Passive Microfluidic Interconnects

by

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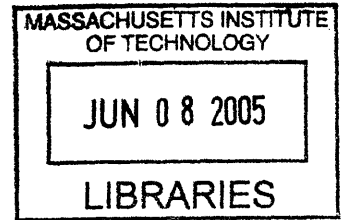
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ABSTRACT

Equipment and procedures were developed to test two passive microfluidic interconnect designs. The first design forms fluid seals by means of interlocking tapered concentric rings held together by the friction forces on the contact surfaces. The second design forms fluid seals by means of thin flared rings of compliant material being compressed into undersized sockets. Interconnects were tested in pairs and arrays. The sealing performance of the first design was found to be highly dependent on the material and surface finish of the features, and the design was found to be largely intolerant to the misalignment inherent in arrays. The second design successfully seals up to 150 psi and is capable of compensating for misalignment to seal in arrays of six interconnects.

Thesis Supervisor: Alexander H. Slocum
Title: Professor of Mechanical Engineering

Acknowledgements

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1   Introduction

This thesis describes the testing of a new type of fluidic seal developed by Mary Kathryn Thompson for her Masters Thesis. Based on the results of the tests, a second generation fluid seal was designed and tested.

Micro Electro Mechanical Systems (MEMS) are used in chemical and biological applications to exploit the benefits of working on the micro scale.[1] The high surface area to volume ratios of the MEMS translate into lower values of space requirement, utility consumption, waste production and power dissipation. Additionally, the high heat and mass transfer rates result in higher yields of products and the setup permits more aggressive and uniform temperature conditions. Another advantage is that the use of toxic chemicals can be minimized because of the small volumes, while safety concerns are reduced by point-of-use of harmful reaction intermediates.

However, providing fluidic connections between MEMS systems and the macro scale world easily and effectively remains a challenge. This is especially true of the microfluidic sealing systems that require non-permanent seals. Ideally, microfluidic ports would be easily connected and disconnected by hand (like LEGO™ blocks), and form fluidic seals without the requirement for clamping forces. Controlling the magnitude and concentration of clamping forces is difficult. Since MEMS devices are small, delicate, and constructed using brittle materials such as silicon, application of clamping forces involves high risks of shattering, chipping, or otherwise damaging the devices. Interconnects for MEMS devices must also have a small footprint and cause minimal design disruption. The sealing features should also be easy to manufacture, and work reliably and repeatably.[2]

Microreactors are a class of MEMS devices that are particularly challenging with respect to reliable and easy to use interconnecting systems. This is because, in addition to the geometric constraints, the interconnects must be chemically inert as well as capable of withstanding high temperature and pressure. Typically, the interconnects must be able to withstand pressures up to 50 atmospheres and temperatures up to 500 degrees Celsius[3]. Another requirement is that the interconnect material must resist corrosion from the chemicals in the system, and must be non-reactive so as to not poison the reactions.

These requirements preclude the use of a large range of materials that are commonly used to fabricate fluid seals.

In order to address the problem of connecting microreactor ports, two designs for reversible fluid sealing mechanisms were developed. The first of these designs, referred to as the *Concentric Ring* interconnect design, was developed by Mary Kathryn Thompson for her Master's thesis at MIT. The second design, referred to as the *Flared Ring* interconnect design, was developed together with Mary Kathryn Thompson. Testing procedures were developed to assess the strength of the fluid seals formed by these two interconnect designs and testing fixtures built to conduct the tests. The results from a series of tests conducted on various models of the two interconnect designs were analyzed and used to refine the designs of the interconnects.

2 Theory

The creation of fluid seals involves blocking potential paths for the fluid to escape from the system. Fluid seals can be created either along a line or along a plane. Every surface has some roughness, comprising a landscape of peaks and valleys rather than a smooth flat plane. When two surfaces are brought into contact, fluid can flow into the small gaps where the peaks of one surface do not completely fill the valleys in the other. Such gaps form channels through which the fluid can flow and leak out through the sealing surface. Though it is difficult to completely eliminate such leakage from seals, the aim of seal design is to minimize this leakage rate.

Applying pressure to compress the sealing surfaces together will cause the material to deform elastically and fill up the fluid channels. However, if the surface roughness is too high, the material may not be able to elastically deform sufficiently to completely block the channels, or the pressure required to obtain such deformation might be so high as to be impractical.

Fluid seals can be obtained by placing intermediate layers of compliant material between sealing surfaces that do not seal elastically. The intermediate layers in such cases are called gaskets. Gaskets are made of highly elastic material. They conform to the sealing surfaces at low values of compressive pressure to form fluid seals. Since most gaskets are made of elastomers, they have low resistance to heat and corrosive chemicals. This is unacceptable for use in microreactors. A compliant yet inert material (for example, gold) must be chosen for gasket seals in applications involving high temperatures and chemical exposure. Alternatively, the surface finish of the sealing surfaces can be enhanced in order to reduce the leakage rate.

Improving the surface finish of surfaces greatly reduces the number of potential paths for fluid leakage. The average height of the peaks and valleys on the material surface is decreased, and hence the material can block fluid channels with less elastic deformation. This means that less compressive pressure is required to form a fluid seal. The compressive pressure required to obtain a fluid seal is related to the surface finish of the sealing surfaces up until a mirror finish quality is obtained. At a mirror finish, atomic contact is achieved between the surfaces and van der Waals forces dominate the

interaction. (These are the interactions used by geckos climbing up walls.) Thus the seal is almost as good as a weld because the two planes are in atomic contact. However, if only an edge and plane are in contact, in the case known as a knife edge seal, the parts will seal and yet not bond to each other.

As the surface finish of the contact surfaces is a crucial factor in determining the efficacy of the seal, it is imperative that the sealing surfaces have the best possible surface finish. The manufacturing technique employed to fabricate pieces greatly influences the surface finish of the product. For example, turning or boring a feature on a lathe results in a better (average as well as best case) surface finish than milling on a milling machine.[4]

Parts can also be processed after manufacture in order to improve the surface finish of critical surfaces. Lapping is a process that can be used to improve metal surface finish. The two mating parts are brought into contact and then turned so both surfaces are worn down to become smoother and conform more closely to the shape of the mating part. A lapping compound can be added on the surfaces to aid this process. The lapping compound is an abrasive that makes wearing down the high points on the surfaces easier. The ultimate surface finish achieved depends on the grade of the lapping compound used.[5]

Other than leakage due to fluid channels in the sealing surfaces or due to corrosion of the sealing material, fluid seals are always imperfect due to diffusion through the sealing material. While diffusion is inevitable, it is also negligible because the rate is very low, and will be ignored for the rest of the discussion.

3   Concentric Ring Interconnect Background

This section provides information necessary to understand the background for the development of the interconnects tested. Sections 3.1 through 3.3 describe work done by Mary Kathryn Thompson for her Master's thesis. Sections 3.4 through 3.5 describe work that she and I did over the summer of 2004. These details are included solely for the completeness of this document.

The Concentric Ring interconnect design was developed to create a fluid seal between two microfluidic ports. The testing of macro and micro scale models yielded positive results for the sealing capacity of the design, and indicated that working on the meso scale would be the most effective way to convert the design into a product.

3.1   Design description

The Concentric Ring interconnects seal by means of sets of interlocking tapered rings. The features are designed to be manufactured on the surface of the substrate with the fluid ports. The concentric rings have a trapezoidal cross section, and the spacing of the rings creates grooves for the rings on the mating part to fit into. When fluid ports surrounded by sets of mating concentric rings are brought in contact, the tapered surfaces of the rings form fluid seals. The features are held together by the friction forces between the contact surfaces. A schematic of the cross-section view of the concentric rings sealing is shown in Figure 1.
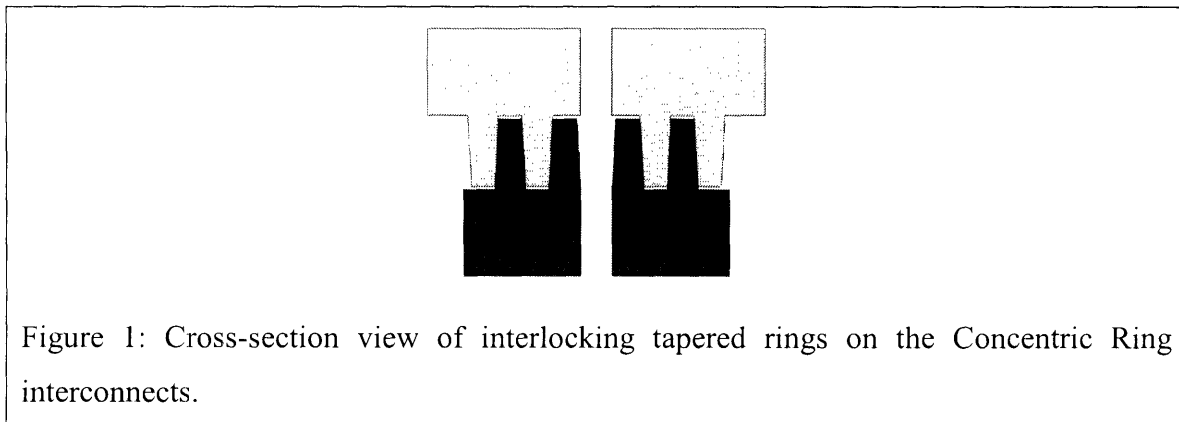
Figure 1: Cross-section view of interlocking tapered rings on the Concentric Ring interconnects.

The variables that control the geometry of the cross-section of the rings (also referred to as teeth) are the average width of the teeth, the taper angle of the teeth, the ratio of the height to average width of the teeth, and the overlap percentage between the teeth and the corresponding grooves. These parameters are shown in Figure 2.

