-R1200) series. The maximum pressure of this mixer is 30 bar (30 kPa) and the maximum temperature is 200°C.

![Figure 2-21: IMM Caterpillar Micro Mixer](image)

### 2.2.3.2 Examples of O-ring Seals in MEMS Research

An example of O-rings in MEMS research devices comes from Yao et al at CalTech who integrated micromachined O-rings in their MEMS devices by depositing silicone at the wafer level. A variety of O-ring geometries were tested with maximum sealing pressures of 10 – 80 psi (70 – 550 kPa above atmospheric) and pull out forces of 0.05 – 0.4 N.

![Figure 2-22: Micromachined O-Rings](image)

### 2.2.4 Ground or Lapped Seals

Ground or Lapped Seals “consist of two parts connected to each other on their ground or lapped surfaces.” For this type of seal, the system relies on very high quality surface finish on all of the mating surfaces by grinding glass or by lapping metal. The better the surface finish, the fewer intrinsic passages for fluid to move through the interface and the less force that is required to compress the surrounding material into blocking those intrinsic passages. Ground or lapped seals can be found in three standard geometries: planar, conical or spherical. The planar seals are formed by placing two smooth, planar surfaces
References

Together. The most common example is slide valves. This configuration seals along a plane. Conical ground seals are created by combining an inner and outer part which fit together and have the same taper. The most common example is the stopper in a glass beaker. This configuration seals along a plane or a line. Spherical ground seals, like those used in ball-valves, are created by placing a sphere inside a conical or cylindrical tube. This configuration seals along a line.

Ground or lapped seals can used for high temperature, high pressure/vacuum applications with good chemical compatibility or resistance. But unlike welded or brazed seals, they can be safely and easily disassembled and reassembled.

One disadvantage of this type of seal is that a very thin layer of vacuum grease must be applied between the two mating surfaces to seal against high vacuum. The grease is a non-Newtonian fluid which fills the remaining passages through the interface that could not be sealed by compression alone. However for many MEMS applications, the use of grease would pose problems with chemical and biological compatibility and could flow or break down at high temperatures.

No examples of ground or lapped seals were found in either commercial or research MEMS devices.

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Chapter 3: Prior Art: Arrays of Interconnects for Microfluidic Systems

As MEMS technology becomes more advanced, research has turned its attention from creating individual devices to developing entire microfluidic systems that have practical research and commercial applications. For example, chemical reactions typically take a minimum of five steps: reactants are combined in a mixer, they are heated up to reaction temperature in a heat exchanger, the react in a catalyst lined reactor, they are cooled in a second heat exchanger and the reaction products are separated in a separator. Similarly, biological samples need to be filtered, washed, combined with reactants, mixed, and concentrated for testing. The individual components (mixers, reactors, separators, etc.) can either be fabricated on a single chip to create an integrated microfluidic system or each component can be fabricated separately and combined to create a modular microfluidic system.

While integrated systems have very low dead volumes, modular microfluidic systems have several advantages over fully integrated lab-on-a-chip devices. First, individual components are easier and less expensive to fabricate than integrated chips. They are also more robust because they are less complicated and have fewer failure modes. Their World-to-Chip modularity makes it possible to quickly connect and disconnect the individual components of the system to replace or repair damaged components. And finally, it increase the flexibility of the systems for research purposes.

One of the biggest challenges of modular microfluidic systems is the difficulty in connecting the individual components to create a reliable mechanical and fluidic seal. As with single World-to-Chip (macro to micro scale) fluid connections, there is no industry standard for arrays of modular interconnects for microfluidic systems. Development for packaging for modular microfluidic systems has followed two basic paths: the creation of arrays of World-to-Chip fluidic interconnects and the creation of Chip-to-Chip fluidic interconnects.

3.1 World-to-Chip Interconnect Arrays for Microfluidic Systems

Arrays of World-to-Chip fluidic interconnects transport fluids between modules using macro scale technology. These interconnections tend to be modified versions of existing commercial technology and thus are relatively robust, reliable and inexpensive. Often, they allow fluids, power and data to be moved into and out of the system using the same mechanism.
The main disadvantages of using arrays of World-to-Chip fluidic interconnects in modular systems are an increase in the total size of the system, dead volumes inside the fluid channels and large pressure drops in the system. There must be space between the modules in the system for the macro scale connector, piping or tubing to fit which can cause a significant increase in the total size of the system. Sudden changes in the size of flow channels (often caused by interconnects) create dead spaces that can trap fluids and lead to contamination between sample runs. Finally, sudden changes in the size of flow channels and very long flow channels (like those between the fluid modules) can cause significant pressure drops in the system which increases the pumping power required to run the system and can decrease the system efficiency.

3.1.1 Examples of World-to-Chip Fluid Interconnect Arrays Available for MEMS Devices

One type of World-to-Chip fluid interconnect arrays is commercially available in microfluidic systems from Mikroglas Chemtech in Mainz, Germany.1 MikroSyn is a full microchemical system that includes both modular microfluidic components, and the pumps, sensors, controls and computer required to operate the system (figure 1). The individual modules are Mikroglas components (glass microreactors, glass micromixers, etc.) in modular packaging with standardized (commercially available) connectors. The end result is a full desktop microreaction system with computer controls. This is an elegant example of microfluidic systems, however the size of both the modules and the interconnections are typical of World-to-Chip arrays and another connection scheme would need to be used to allow this system to be used for hand held (portable) applications.

![Mikroglas Microchemical System](image)

Figure 3-23: Mikroglas Microchemical System

3.1.2 Examples of World-to-Chip Fluid Interconnect Arrays in MEMS Research

An example of World-to-Chip fluid interconnect arrays in MEMS Research comes from the Institut fur Mikroverfahrenstechnik Forschungszentrum Karlsruhe. Micromixers, micro reactors, cooling passages and micro heat exchangers are all packaged in stainless
Examples of World-to-Chip Electrical and Fluid Interconnect Arrays in MEMS Research

steel housings with standard macro scale tubing quick connects.\textsuperscript{2} The individual components snap together to form a 2D microreaction system for explosive mixtures of hydrogen and oxygen. The system is suitable for very high temperature / pressure applications and has very good chemical compatibility, however it still has relatively low component density and devices can only stack in two dimensions.

![Figure 3-24: Karlsruhe Modular Microchemical Reaction System](image)

\textbf{3.1.3 Examples of World-to-Chip Electrical and Fluid Interconnect Arrays in MEMS Research}

The next two examples of World-to-Chip packaging include both electrical and fluidic interconnect arrays. The first example is from Sandia National Laboratories which modified standard dual inline packaging to include both electrical and fluid interconnects to create an Electro-Microfluidic Dual Inline Packaging (EMDIP) scheme.\textsuperscript{3,4}

Two versions of EMDIP were built. The first version modified standard electronics packaging by drilling fluid ports into a standard ceramic DIP and using a silicone insert to create a fluid seal and transport the fluid from the electro-microfluidic device to the fluid ports in the DIP (figure 3). The second version uses a custom designed acrylic lead frame with PEEK flow channel insert and is held together with adhesive tape (not shown).

Both versions of the EMDIP employ three channel sizes to take the flow from the macro to meso to micro scale and then back up again. This eliminates the creation of large dead spaces in the fluid channels and minimizes the pressure drops due to large changes in channel size. Two patents were granted for this technology. Patent # 6,443,179 was granted September 3, 2002 and patent #6,548,895 was granted April 15, 2003.
3.2 Chip-to-Chip Interconnects for Microfluidic Systems

Chip-to-Chip fluidic interconnects transport fluids between modules on the micro scale. The primary advantages to these interconnects is the size. In the best case, microfluidic systems that use Chip-to-Chip (micro to micro scale) interconnects are the same size as their integrated counterparts. The length of non participatory channels in the system is minimized and dead volumes are minimized or eliminated.
The primary disadvantage is that the interconnections have to be designed with the individual components and in many cases have to be built using micro fabrication techniques. This can increase cost and complexity. Fluid and electrical connections are often created using separate mechanisms. And there is a greater chance of damaging the microscale components during assembly. Nevertheless, technology will continue to move towards smaller and smaller modular systems if possible.

3.2.1 Examples of Chip-to-Chip Fluid Interconnect Arrays Commercially Available for MEMS Devices

One example of commercially available Chip-to-Chip fluid interconnects comes from Ehrfeld Mikrotechnik in Wendelsheim, Germany. Ehrfeld offers a series of microfluidic components including reactors, mixers, pumps and heat exchangers packaged in stainless steel with module to module connections. In addition to component modules, there are also inlet / outlet modules and cap modules to allow (or prevent) fluid from entering and leaving the components. The microreaction system also includes an assembly board which allows the modules to be secured. There is no information available on the sealing mechanism or the electrical inputs.

Although the system is elegant, effective and extremely compact, it is also currently prohibitively expensive to purchase and use.

