

Design of a Dynamic Seal to Enable Movement of a Film or Filament Through a Pressurized Space While Maintaining a Desired Pressure

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Abstract

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The solid-state process has been successful in creating microcellular foams in a number of thermoplastic polymers using sub-critical CO₂ in the 3-5 MPa range. In this process, the polymer film or sheet is first saturated with CO₂ in a pressure vessel. The gas saturated film is then removed from the pressure vessel and heated in a suitable medium (liquid or hot air, for example) to create a microcellular structure. The time elapsed from depressurization of the pressure vessel to the heating of the gas-saturated film, known as the *desorption time*, is an important process parameter that can range from a few minutes in a laboratory environment to tens of minutes in a manufacturing environment. During the desorption time, the absorbed gas leaves the polymer film. This loss of gas becomes increasingly critical as the film thickness is reduced; polymer films below 0.010" in thickness are difficult or impossible to foam in the current solid-state batch process. To successfully foam thin films it is necessary for the polymer to retain the absorbed gas prior to heating. Towards this end, a dynamic seal was developed that allows a polymer film/filament to be smoothly drawn out of a pressure vessel while preventing the pressurized gas from escaping. The dynamic seal is based on the idea of using a liquid (e.g. water) to provide a seal and keep the pressurized

gas from escaping. The liquid thus experiences the same pressure as the gas. Liquid leakage is controlled to within an acceptable limit by designing sufficient resistance to the flow.

The early prototypes showed that a pressure of 5 MPa could be maintained in a small pressure vessel, while a 0.4 mm nylon fishing line was drawn through the seal. Water was used to seal the gas in this case. It was quickly apparent that the key challenge would be to move, or draw, the polymer film through the seal mechanism while keeping its integrity. The key is to strike a balance between sealing forces that keep the leakage rate low and the frictional forces that result on the polymer film as it moves through the seal.

To explore the parameters that influence the seal performance, a lab scale pressure vessel and seal mechanism was designed, built, and tested. The vessel with 5 inch ID and 7.5 inch OD was machined from Aluminum. The vessel had a clear polycarbonate window of 3.5 inch diameter embedded in the lid, with 30 LEDs illuminating the inside of the vessel. This design allowed for visual observations of the seal performance during testing. Various materials, both porous and nonporous, were investigated as seal materials. It was found that porous materials allow more control over the leakage rate and the resulting pressure drop in the flow direction is linear, consistent with Darcy's law. However, the porous material (a polyurethane foam) resulted in a large frictional force on the film. On the other hand, the nonporous material (also a PU foam but with a closed-cell structure) resulted in reduced frictional resistance on the film, however there was less control on the leakage rate and the pressure distribution was non-linear.

The basic feasibility of the dynamic seal idea was demonstrated. Further work is needed to reduce the frictional resistance on the polymer film and reduce leakage rates. Several ideas for improving the seal are presented.

Contents

1	Introduction	1
1.1	Solid State Foaming	1
1.1.1	The Solid-State Microcellular Process	1
1.1.2	Unique Microstructures	2
1.2	Physics of the Process	6
1.2.1	Thermodynamics	6
1.2.2	Diffusion	7
1.2.3	Solid Skin	7
1.3	Necessity for a Dynamic Seal - Continuous Process	8
2	The Dynamic Seal	11
2.1	Facets of a Dynamic Seal	11
2.2	Early Ideas	12
2.3	The Idea of a Controlled Leak	13
2.3.1	Working Prototype	14
2.3.2	Blocking Liquid	15
3	Seal Materials	17
3.1	Porous vs. Nonporous Materials	17
3.1.1	Nonporous Materials	17
3.1.2	Porous Materials	19
3.1.3	Analysis	20
3.2	Experiments	21
3.2.1	Experimental Methods	21
3.2.2	Results	24
3.2.3	Conclusions	26
3.2.4	Experiment with Fishing Line	27
4	Design of a Pressure Vessel - Dynamic Seal System	29
4.1	Vessel Design	29
4.1.1	Machining Operations	36
4.2	Stress Analysis	39
4.2.1	Stress in a Thick-walled Vessel	39

4.2.2	Cap Bolts	40
4.2.3	Bolt Pattern for the Plates	40
4.2.4	FEA	43
5	Design of the Dynamic Seal	47
5.1	Porous Seals	47
5.2	Edge Clamping Problems	50
5.3	Flow Visualization Through Porous Seals	54
5.4	Back to Closed Cell Seals	60
5.4.1	Microcellular Sheet as Seal Material	62
6	Pressure Measurements	67
6.1	Pressure Inside the Seal	67
6.1.1	Hardware	67
6.1.2	Electronics	73
6.2	Preliminary Results	76
7	New Ideas and Future Work	81
7.1	Cutting Channels	81
7.2	Combining Porous and Nonporous Materials	85
7.3	High Viscosity Fluids/Gels	86
7.4	A New Idea: Self-Sealing Design	87
7.4.1	Experimental Verification	90
7.5	Overall Conclusions	93
	Appendix A List of Programs Used	99
	Appendix B MATLAB Code	101
	Appendix C Arduino Microcontroller Code	103
	Appendix D Flow Experiments Data	109
	Appendix E Patent Application	111

List of Figures

1.1	Sequential diagram illustrating the three main steps in the solid-state microcellular process.	2
1.2	PET saturated at 5 MPa and room temp. Foamed at 80°C. [Kumar, unpublished]	3
1.3	PLA saturated at 2 MPa. Foamed at 80°C. [Kumar, unpublished]	3
1.4	COC (Cyclic Olefin Copolymer) at 73% relative density. Saturated at 5 MPa, foamed at 70°C. [Kumar, unpublished]	4
1.5	Polycarbonate saturated at 5 MPa, foamed at 100°C. Right: zoom on cell wall showing a nano-structure [5]	4
1.6	HDPE saturated at 3 MPa, foamed at 105°C. [Kumar, unpublished]	5
1.7	ABS saturated at 5 MPa, foamed at 60°C. [Kumar, unpublished]	5
1.8	PEI (Polyethylenimine) saturated at 5 MPa, foamed at 180°C. This high-temperature thermoplastic produces a nanometer order structure under some conditions. Here, the average cell size is 120 nm. The structure is open-porous and permeable to fluids. [Kumar, unpublished]	6
1.9	Effects of desorption on the microstructure of the foam. The outer layer is a solid skin, then there is a transition region of large cells, and finally the uniform core in the inner part of the sample.	8
1.10	Concept of a continuous solid-state microcellular foaming process wherein a roll of raw polymer is continuously passed through a pressurized space for gas sorption and foamed as it exits through the second dynamic seal.	9
2.1	A dynamic seal design using rubber rollers to seal the gas but allow a film or fiber to pass through	12
2.2	A dynamic seal design where the film is clamped by two soft (foam) materials that provide the seal. The film enters the pressure vessel below (not seen) through the slit.	13
2.3	A schematic of the dynamic seal idea utilizing a soft sealing material and a liquid to block the gas from escaping. This concept was filed in a provisional patent application in March 2013.	13