Figure 2: Parameters that affect the geometry of the concentric rings.
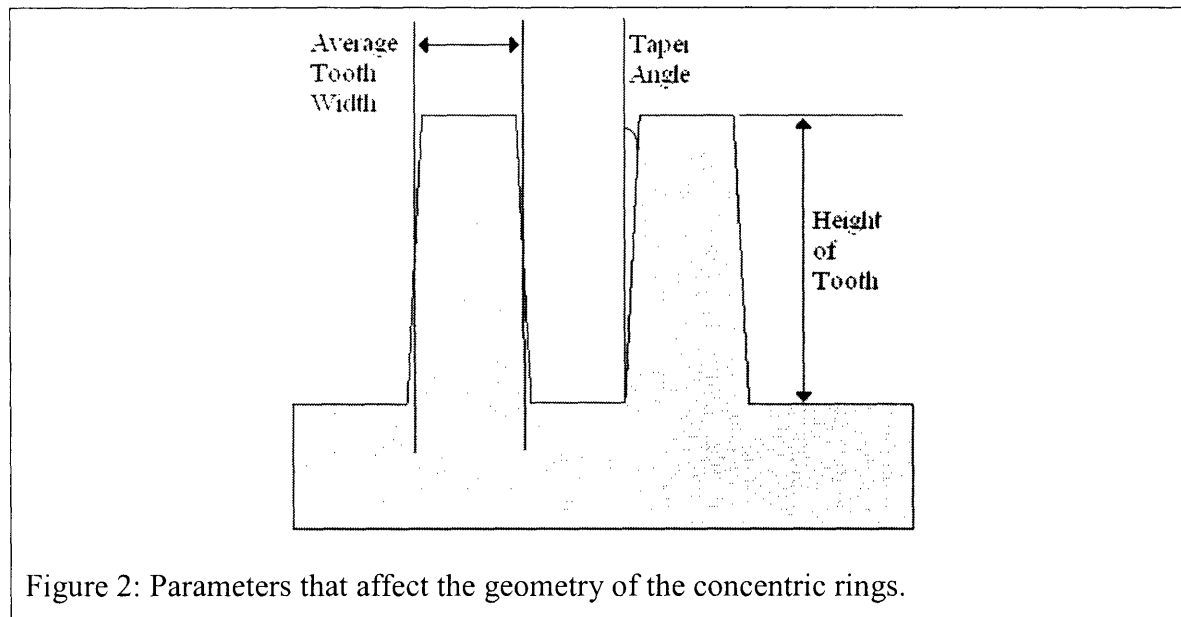
It is important that the fluid seal is formed along the tapered surfaces, so that the surface finish of the bottom of the groove does not matter for the seal to be effective. In order to ensure that the tapered surfaces are the sealing surfaces, the top of the teeth must not contact the bottom of the grooves. Hence the teeth are constructed to be slightly oversize for the corresponding groove. The percentage by which the width of the top of the tooth exceeds the width of the bottom of the groove is referred to as the overlap percentage.

Another important consideration in the design is the tolerance of the interconnects to misalignment between the mating parts. This is the capability of the features to flex and form a fluid seal when the two fluid ports are not coaxial. The rings of the interconnects are like axisymmetric cantilevered beams. They are extremely strong in compression but not in tension or shear. The lower the average tooth width, the thinner and more flexible the beams are. If the taper angle is high, the teeth are stiffer because the

base width is high compared to the width at the top of the tooth. Finally, if the height to average width ratio of the teeth is high, the teeth are more flexible. A ring that is too stiff may be unable to compensate for manufacturing misalignment. However, if a ring is too flexible it may tend to deflect excessively and break off at the base. A balance between these two extremes must be found for the interconnects to work successfully.

The concentric rings must be self aligning, and easy to align by hand. The ease of alignment depends on the difference between the width at the top of the tooth and the width at the top of its mating groove. This distance is controlled by the taper, aspect ratio of the teeth, and average tooth width. The overlap percentage is essential for proper sealing, but takes away from the extent to which the interconnects are self-aligning; a trade-off must be made between these conflicting interests.

The taper angle of the teeth is an important factor for the locking properties of the rings. A "self-holding" taper is defined as the case "where the angle of taper is only 2 or 3 degrees", offering "considerable frictional resistance to any force tending to turn or rotate" the shank relative to the socket, and a "self-releasing" taper refers to a taper angle of "over 16 degrees".[6] A self-holding taper might make disassembly of the components difficult whereas a self-releasing taper offers negligible frictional resistance, which means that the interconnects would not hold together without a clamping force. A taper angle between these two extremes would be expected to enable the interconnects to hold together without clamping and yet remain amenable to easy disassembly.

Since MEMS devices are small, the interconnection features must also be small in order to accommodate the necessary concentration of fluid ports on a device. The inside diameter of the inner ring determines the footprint occupied by the interconnecting features, and must be minimized while keeping in mind the difficulty of machining microscale features.

A finite element model of the design in ANSYS predicted the optimum values of the tooth taper angle, height to average width aspect ratio, and overlap percentage to be 7 deg, 3:1, and 5% respectively. Varying the inside diameter of the inner ring while keeping the other parameters same can yield micro, meso, or macro scale sealing features.

3.2   Macro scale interconnects

A series of macro scale delrin interconnects of this design were built by Mary Kathryn Thompson to provide proof of concept of the sealing mechanism. Rings with a taper angle of 7 degrees and aspect ratio slightly lower than 3:1 were machined onto 2" diameter delrin cylinders by turning the pieces on a lathe. The delrin interconnects are pictured in Figure 3.

Figure 3: Delrin interconnects, single pair and in an array of six. Photo credit Mary Kathryn Thompson.
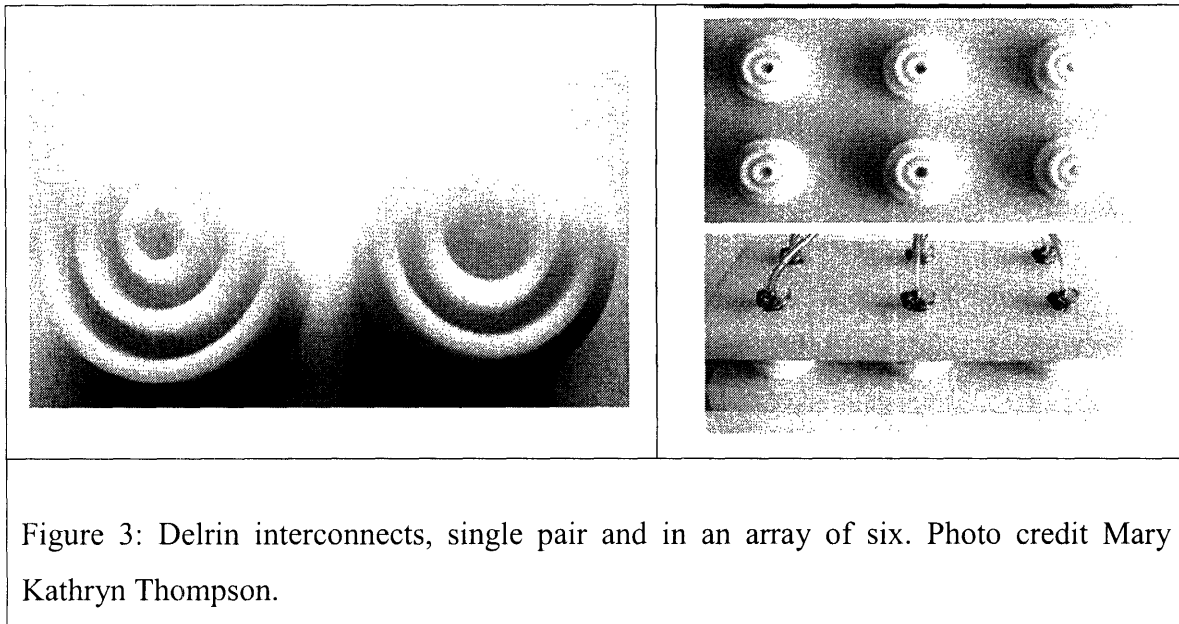
The interconnects were tested in single pairs and in an array of six pairs. The single pairs sealed up to 80 psi with clamps. The arrays were tested after preloading with an arbor press and clamping the pieces together. A four-unit interconnect array was able to seal up to 20 psi with no leaks, and a six-unit interconnect array had two units leaking at 20 psi and the remaining four units holding to 80 psi.[7]

The observation that four of the six interconnects in an array could seal indicated that the interconnects could cope with a little misalignment but not enough to seal in an array of six. It would be desirable to increase the tolerance to misalignment so that the interconnects can seal effectively when arranged in arrays of larger numbers.

Clamping the interconnects together in order to obtain a seal is undesirable as this can stress the material. However, delrin is a good bearing material, and is therefore not

suited to provide good friction seals. It was used to fabricate the units for the proof of concept of this sealing mechanism because of its availability rather than because of desirable material qualities. Thus the necessity to clamp the delrin interconnects was a consequence of the choice of the material rather than a reflection on the robustness of the design, and the results were encouraging enough to warrant future work with this design.

3.3   Micro scale interconnects

Micro scale Concentric Ring interconnects were also fabricated by Mary Kathryn Thompson. The concentric ring features were etched onto silicon wafer. The desired dimensions were as follows: inside diameter of the inner ring 400 μm; tooth height 40 μm; tooth width at the tip 15 μm; taper angle 7 degrees; and percent overlap 5%.

However, since only the parameters on the photo mask can be defined and the variations in the fabrication process are large, 28 interconnect pairs with varying ring thicknesses were machined per wafer to maximize the probability of at least one interconnect pair resulting in the correct geometry.

The resulting interconnect features were too small to align by hand and eye. An alignment jig was constructed to try to align the rings, but alignment still remained difficult. The features had to be aligned by hand while observing them under a microscope and a preload had to be applied in order to get the features to engage. The rings separated as soon as the preload was removed.

3.4   Flexible interconnects

In an attempt to address the inability of the delrin macro scale interconnects to seal in arrays, the design was modified to make the teeth more flexible. The trapezoidal cross section of the teeth causes stress concentrations at the corners of the base of the teeth. This makes the teeth tend to break off at that point instead of flexing to cope with misalignment. The addition of a radius on the base of the teeth (as shown in Figure 4) greatly increases the flexibility of the teeth.
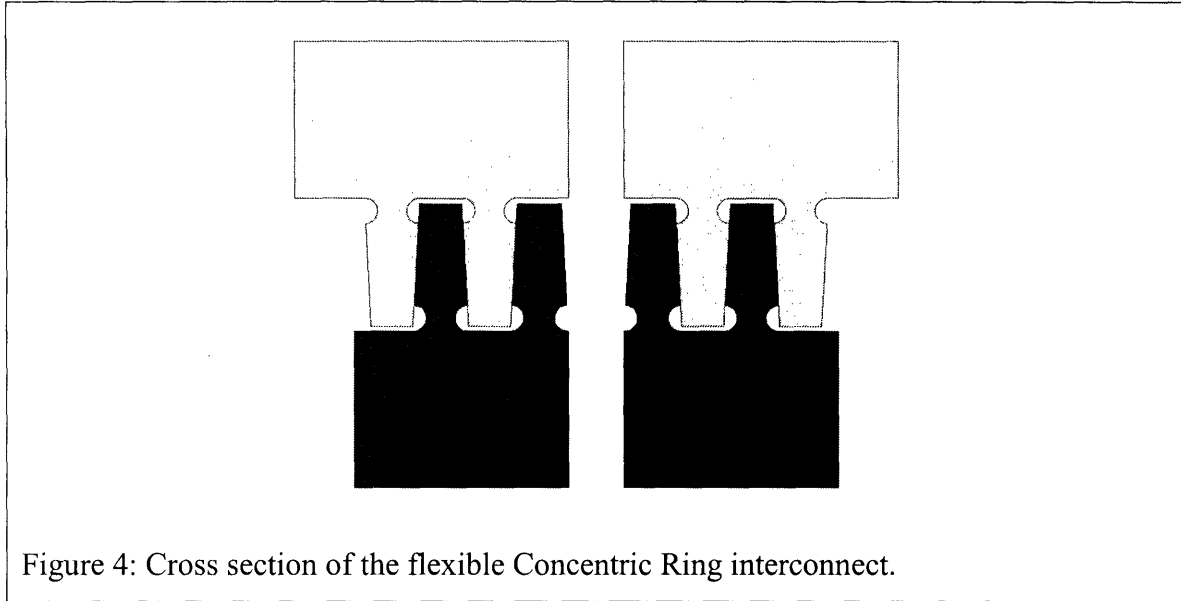
Figure 4: Cross section of the flexible Concentric Ring interconnect.