Figure 3-27: Ehrfeld Microreaction System

O’Connor et al. from Nanostream, Inc. were granted patent # 6,536,477 on March 25, 2003 for a general modular microfluidic system. “This invention describes modular microfluidic systems and microfluidic coupling devices that are used to connect microfluidic modules and to transport fluid into a microfluidic device, bring fluid off of a microfluidic device, or transfer fluid between two devices.” Each module in the proposed system is “adapted for rapid attachment to or detachment from one or more other modules, and is self-contained for performing a desired function independently of each other module.” However, no details were provided for the fluid or electrical interconnects to be used in such a system.
3.2.2 Examples of Chip-to-Chip Fluid Interconnect Arrays in MEMS Research

An example of vertical (stacked) Chip-to-Chip interconnect arrays comes from Nagoya University in Japan. In this example, a set of modular micro scale devices (referred to as Biochemical Integrated Circuit Chips or ICs) including pumps, valves, reactors and concentrators were combined to create a total biochemical system. The chips were vertically stacked in a plastic holder unit with silicon rubber films (gaskets) sandwiched between the chips to create a fluid seal. Preload for the gaskets was provided by a spring inside the holder unit. The system was found to be leak tight up to 420 kPA.

The primary advantage in this type of system is that microfabrication techniques favor the creation of wide, thin devices with vertical through holes. The ability to vertically stack chips can significantly reduce the surface area that the microfluidic system occupies. However, the use of gaskets prevents this type of system from being used for high temperature, high pressure applications.
An example of a microfluidic breadboard comes from Gonzalez et. al at the University of California, Davis. In this system, a microfluidic breadboard was created by using specially developed “interconnecting finger joints.” Interlocking finger joints (left) have two sets of rectangular interlocking structures. When two silicon wafers with etched channels are brought together, a leak tight fluid passage is created by compressing a silicone rubber gasket between the two faces which are held together by the frictional forces between the fingers. The seal was leak tight to 20 psi.

While this system is very flexible in the arrangement of fluidic channels that can be created and used, it is limited in operating temperature and pressure.

3.2.3 Examples of Chip-to-Chip Electrical and Fluid Interconnect Arrays in MEMS Research

One example of Chip-to-Chip arrays that combine both electrical and fluid interconnects comes from the University of Twente in Enschede, The Netherlands. This microfluidic system involves the modular hybrid integration of components used for measurement of ammonia concentrations in aqueous solutions. The components modules are created by gluing the microfluidic components into a module housing. The electrical connections between the component and the module housing are made “by means of copper tracks that pass through the module housing and encapsulated wire bonds.” The base plate that the modules plug into is created from a stack of three plates expoxied together. The top plate is a printed circuit board with the electrical leads for the system. The second and third plates are a silicon wafer with etched channels and holes and a pyrex wafer to cap the channels. When the modules and base plate are complete, the modules can be inserted into the base plate. Fluidic connections between the modules and the base plate are formed using o-rings. Mechanical and electrical connections are created by soldering the two together at solder joints.

While this packaging scheme uses a combination of very simple and robust components, it does not employ truly reversible interconnects since the modules must be desoldered to be removed.
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Chapter 4: Reversible Microfluidic Interconnects

The interconnects that have been developed for either World to Chip or Chip to Chip interfacing are sufficient for most applications, however there is still no good option for reversible, Chip to Chip interconnects for high temperature, high pressure applications such as chemical microreactor systems. High temperatures and the harsh chemicals preclude the use of most gaskets and standard compression seals can break the fragile micro scale devices. The goal of this project was to develop a reversible, high temperature, high pressure microfluidic interconnect for use in microreactor systems.

4.1 Functional Requirements

The proposed interconnect had series of performance goals and functional requirements which were determined from feedback from microreactor users and designers. The functional requirements included:

- High Operating Temperature (-40°C and 500°C)
- High Operating Pressure (up to 50 atm)
- Minimal Leakage Rate
- Good chemical resistance
- Quick, easy reversible assembly
- Relatively cheap and easy to manufacture
- Good thermal isolation
- Low dead volume
- Minimal design disruption

4.1.1 High Operating Temperature and Pressure

Chemical reactions take place over a huge range of temperatures and pressures. Most interconnects that are currently available operate at temperatures between -40°C and 200°C and at pressures between 0 and 5 atmospheres (0.5 MPa). For any new interconnect design to be able to compete with prior art in the field, it would also have to function in these temperature and pressure ranges. Therefore, these ranges are the minimum operating ranges for any new interconnect design.

However, many microreactor systems operate at temperatures of up to 500°C and at pressures up to 50 atmospheres (5 MPa), therefore these ranges are the preferred operating ranges for the new interconnect design.
4.1.2 Leakage Rate

Many of the chemicals used in chemical reactor systems are extremely hazardous (flammable, corrosive, carcinogenic, and occasionally fatal in large quantities). While microchemical systems limit the quantities of chemicals used, and thus minimize the potential damage due to a catastrophic failure of the reactors, it is also important to limit the average leakage rate of the interconnects over time.

It is preferable that any interconnect designed for microreactor systems create a seal as close to hermetic as possible, with undetectable or barely detectable leakage rates.

4.1.3 Chemical Resistance

Common chemicals used in microreactors include:

- Methylene Chloride
- Hexanes (and other hydrocarbons like octane, heptane, etc.)
- Toluene
- Acetone
- Ethyl Acetate
- Acids (HCl, sulfuric, nitric, phosphoric, acetic)
- Bases (NaOH, KOH, sodium dibasic)
- Alcohols (isopropyl, ethyl, methyl)

While it is important for microreactor components to be resistance to the specific chemicals used in their respective systems, microfluidic interconnects should be resistant to as many (if not all) of the chemicals used in microreactors in general as possible.

4.1.4 Quick and Easy Assembly

Many microfluidic components require extensive work and complicated equipment (either optical microscopes or mechanical jigs) to align before connecting to create a reversible seal. Permanent fluid seals require extra time and hardware to bond, weld or solder the components together. To save time, energy and the use of extra equipment, it is preferable that the proposed interconnect be passively actuated and self aligning so the components can be snapped or pressed together as quickly as possible.

4.1.5 Thermal Isolation

Silicon has very high thermal conductivity (124 W/mK) which means that large quantities of heat can be transferred from a microreactor at very high temperatures to lower temperature components in the system through the interconnect if the interconnect also
has a high thermal conductivity, large contact area or small height/thickness. Interconnects should be designed to minimize the heat transfer between high and lower temperature components.

4.1.6 Low Dead Volume

To minimize pressure drops and the possibility of contamination in the system, the interconnects should have the lowest dead volume possible.

4.1.7 Minimal Design Disruption

Even the simplest microscale device takes a great deal of time and money to fabricate and every new feature that is added to a device also adds one or more masks and dozens of fabrication steps. To save time and money and to prevent the microreactor components from having to be completely redesigned, the interconnect design was to have either no impact or a very minimal impact on the microreactor component design and fabrication.

4.2 Design Parameters

As discussed in Chapter 1, a variety of design factors or parameters must be considered before a design can be completed. These include:

- Interconnect Size (World to Chip vs. Chip to Chip)
- Interconnect Type (Permanent vs. Demountable)
- Interconnect Material
- Interconnect Seal Type
- Interconnect Geometry

4.2.1 Interconnect Size

Chip to Chip interconnects were chosen because of the low pressure drops, low dead volume and compact packing possible associated with micro scale arrays. However, several considerations had to be balanced when deciding the scale of the interconnect. The flow channels for the fluid needed to remain on the micro scale to reap the benefits of low pressure drops and low dead volumes. The mating interconnect features had to be large enough to be assembled by hand without the use of microscopes and alignment stages. However, if the interconnect features were too large, they would force the microreactor components to be larger than necessary, wasting resources and space. The result was a meso to meso scale interconnect with micro scale flow channels.
4.2.2 Interconnect Type (Permanent vs. Demountable)

Since one of the functional requirements of the interconnect was easy and quick assembly/disassembly, the interconnect design had to be reversible (demountable).

4.2.3 Interconnect Material

The interconnect material was dictated by the required operating temperature, operating pressure and chemical compatibility of the microreactor systems. In general, plastics, metals, glasses and semi-conductors like silicon can be used with or in conjunction with microfabrication processes. However, the melting point of plastics is less than the maximum desired operating temperature of the microreactors, while the melting (or softening) point of glasses, metals and semiconductors are all much higher (see Table 4.1). For this reason, plastics were rejected as both the material for the mechanical structure of the interconnect and also for gasket materials to create the fluid seal.