2.4	Prototype vessel and dynamic seal. It was able to successfully seal 0.60 MPa CO_2 pressure while allowing a PEI film of 0.15 mm thickness to pass through freely with very low friction. The blocking liquid was water which leaked at a very low rate, as seen by the drops present on the film.	14
2.5	Left: setup of the prototype experiment. Right, view of the inside through the clear polycarbonate window. The PEI film is rolled inside the cavity. . .	14
3.1	Coordinate system defining the fluid problem.	17
3.2	Liquid deforming the seal material to create a pocket. This large fluid volume has a much smaller pressure drop.	19
3.3	A porous seal material allows flow within. The fluid forces on this material are very different than those on the nonporous material.	20
3.4	Aluminum experimental jig. Fluid is pushed by a controlled pressure through the small orifice at the center and flows through the porous disk to the outside edge exposed to the atmosphere. The porous material is adhered to the aluminum by contact cement on both faces. The distance between the plates was controlled by adjusting the nuts.	22
3.6	Problem definition. The domain is a disk with inner radius $r_1 = 3$ mm and outer radius $r_2 = 22$ mm. The inner boundary has an applied pressure p_1 and the outer boundary is at p_2 taken as 1 atmosphere.	22
3.5	Experimental setup diagram. Pressurized water flows radially through the porous material and is collected and weighted using a digital scale.	23
3.7	Plot of predicted pressure versus radius in the porous disk. The gradient is steepest at $r = r_1$	24
3.8	Polyurethane open cell foam of 1/16 inch (1.59 mm) thickness, 20 lb/ft ³ density. The sample was compressed to 1.0 mm and the water was allowed to leak for 2.0 minutes at each pressure. $R^2=0.989$	25
3.9	Silicone open cell foam of 1/8 inch (3.18 mm) thickness, 12 lb/ft ³ density. The sample was compressed to 1.4 mm and the water was allowed to leak for 3.0 minutes at each pressure. $R^2=0.974$	25
3.10	Natural Gum rubber open cell foam of 1/8 inch (3.18 mm) thickness, 30 lb/ft ³ density. The sample was compressed to 1.0 mm and the water was allowed to leak for 5.0 minutes at each pressure. $R^2=0.943$	26
3.11	Polyurethane open cell foam of 1/4 inch (6.35 mm) thickness, 1.8 lb/ft ³ density. The sample was compressed to 2.0 mm and the water was allowed to leak for 1.0 minute at each pressure. $R^2=0.997$	26
3.12	A 0.4 mm nylon fishing line was threaded through the center hole and out between the porous seal material (1.8 lb/ft ³ polyurethane foam). A pressure of 1.62 MPa was maintained at the center of the seal and the filament was drawn by hand easily with only a few water drops of leakage.	28

4.1	Top: CAD representation of the full size prototype vessel. The polymer (yellow) enters through the bottom seals, goes up through a slit in the bottom of the vessel, then returns through the same slit and exits on the opposite side. Bottom: Detail view of the polymer entering in and out of the slit, all between the seal material (red).	30
4.2	CAD sketch of the vessel body with dimensions in inches.	31
4.3	Exploded view of the pressure vessel CAD model.	32
4.4	White LEDs in the cap illuminate the vessel so that the vessel interior can be seen through the clear window.	33
4.5	View into the vessel showing the slit on the bottom through which the polymer can enter and exit the pressure vessel.	34
4.6	Isometric CAD view of the vessel to be built.	35
4.7	Side view photograph of the complete assembled vessel. It has been tested at the full CO_2 tank pressure of 5.17 MPa and no leaks were found. The gas was left pressurized overnight and no drop in pressure was observed.	35
4.8	Inlet port to the vessel. The system is equipped with a mechanical pressure gauge, and a 3-way valve that allows venting through a porous brass plug (seen behind the gauge).	36
4.9	Machining of the vessel cap. A Trakk 2-axis mill was used; the Z-axis was controlled manually.	37
4.10	Vessel boring setup on the HAAS TL-3 CNC lathe.	38
4.11	Inside view during the boring operation. The passes are very small (approximately 0.015") and the rotation speeds less than 80 RPM on average. The liquid seen is lubricant/coolant which also helps wash away the chips.	38
4.12	Diagram of the stress directions for the thick-walled pressure vessel equations. From www.engineeringtoolbox.com , used with permission.	39
4.13	Problem definition and set-up, cut in half at $x = 0$ by symmetry. Each element has area A and boundary at x_i except for the first element.	40
4.14	Equal spacing between supports leads to highly uneven load sharing.	42
4.15	Support pattern as given by the MATLAB code solution. The stress is even in all members.	43
4.16	Equidistant bolt pattern. The bolts closest to the center (the slit in figure 4.1) take the most load.	44
4.17	Bolt pattern generated by theory. The stress is much better shared between the bolts.	45
5.1	View of the bottom half of an example seal setup. The porous material (white) is the working seal, while the black rubber (neoprene) serves to block flow transversely.	47
5.2	First seal design. The material used is 1/4 inch 1.8 lb/ft ³ polyurethane open-celled foam. Along the sides, 0.75 inch strips of 1/16 inch thick neoprene rubber are attached for providing structural support and for sealing the flow transversely.	48

5.3	Seal material setup. The material used is 1/32 inch 20 lb/ft ³ polyurethane open-celled foam. Along the sides, 1.5 inch strips of 1/32 inch thick neoprene rubber are attached for sealing the flow transversely.	49
5.4	When a linear elastic solid (red) is pressed against a rigid body (circle) it will not conform exactly to its shape if the curvature is too large. Instead, gaps will be made through which fluid can leak (blue).	50
5.5	A low curvature wedge designed to seal the edges of the seal material.	51
5.6	Photo of the polycarbonate V-shaped piece used as a barrier for transverse flow. Since the edge does not have infinite curvature as does a rectangle corner, it was hypothesized that the seal material would conform to the shape and not produce any gaps.	52
5.7	1/4 inch 1.8 lb/ft ³ polyurethane open-celled foam seal without side supports. Instead, adhesives were used to provide the barrier for transverse flow (seen as the pink leftover streaks).	53
5.8	Instead of flat clamping plates, a smooth curve is machined such that the separation distance tapers down and squeezes the material sufficiently to block flow through that region.	54
5.9	Curvature machined in the polycarbonate plate to clamp the seal material edges.	55
5.10	End view of the assembly. Note that the seal material (white) is highly compressed at the edges; this blocks transverse flow, i.e. seals the edges effectively.	55
5.11	Top view of the assembly. The pressurized fluid comes in through the fitting in the center and then flows along the length of the seal. The polymer film sandwiched in the seal is 0.15 mm thick PEI.	56
5.12	Flow visualization using red dyed water. The valve was only slightly open so that it restricted the flow rate.	57
5.13	Identical setup except that the valve was opened further.	58
5.14	Close up of the flow around the seal edges. Note that the far edges are still white due to the clamping technique which kept the red fluid out of that region.	59
5.15	Porous open-cell seal material replaced with non-permeable closed-cell polyurethane. The friction was reduced significantly.	60
5.16	A gap occurring in a closed-cell seal material due to high curvature at the pinch zone is not a problem since the fluid cannot flow normal to the surface and is therefore sealed.	61
5.17	Exploded view of a closed-cell seal material implementation. The bumps on the clamping plates are sufficient in shutting off the transverse flow.	62
5.18	A US quarter was pressed into a PET microcellular sheet from MicroGREEN™ using a vise. The detail is permanently imprinted on the sheet.	63
5.19	Photo of 2 mm microcellular PET sheets from MicroGREEN™ used as seal material. A 1.0 inch wide by 0.0125 inch thick ABS film strip was fed through the center. The clamping pressure forced the microcellular material to conform to the shape of the film strip.	64