The delrin interconnects were modified by adding a radius of diameter 1/8th inch along the base of all the interconnects on one side of the array. When the array was clamped together and tested, all six interconnects sealed to 80 psi (which was the limit of the pressurized air supply we had at the time). This suggests that adding a radius to the base of the teeth does in fact allow them to flex enough to adjust for misalignment and seal in arrays of six interconnects.

3.5   Conclusions

The conclusion from the observations of the Concentric Ring interconnects was that even if the design of micro scale interconnects was further refined and the units tested and proven to work, they would still be unviable in commercial applications due to the difficulty in aligning the features. In addition, fabrication of such features in silicon is expensive, and incorporating these features on the surface would require redesigning the masks for the original microreactor systems.

It became clear that usability of the features was a key factor to ensure their success as a product. Therefore, the construction of meso scale interconnects was planned. These would be small enough to fit around fluid ports and yet large enough to see and align by hand. In addition, these could be machined by conventional macro

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machining, which is cheaper, easier and more reliable than micro fabrication. The project now focused on scaling the design to the meso scale, creating meso scale interconnects and testing their viability as sealing features for MEMS fluid ports.

4   Concentric Ring Interconnect Tests

Simultaneously with the construction of meso scale interconnects, plans were developed for effective testing of the interconnects when they were completed. The testing objectives were determined and procedures were designed to ensure that these objectives would be met.

4.1   Testing objectives

The purpose of testing the Concentric Ring interconnects was to understand their performance in terms of maximum seal pressure and repeatability of seals as a function of the preload applied and force required to pull them apart. Tests were designed to determine:

a. Minimum preload for interconnects to seal to a pre-determined fluid pressure
b. Relationship between preload applied and seal pressure (if any)
c. Force required to pull sealed interconnects apart
d. Repeatability of seals
e. Leakage rates of interconnects

In the context of developing viable designs for quick connection and disconnection of the ports of microreactors, the preload to be applied is crucial. Lower preloads are preferred as this will minimize the risk of damaging fragile silicon microreactors. One of the major goals of the designs for the interconnects is that the units be preloadable by hand. Hence it is important to determine the minimum preload required to ensure a reliable seal. To ensure that the interconnects are reversible, it is also important for disassembly to be easy, and so the force required to disassemble the interconnects must also be determined.

The design of the interconnects depends on friction forces between the contact surfaces to hold the two units together. Thus, it is possible that there is a threshold preload value that is necessary to engage the features, and the pressure that the seal can hold does not directly depend on the preload applied beyond that threshold value. However, if the preload applied determines the eventual seal pressure, then the

relationship between these two factors, the preload applied and the seal pressure, must be understood to enable the design of sealing features appropriate for the intended application.

The formation of a fluid seal by the interconnecting features must be repeatable so that they survive multiple seal/disconnect cycles. This means that testing the interconnects for a number of seal/disconnect cycles is important to make sure that the seals produced are repeatable.

Once the above data are collected, comparison across material, aspect ratio and taper angle can yield an understanding of parameters to be used:

a. Material – Aluminum Vs Stainless Steel vs. Titanium Nitride coated Steel

b. Height:Width ratio of teeth – 3:1 vs. 5:1

c. Taper Angle of teeth – 3 vs. 5 vs. 7 degrees

The optimal combination of all these factors can then be used to design a unit that requires the minimum preload for the maximum seal pressure held, subject to the constraint that the interconnects be easy to preload and disassemble by hand.

4.2   Testing Procedures

Testing procedures were developed to ensure that the interconnects were tested in an effective and efficient manner to gather the data necessary to gage their ability to seal against pressurized fluid.

4.2.1   Test for Fluid Seal

A bubble test with a pressurized nitrogen source served as a quick go/no-go gate to determine whether the interconnects held a fluid seal. The procedure for the bubble test is:

1. Fit interconnects with pipe fittings

2. Apply preload to interconnects

3. Attach interconnects to tubing from nitrogen cylinder

4. Immerse interconnects in water

5. Increase pressure and observe for formation of bubbles
6. Note pressure at which bubbles form

A pressure gauge was connected to the end of the tubing where the interconnects would be attached for subsequent testing. The pressure drop in the tubing was measured and a correction factor was used to determine the sealing pressure of the interconnects. A picture of the bubble test setup is shown in Figure 5.

Figure 5: Picture of the experimental setup for bubble test.
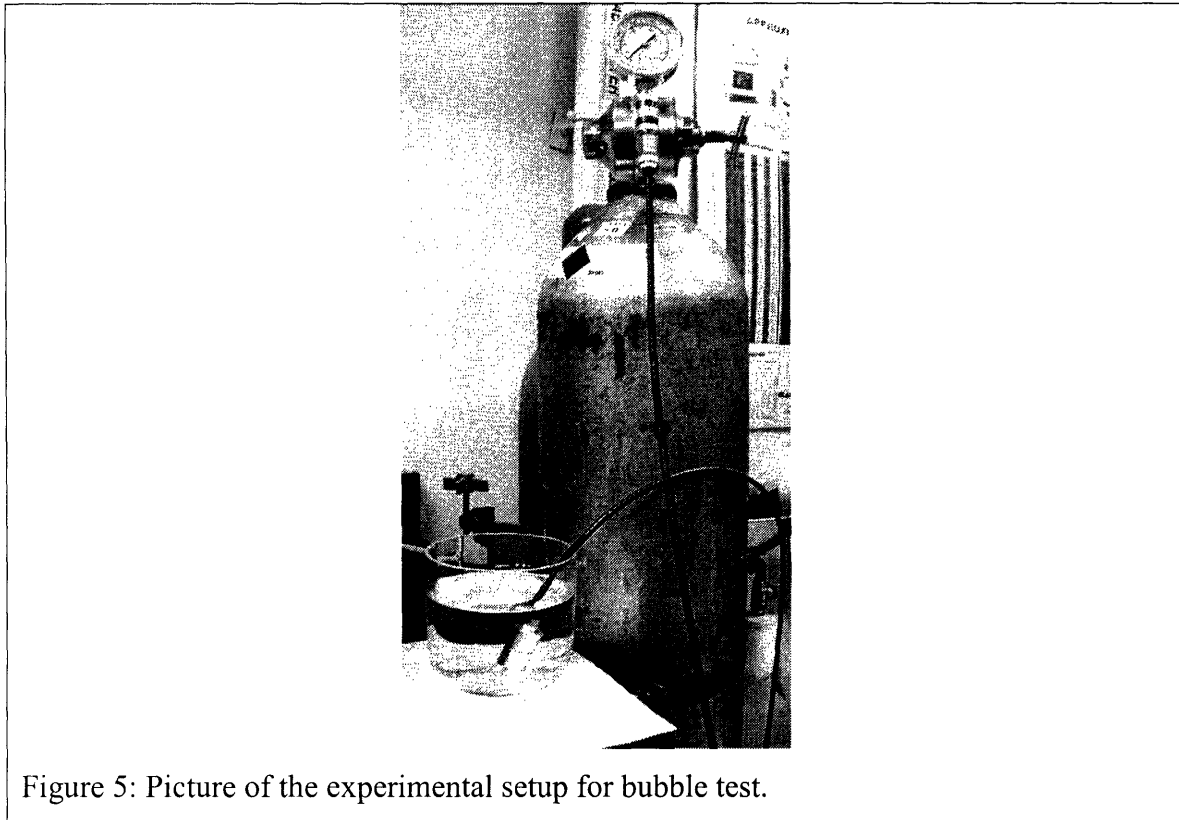
4.2.2 Test for Minimum preload

Data about the minimum preload required to obtain a given seal pressure of 125 psi was gathered. This was the maximum pressure available from the nitrogen supply at the time of testing. The procedure for the minimum preload test is:

1. Start at 10 N clamping force
2. Apply pre-load, test for seal and note seal pressure

3. If seal does not hold till maximum nitrogen pressure, increase pre-load by 10 N

4. Repeat until seal holds for maximum nitrogen pressure

4.2.3   Test for Repeatability

Another important factor to be determined was the repeatability of the seals formed by the interconnects. The procedure developed to ensure that the interconnects formed seals reliably and repeatedly is given below. In addition, the force required to separate the interconnects was also to be noted to determine variation, if any, in that parameter. The procedure for the test for repeatability is:

1. Apply preload of pre-determined minimum value

2. Make sure seal holds by bubble test

3. Measure force required to take interconnects apart

4. Repeat for ten successful trials, or until failure to seal

5   Large Meso scale Concentric Ring Interconnects

This section describes work conducted under the UROP program in Summer 2004 and is included only for the completeness of this document.

The meso scale Concentric Ring interconnects aimed to use conventional machining techniques to fabricate features that could be used in microreactors. A set of interconnects was machined to provide proof of concept; they were tested and found to seal to pressures of 125 psi.