Table 4-1: Melting Points of Various Microfabrication Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Melting Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plastics</td>
<td></td>
</tr>
<tr>
<td>Polyethylene</td>
<td>~100°C</td>
</tr>
<tr>
<td>Polyetheretherketone (PEEK)</td>
<td>~350°C</td>
</tr>
<tr>
<td>Glass</td>
<td></td>
</tr>
<tr>
<td>Borosilicate</td>
<td>~800°C</td>
</tr>
<tr>
<td>Quartz (softening point)</td>
<td>~1700°C</td>
</tr>
<tr>
<td>Metals</td>
<td></td>
</tr>
<tr>
<td>Gold (pure)</td>
<td>~1000°C</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>~1400°C</td>
</tr>
<tr>
<td>Semiconductor</td>
<td></td>
</tr>
<tr>
<td>Silicon (pure)</td>
<td>~1400°C</td>
</tr>
</tbody>
</table>

Next, chemical compatibility of a number of materials was examined and the most inert structural materials available for use with microfabrication techniques were borosilicate (pyrex) glass, quartz glass, stainless steel and silicon (see Table 4.2). Silicon was chosen for the interconnect substrate because there are more tools available for the microfabrication of silicon than for the other materials, making the interconnects easier and less expensive to produce. Gold was identified as a potential gasket material because it is very soft and also chemically inert.
### Table 4-2: Chemical Compatibility of Various Materials to Selected Chemicals

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Borosilicate Glass</th>
<th>Quartz</th>
<th>Aluminum</th>
<th>Copper</th>
<th>Gold</th>
<th>Stainless Steel</th>
<th>Silicon</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methylene Chloride</td>
<td>A</td>
<td>A</td>
<td>C</td>
<td>B</td>
<td>A</td>
<td>B</td>
<td>A</td>
</tr>
<tr>
<td>Hydrocarbons (Hexanes, Octane, Heptane)</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
</tr>
<tr>
<td>Toluene</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
</tr>
<tr>
<td>Acetone</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>A</td>
<td>B</td>
<td>A</td>
</tr>
<tr>
<td>Acids</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HCl</td>
<td>A</td>
<td>A</td>
<td>D</td>
<td>D</td>
<td>D</td>
<td>D</td>
<td>A</td>
</tr>
<tr>
<td>Sulfuric Acid</td>
<td>A</td>
<td>A</td>
<td>D</td>
<td>D</td>
<td>A</td>
<td>B to D</td>
<td>A</td>
</tr>
<tr>
<td>Nitric Acid</td>
<td>A</td>
<td>A</td>
<td>D</td>
<td>D</td>
<td>A</td>
<td>D</td>
<td>A</td>
</tr>
<tr>
<td>Phosphoric Acid</td>
<td>D</td>
<td>D</td>
<td>C</td>
<td>D</td>
<td>A</td>
<td>B to D</td>
<td>A</td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>A</td>
<td>A</td>
<td>B</td>
<td>B</td>
<td>A</td>
<td>B</td>
<td>A</td>
</tr>
<tr>
<td>Bases</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NaOH</td>
<td>A to D</td>
<td>A</td>
<td>D</td>
<td>A to D</td>
<td>A</td>
<td>B</td>
<td>D</td>
</tr>
<tr>
<td>KOH</td>
<td>A to D</td>
<td>A</td>
<td>D</td>
<td>B</td>
<td>A</td>
<td>A</td>
<td>D</td>
</tr>
<tr>
<td>Sodium Dibasic</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>A</td>
</tr>
<tr>
<td>Alcohols (isopropyl, ethyl, methyl)</td>
<td>A</td>
<td>A</td>
<td>A to B</td>
<td>A to B</td>
<td>A</td>
<td>A to B</td>
<td>A</td>
</tr>
</tbody>
</table>

#### 4.2.4 Interconnect Seal Type

Recall from Chapter 2, there are three types of demountable seals: compression seals without gaskets, compression seals with gaskets or o-rings, and ground or lapped seals. Compression seals, with or without gaskets, require a very soft material (like a thermoplastic) to deform and fill all potential fluid leakage paths. Since plastics were eliminated as a possible material for use in the system, a ground or lapped seal was chosen for the interconnect sealing mechanism. A compression seal with a deposited gold gasket layer was chosen as a counter measure in the event than a ground or lapped seal could not be achieved.

#### 4.2.5 Interconnect Geometry

The fluid inlet/outlet ports in microfluidic devices tend to be little more than through holes in the top layer of the device that connect to the fluid channels inside. These holes are either circular with straight sidewalls made using Reactive Ion Etching (RIE) or Deep Reactive Ion Etching (DRIE), or the holes are pyramidal pits made using potassium hydroxide (wet chemical etching). Two different interconnect geometries were chosen to interface with each of these types of fluid ports.

A concentric ring coupler using Reactive Ion Etching (RIE) to create a set of interlocking circular rings was chosen as the primary interconnect geometry. An interlocking pyramid coupler using wet chemical etching (KOH) etching was chosen as a second option for the interconnect geometry.
4.3 Concentric Ring Design

The micro fabricated equivalent of a conical ground seal can be achieved by using a directional reactive ion etch to create slightly tapered concentric rings in a pair of silicon substrates. In the design shown below, each fluid port is surrounded by two rings which come together to form three sealing faces (although the number of rings is variable). Having more than one sealing face increases the redundancy in the system and can reduce or eliminate leakage in the event that the first seal fails. The taper on the sidewalls of the rings make the devices self-aligning and allow the leakage rate to be decreased as the preload on the interconnect is increased. The taper also prevents the rings from bottoming out in their respective grooves, which means that the surface finish is only important on the tapered faces and not on the wafer surface and loosens some of the manufacturing tolerances.

Although the surface finish using RIE should be good enough to seal the interface and prevent leakage, a better surface finish with different geometry might be achieved using wet etching with KOH.

![Figure 4-32: Schematic for Concentric Ring Coupler](image)

4.4 KOH Pyramid Design

The micro fabricated equivalent of a plane ground joint can be achieved by placing fluid vias through planar surfaces created by KOH etching. Potassium hydroxide (KOH) etches (100) silicon along the (111) atomic plane. The result is a mirror perfect surface finish on a plane 54.7 degrees from vertical. The anisotropic KOH etch can be used to create both pyramids (projecting above the surface of the wafer) and pyramidal pits.

A KOH wet etch is self-terminating when the four angled faces meet. If the wafer is not deep enough to allow the etch to self-terminate, a through hole is created through the silicon wafer. This is the easiest way to create a single fluid port through a KOH interconnect (figure 2.2 right). In this case, a gasket material would need to be used to prevent leakage at the corners where the four angled faces meet. However, if the etch is allowed to self- terminate, (D)RIE can then be used to drill up to four fluid ports per
Kinematic KOH Pyramid Interconnect

pyramid (one per side). Since the fluid connections would be along the four faces of the pyramid instead of along the top of the pyramid/bottom of the pit, there is no need for the four corners of the pyramids to interface exactly and no need for a gasket (figure 4.2 left).

![Top View](image1)

![Side View](image2)

**Figure 4-33: Schematic of KOH Fluid Ports**

The four port design is a planar version of Slocum’s “Kinematic Coupling Fluid Couplings and Method” presented in US Patent #5,683,118. Slocum’s design was perfectly constrained by using spheres with fluid ports on one half of the coupler and planes with fluid ports on the other half. Since the KOH ports use planes on both sides of the coupler, they are not perfectly constrained and an applied pre-load is required to average out misalignment. For this reason, the four port KOH couplers can be referred to as “quasi-kinematic fluid couplings.”

Three KOH interconnect designs were envisioned for use with either type of fluid port: a kinematic KOH pyramid interconnect, a KOH interposer interconnect and a flexible KOH pyramid interconnect.

### 4.4.1 Kinematic KOH Pyramid Interconnect

The proposed kinematic KOH pyramid interconnect is a Chip to Chip interconnect which allows the pyramid from one device to mate directly with the pyramidal pit in another device (figure 4.3). This design has the advantage of zero dead volume in the fluid channels and very compact spacing of microfluidic components. However the pyramids can be difficult to fabricate and the interconnect may have very high heat transfer rates if the faces of both devices touch along their entire lengths.
4.4.2 KOH Interposer Interconnect

The proposed KOH interposer design would use a third device between the two component faces to form an interconnect. The major advantage of this design is that the complicated pyramids can be fabricated on a separate device, while the simple pyramidal pits (which may already be part of the component design) are fabricated on the component wafer. This design will also have lower heat transfer rates than the kinematic version. The main disadvantage is that it is a more complicated interconnect, with more components to fabricate, assemble and repair if necessary.

4.4.3 Flexible KOH Pyramid Interconnect

The last proposed design is for a flexible KOH pyramid interconnect. Unlike the concentric ring design, the KOH pyramids and pyramidal pits cannot deflect to accommodate slight misalignments in either the manufacturing process or in the final alignment of the devices. This interconnect design would have the pyramids on one of the mating wafers be suspended on flexible cantilever beams. This would allow the pyramids the flexibility to connect with their respective pyramidal pits in spite of small misalignments. The major disadvantage to this design is the complexity of the fabrication process to create the flow channels through the cantilevers and into the pyramids.
Flexible KOH Pyramid Interconnect

Figure 4-36: Schematic of Flexible KOH Interconnect
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Chapter 5: Mathematical Modeling of Concentric Ring Coupler

Analytical and finite element models were created for the concentric ring coupler to verify proof of concept before investing the time and money into building macro or micro scale versions of the interconnects.