5.20	Close up of the microcellular seal material. The imprint on the surface is more visible.	65
6.1	Broad view of the system built to capture pressure data inside the dynamic seal.	68
6.2	Photo showing a hole through the seal material. The fluid pressure at that location is thus measured.	68
6.3	The holes go all the way through the aluminum plates and connect to the compression fittings on the other side.	69
6.4	Fittings concentric to the small test holes. Copper tubes transfer the fluid pressure to the pressure sensor in order to be read.	69
6.5	Isometric view of the line router device CAD model.	70
6.6	Section view showing the internal connections.	70
6.7	Diagram showing the working principle. A steel ball is pressed against the opening by a set screw to close off the fluid. When the fluid pressure is to be read, the set screw is backed off and the ball is pushed back by the fluid and can enter the channel (below) that routes to the sensor.	71
6.8	The finished product with sensor, screws, balls, and fittings assembled. . . .	72
6.9	Display panel made on prototype board. This is how the user interacts with the data acquisition system.	73
6.10	Diagram of the data acquisition process.	74
6.11	Sample data file from the acquisition system. Each experiment collects N data points and stores them in a columns as CSV format. The first row entry of each column is chosen by the user before the data collection is begun.	75
6.12	Pressure distribution experiment 1.	76
6.13	Pressure distribution experiment 2.	77
6.14	Pressure distribution experiment 3.	77
6.15	Pressure distribution experiment 4.	78
6.16	Pressure distribution experiment 5.	78
7.1	Due to uneven bolt clamping, the fluid may be squeezed out of certain areas which leads to non-uniformity in flow and pressure drops.	82
7.2	A serpentine style cut guides fluid along the width of the seal. Since the arc length of the curve is large, the resistance to fluid flow will be large and thus the leak minimized.	83
7.3	A criss-cross pattern style utilizing the same idea.	84
7.4	A fishbone style pattern.	84
7.5	A possible seal design using both porous and nonporous material in order to obtain the benefits of both.	85
7.6	A nonporous material bounded by porous materials on either side. This seal material arrangement can make use of the advantages of both material behaviors.	86
7.7	A grease product tried as blocking fluid for the dynamic seal.	87

7.8	An example where the fluid from the vessel is allowed to enter the space between the clamping plates and the seal material. Thus, the seal material experiences forces from fluid pressure on either side.	88
7.9	A look into the pressure distribution on the seal material if it was rigid and flat. Since the fluid between the seal material and polymer is flowing, the pressure drops due to wall shear (from viscosity).	89
7.10	Since the seal material is deformable, the net pressure force presses it closer to the polymer film, decreasing the gap and choking the flow.	90
7.11	A solid rod is loosely fed through a soft tube which is adhered to a rigid pressure vessel. The only path for the vessel's fluid to escape is through the space between the solid rod and the inside of the flexible tube.	91
7.12	Net force diagram of the cylindrical setup. The pressure difference between the two sides of the tube work to collapse it onto the rod and reduce the flow.	91
7.13	Photograph of the parts used in the experiment.	92
7.14	Closeup view of the experimental setup. Left: rubber tube is attached to the acrylic tube wall using epoxy and contact cement. Right: solid rod protruding all the way through the tube close to the brass fitting (pressure inlet).	92

List of Tables

D.1	Polyurethane (dark gray) open cell foam of 1/16" (1.59 mm) thickness, 20 lb/ft ³ density.	109
D.2	Silicone open cell foam of 1/8" (3.18 mm) thickness, 12 lb/ft ³ density.	110
D.3	Natural Gum rubber open cell foam of 1/8" (3.18 mm) thickness, 30 lb/ft ³ density.	110
D.4	Polyurethane (white) open cell foam of 1/4" (6.35 mm) thickness, 1.8 lb/ft ³ density.	110

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Chapter 1

Introduction

1.1 Solid State Foaming

Conventionally, foamed polymers are produced by introducing a blowing agent (physical or chemical) into a polymer melt and expanding to produce a cellular material. Typically the cells are on the order of 10^{-4} m. This is too large for applications where the material thickness is on the same order. In the 1980s, a challenge was given by the packaging industry (companies Kraft and Kodak) to reduce the cell size to the micrometer range or below such that 100+ cells fit across the thickness of a typical packaging polymer sheet. A research team at the Massachusetts Institute of Technology led by Professor Nam Suh responded to the challenge and conceived a process for creating microcellular polymer foams. They were granted US Patent #4,473,665 for their work which is now referred to as the "solid-state microcellular process" [1].

1.1.1 The Solid-State Microcellular Process

The solid-state microcellular process is very simple (wherein lies its beauty) and consists of three main steps which are summarized in figure 1.1:

1. Gas absorption: a thermoplastic polymer is placed in a high pressure gas environment.
2. Pressure release: the polymer is removed from the high pressure environment, and desorption begins.
3. Foaming: The polymer temperature is elevated close to its glass transition temperature: cell nucleation and growth occurs to create the cellular material.

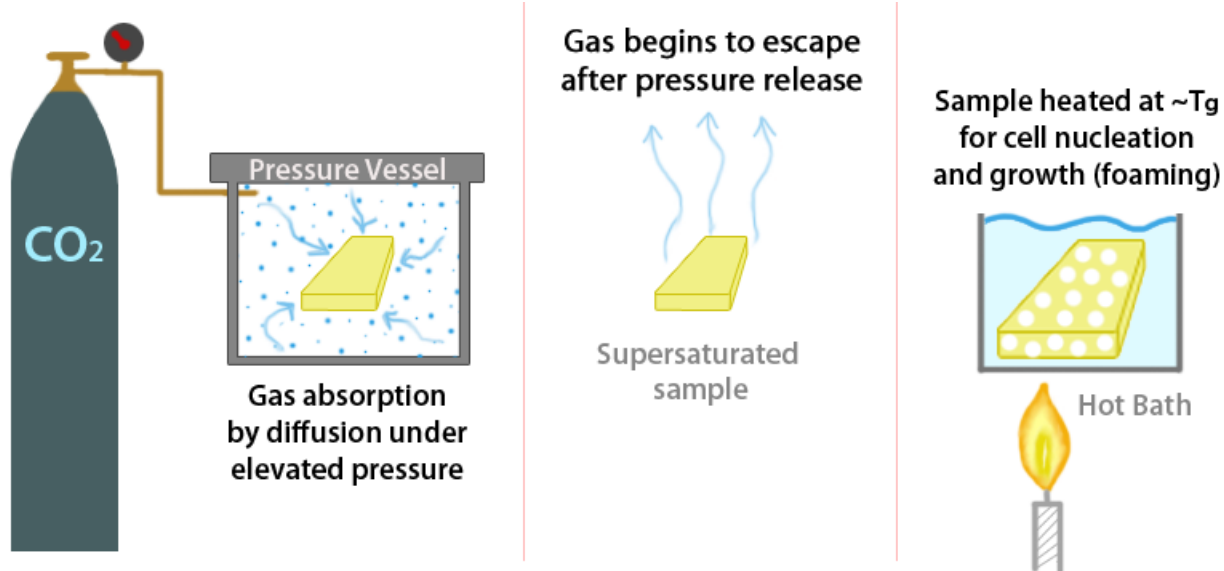


Figure 1.1: Sequential diagram illustrating the three main steps in the solid-state microcellular process.