5.1   Interconnect details

To provide proof of concept at the meso scale, a set of Concentric Ring interconnects were machined in aluminum. Aluminum was chosen because it has higher friction than delrin. This implies that once preloaded, the interconnects would require less force to maintain the seal. Also, being a relatively soft metal, aluminum would elastically deform to seal even in the absence of a great surface finish. The units were manufactured on a high speed CNC mill. The concentric rings were machined with a carbide tapered miniature end mill from Harvey tool with 3 degree taper angle and 0.015" end diameter. The taper angle was fixed at 3 degrees because that was the geometry of tapered end mill available; end mills with larger taper angles had a larger minimum-end diameter. The aspect ratio of the teeth was fixed at 3:1 because that was the optimum taper value obtained from finite element analysis. Though the features were small, they were machined on 1 inch stock to enable the use of the 1/8th NPT fittings used previously for the delrin interconnects in the pressurization tests. Through-holes were drilled in the interconnects in for the insertion of NPT fittings. The aluminum interconnects fabricated are pictured in Figure 6.
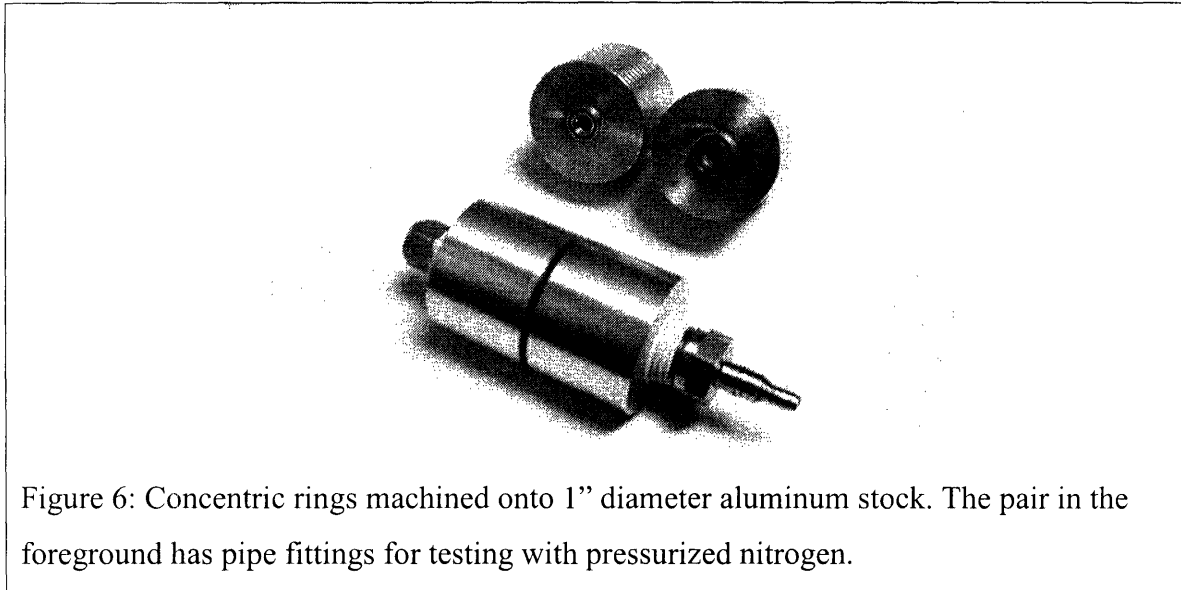
Figure 6: Concentric rings machined onto 1" diameter aluminum stock. The pair in the foreground has pipe fittings for testing with pressurized nitrogen.

5.2   Testing the interconnects

The interconnects were held in a vise and fitted with 1/8th NPT pipe fittings. One piece of an interlocking pair was fitted with a nozzle to connect to the pressurized nitrogen supply, and the other was fitted with a plug. Since the features were machined on round stock, it tended to spin in the vise and made the fitting process difficult.

Preload was applied to the interconnect pair by pressing them together in a vise. A tube was put around the NPT nozzle to transfer the force so that the preload was applied on the 1" aluminum stock, not through the nozzle. Similarly, a piece of Lexan with a hole in it was used on the other side to ensure that the force was applied on the aluminum and not on the NPT plug. Initial trials did not use an alignment tube for the two interconnects. However, this led to the interconnects getting pressed together at an angle because of kinking of the pieces in the vise jaws. This also damaged the delicate concentric rings and resulted in seal failure.

An alignment tube was then used to ensure that angular misalignment did not occur during the preloading of the interconnects. The apparatus used for aligning the interconnects is shown in Figure 7.

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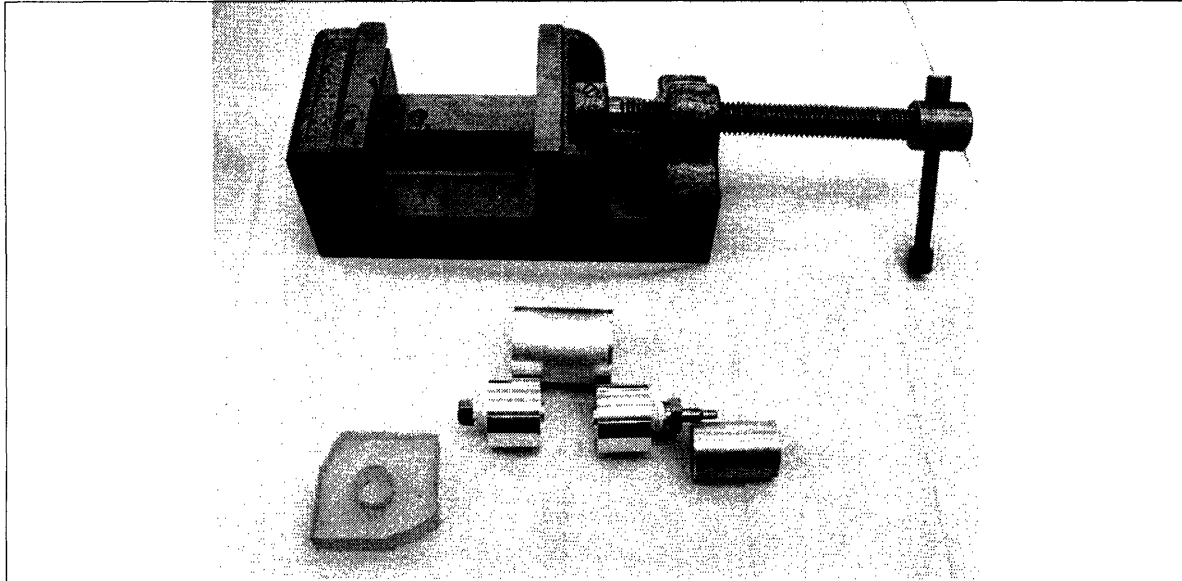
Figure 7: Picture of vise with interconnects and alignment tube

The interconnects that had been assembled with a preload could not be disassembled by hand. Hence the heads of two flathead screwdrivers were inserted into the gap between the interconnects and used as lever arms to pry them apart. This approach meant that the alignment sleeve could not be used during disassembly, leading to the risk of deforming of the delicate interlocking rings and thereby preventing them from sealing effectively again. This procedure also held the risk of scratching or otherwise marring the surface during disengagement.

5.3   Results

The interconnects were testing for seal pressure with nitrogen. One pair sealed to 125 psi on the first try, but never again. Another pair was tested multiple times (details in Appendix D): this pair sealed on the first trial and two subsequent trials to 125 psi, but again, the results were not reliably repeatable. This was probably due to the inability to properly preload the interconnects and the damage done to the sealing surfaces during the disassembly of the sealing pairs.

6 Small Meso scale Concentric Ring Interconnects

The aim of this set of interconnects was to use conventional macro scale machining to create interconnecting features small enough be used on a micro scale device. This involved reducing the diameter of the innermost ring to a size corresponding to the diameter of fluid ports in microreactors. The average width of the teeth was the same as in the large meso scale interconnects.

6.1 Interconnect details

Units varying in the different parameters as shown in Table 1 were ordered. In general, a single parameter was varied at a time in order to be able to easily identify the cause in case of a failure of function.

Number of teeth	Aspect Ratio	Taper Angle	Material
4	3:1	3	Aluminum
4	3:1	3	Steel
4	3:1	3	Steel, TiN coated
4	5:1	3	Steel
4	5:1	5	Steel
4	5:1	7	Steel

Table 1: Details of small meso scale Concentrc Ring interconnects to be tested.

Units with teeth of a 3:1 height to width aspect ratio had been proven to work both in aluminum and delrin. 3:1 was also the optimized value obtained from finite element analysis. However, taller teeth can deflect more and accommodate misalignment better. Therefore, some interconnects with teeth of 5:1 aspect ratio were ordered for comparison.

The taper angle of 3 degrees had been proven to work for meso scale features in

aluminum. The taper angle of 7 degrees had been successfully tested in delrin and was the optimum value as per the finite element analysis. A taper angle of 5 degrees is more of a self-releasing taper than a taper angle of 3 degrees; therefore, it was hoped that rings with a taper angle of 5 degrees might seal and require lower force for separation. Therefore interconnects with taper angles of 3 degrees, 5 degrees, and 7 degrees were ordered.

Interconnects were ordered in stainless steel because it is a largely inert material, and can be used with a large number of chemical reactants without poisoning the reaction. In the unit samples ordered, stainless steel was chosen, because being less reactive, it would be suitable for use in more reactions. (Microreactors have been manufactured from stainless steel foils[8], and during the development of the small meso scale interconnects it was assumed that they could be used in such microreactors. Since then, our chemical microreactor collaborators[9] have informed us that the steel microreactors experience problems in form of contaminated reactions. However, steel is still an acceptable material for many reactions, though not for as wide a range of reactions as silicon.)

Two different kinds of stainless steel were used for the mating parts of an interconnecting pair in order to prevent galling. Coating the steel with titanium nitride (TiN) would result in an even harder surface as well as a smoother surface finish. In view of the fact that TiN coating is chemically resistant – "highly inert to acids, bases, solvents, caustic, etc."[10] – a TiN coating was added on some of the steel units courtesy of BryCoat[11]. The TiN coating was on the order of a micron thick, (the thinnest coating possible) so that the feature dimensions were not affected by the coating. Units constructed with aluminum were also ordered to serve as controls, as they had been tested in previous experiments.

In conclusion, a large number of interconnects with features varying over these factors were machined for testing in order to optimize across the range of variables. Some of the interconnect types are pictured in Figure 8.
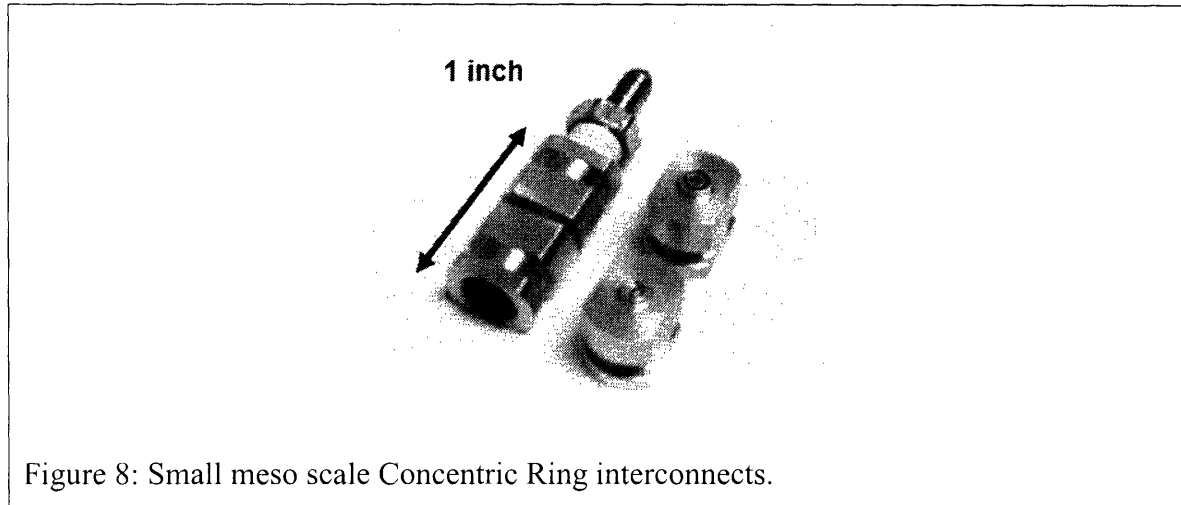
Figure 8: Small meso scale Concentric Ring interconnects.