5.1 Geometric Parameters

Before any modeling could be done, geometric parameters had to be defined and set.

5.1.1 Taper Angle

The concentric ring coupler was supposed to be able to hold a fluid seal using just the frictional forces along the connecting faces without any other constraining mechanisms, but still be easy to disassemble. Since the taper angle, in part, determines the frictional forces between the mating faces, the angle was chosen to be a compromise between a locking and non-locking taper.

The Machinery’s Handbook defines a “self-holding” or locking taper as being one “where the angle of the taper is only 2 or 3 degrees, [and] the shank of a tool is so firmly seated in its socket that there is considerable frictional resistance to any force tending to turn or rotate the tool relative to the socket.” A “self-releasing” or non-locking taper is one “over 16 degrees.” (p.904) The taper angle can be difficult to accurately control in microfabrication, so an acceptable range of taper angles was defined to be any constant angle between 5 and 15 degrees, with a nominal value of 7 degrees used for modeling and analysis.

5.1.2 Ring Diameter

The ports of most microfluidic devices range from 100 to 400 microns in diameter. To accommodate the maximum number of potential devices that the coupler could be used with, the inner diameter of the inner most ring was set to 400 microns with the diameters of the other rings defined by other geometric parameters.
5.1.3 Number of Rings

For interlocking rings systems shown in figure 5.1, a small force applied on the face of the interconnect causes a large reaction force on the faces of the rings. The primary result of this force is deflection of the outer most rings with compression of the inner rings as a secondary effect.

The use of two rings (one on each device), may not form a fluid seal if both the inner and outer deflect and the interface between them does not compress. The countermeasure for this system failing is to add another ring.

The use of one ring and one groove (one ring on one face and two rings on the mating face) creates two faces with compressive forces. The inner ring will compress while the outer rings will deflect. This is the minimum number of rings to be sure of creating a fluid seal. The counter measure for this system failing is to add another ring.

The use of two pairs or rings (two rings on each device) creates three faces with compressive forces and is the potential ideal situation. The inner rings will compress while the outer rings will deflect.

The use of two rings and two grooves (two rings on one face and three rings on the mating face) creates four faces with compressive forces. The inner three rings will compress while the outer two rings will deflect, however there is a risk that the middle ring will not compress enough. This could lead to misalignment or the failure to create a fluid seal. The counter measure for this system failing is to add a gasket material.

The use of three rings and two grooves (three rings on each face) creates fives faces with compressive forces. It is probable that this configuration will not be able to accommodate mismatches and/or large deflections. In this case, a tight fluid seal will not be created on all (any) of the faces, resulting in a labyrinth seal which relies on successive pressure drops to minimize the leakage rate instead of the elimination of leakage paths.

Since two rings and one groove (two rings on each device) appears to have the least risk, this geometry was used for both the analysis and test devices.
5.2 Analytical Model

As stated above, a small downward force on the top half of the interconnect causes a large horizontal force on the rings on the lower half of the interconnect. From Newton's Second Law, the downward force on the top ring (or tooth, referring to the cross sectional appearance), must be equal to the sum of the upwards reaction forces from the bottom teeth.

\[ F_{\text{down}} = F_{\text{up}} \]  

From trigonometry, the tangent of the taper angle is equal to the horizontal reaction force divide by the vertical reaction force. Or rewritten, the horizontal reaction force is equal to the vertical reaction force divided by the tangent of the taper angle.

\[ F_{\text{side}} = F_{\text{up}}/(\tan \theta) \]  

Pressure is defined as force divided by area. The horizontal pressure on the tooth of interest is equal to the horizontal force divided by the area of contact between the top and bottom tooth. That contact area is the length of contact between the two teeth (which we will approximate as being equal to the height of the tooth \( L \)) times the depth of the tooth \( b \).

\[ P_{\text{side}} = F_{\text{up}}/(Lb \cdot \tan \theta) \]
Each ring is an axisymmetric entity and can be thought of as a trapezoidal cross section revolved around a central axis. For thin slices of the ring, the cross section can be approximated as a beam with a constantly varying cross section and a uniform pressure applied along a section of the beam. For simplicity, the ring section (or tooth) will be modeled as a beam with length equal to the tooth height $L$, height equal to the average width of each tooth $H$, depth equal to the thickness of the ring slice $b$, and a constant pressure $P_{\text{side}}$ applied along the entire length of the beam.

These simplifications allow us to use beam bending equations to approximate the displacement and maximum stress in each tooth under loading conditions. The deflection of the beam at the tip of the tooth (end of the beam) is given by:

$$w(L) = \frac{-(P_{\text{side}} \cdot L^4)}{8 \cdot E \cdot I} \quad (4)$$

where $E$ is the Young's Modulus of the material (169e3 MPa for Silicon) and $I$ is the moment of inertia. For a square beam with constant cross section, the moment of inertia is given by:

$$I = \frac{bH^3}{12} \quad (5)$$

The maximum stress in the beam is given by:

$$\sigma_{\text{max}} = \frac{L^2 P_{\text{side}}}{2b} \quad (6)$$
The user should be able to apply the initial downward (pre-loading) force on the interconnect by hand. This value can be estimated as 3.5 pounds force or 15 Newtons. If each device is approximately 1 cm x 1 cm, the applied pressure is 0.15 MPa. The thickness of a thin slice of the ring (depth of the beam) can be approximated as 3.5 microns (1/360th of the perimeter of the ring). Using angles ranging from 5 to 15 degrees, average tooth widths ranging from 5 to 20 microns, and a tooth height of 50 microns, the maximum deflection and stresses were calculated. The results are shown in Table 5.1.

<table>
<thead>
<tr>
<th>Angle (degrees)</th>
<th>Length (um)</th>
<th>H Avg (um)</th>
<th>Mom Inert. (um^4)</th>
<th>P down (N)</th>
<th>P side (Mpa)</th>
<th>Deflection (um)</th>
<th>Stress (Mpa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>50</td>
<td>5</td>
<td>36.46</td>
<td>0.15</td>
<td>0.34</td>
<td>0.0436</td>
<td>122.70</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>15</td>
<td>984.38</td>
<td>0.15</td>
<td>1.03</td>
<td>0.0048</td>
<td>368.10</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>20</td>
<td>2333.33</td>
<td>0.15</td>
<td>1.37</td>
<td>0.0027</td>
<td>490.79</td>
</tr>
<tr>
<td>10</td>
<td>50</td>
<td>5</td>
<td>36.46</td>
<td>0.15</td>
<td>0.17</td>
<td>0.0217</td>
<td>61.23</td>
</tr>
<tr>
<td>10</td>
<td>50</td>
<td>15</td>
<td>984.38</td>
<td>0.15</td>
<td>0.51</td>
<td>0.0024</td>
<td>183.70</td>
</tr>
<tr>
<td>10</td>
<td>50</td>
<td>20</td>
<td>2333.33</td>
<td>0.15</td>
<td>0.69</td>
<td>0.0014</td>
<td>244.93</td>
</tr>
<tr>
<td>15</td>
<td>50</td>
<td>5</td>
<td>36.46</td>
<td>0.15</td>
<td>0.11</td>
<td>0.0144</td>
<td>40.69</td>
</tr>
<tr>
<td>15</td>
<td>50</td>
<td>15</td>
<td>984.38</td>
<td>0.15</td>
<td>0.34</td>
<td>0.0016</td>
<td>122.07</td>
</tr>
<tr>
<td>15</td>
<td>50</td>
<td>20</td>
<td>2333.33</td>
<td>0.15</td>
<td>0.46</td>
<td>0.0009</td>
<td>162.77</td>
</tr>
</tbody>
</table>

The simple analytical model shows that even under relatively small loads, the teeth will still show small deflections and the maximum stresses are well below the yield stress of the silicon (2800 MPa). So to first order, the concentric ring design should work.

**5.3 Finite Element Model**

A fully parameterized two dimensional axisymmetric finite element model was created to better understand the behavior of the mating concentric rings and optimize the interconnect geometry using ANSYS.
5.3.1 Model Units

Numerical modeling of micro scale devices has some unique problems. Most numerical simulation packages have limits on the number of significant figures they carry for calculations. If any value in the model is either higher or lower than that number of significant figures, it is considered to be “zero” or “infinity” by the program and carries the numerical singularities associated with those two values. Most often, the program will simply return an error and terminate the simulation.

To eliminate this problem and keep calculations within acceptable ranges, ANSYS has developed the micro MKS system (microns, Kelvin, seconds). This system of units was used for this analysis so all displacements are in microns, and stress is measured in MPa.