In the first step, the gas utilized for this research is Carbon Dioxide (99.9 % purity) although Nitrogen has also been investigated originally at MIT [2]. In the second step, the pressure is released to the atmosphere and the desorption time is used as a parameter for different foaming structures and skin characteristics [3]. In the third step, the thermoplastic polymer is brought to a temperature close to the glass transition temperature of the system. It is important to note that the glass transition temperature is decreased by the presence of the absorbed gas, thus the T_g in question is lower than that of the virgin material. At this temperature, the thermodynamic instability created by the supersaturated gas gives rise to cell nucleation and cell growth. The cell densities are large, ranging from $10^8/cm^3$ in PS-N₂ system to $10^{14}/cm^3$ in PEI-CO₂ system [4, 5]. The polymer is never melted during the process; it is actually far from its melting temperature: here lies the uniqueness of the solid-state microcellular process.

1.1.2 Unique Microstructures

Unique microstructures can be produced with this process by varying the parameters: polymer type, saturation pressure, saturation temperature, desorption time, foaming temperature, and foaming time. Following are some examples.

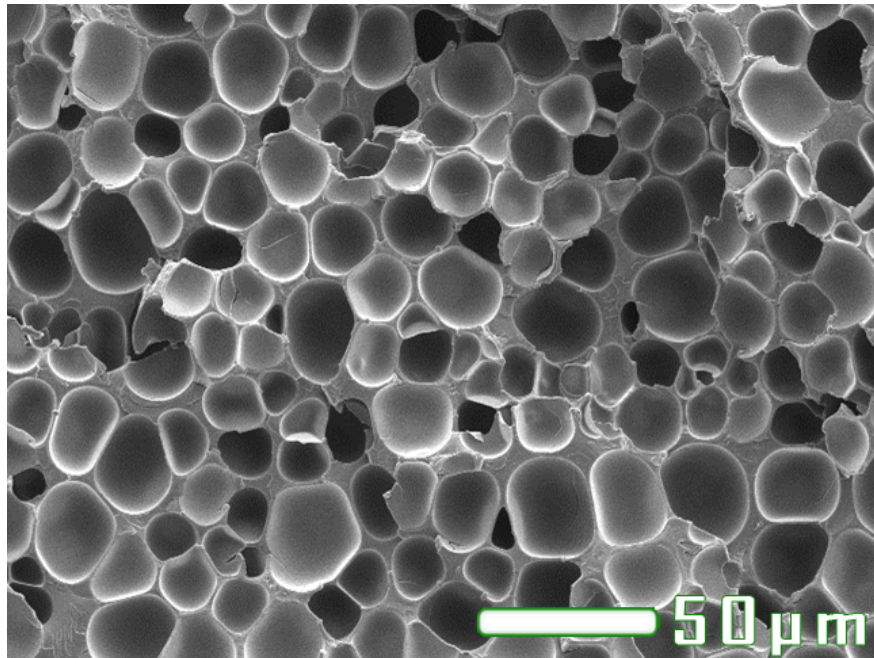


Figure 1.2: PET saturated at 5 MPa and room temp. Foamed at 80°C. [Kumar, unpublished]

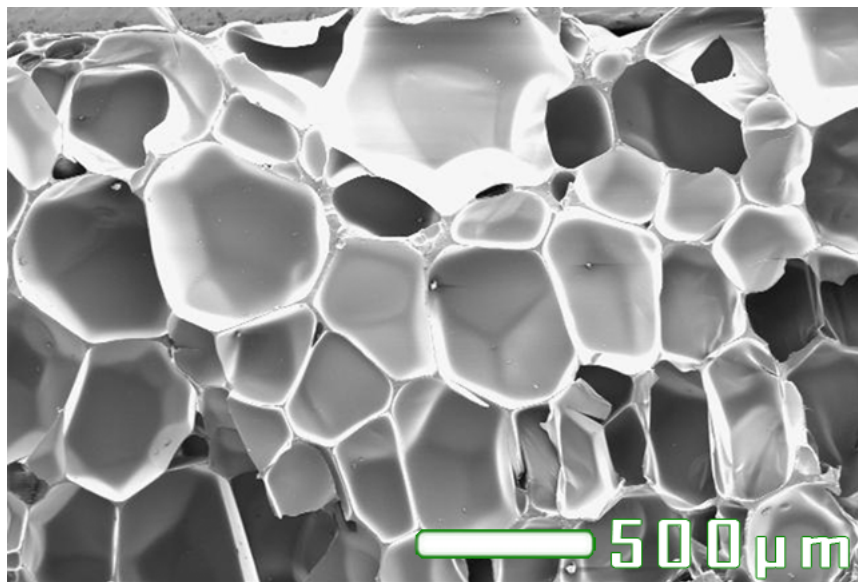


Figure 1.3: PLA saturated at 2 MPa. Foamed at 80°C. [Kumar, unpublished]

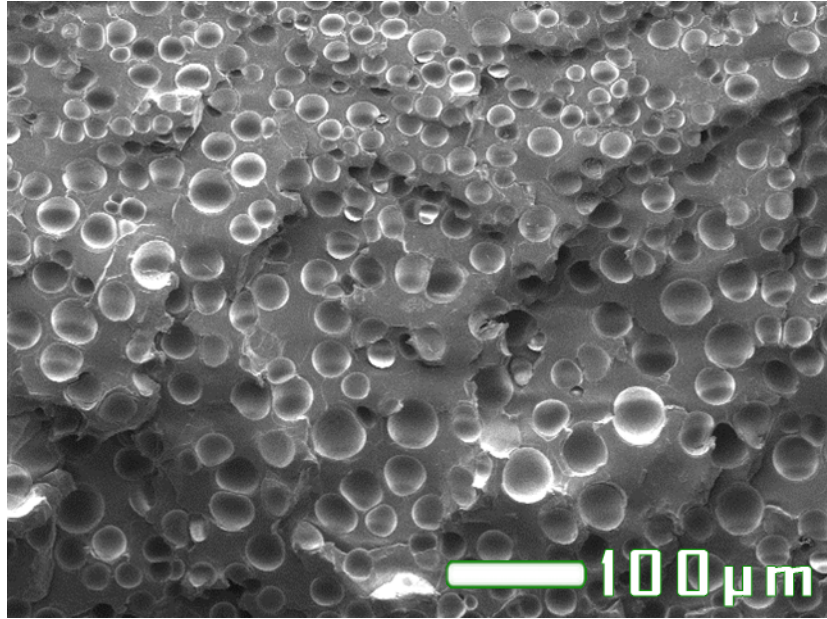


Figure 1.4: COC (Cyclic Olefin Copolymer) at 73% relative density. Saturated at 5 MPa, foamed at 70°C. [Kumar, unpublished]