6.2   Features on the interconnects

In order to eliminate some of the problems that came up while testing the large meso scale interconnects, several features were added to the stock on which the interconnecting rings were machined.

A pair of parallel flats were added to the interconnects in order to make the pieces easier to hold in a vise. This helped to get a better grip and stop the part from spinning in the vise during the insertion of pipe fittings. This also prevented the cylindrical pieces from rolling off the working surface.

The rings were machined on ½ inch rather than 1 inch diameter stock. This avoided wastage of material and was closer to the scale expected by the customer. Thus it better conveyed the look and feel of the intended final product. 1/16th NPT fittings were used to go with the smaller diameter stock.

To avoid angular misalignment of the features during preloading, a method was required to ensure that the preload forces would only be applied normal to the plane with the interconnecting rings. Hence the interconnects needed features that would allow them to be put together and pulled apart by applying only normal forces. To this end, a groove was added near the base of the interconnects. This ensured that the interconnects could be inserted into appropriately shaped jaws and then rotated to lock in place (like bayonets). These features are highlighted in Figure 9. The jaws could then be moved to manipulate the interconnects while only applying forces normal to the plane of the sealing faces.

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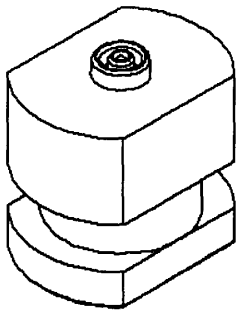
Figure 9: An isometric view drawing of the bayonet features on an interconnect.

6.3   Testing fixture

The addition of bayonet features ensured the application of forces purely normal to the sealing plane during preloading and disassembly. The easiest way to measure the force required to preload and separate the two halves of an interconnect pair was to use a machine that could apply compressive and tensile forces. The ZWICK machine (a smaller version of an INSTRON tensile test machine) met these criteria and was used for further testing of the interconnects. The ZWICK has two posts for the attachment of jaws, each with a pin and bar interface as shown in Figure 10.
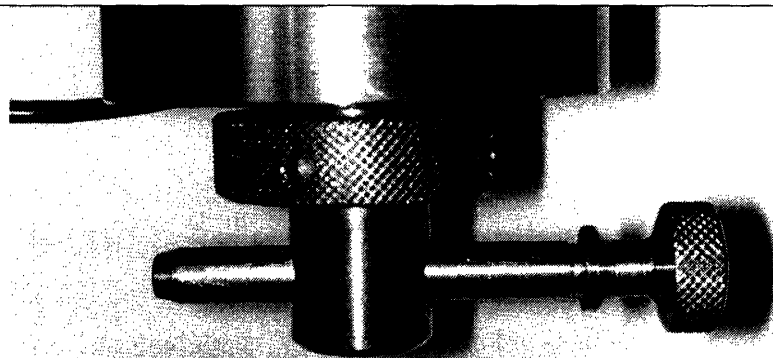
Figure 10: Picture of ZWICK-jaw interface. The ZWICK post and pin are respectively 20 mm and 8 mm in diameter.

A pair of jaws (drawings shown in Figure 11) were designed to hold the interconnects in the ZWICK. The rectangular hole allows the interconnects to be inserted in place to the level of the groove. At this point, they can be twisted 90 degrees to lock in place vertically. There was a possibility that if the posts of the ZWICK were not perfectly coaxial, the rings would be misaligned and unable to center themselves. To address this possibility, the rectangular shape allows sliding movement along the x and y axes in order for the interconnects to self align during pre-loading.
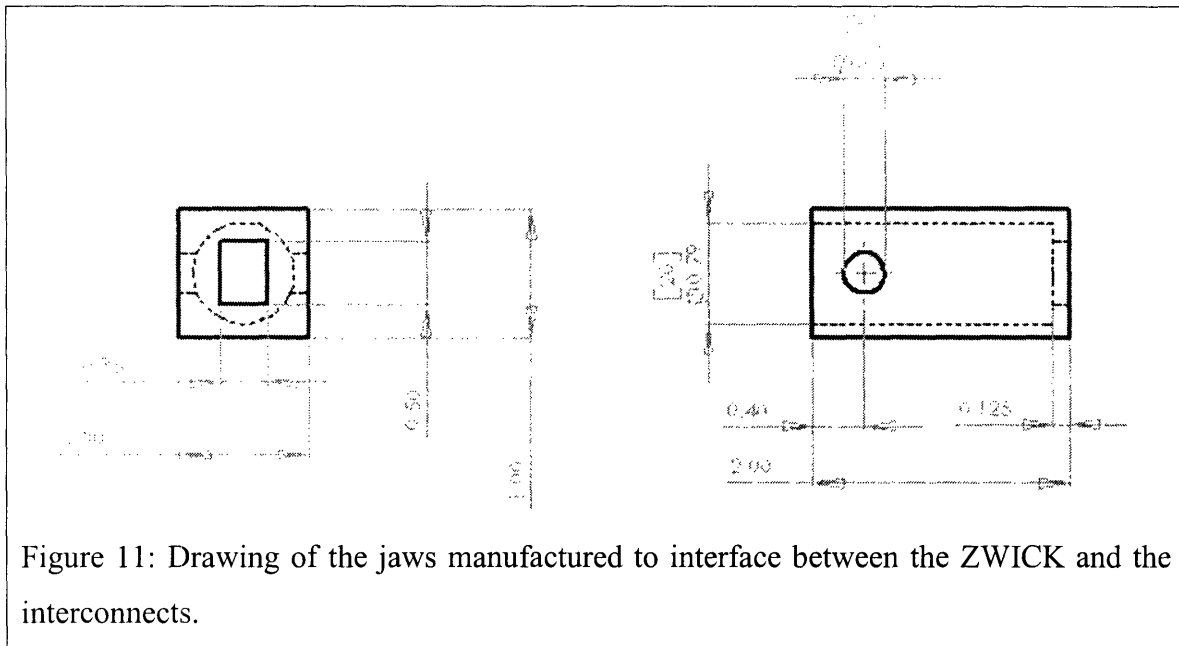
Figure 11: Drawing of the jaws manufactured to interface between the ZWICK and the interconnects.

The jaws were machined out of aluminum due to the ease of processing. One of the jaws had to be made a couple of inches longer than shown in the drawing because the ZWICK posts have a minimum distance of approach, and the shorter jaws could not get close enough to compress the interconnects together.

A picture of the actual experimental setup is shown in Figure 12.
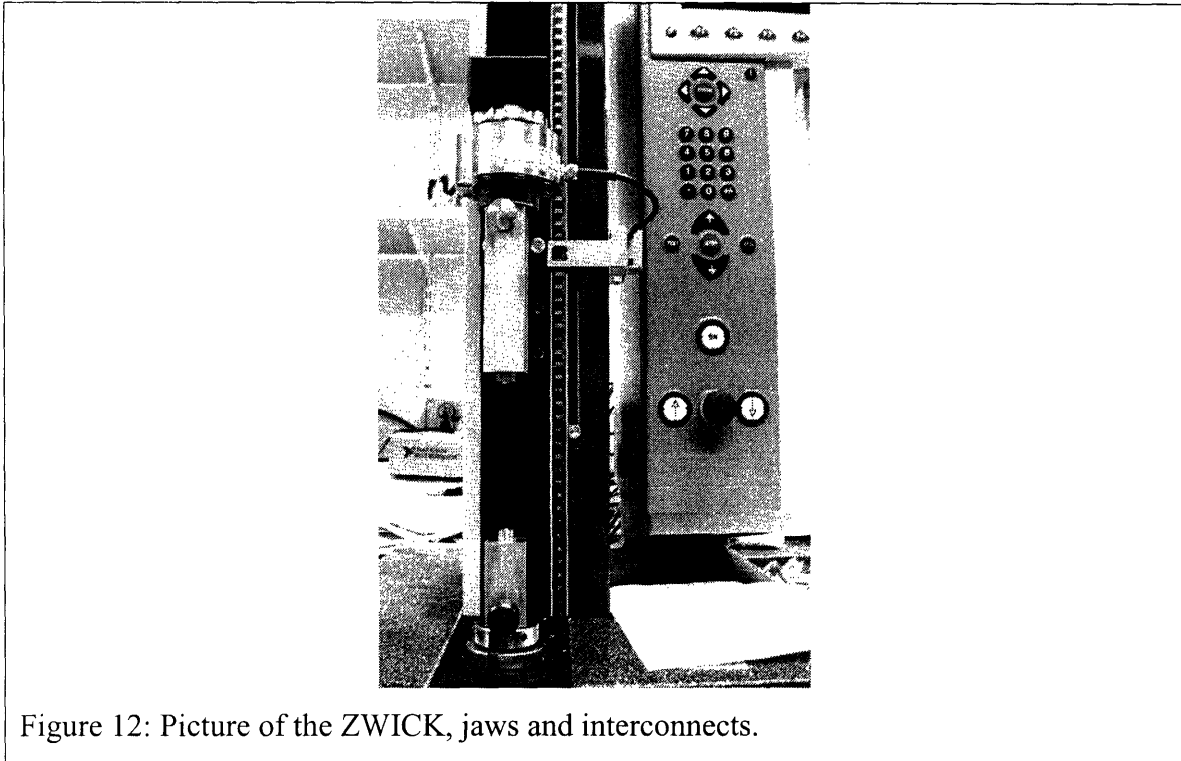
Figure 12: Picture of the ZWICK, jaws and interconnects.

6.4 Tests and results

The 5 variants of 4-toothed Concentric Ring interconnects are listed in Table 1; It was determined that for each of these variants, one pair would be tested for the minimum pre-load and repeatability. In case this pair became damaged during the testing process, another pair would then be tested for repeatability.

Table 2 summarizes the results for interconnects tested and whether they held fluid seals. The aluminum Concentric Ring interconnects worked for 30 N preload. However, none of the others materials yielded seals, even for preloads of up to 500 N.

Material	Aspect Ratio	Taper Angle	Seal pressure	Preload
Al	3:1	3 deg	125 psi	30 N
Steel, TiN coated	3:1	3 deg	n/a	150 N
Steel	5:1	3 deg	n/a	500 N

Table 2: Results of the small meso scale Concentric Ring interconnect sealing tests.