Table 5-4: Micro MKS System of Units

<table>
<thead>
<tr>
<th>Mechanical Parameter</th>
<th>MKS Unit</th>
<th>Dimension</th>
<th>Multiply by This Number</th>
<th>To Obtain μMKS Unit</th>
<th>Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length</td>
<td>m</td>
<td>m</td>
<td>10^6</td>
<td>1</td>
<td>μm</td>
</tr>
<tr>
<td>Force</td>
<td>N</td>
<td>kg m / s^2</td>
<td>10^6</td>
<td>1</td>
<td>N</td>
</tr>
<tr>
<td>Time</td>
<td>s</td>
<td>s</td>
<td>1</td>
<td>1</td>
<td>s</td>
</tr>
<tr>
<td>Mass</td>
<td>kg</td>
<td>kg</td>
<td>1</td>
<td>1</td>
<td>kg</td>
</tr>
<tr>
<td>Pressure</td>
<td>Pa</td>
<td>kg / m / s^3</td>
<td>10^-3</td>
<td>1</td>
<td>MPa</td>
</tr>
<tr>
<td>Velocity</td>
<td>m / s</td>
<td>m / s</td>
<td>1</td>
<td>1</td>
<td>m / s</td>
</tr>
<tr>
<td>Acceleration</td>
<td>m / s^2</td>
<td>m / s^2</td>
<td>1</td>
<td>1</td>
<td>m / s^2</td>
</tr>
<tr>
<td>Density</td>
<td>kg / m^3</td>
<td>kg / m^3</td>
<td>10^-18</td>
<td>1</td>
<td>kg / m^3</td>
</tr>
<tr>
<td>Stress</td>
<td>Pa</td>
<td>kg / m / s^2</td>
<td>10^-6</td>
<td>1</td>
<td>MPa</td>
</tr>
<tr>
<td>Young’s Modulus</td>
<td>Pa</td>
<td>kg / m / s^3</td>
<td>10^-12</td>
<td>1</td>
<td>MPa</td>
</tr>
<tr>
<td>Power</td>
<td>W</td>
<td>kg m / s / s^3</td>
<td>10^-3</td>
<td>1</td>
<td>kW</td>
</tr>
</tbody>
</table>

5.3.2 Solid Model Geometry

The finite element model assumes that all rings on both halves of the interconnect have the same cross sectional properties and that the distance between the rings (the width of the grooves) is smaller than the ring that is to fit into that space (or groove). By forcing a ring that is too large into a groove that is too small, we are able to ensure compressive forces on the mating faces and that the ring will not bottom out in the groove. It also assumes a perfect (smooth) surface finish on all solid model entities.

Six geometric parameters were defined to generate the interconnect geometry: the inner diameter of the innermost ring, the height of each tooth, the width of each tooth at its tip, the taper angle for each tooth, the percent difference between the width of the ring and its mating groove and the downward pressure on the top half of the coupler. These parameters were used to calculate the x and y locations for key locations along the cross section (like the outer bottom edge, outer top edge, inner top edge and inner bottom edge of a ring) in a series of arrays.
Material Properties

The geometry for the model was created using bottom up solid modeling. The arrays of x and y values were used to generate key points. Lines were then used to connect those key points in the order they were generated and areas were created from those lines. All sharp corners were given a radius of 1 micron to prevent numerical singularities that would result in the appearance of excessive stress concentrations.

Because the geometry is fully parameterized, new array parameters are calculated each time a parameter is altered and the geometry updates perfectly each time.

5.3.3 Material Properties

Material properties for the model were assigned based on bulk single crystal silicon because the presence of dopants in the semiconductor and the etching process have a negligible impact on the mechanical properties of the material.

5.3.4 Finite Element Model

The element mesh automatically generated using a smartsize of 4 and plane183 elements. After the first set of elements was produced, the lines along the potential interface between the two couplers were selected and target169 and contact172 elements were applied along those lines.

5.3.5 Loads and Constraints

A zero displacement constraint in the x and y directions was placed on the bottom line of the bottom coupler. The top coupler was loaded in two steps. In the first step, a downward displacement constraint was applied to the top surface to put the two contact surfaces inside the pinball region of the contact elements. In the second step, the displacement on the top surface was replace with a pressure along the top line coupler to act as a pre-load for the system.

5.3.6 Results

Post processing returned six parameters and four plots. The first three parameters returned were the maximum von Mises stress, the maximum shear stress and the minimum shear stress. These were calculated to ensure that the structure would be under less than 1% of the maximum yield stress. The last three parameters were the contact pressures along the three planes of contact. These values were calculated by summing and averaging the values over the nodes on those lines. The values of these six parameters were used to optimize the coupler geometry. The four plots returned were for the deflection in the x direction, von Mises stress, XY shear stress and contact pressure were generated for the optimized geometry (including tooth height and width, taper angle and degree of interference).
Results

The results below are for an interconnect with the following values:

- Inner Diameter of Innermost Ring - 400 um
- Tooth Height - 40 um
- Tooth Width at Tip - 15 um
- Taper Angle - 7 degrees
- Percent Difference between Ring and Groove - 5%
- Downward Pressure - 200e-3 MPa

5.3.6.1 Displacement Plots

The displacement plot (figure 5.4) validates the earlier assumptions about the behavior of the meshing teeth. The inner tooth moves in the negative x direction while the outer tooth moves in the positive direction while both inner teeth compress very slightly. For this configuration, the maximum displacement is 0.0035 um. The results from the analytical model predicted a displacement between 0.0027 and 0.0048 um for this configuration which shows very good agreement.

Figure 5-40: Horizontal Displacement (um)
5.3.6.2 Stress Plots

The von Mises stress plot (figure 5.5) shows that the highest stress concentrations are located at the root of the teeth, where the rings connect with the rest of the silicon substrate, and at the tips of the teeth where they contact the other surfaces. The maximum stress in the models is 30.548 MPa, which is two orders of magnitude lower than the yield stress of the material. The strain for this model is 0.01%, which is ten times less than the maximum “safe” strain.

![Von Mises Stress Plot](image)

Figure 5-41: Von Mises Stress (MPa)

5.3.6.3 Contact Stress Plots

The contact pressure plot (figure 5.6) shows that the average contact pressure is on the order of 20 MPa with even this very small pre-load and indicates an ability to seal against very high fluid pressures.
Figure 5-42: Contact Pressure
Chapter 6: Macro Scale Devices

A series of macro scale concentric ring interconnects were built to ensure that the sealing mechanism was feasible for both single interconnects and arrays of interconnects.

6.1 Single Interconnects

A series of single concentric ring interconnect were made out of 2” delrin cylinders. The rings and grooves were nominally 1/3” thick and 3/8” of an inch deep. The top interconnect had two rings and one groove. A 1/8” diameter hole was drilled through this interconnect. The bottom interconnect had two rings and two grooves.

![Figure 6-43: Single Interconnect Halves](image)

6.1.1 Interconnects with Straight Sidewalls

The first set of interconnects that were made did not have tapered sidewalls and were made using a CNC mill. The rings of the first pair were 2/1000” thicker than their corresponding grooves. The rings of the second pair were 3/1000” thicker than their corresponding grooves.

The interconnects were assembled using an arbor press to compress the rings into the corresponding grooves. The fluid seal was tested by pumping pressurized air into the top half of the coupler, submerging the coupler pair and looking for bubbles leaking out of the interface between the two halves.

The first interconnect showed significant leakage at relatively low pressures (~ 10 to 20 psi). The second interconnect had a much lower leakage rate when pressurized up to 60 psi, but still did not create a fluid seal at low pressures. It was also very difficult to disconnect once the two halves were pressed together and difficult to get the two halves to align without one half being at a slight angle to the other. To correct some of these difficulties, a series of interconnects with tapered sidewalls were made.
6.1.2 Interconnects with Tapered Sidewalls

The second set of macro scale interconnects were manufactured with 7 degree tapers on the ring sidewalls and made using a CNC Lathe. The use of a lathe instead of a mill for these interconnects significantly improved the surface finish on the rings and allowed the tapered sidewalls to be created without having to increase the size of the interconnect geometry.

The tapered interconnects were tested using the same method as before. Although an arbor press was not required to assemble the halves of the tapered couplers, a pre-load was applied using a C-clamp. The C-clamp was then loosened until it was applying no force on the interconnect but was able to prevent the two halves from separating. Under these conditions, the interconnect held a fluid seal under 80 psi. However, without the presence of the C-clamp, eventually the coupler would have separated and leaked.

This confirmed that both the taper and the surface finish are important elements in creating a fluid seal. The next step was to confirm that these interconnects could be used in arrays for devices with multiple inlet / outlet ports.