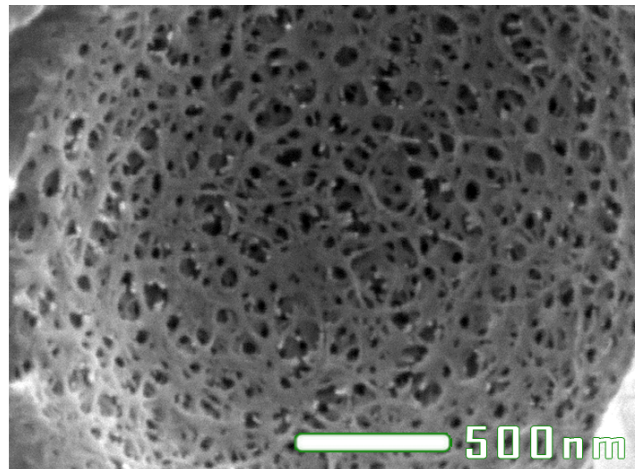
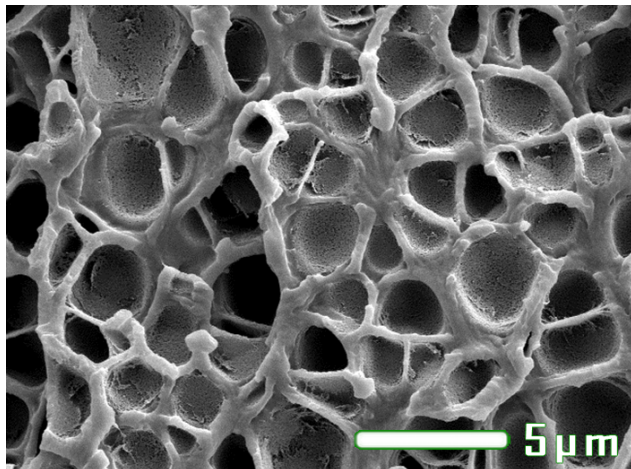


Figure 1.5: Polycarbonate saturated at 5 MPa, foamed at 100°C. Right: zoom on cell wall showing a nano-structure [5]

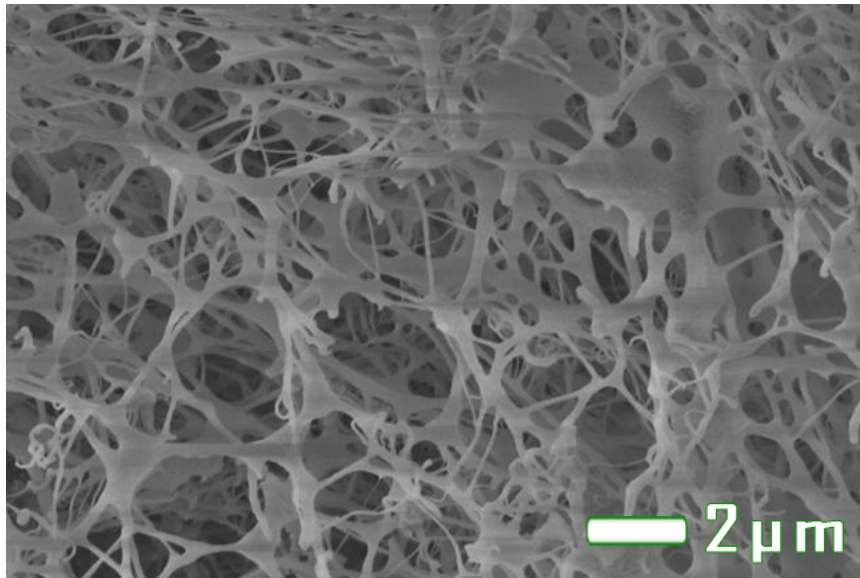


Figure 1.6: HDPE saturated at 3 MPa, foamed at 105°C. [Kumar, unpublished]

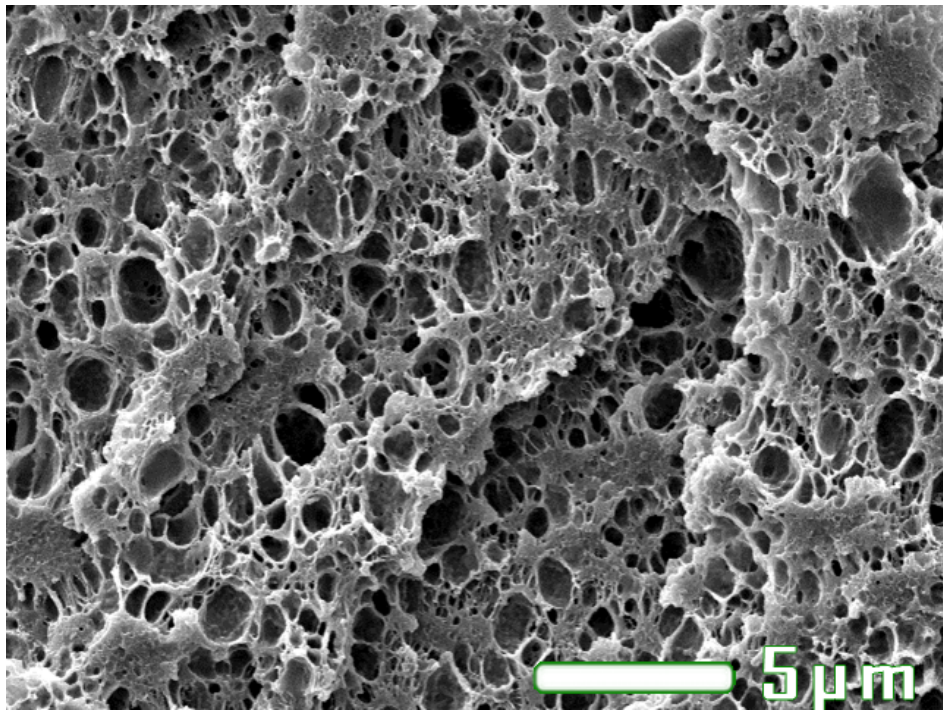


Figure 1.7: ABS saturated at 5 MPa, foamed at 60°C. [Kumar, unpublished]

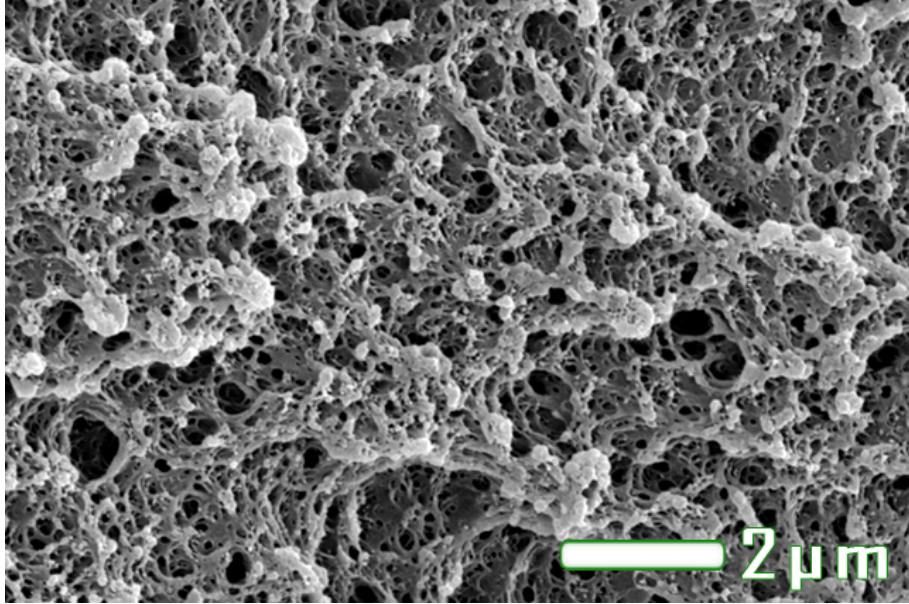


Figure 1.8: PEI (Polyethylenimine) saturated at 5 MPa, foamed at 180°C. This high-temperature thermoplastic produces a nanometer order structure under some conditions. Here, the average cell size is 120 nm. The structure is open-porous and permeable to fluids. [Kumar, unpublished]