Since the units with 3 degree taper angle did not perform well, the interconnects with 5 and 7 degree taper angle were not tested. There were two reasons for this; for one the increased taper angle implied less of a locking taper, and so there was no reason to believe they would succeed where the interconnects with taper angle of 3 degree had failed, and secondly, in case the testing procedure was causing damage to the interconnects, it appeared prudent to hold off testing them until the problem was identified and fixed.

In order to gain insight into the reason behind the failure of the interconnects to seal, tested and untested interconnects were examined under a microscope. Interconnects in aluminum, steel, and titanium nitride coated steel were examined. There were vertical ridges up the tapered walls on all the interconnects. The features were manufactured with an end mill instead of by turning on a lathe, and the vertical marks observed on the tapered surfaces are indicative of tool chatter while milling the features. These flaws form leakage paths for the fluid. The aluminum interconnects looked the best, and they also had the most signs of wear. This suggests that since aluminum is a soft material it deforms and wears down so that the two surfaces mate closely and form seals. Steel and titanium nitride, being harder materials, do not exhibit such behavior. Thus, the surface finish on those interconnects needs to be much improved for a fluid seal to be formed.

Electroplating, electropolishing and lapping are processes that can be used to improve surface finish of metals. Electroplating is likely to have the same effects as coating with titanium nitride. The titanium nitride coated interconnects have a smoother overall surface finish, but the chatter marks are not entirely covered. Hence this option is unlikely to work. Electropolishing takes away material from the surface, but this would both reduce the overlap percentage (which would be undesirable) and probably not smooth out the chatter marks. Therefore lapping seems to be the most effective way to improve the surface finish on the sealing surfaces.

7   Flared Ring Interconnect Background

The lack of flexibility of the Concentric Ring interconnects led to the idea of reversing the trapezoidal cross section to make the structure more flexible. The Flared Ring interconnect grew out of the thought of a hollowed out inverted trapezoidal ring. This is a result of the application of the principle of reciprocity.

The Flared Ring interconnects are interposer type interconnects. This is a benefit because they provide a way to interconnect simple fluid ports, without the need to machine features on the surface of the device. This minimizes the impact on microreactor design.

7.1   Design description

The Flared Ring interconnects seal by the compression of thin flared rings at either end of the interconnect. Each flared ring is inserted into a straight socket and forms a compression seal. When the system is pressurized, the ends of the flared rings press harder against the inner surface of the sockets, and this increases the frictional force that holds the interconnect in place, making the seal stronger. This is an application of the principle of self-help. A schematic of the cross-section view of a flared ring inserted into a fluid port is shown in Figure 13.
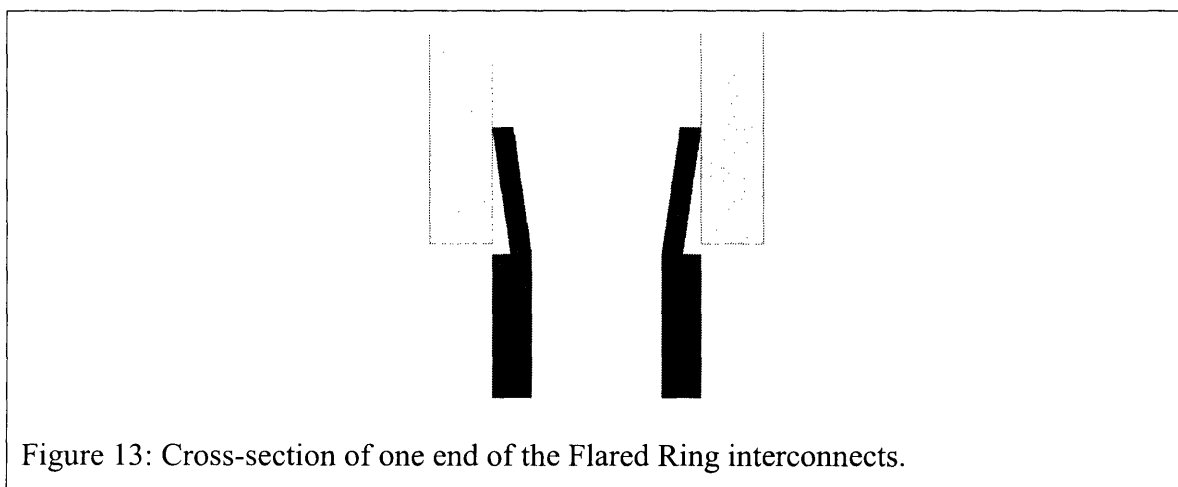
Figure 13: Cross-section of one end of the Flared Ring interconnects.

The important geometric parameters in the design are the length and thickness of the flared rings, and their taper angle.

The longer and thinner the rings are, the more flexible they are, but it is difficult to control tolerances. They should not be made so long and thin as to break easily or permanently deform when subjected to pressure. The difference between the maximum outer diameter of the flared ring and the minimum inside diameter of the socket it is inserted into determines the pressure exerted on the wall as contact stress and the insertion forces.

The taper angle in this design exists only to define the region of contact, and to makes the flared rings flexible. The reason for incorporating the taper angle into the design parameters is different compared to that in the Concentric Ring design, and thus the considerations of self-holding and self-releasing tapers no longer apply.

This design requires the interconnects to be made of compliant material that can compress to fit into a slightly undersize socket and adapt to the irregularities of the socket surface. In addition, the material must be chemically unreactive and capable of withstanding high temperatures.

8 Flared Ring Interconnect Tests

8.1 Testing objectives

The purpose of testing the Flared Ring interconnects was mainly to provide proof of concept and confirm that the design would hold fluid seals.

The relationship between the internal diameter of the socket and the external diameter of the flared ring required for a fluid seal to hold was to be determined. It is expected that higher insertion forces would be required to force the flared rings into smaller sockets, and so the minimum socket diameter that would ensure a fluid seal for a given flared ring diameter was also to be determined.

The interconnects were also to be tested for repeatability of sealing.

8.2 Testing Procedures

The Flared Ring interconnects were to be tested for sealing capability and repeatability. Proof of sealing was provided by bubble tests, the procedure for which is outlined below. The procedure for the bubble test is outlined below-

1. Insert interconnects into test sockets
2. Attach the assembly to tubing from nitrogen cylinder
3. Immerse interconnects in water
4. Increase pressure and observe for formation of bubbles
5. Note pressure at which bubbles form

The test for repeatability involved testing each interconnect ten times or until failure to seal.

9   Single sided Flared Ring interconnects

To act as an interposer between two fluid channels, the interconnect must have flared rings at both ends. To provide proof of concept, however, testing a piece with a flared ring at only one end would suffice. Hence single sided Flared Ring interconnects were manufactured and tested.

9.1   Interconnect details

Single sided Flared Ring interconnects with a maximum ring diameter of 0.226 inches were machined by Mary Kathryn Thompson to provide proof of concept for the Flared Ring design. The interconnects were turned on a lathe from ¼" diameter tubing of a high temperature resistant plastic polyetheretherketone (PEEK). The outside taper was turned on the lathe, and the inner taper was then obtained by drilling with a tapered end mill. The height of the ring was 0.205 inches and the ring thickness was 0.023 inches.

9.2   Testing fixture

An aluminum testing socket was made in ½ inch diameter stock with a socket of 0.222 inch diameter. The other side was fitted with a 1/16" NPT nozzle.

9.3   Tests and results

An arbor press was required to fit the Flared Ring interconnect into the testing socket at first, but it could be maneuvered (wriggled) out by hand. After a few trials, the Flared Ring interconnect could also be inserted into the test socket by hand. The interconnects could be wriggled in and out of the hole but could not be pulled straight out of the hole using a single swift motion. Thus, the ease of disassembling the interconnect does not reflect on its sealing capability. On the bubble test the single sided Flared Ring interconnect held 130+ psi pressure with no leaks, justifying further work on the design. The single sided Flared Ring interconnects and test socket are pictured in Figure 14.
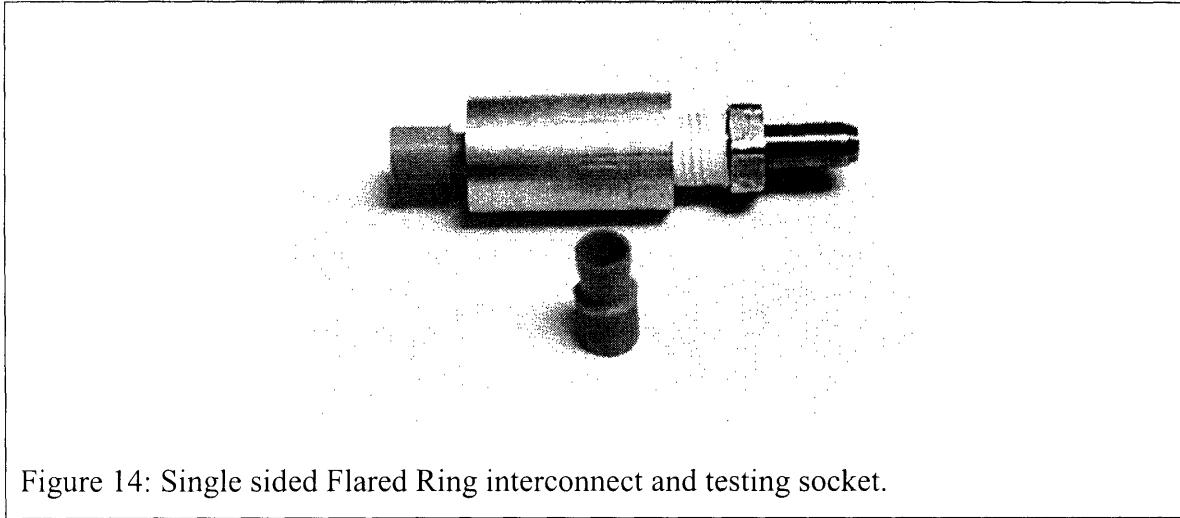
Figure 14: Single sided Flared Ring interconnect and testing socket.

10 Double sided Flared Ring interconnects

The success of the single sided Flared Ring interconnects called for proof of concept tests to be done on the double sided design in order to publish these results as soon as possible.

10.1 Interconnect details

Because of time constraints, an order was placed for a set of Flared Ring interconnects with the double sided version of the design that had been tested and proven to work successfully in the single sided Flared Ring experiments. The double-sided interconnects had maximum ring diameter of 0.25 inches on either side of the unit. Figure 15 shows a drawing of the design.