6.2 Interconnect Arrays

Two arrays of macro scale concentric ring interconnects were manufactured. The top interconnect halves for both arrays was made of delrin and had two rings and one groove. The bottom halves for the first array had two rings and two grooves while the coupler for the second array had two rings and one groove. Both the top and bottom couplers had 1/8” NPT threaded through holes drilled through the center.

The baseplates to hold the interconnect halves were made from nylon on a CNC mill. Each plate had six circular depressions 1.95” in diameter with a 1/4” through hole through the bottom. The interconnect halves were press fit into these depressions. The through holes allowed fluid tubing and fittings to pass through the base plate.

The interconnect arrays were also tested using the bubble method. Threaded plugs were inserted into the bottom interconnect halves to prevent fluid leakage. Threaded barbed hose fittings were inserted into the top interconnect halves and connected to a pressurized air manifold. The two halves of the baseplates were pre-loaded, clamped together and submerged to determine the extent and location of air leakage.
Interconnect Arrays

Figure 6-44: Interconnect Arrays

The interconnect array with two rings and two grooves (five rings total) was only able to seal up to 20 psi with four, five or six interconnects in the array. As the pressure increased to 80 psi, eventually all but two interconnects leaked in each array.

The interconnect array with two rings and one groove (four rings total) and four interconnects was unable to seal when the baseplates were pre-loaded by hand and not clamped together. When pre-loaded using the arbor press and not clamped together, the four interconnect array was able to seal up to 20 psi with no leakage. (Testing above 20 psi was abandoned due to noise.) The six interconnect array (one clamp per interconnect) showed leakage at two of the interconnects by 20 psi but was able to seal four of the six interconnects up to 80 psi.

These results indicate that arrays of concentric ring interconnects are feasible as long as the deformation of the interconnect rings are able to compensate for mismatch in alignment. The pre-load necessary to seal against significant pressures may also be much larger than initially anticipated. Nevertheless, the results were very promising and supported the creation of microscale interconnects.
Chapter 7: Micro Scale Interconnects

The design and fabrication of the micro scale concentric ring interconnects was more complicated than their macro scale counterparts because the variations in the fabrication process relative to the tolerances of the devices are much larger and the materials used are much more brittle.

7.1 Micro Fabrication Techniques

A brief overview of microfabrication techniques is presented here to explain some of the design decisions for the concentric ring couplers. For more information on microfabrication, see Microsystem Design by Steven D. Senturia.

Unlike conventional macro scale machining processes, micro scale fabrication allows for material to both be added and removed. Silicon compounds like silicon dioxide and nitride can be grown on a silicon wafer surface by consuming some of the surface silicon. Silicon compounds, polysilicon, and metals can be deposited on the wafer surface via sputtering or removed from the wafer surface using chemical etching, ion bombardment or a combination of the two.

Most micro scale geometry is defined using photolithography. In this process, the wafer is coated with a photosensitive polymer (positive photoresist) and exposed to UV light through a photo mask. Parts of the mask allow the light to pass through while other parts prevent the polymer (or photoresist) from being exposed to the light. The exposed polymer becomes soluble in a chemical developer and is removed while the unexposed resist remains, forming a patterned layer on the surface of the wafer which protects it from other microfabrication processes like deposition or etching. Since the pattern is two dimensional, the end result is that most micro fabrication processes create extruded two dimensional features.

For example, the creation of concentric rings on the surface of a silicon wafer consists of eight main steps (figure 7.1). First, a layer of silicon dioxide is grown on the surface of the wafer. This layer will serve as a hard mask for the main etching step. Next a layer of photoresist is deposited on top of the oxide layer. The resist is then patterned and the oxide layer is etched using the resist pattern. The resist is removed and the silicon is etched using the oxide pattern, Finally, the oxide mask is removed.

The pattern on the photomask can be defined with micron (and in some cases, sub-micron) accuracy. However, the mask (whether made from photoresist or oxide) is eroded during the etching process (albeit slower than the substrate being etched) so the protected area on the substrate may decrease over time. Also, no etching process is strictly anisotropic so the substrate is also being etched from the sides as well as from the top. The rates that control
the etching of the mask and of the substrate are a function of temperature, pressure, chemicals used, concentrations of those chemicals, RF power, voltage across the substrate the geometry, the geometry of the unmasked substrate (both in terms of total exposed substrate and the distribution of that space) and the depth of the etch. So the same recipe can produce very different results on two different masked wafers, and occasionally will produce slightly different results on the same masked wafer on two different days.

These variations in micro fabrication mean that nominal values for the concentric ring geometry can be specified but the actual geometry produced may vary significantly from the expected geometry. This variation was a major factor in the design of the micro scale concentric ring interconnects and the mask to create them.

7.2 Design of Micro Scale Interconnects

7.2.1 Inner Ring Geometry

Recall that the geometry for the finite element model was fully defined by five parameters: the inner diameter of the innermost ring, the height of each tooth, the width of each tooth at its tip, the taper angle for each tooth, and the percent difference between the width of the ring and its mating groove. Since the finite element analysis predicted the optimal nominal values for the geometric parameters, the same nominal values were desired for the design of the micro scale interconnects:

- Inner Diameter of Innermost Ring - 400 um
- Tooth Height - 40 um
Outer Ring Geometry

- Tooth Width at Tip - 15 um
- Taper Angle - 7 degrees
- Percent Difference between Ring and Groove - 5%

However, only the parameters on the photo mask can be strictly defined. These include:

- Inner Diameter of Innermost Ring - 400 um
- Initial Thickness of Photoresist Rings on Top Coupler
- Initial Thickness of Photoresist Rings on Bottom Coupler

Tooth height is roughly the same as etch depth, and controlled by timing the etch process. It is controllable to within 5%. Taper angle is determined by the ratio of anisotropic to isotropic etching and controlled by the chemical concentrations (which product isotropic etching) and the RF power which accelerates ions at the substrate (and produces anisotropic etching). It is controllable to within 5 degrees. However, the percent difference between ring and groove thickness is a function of two unknowns: the final thickness of one set of teeth (or rings) at the top and the final thickness of the grooves on the other set of teeth (or rings). Since this parameters is what determines the horizontal compressive forces on the ring sidewalls which in turn determines the quality of the fluid seal created, it could not be left to chance.

Figure 7-46: Ring Parameterization Schematic

The initial photoresist ring thickness for the bottom chip of the interconnect pair (A) was set to 15, 20, 25, and 30 um (figure 7.2). The initial photoresist ring thickness for the top chip of the interconnect pair (B) was set to the bottom ring thickness plus -4, -2, -1, 0, 1, 2 and 4 um. This resulted in 28 pairs of per wafer with the maximum probability of having at least one interconnect pair with the correct geometry to create a fluid seal.

7.2.2 Outer Ring Geometry

Unlike the macro scale interconnects, the micro scale version are too small to be manipulated by hand so they had to be mounted on a chip that was large enough to be manipulated by hand. A chip size of 1 cm x 1 cm was chosen to allow all 28 pairs of chips to fit on a 4” silicon wafer and still be manageable.
The process to create the concentric rings involves removing all of the silicon around the rings. The rings themselves occupy very little area compared to a square centimeter, so removing all of the silicon on the wafer surface except the sealing rings would create a huge load on the etching reactants and on the plasma, slowing the etch rate and causing problems with etch uniformity. To minimize the plasma loading, an outer set of rings was added to the design. Each outer ring covers 47% of its 1 cm x 1 cm chip surface, reducing the plasma loading by almost half. There is 200 μm between the outer diameter of the outer ring of the bottom chip and the inner diameter of the outer ring of the top chip to prevent these features from interfering with the inner rings.

**Figure 7-47: Solid Model of Micro Scale Interconnect Chip Layout**

The outer rings also have a structural function on the interconnect chips. The inner rings of the interconnect are nominally 40 μm tall. Since the two sets of rings cannot mate completely, the distance between the two faces of the chips will be between 40 and 50 microns apart. Silicon, like many other brittle materials, is very strong in compression so if the chips are loaded at or near the inner rings, the devices should be able to withstand relatively high forces. However, if the outer rings were not present and the chips were loaded at the edges far away from the support of the inner rings, the chips could deflect the full distance between the chips before reaching the support of the other chip (figure 7.4). Since this applies a tensile load to the chip until it reaches a support, it is possible that the chip would break.

**Figure 7-48: Loading of Interconnect Chips With (right) and Without (left) Outer Rings**
The outer rings prevent the chip from deflecting more than 5 or 10 microns before again becoming a compressive load and thus reduce the possibility of breaking the devices during testing and use. A detailed cross section of an assembled micro scale concentric ring interconnect is shown in figure 7.5.