1.2 Physics of the Process

1.2.1 Thermodynamics

When a pure solid is placed in a gas environment, there are initially zero gas particles in the solid while the environment has some positive gas concentration N_0 . From statistical thermodynamics, we know that at equilibrium there is a probability of finding a gas particle in any state (such as dissolved into the solid) quantitatively given by the Boltzmann factor [6]

$$P(s) = \frac{1}{Z} e^{-(E(s) - \mu N(s))/kT} \quad (1.1)$$

Where $P(s)$ is the probability P of a system in a microstate with energy E and particle count N . Z is the partition function and μ the chemical potential. From this we can see that, at equilibrium, the solid will become saturated with gas at a concentration $0 < C < N_0$ depending on the properties of the solid, temperature, and gas pressure i.e. $E(s)$, T , and μ . For example, a metal will have a very small concentration since the energy required for a particle to be in the dissolved state is very large. In thermoplastic polymers, the energy is smaller and appreciable gas can be absorbed. For example, PLA and PEI can absorb 10% of their mass in CO_2 at 5 MPa [7, 8].

1.2.2 Diffusion

While thermodynamics quantifies equilibrium states, it does not tell anything about the transient behavior of the system. In the simplest case, Fick's law of diffusion relates the diffusive flux \mathbf{J} to the concentration gradient

$$\mathbf{J} = -D\nabla C \quad (1.2)$$

where D is a second order tensor which linearly maps the concentration gradient vector to the flux vector. If there are no chemical reactions, mass conservation requires the flux divergence to decrease the concentration in the solid:

$$\nabla \cdot \mathbf{J} = -\frac{\partial C}{\partial t} \quad (1.3)$$

If the material is isotropic, $D = DI$ where I is the identity tensor. Furthermore, if D is a constant the formula can be simplified to yield the parabolic partial differential equation also referred to as "the heat equation"

$$D\nabla^2 C - \frac{\partial C}{\partial t} = 0, \quad D > 0 \quad (1.4)$$

This model has been used with high degrees of success by many researchers. A short list of such works is given in the references [3, 5].

1.2.3 Solid Skin

In the solid-state process, there is a finite time in Step 2 where the gas can desorb from the polymer. From equation 1.4, the gas concentration at the edge of the polymer decreases instantaneously when the ambient concentration is dropped. Thus, the outside edges of the polymer remains solid after foaming as there is not sufficient dissolved gas concentration for foaming. This model was explored by Weller's PhD work [3]. The longer the desorption time in step 2, the larger the skin.

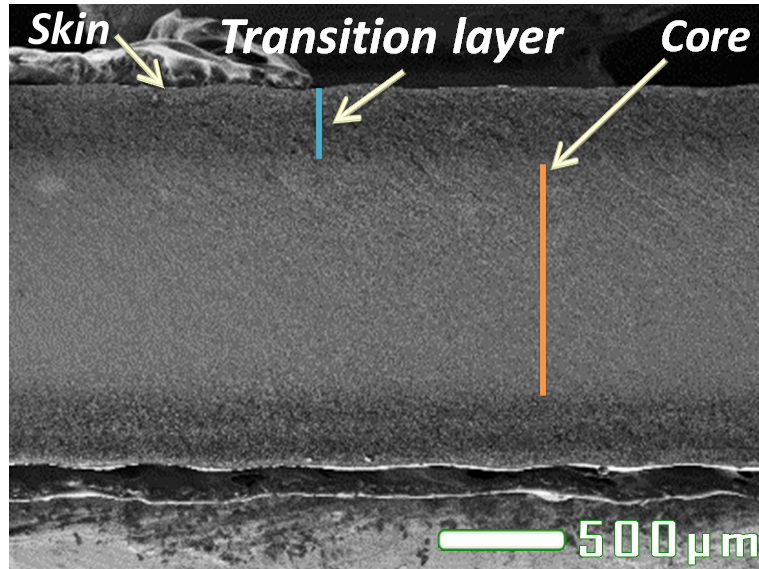


Figure 1.9: Effects of desorption on the microstructure of the foam. The outer layer is a solid skin, then there is a transition region of large cells, and finally the uniform core in the inner part of the sample.

Thin Films If the polymer is very thin, the solid skin can be a significant fraction of the total thickness. By performing dimensional analysis on equation 1.4, the diffusion coefficient is found to have units:

$$D \left[\frac{\text{length}^2}{\text{time}} \right] \quad (1.5)$$

For 1D diffusion (approximated by geometries where width \gg thickness) the only length scale is the thickness of the film. Thus, the diffusion time (of arbitrary definition) must vary as

$$\text{time} \propto \text{thickness}^2 \quad (1.6)$$

Thus, for materials of small dimension (such as thin films or filaments) desorption occurs very rapidly and the time spent in Step 2 of the solid-state microcellular process must be very small. For example, a COC film of 1mm thickness saturated at 5 MPa would lose about half its dissolved CO_2 in 2.5 minutes [8]. As the dimension is further reduced, this time scale rapidly approaches zero.

1.3 Necessity for a Dynamic Seal - Continuous Process

In order to make large quantities of foam, a batch process is used to perform the solid-state foaming. In a typical example, a sheet roll is placed in a large pressure vessel for gas absorption. The roll is then removed and the polymer is unrolled and foamed continually.

The time required to foam the entire roll from one end to another is

$$\text{roll foam time} = \frac{\text{length of roll}}{\text{draw speed}} \quad (1.7)$$

The draw speed is limited by heat transfer and cell growth rate, while the length of the roll must be maximized for economical efficiency. Therefore, the time to foam an entire roll becomes appreciable. For thin geometries as described above, the gas is lost too quickly for this process to be viable. It is therefore necessary to design a method for drawing the polymer out of the pressure vessel through a seal and immediately foamed, while the rest of the roll remains in the pressure vessel at the elevated pressure.