All fillets radius .025 in

5 degree taper

Figure 15: Drawing of double sided Flared Ring interconnect.
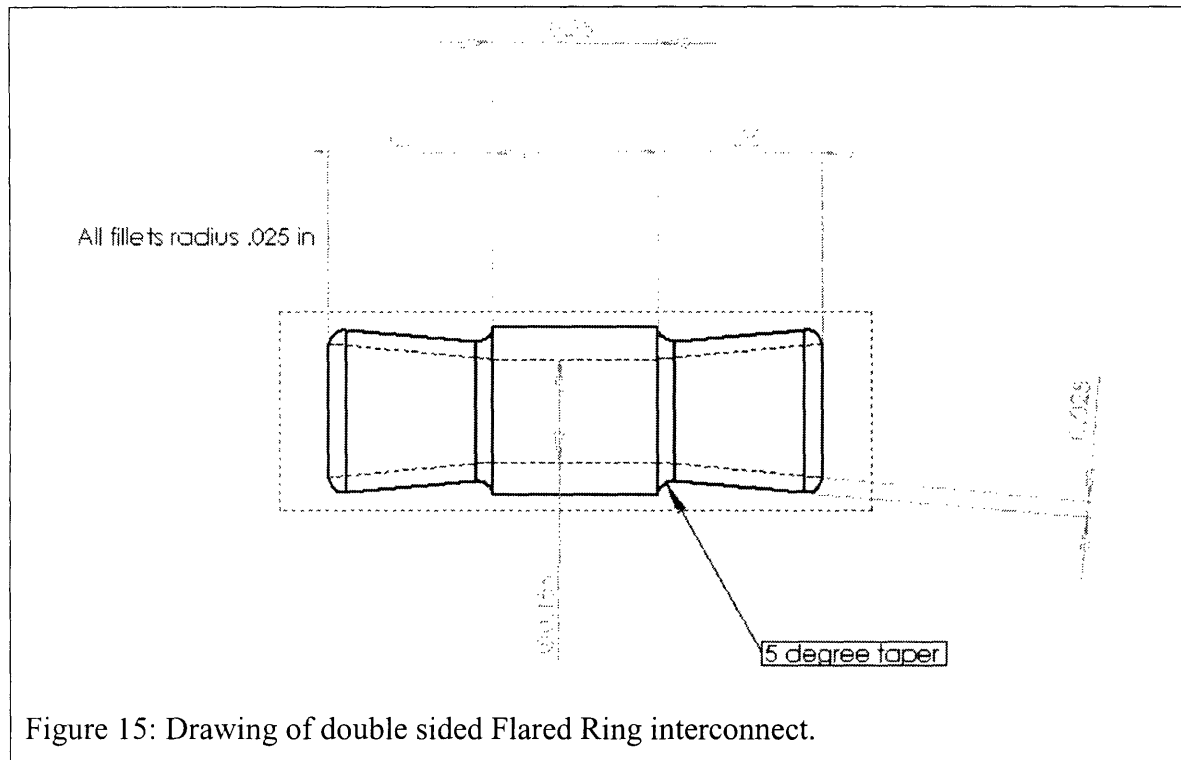
10.2   Testing fixture

A close examination of several interconnects showed that they deviated significantly from a circular shape and that the flared rings on either side were unequal in diameter (parameter measurements given in Appendix C). So the flared ends must deform enough when pushed into the socket to be forced to form a circle of the socket diameter. This means that the socket diameter should be at least as small as the average diameter of the flared ring.

A set of bores with increasing diameters was machined in aluminum to allow the interconnects to be tested for their ability to hold a seal. The drawings for the part are shown in Figure 16. The parts were machined on the computer display lathe. The diameters of the plug-type units ranged from 0.246 to 0.243 inches, and the diameters of the tapped-type ranged from 0.244 to 0.241 inches. Fittings that have the capability to quick connect to the tubing were used rather than NPT fittings. The sockets in these pieces were rounded and chamfered in order to make the compression of the flared rings easier. Pictures of the working interconnects and fittings are shown in Figure 17.

Figure 16: Drawings of the testing sockets manufactured for the double sided Flared Ring interconnects. To the left is the bore with a tapped hole for the pipe fitting so that the system could be pressurized; to the right is the plug for the other end of the double sided Flared Ring interconnects.

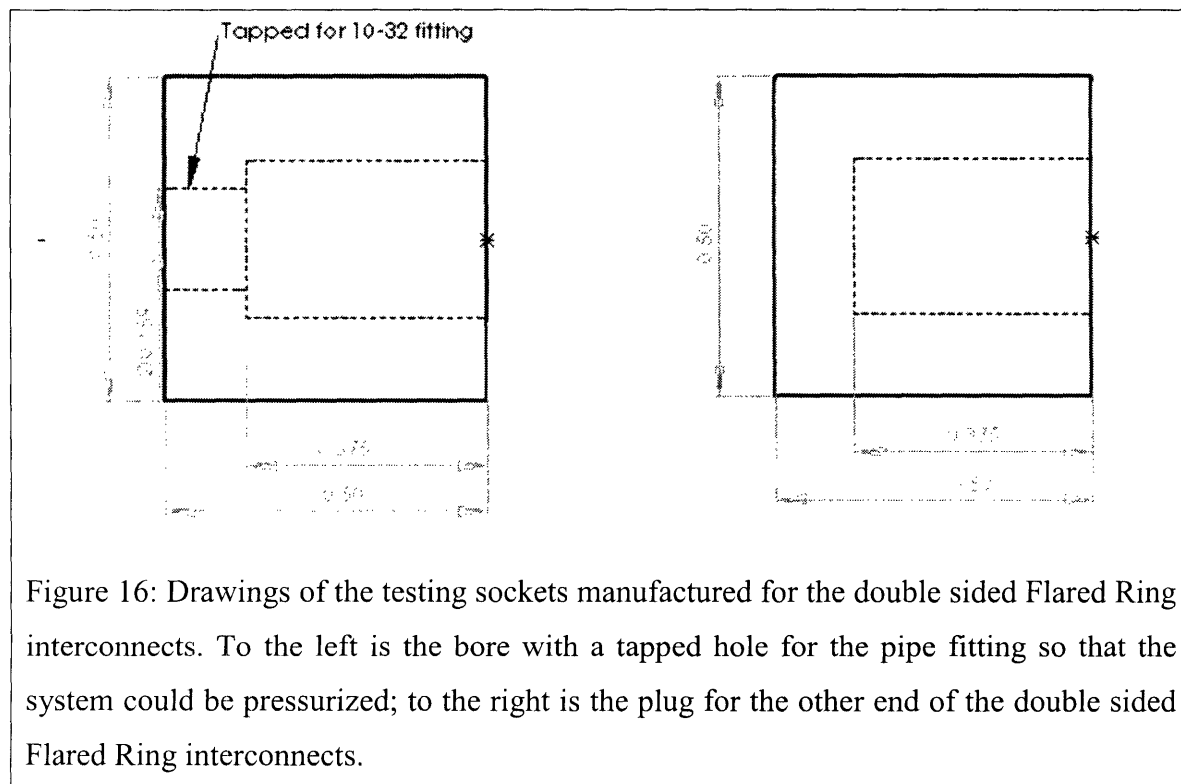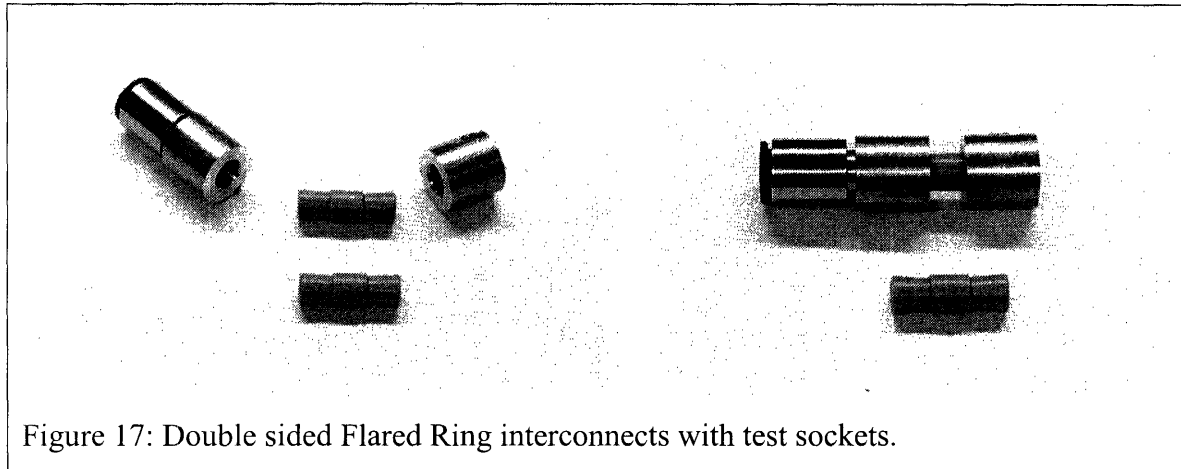Figure 17: Double sided Flared Ring interconnects with test sockets.

10.3   Tests and results

The Flared Ring interconnects were inserted in the test sockets discussed in the previous section. The two rings on the end were of different sizes, and each was inserted into a socket of known diameter until all leaking stopped. The minimum diameter of the interconnects was 0.2457 inches for Side #1 and 0.2442 inches for Side # 2. The results of the bubble tests are tabulated in Table 3.

Trial #	Side #	Bore Diameter	Result
1	1	0.245"	No leak at 20 psi
	2	0.243"	Leak at 20 psi
2	1	0.245"	Small leak at 80 psi
	2	0.242"	No leak at 120 psi
3	1	0.244"	No leaks at 150 psi for >10
	2	0.242"	minutes

Table 3: Results of the double sided Flared Ring interconnect sealing tests.

Thus, the minimum diameter socket required for the flared ring to seal was

roughly 0.002 inches smaller than the flared ring diameter. The rings also seal in smaller diameter sockets.

10.4   Interconnect arrays

The double sided Flared Ring interconnects were tested in an array of six to gage their capability to adjust to misalignment.