![Figure 7-49: Detailed Cross Section of Final Micro Scale Interconnect](image)

7.3 Design of Micro Scale Testing Procedure

7.3.1 Testing Geometry

Although the halves of the final interconnects will have through holes to allow fluid to pass from one device to another, the test devices to show that the interconnects work do not. As with the macro scale models, the micro scale concentric ring interconnects will be testing by supplying pressurized air into one half of the interconnect, submerging the interconnect in water and looking for bubbles. For this reason, only the top halves of the test interconnects have through holes in them.
7.3.2 Alignment Device

Although the interconnect was designed to be self-aligning, both sets of rings on the devices have to get close enough to drop into place. To help facilitate this initial alignment, a kinematic alignment jig was designed.

The basic principle behind the kinematic alignment jig is that all 6 degrees of freedom of the interconnect chips need to be perfectly constrained in a very repeatable manner. The chip is placed on a sloped plane (either a surface or a plane defined by three points). This prevents the chip from rotating into the resting plane (2 rotational degrees of freedom) or traveling through the resting plane (1 translational degree of freedom). The other three degrees of freedom are constrained by placing three posts along the bottom edges of the chip. The post on the bottom prevents the chip from sliding down, while the pre-load from gravity (resulting from the sloped plane) prevents the chip from moving up (1 translational degree of freedom). The other two posts prevent the chip from moving sideways and from rotating in the resting plane (1 translation and 1 rotational DOF). Since the chip touches the alignment jig at three points on the resting plane and three lines on the posts, it cannot be over constrained and the chip should fall into the same place every time.

The configuration is relatively stable because both instant centers of rotation created by the placement of the posts are above the center of gravity of the chip (figure 7.7). If the posts were placed such that one or both instant centers of rotation were below the center of gravity, small perturbations could dislodge the chip from its resting location.

It was assumed that once the chips were nominally aligned using the jig, gently tapping the device would cause the rings to shift enough to drop into place.
The actual alignment jig was made from a small piece of aluminum L-extrusion. Three ball bearings 1/16” in diameter were placed on the surface on the aluminum to create the resting plane for the chips. Three 1/6” diameter steel rods were used for the alignment posts. Since the devices require a pre-load to seal and mated pairs may need to be moved from microscope to microscope without losing the alignment, a T-shaped clamping device was also added. The clamp is made from 1/8” thick aluminum sheet with indentations removed to allow the alignment posts to pass through. The clamp is attached with screws using threaded holes in the aluminum extrusion.

7.3.3 Alignment Geometry

Although the alignment jig should work for all square devices, there was a concern that the chips could still slip from their alignment posts if jostled and that the surface finish caused by separating the devices from their original wafer could cause unacceptable variations in the alignment procedure. To eliminate both of these concerns, alignment features were also added to the chip design. Each chip needed exactly three rectangular areas to be removed from two sides of the chip. However, since one chip sits on the jig face up and the other face down, a fourth area was needed to ensure both chips would
connect with all three posts. These areas were to be removed using deep reactive ion etching (DRIE) which produced much better surfaces than using more common tools like the die saw.

![Alignment Feature Schematic](image)

**Figure 7-53: Alignment Feature Schematic**

### 7.4 Micro Fabrication

The fabrication of the micro scale components can be separated into two basic categories: the creation of shallow features and the creation of deep through features. The inner and outer rings that make up the portion of the interconnect that creates the fluid seal are relatively shallow etches (~ 40 um) and can be done using the LAM490B, a reactive ion etching machine. However, the fluid manifold and the alignment features on the edges of the chips must be done using STS2, a deep reactive ion etching machine. Because these features are created using different machines and at different times in the fabrication process, they can be divided into two photomasks.

#### 7.4.1 First Mask Design

The first photomask contains 52 chips; 26 pairs of concentric ring coupler halves defined by a 1 cm x 1 cm perimeter and outer and inner ring pairs. In the upper left hand corner of each chip there is a letter and a series of numbers which indicate whether it is a top or bottom half of a coupler and which parameterized pair it belongs to. For example, B26 is the bottom half of coupler pair 26 while T26 is the top half for the same coupler. In the center of the mask on the left and right sides, there are two black squares. These are the mask alignment marks which allow subsequent masks to be aligned to features on the wafer surface. Although it is not visible, there are tiny white lines that form a cross in the center of the squares. The outer half of each of the lines that form the cross are 20 um thick while the inner half of each of the lines is 10 um thick. The location of the alignment marks is dictated by the machine that performs the UV exposure and cannot be changed. For this mask, the alignment marks fell directly on top of two pairs of chips which had to be removed so the alignment marks could take their place. This is the reason that 26 pairs, instead of the 28 originally envisioned.
The chips are laid out on the mask so that each chip is as close as possible to its corresponding mate. For example, chip T1 is the first chip in the upper left hand corner of the mask. Chip B1 is directly below it. Chip pairs were kept together so that etching variations across the wafer would not prevent interconnect pairs from mating properly.

The first photo mask is a positive photomask and areas in black represent regions protected by photoresist.

**Figure 7-54: First Photo Mask**
7.4.1.1 Second Mask Design

The second photo mask contains circles 200 um in diameter for the top chips to create the fluid inlets and three rectangles per chip to create the alignment features for use with the alignment jig.

The photomask shown in figure 11 is an early version of the photo mask that was never made. A new version of the photomask will be designed in the future with alignment marks. Each chip will also have a thin border around it so the chips can be separated from their wafer during the through etch instead of having to use the die saw after fabrication is complete. This will save time, money, create more accurate chip surfaces and prevent possible die saw damage.

The second photo mask is a negative mask and the black areas represent areas where there will not be photoresist.
7.4.2 Process Plan

As indicated above, the second photo mask was never used. Micro fabrication focused primarily on creating the inner concentric rings with a uniform taper and excellent surface finish so the process plan includes micro fabrication steps up to but no further than the etching step for the concentric rings.

The process begins with pristine 6" single-side polished silicon wafers. Since the interconnects are purely mechanical devices, the doping is unimportant. The wafers are cleaned in a three step process called an RCA clean and then placed in an oven for 7 hours to grow 1.5 um of silicon dioxide on the wafer surface. The wafers are then cleaned again.
and placed into Concept 1 where another 1.5 um of oxide is deposited. The wafers are then coated with HMDS (a photoresist adhesion promoter), coated with photoresist, baked for a half an hour, exposed, developed and baked again to cure the resist. The wafers are then placed in the AME5000 which etches the silicon dioxide, creating a hard mask for the concentric rings. The photoresist is then stripped in the asher and cleaned again. Finally, the wafers are placed in the LAM490B which etches the concentric rings. An optional cleaning etch can be done in either the LAM480B (reaction ion etch) or STS2 (deep reactive ion etch) to improve the surface finish of the rings.

References

Chapter 8: First Stage Micro Fabrication Results

The goals for the first stage of micro fabrication were to create the tapered sidewalls for the concentric rings, ensure a good surface finish on those sidewalls, ensure that the rings fit together, and determine if the interconnects are self-aligning. Only when all of these goals were met would the second stage of micro fabrication to create pressurized test devices begin.

8.1 Recipe Optimization

The concentric ring etch uses a modified version of the Deep Silicon etch on the LAM490B. This recipe uses 96 sccm of chlorine gas and 134 sccm of helium gas at 400 mTorr of pressure and 200 W of RF power with a 0.5 cm electrode spacing. The recipe has three main steps: a 30 second stability step, an etch step, and a cool step with the same parameters as the etch step but without the chlorine gas or the RF power. The chiller was set to hold the temperature in the etching chamber at 15°C.

Previous users had reported that this recipe used with a 6 minute etch step had an etch rate of 0.5 um/sec, produced a five degree sidewall taper and taper uniformity within 2%. The selectivity of the silicon dioxide to silicon was roughly 40:1.

The selectivity of silicon dioxide to silicon is relatively stable at temperatures below 15°C, but that selectivity starts dropping rapidly above 15°C. The longer the etch cycle, the greater the temperature increase in the etching chamber. For this reason, the concentric ring recipe kept all the parameters the same but the etch step was shortened to 4 minutes with a 2 minute cooling cycle in between. It takes just over two hours to etch 40 um.

The LAM490B is a reactive ion etching machine (RIE) and is intended for relatively shallow etches. The deepest features that had been etched in the machine were 25 um deep. So the creation of 40 um tall rings was known to be pushing the limits of the machine.

8.1.1 First Iteration: 15C LAM Etch

The first attempt to etch the concentric rings, the wafer used a 1um thick oxide mask. The chiller temperature was left at 15°C but the temperature in the etching chamber climbed to or above 16.5°C by the end of the etch steps. No photographs of this iteration are available because the oxide mask on the wafer had been completely etched away before the wafer was even halfway through its etch. After the mask was gone, there was no hope of obtaining the desired geometry and the wafer was discarded.
To determine the cause of the mask failure, another wafer was etched for five cycles (instead of the full twenty). Measurements showed that for the concentric ring masks, the selectivity was closer to 20:1 and the etch rate was just under 0.5 um/sec.