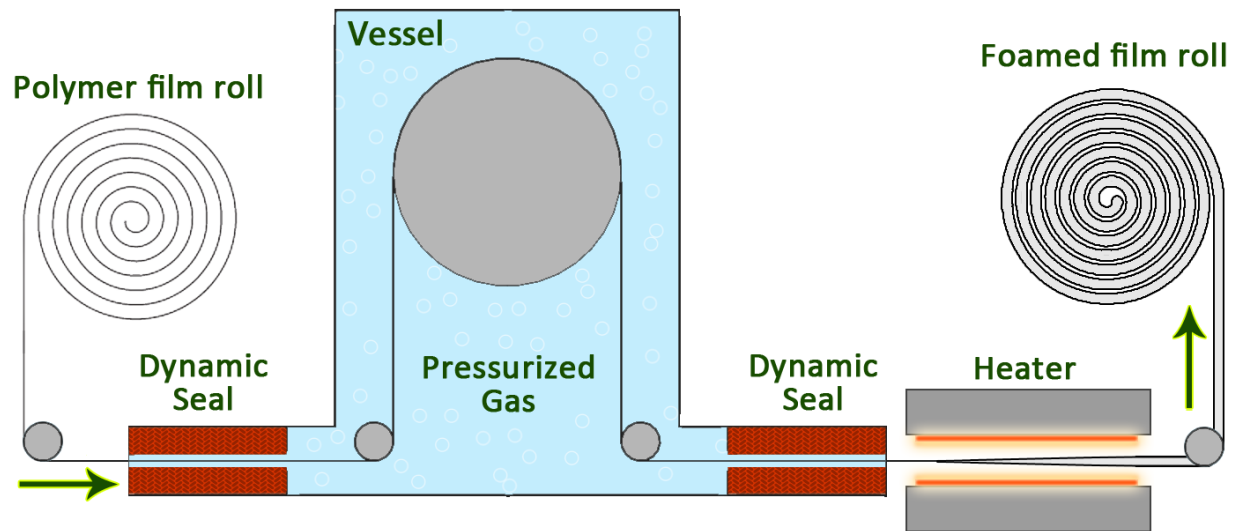


Figure 1.10: Concept of a continuous solid-state microcellular foaming process wherein a roll of raw polymer is continuously passed through a pressurized space for gas sorption and foamed as it exits through the second dynamic seal.

If such a seal can be made, it would create a continuous process for solid-state foaming thin films and fibers. This is the scope of the research covered in this thesis.

Chapter 2

The Dynamic Seal

Pressure seals are found in various applications in engineering. For example, sealing off the lid to a pressure vessel is typically done by an O-ring which is a static seal meaning that there is no relative movement along the seal. Dynamic seals are usually found in shaft systems where a pressure vessel wall must be pierced by a shaft to provide torque transfer between the inside and outside of the vessel. These are radially symmetric and movement is along the azimuthal direction of the shaft. The present task is to design a seal that permits movement of a thin polymer film through an opening, meaning that the direction of movement parallel to the pressure gradient (moves from inside to outside of the vessel as opposed to just rotating like a shaft) and the geometry is not necessarily circular as the case of a shaft; it could be a variety of polymer film thicknesses with high aspect ratio rectangular cross sections. This has been an unsolved problem since the conception of the solid-state microcellular foaming process in the 1980s.

2.1 Facets of a Dynamic Seal

A dynamic seal is a device that allows a solid to freely pass through while blocking a fluid. This necessitates a pressure drop across the seal. The pressure force acting on a volume of fluid can be written as

$$\mathbf{F} = - \oint_{\partial V} p \, d\mathbf{S} \quad (2.1)$$

Using the Divergence Theorem, this can be converted to

$$- \oint_{\partial V} p \, d\mathbf{S} = - \iiint_V \nabla p \, dV \quad (2.2)$$

Therefore, the total pressure force on a fluid particle per unit volume is the gradient of pressure

$$\mathbf{F} = -\nabla p \, dV \quad (2.3)$$

It is now apparent that if a fluid forms a connected space between the inside and outside of the pressure vessel, there will be flow. One approach is to disconnect the inside and outside

fluid by blocking the path using a solid (as is typically done with an O-ring). However, the requirement is that the seal is “dynamic”, i.e. permits slipping; excessive clamping forces increase friction between the seal and the material to be passed through. This is undesirable as it increases drawing forces and stress on the polymer itself. A second approach is to allow fluid leakage and control it to acceptable levels. This was the method developed in the present work.

2.2 Early Ideas

The idea is to design a seal that provides a very large resistance to fluids but a very small resistance to the solid being passed through. One of the first concepts was to use rollers as shown in figure 2.1.

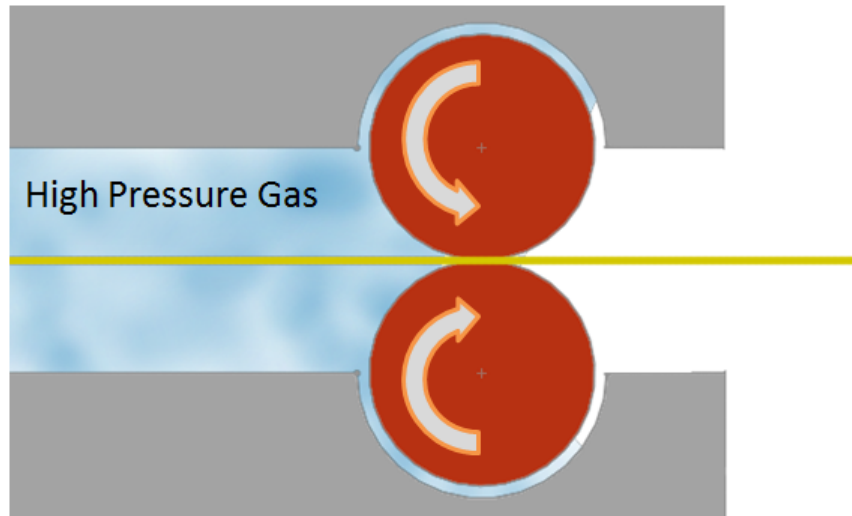


Figure 2.1: A dynamic seal design using rubber rollers to seal the gas but allow a film or fiber to pass through

The gap between the rollers and the vessel walls is to be made very small such that the fluid leak there can be controlled. This can be done by using soft materials to block the gap, or to reduce the gap enough that the wall shear equals the pressure gradient, similar to the working principle of journal bearings (e.g. car engine crankshaft/connecting rod oil bearing).

The problem with this design is sealing the ends in the 3rd dimension (out of the page). Attempts to resolve this issue were unsuccessful as it introduced excess complexity to the design and gave rise to reliability issues. It is precisely the same problem that plagues the Wankel Rotary Engine - creating effective face seals is a difficult task.

2.3 The Idea of a Controlled Leak

A design was conceived where the polymer film or fiber is clamped by a soft material that blocks the flow of fluid, but held lightly enough for the polymer to be free to slide through.



Figure 2.2: A dynamic seal design where the film is clamped by two soft (foam) materials that provide the seal. The film enters the pressure vessel below (not seen) through the slit.

To guarantee that the gas does not leak, a liquid is used to block the opening as shown in figure 2.3. This will be referred to as the “blocking liquid”.

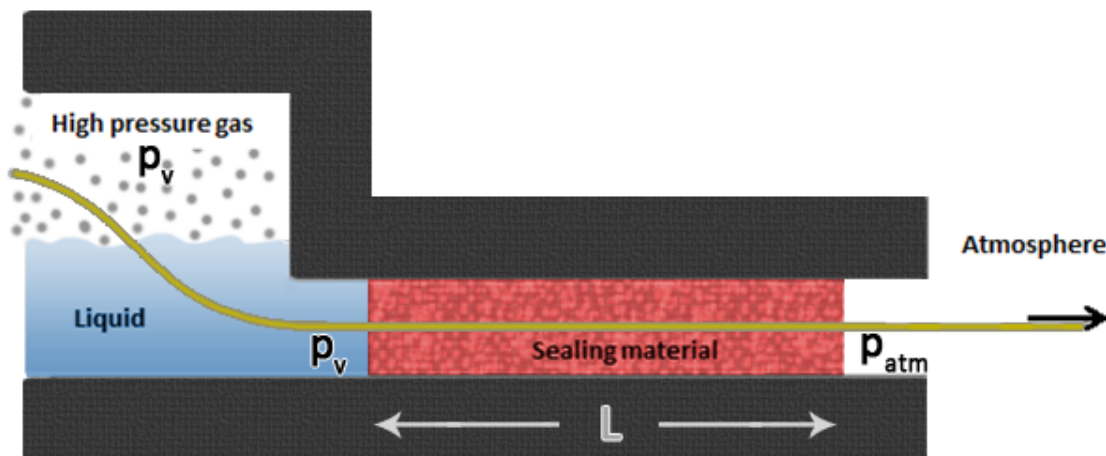


Figure 2.3: A schematic of the dynamic seal idea utilizing a soft sealing material and a liquid to block the gas from escaping. This concept was filed in a provisional patent application in March 2013.