10.5   Testing fixture

Arrays of sockets were made on a milling machine to enable the testing of the interconnects. Two array plates were made, one with through-holes for the pipe fittings and one with only the sockets for the flared rings to fit into. The drawings for the array with through-holes for pipe fittings are shown in Figure 18.
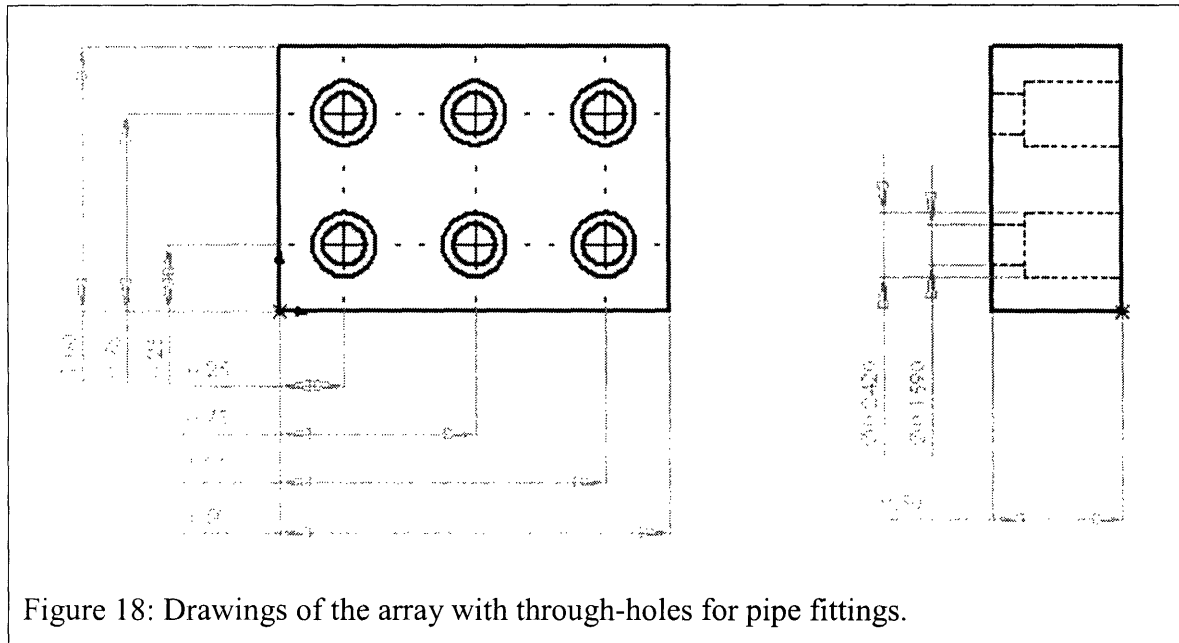

Figure 18: Drawings of the array with through-holes for pipe fittings.

The holes were drilled first with a 0.1590 inch diameter drill, (which is the diameter of the through-holes), then enlarged with a 0.2380 inch diameter drill, and then

reamed with a 0.2420 inch diameter reamer. The finished pieces, with interconnects inserted in the sockets, are shown in Figure 19.
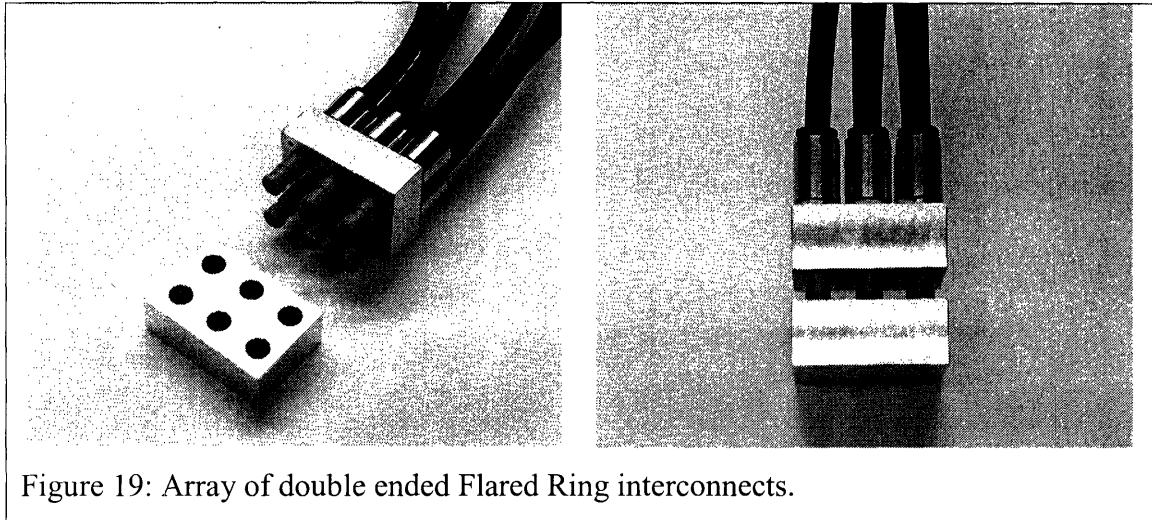

Figure 19: Array of double ended Flared Ring interconnects.

10.6   Tests and results

Out of the twelve seals formed in an interconnect array of six holes tested, eleven seals held 120 psi pressure on the first trial. Repeating this test with the interconnects in different holes led to the same result, however the leak in this case involved neither the same socket nor the same interconnect as before. This indicated that the design could elastically average 11 out of 12 seals.

Four trials into the array testing, however, all 12 seals held 120 psi. This indicates that the interconnects might have to be "broken in" before they reach their optimum performance, or better manufactured to avoid the need to wear-in. This corresponds with the experience that the single sided Flared Ring interconnects needed to be fit into the test socket with an arbor press for a few trials before they could be inserted by hand.

The observation that the interconnects functioned well despite the fact that the sockets in the array were not line bored or CNCed, and had a mere reaming surface finish (as opposed to boring), suggests the interconnect design is robust enough to compensate for misalignment of sockets due to machining variations.

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11 Conclusions and Recommendations

11.1 Concentric Ring interconnects

The Concentric Ring design shows promise. The material used to construct the Concentric Ring interconnects appears to be critical to their function. It is possible that lapping and improving the surface finish will improve the performance of interconnects made from different metals. In addition, a test of cross material pairs (PEEK/steel) would be informative and yield a better understanding of the factors critical for holding fluid seals. Six aluminum interconnects could be arranged in an array baseplate and tested. Once this unit is operational, it can be tested using helium testing to determine the leakage rates for the seals. If the units work in steel, hot embossing pyrex or other glass can make them usable in actual microreactors.

11.2 Flared Ring interconnects

The Flared Ring interconnects performed to specifications, both as single units and when assembled in arrays. The next step would be to optimize the ANSYS model of these units and compare it against the closed form solution. Another area to investigate is the possibility of manufacture using injection molding to also obtain smaller versions. It should be kept in mind that the interconnects that were tested needed to be 'broken in', and this may also be required for molded units. Thus a step of 'breaking in' may have to be incorporated into the manufacturing process to prepare the interconnects for peak performance. This would not be a desirable step, however, and thus attention should be paid to the design of the mold and process to prevent the need for wear-in of the interconnects.

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Appendix A: ZWICK Jaws Fabrication Insights

The jaws were machined from one inch square bar aluminum stock. The stock was mounted in the lathe in a self centering four jawed chuck. A ¼ inch diameter through-hole was drilled in the piece. End mills of ½ inch and ¾ inch diameter were used in succession to widen the hole to a depth of 1 ¾ inches. The piece was removed from the lathe and fixed on a mill to drill the 8 mm holes. The hole on the lower side tends to turn out undersize for the pin when an 8 mm drill is used, while the hole on the upper side is perfect. So the piece is reversed and the holes drilled through the other way, so that the pin fits through both holes. The 8 mm holes must be drilled before finishing the 20 mm hole so that 20 mm hole does not have burrs on the inside. The piece was put back in the lathe and a 20 mm drill was used to drill to a depth of about an inch so that the post of the ZWICK would fit. The bottom of the hole was squared with a boring bar so that the two surfaces, that the groove fits around, would be parallel. The piece was mounted on a CNC mill and the rectangular feature, centered around the center of the existing ¼ inch through hole, was machined with a 3/32 end mill. The part was then put back in the lathe and faced until the interconnect could turn and lock in place.

Appendix B: ZWICK Insights

Testing for the preload involves using the ZWICK. The ZWICK is a versatile machine that cannot be operated by hand. The operator can control the motion of the moving jaw by a button. Unfortunately the speed of the motion of the jaw is proportional to the amount of time the button is held down, and there is no good way to get a feel for what kind of force it applies on the specimen between the jaws in order to move at a certain speed (as opposed to a vise, where you have a physical feel of the force you are applying).

To apply preload, the ZWICK is set in compression test mode. The maximum compression force is set to the value of the preload to be applied. The next parameter to input is the initial displacement of the jaws. This is tricky because the jaws move directly to that position at a rather high speed. Hence it is inadvisable to have the test pieces touching at the initial position, because the ZWICK may move too fast and crush the delicate interlocking features in case of even the slightest misalignment of the interconnects. One can set the rate at which the jaws move together during testing, but it was unclear if the interconnects were actually getting aligned.

It is possible that an alignment tube would have helped. One was constructed but not used, because using the alignment tube would obscure the view of the unit. So the jaws were brought as close as possible without having the interconnects touch, and then moved by the ZWICK by pulsing on the button to align the rings within each other. Then one can override the initial displacement position and have the test run from the position the jaws were in after the manual adjustment. The ZWICK logs a strain Vs. preload graph. From the graphs generated, the ZWICK wasn't actually accurate at stopping at the cutoff point specified. The strain graphs seemed to show strain hardening, which meant that it became harder and harder to get a given displacement, even with increasing preload. But the slope of the graph did not increase monotonically, so it is not clear what is actually happening. Once one has preloaded the units one can get to try to take them out of the ZWICK. Unfortunately, the ZWICK's compression program includes returning back to the initial position after the test is done. So if one has the bayonet features on both the parts engaged, the ZWICK pushes them together and then tears them apart again.. To avoid this, one has to put one of the interconnect units of a pair on top of the carefully machined jaws so that it lifts into the air after preloading because it sticks to the other half.

To pull things apart, the ZWICK is set for tension testing and then run. The highest point on the graph is the force required to pull the interconnects apart. The larger challenge was trying to get both sets of bayonet features engaged. Generally one could disconnect the specially machined jaw from the ZWICK and get it engaged on one relevant bayonet feature, and then move the ZWICK post down until it pins the jaw on again. Once again, the lack of a hand crank makes this difficult. But it is achievable, and it does give numerical results.

Appendix C: Flared Ring Variation Data

In order to determine the size of the holes for the bores that the flared rings would be inserted into, the diameters of the rings of 5 interconnects were measured.

Piece #	End #	Trial 1	Trial 2	Trial 3	Trial 4	Average
1	1	.2471	.2469	.2465	.2469	.24685
	2	.2450	.2446	.2450	.2449	.24488
2	1	.2461	.2464	.2462	.2462	.24623
	2	.2449	.2446	.2446	.2447	.24470
3	1	.2467	.2466	.2466	.2467	.24665
	2	.2452	.2452	.2453	.2452	.24523
4	1	.2461	.2457	.2461	.2462	.24603
	2	.2444	.2445	.2442	.2442	.24433
5	1	.2465	.2466	.2461	.2462	.24635
	2	.2446	.2446	.2446	.2447	.24463

In conclusion, the minimum average diameter for Side #1 was 0.2457 and for Side #2 was 0.2442 inches.

Appendix D: Large Meso scale Concentric Ring Results

Interconnect #	Trial #	Sealing Pressure (psi)
2	1	120
2	2	50
2	3	40
2	4	40
2	5	40
2	6	20
2	7	<20
3	1	120+
3	2	40
3	3	30
3	4	120+
3	5	50
3	6	120+
3	7	40
3	8	30
3	9	20
3	10	30
3	11	70
3	12	40
3	13	60
3	14	30
3	15	20