8.1.2 Second Iteration: 10C LAM Etch

For the second iteration, a combination of 1.5 um of thermally grown oxide and 1.5 um of deposited oxide were used as the hard mask. The chiller temperature was also set to 10°C to prevent the temperature dependent selectivity variation. After the etch, the wafer is separated into chips using the die saw. The chips are washed using acetone, methanol and isopropanol and then examined under optical and scanning electron microscopes.

Figure 8.1 shows a cross section of the bottom half of pair #1. The rings are 36.8 um tall with a taper angle of 9.1 degrees. The rings are 7.3 um thick at the tip and 19.1 um thick at the base. The shape of the rings are very good, but the surface finish is not ideal. There seem to be three regions of roughness on the sidewalls. At the top, there is a thin ring that seems to be under etched. In the middle, the surface finish looks very smooth. At the bottom, there is a region of vertical extrusions. It was unclear if the surface roughness present was a function of the etching or if it was a result of using the die saw to cut the chip in half. More samples were needed to determine the source of the roughness so it could be eliminated in future devices.

Figure 8-57: SEM Image of Second Iteration

Figure 8.2 shows sections of the top (left) and bottom (right) halves of chip pair #19 taken with a 5x power optical microscope. Polished silicon is an excellent reflector and all smooth areas on the wafer surface appear to be perfectly white. The dark areas on the chip are roughened areas filled with tiny silicon cones. They scatter the light and thus appear to be black which is why areas with large concentrations of these features are referred to as “black silicon.” A close up view of these cones can be seen in the lower lefthand corner of figure 8.1 where the outer ring meets the surface of the substrate.
Third Iteration: 5C LAM Etch

![Optical Microscope Images T19 and B19](image)

**Figure 8-58: Optical Microscope Images T19 and B19**

These unwanted silicon protrusions could prevent the two halves of the interconnect from mating fully and creating a fluid seal. The next iteration of devices were devoted to finding a way to improving the surface finish of the rings and eliminating the black silicon.

### 8.1.3 Third Iteration: 5C LAM Etch

The third and all subsequent iterations also used a 3 um thick thermal/deposited oxide hard mask. The etch recipe was the same as the previous iterations, however the chiller temperature was set to 5°C. The etched wafer was again separated into chips using the die saw after the etching step. However, this time only half of the chips were cut in half with the die saw. The other half were left whole to be sectioned using a diamond tipped scribe. The chips were again washed using acetone, methanol and isopropanol and then examined under optical and scanning electron microscopes.

Although the only difference between the second and third iterations is a 5°C decrease in temperature, the black silicon in the third iteration seems to be almost as tall as the rings, where before it was only roughly one quarter as tall. There also seems to be more of it. It is impossible to tell if the surface finish of the rings has changed because the view is blocked by the silicon stalactites protruding from the substrate surface.

The silicon dioxide hard mask can be seen as a thin white layer on top of the rings in the right hand image in figure 8.3. There is no indication that this mask is being undercut and the source for the top layer of roughness on the second iteration of rings is still unclear.
Fourth Iteration: LAM Etch with LAM Cleaning

The cross section that was created using the die saw was compared to the cross section that was created using the diamond tipped scribe to cleave the wafer (not shown). The die saw can distort the actual cross section of the rings and scatter debris onto the ring surfaces. So it is still possible that the surface roughness seen in the second iteration was at least partially due to the die saw.

8.1.4 Fourth Iteration: LAM Etch with LAM Cleaning

Decreasing the etch temperature clearly did not improve the surface finish or decrease the size or quantity of the black silicon, so the next two iterations focused on ways to remove the black silicon after it had formed and improve the surface finish.

The fourth iteration followed the same etching procedure as before (3 μm hard mask, chiller temperature of 5°C), but received a secondary etching treatment after the creation of the concentric rings. This treatment involved a 90 second isotropic etch in the LAM490B using 190 sccm SF₆ and 19 sccm O₂ at 300 mTorr of pressure and 130 Watts.

Figure 8.4 shows that this cleaning etch was very successful in reducing the size and quantity of black silicon while preserving the uniform taper on the ring sidewalls. This is more easily seen in the right ring of the cross section SEM picture because much of the surface that was damaged by the die saw has actually broken off to reveal the natural shape beneath. The surface finish, while not ideal, has also been much improved.
While the cleaning etch in the LAM is very promising, it did show some odd directional behavior. Figure 8.5 shows top views of chips T21 (left) and B21 (middle) from an optical microscope. Notice that in both photographs there are four white areas between the rings at approximately 45 degrees from vertical. A close-up of these areas is shown at the left of figure 8.5.

It is unknown if the black silicon is simply being removed faster along these two axes than elsewhere on the wafer or if the directional nature of the etch is actually changing the cross section of the rings. The etch should be perfectly isotropic, etching equally in all areas, so there is no good explanation of why this phenomenon is occurring or whether or not it will adversely impact the functionality of the interconnects.
8.1.5 Fifth Iteration: LAM Etch with STS Cleaning

The fifth iteration followed the same etching procedure as before, but received a secondary etching treatment after the creation of the concentric rings. This treatment involved a 15 second isotropic etch in STS2, a deep reactive ion etching machine, using SF$_6$ and O$_2$ chemistry.

Figure 8.6 shows that while this cleaning etch is successful in decreasing the size and quantity of the silicon protrusions, it is less successful than the cleaning etch performed in the LAM490B. Both the die sawed and cleaved cross sections also indicate that the STS cleaning etch causes a slight decrease in the taper angle of the rings near the tip. Although this may not be enough to prevent the rings from sealing, a more uniform taper is still preferable.

8.1.6 Seventh Iteration: Deep Reactive Ion Etching

The last iteration was an attempt to create the tapered sidewalls using a modified shallow etch repose for STS2, a deep reactive ion etching (DRIE) machine. The wafer was etched for 26 minutes and followed by a 15 second SF$_6$ cleaning etch.

Although this recipe created smooth sidewalls and no black silicon, it also did not create a detectable taper on the ring sidewalls.
8.2 Interconnect Assembly

The next step was to determine if the halves of the interconnects could be assembled and if they were self aligning. Since the devices created using the fourth recipe iteration (LAM etch with LAM cleaning) showed the best sidewall profiles and the least amount of black silicon, those devices were used for this purpose.

The concentric ring interconnects were designed to be self-aligning without the use of jigs or other alignment devices. Two chips, when put together and moved gently in the palm of ones hand, will eventually drop into place. However, the chips can still move relative to each other after you have felt the drop. This is because the outer rings of the chips with 200 um of clearance between the two have dropped into place but the smaller, inner rings have not.

This procedure can be repeated using the alignment jig discussed in the previous chapter. Again, gently tapping the jig will cause the outer rings to drop into place, but the inner rings will not drop.

The reason for this is unclear. It is possible that the tolerance of the rings is so small, that they have to be aligned before they can drop. It seems more likely, however, that there is something on which the rings are resting which prevents them from dropping into place as they pass over each other.
Since the rings would not align themselves, the rings were aligned manually using a small screwdriver to manipulate them while looking at the cross section of the pair through an optical microscope. Once the rings were relatively well aligned, pre-load was applied by pushing on the outer surface of the top coupler with the screwdriver as hard as possible. (The clamp of the alignment jig was unable to apply enough pre-load to force the interconnects together.)

The assembled interconnect is shown in figure 8.9. Optical microscopes have a very shallow depth of field so only objects in the same plane will appear in focus. In this case, the bottom chip is in focus, however the front surface of the top chip is further back than the front of the bottom chip so it appears blurry. This is also why the rings appear to be misaligned, even though they are more than half engaged. When the pre-load was removed, the rings separated immediately.

It can be concluded from this that in their current state, the interconnects are not self-aligning and cannot be held together by friction, but are much more resistant to compressive loading than expected.
Chapter 9: Conclusion

9.1 Conclusions

It is too early in this work to be able to usefully draw any conclusions about whether or not the interconnects will function on the micro scale as they were designed. However, it has been shown that the concentric ring interconnects function on the macro scale as they were designed. The micro scale geometry, while not yet perfected, can be fabricated. The interlocking rings, while not yet self-aligning, do align, and while they may not seal, they do engage. Moreover, they have been shown to be much stronger than anticipated. Perhaps most importantly, we conclude that none of the work done up to this point indicates that the interconnects will not function as designed and much of it points to future success.

9.2 Future Work

In the short term, work will continue on optimizing the micro fabrication process for the concentric ring features by reducing or eliminating the black silicon and improving the surface finish. Once this geometry has been optimized, the second mask with the testing and alignment features will be redesigned to eliminate the need for the die saw and test interconnects will be made.

At the same time, work will continue to find out why the interconnects are not self-aligning and to redesign them if necessary to ensure that they are self-aligning.

If the attempts to create a single, fully functional concentric ring interconnect are successful, work will begin on design and fabrication of single micro scale KOH fluid interconnects and arrays of concentric ring interconnects for a Ph.D. project.
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