2.3.1 Working Prototype

A small prototype was built to test out the idea. It is a small vessel made of clear polycarbonate. The sealing material used was 0.1 inch thick closed-cell polyurethane foam. Figure 2.4 shows the setup.

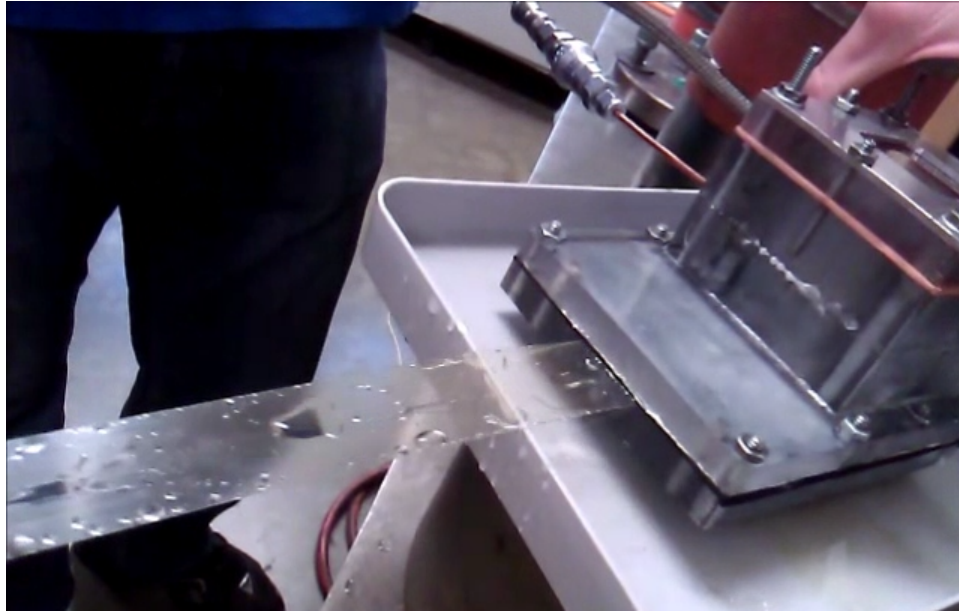


Figure 2.4: Prototype vessel and dynamic seal. It was able to successfully seal 0.60 MPa CO_2 pressure while allowing a PEI film of 0.15 mm thickness to pass through freely with very low friction. The blocking liquid was water which leaked at a very low rate, as seen by the drops present on the film.

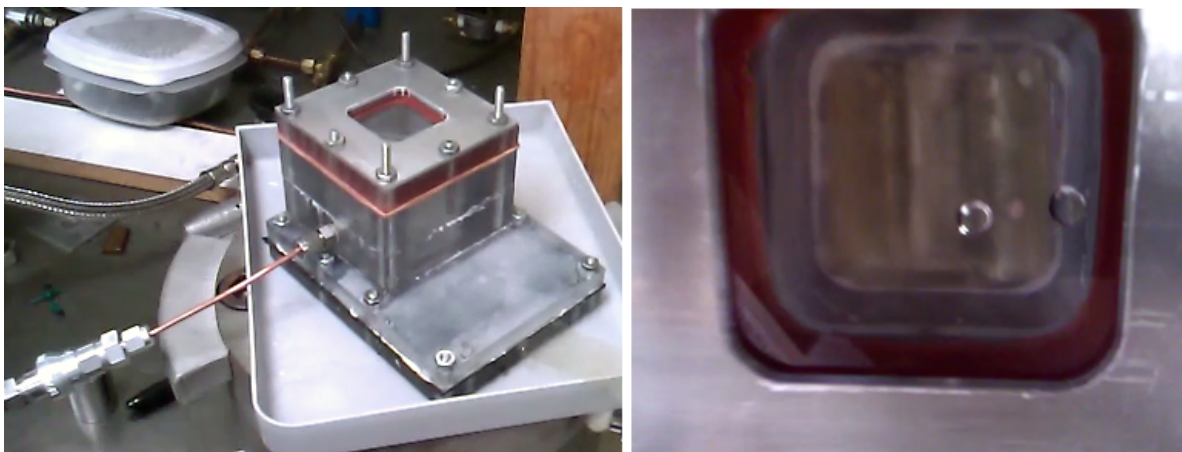


Figure 2.5: Left: setup of the prototype experiment. Right, view of the inside through the clear polycarbonate window. The PEI film is rolled inside the cavity.

It was observed that before pressure was added to the vessel, the PEI film was immovable due to high friction from the large clamping force applied to the plates by the bolts. However, when sufficient pressure was applied, the film became loose and very easy to remove using only index finger and thumb. This result initiated further interest in studying this design and increased confidence that the dynamic seal is viable. When the pressure was further increased in this prototype beyond 1.0 MPa, the seal failed in the following way: the fluid pressure was large enough to push apart the two seal halves and create a channel in the center through which the fluid rapidly leaked.

2.3.2 Blocking Liquid

The idea of using a liquid between the gas and dynamic seal in figure 2.3 is a hallmark of the design. Not only does it prevent the gas from escaping through the seal, it also lubricates the polymer film inside the seal and substantially reduces the leakage rate due to its higher viscosity. This will be shown in more detail later.

Concerns A pure liquid cannot exist in equilibrium with another gas present; some of the gas will dissolve in the liquid. The molality of the dissolved gas m is related to the partial pressure of the gas outside P and the Gibbs energy difference between the gaseous and aqueous states ΔG° by a result known as “Henry’s law” [6].

$$m = \frac{P}{P^\circ} e^{-\Delta G^\circ/RT} \quad (2.4)$$

Where the G° and P° denote properties at standard conditions (101.325 kPa, 293.15 K). Another aspect in considering when choosing the blocking liquid is toxicity, availability, and adhesion. The fluid should be inexpensive and readily available, safe, and not stick to the polymer as to contaminate it. It was observed in experiments that water is very suitable, as it did not dissolve significant CO_2 (very low amount of “fizz” observed) and could be very easily wiped off the polymer. For this reason, water was used as the blocking liquid in this work.

Chapter 3

Seal Materials

After the initial success of the first prototype, it became clear that a pivotal role in the design is played by the sealing materials. The pressure must be dropped along the materials, and the polymer must slide between them: therefore, the mechanics of the process is largely controlled by the choice of materials. A few options were investigated through both analysis and experimentation.

3.1 Porous vs. Nonporous Materials

The main types of materials considered are porous (permeable) and nonporous (non-permeable) foams. These have similar mechanical properties but very different fluid interaction.

3.1.1 Nonporous Materials

If a nonporous material is used, the liquid must flow between the polymer (moving at a speed u_0) and the seal material surface.

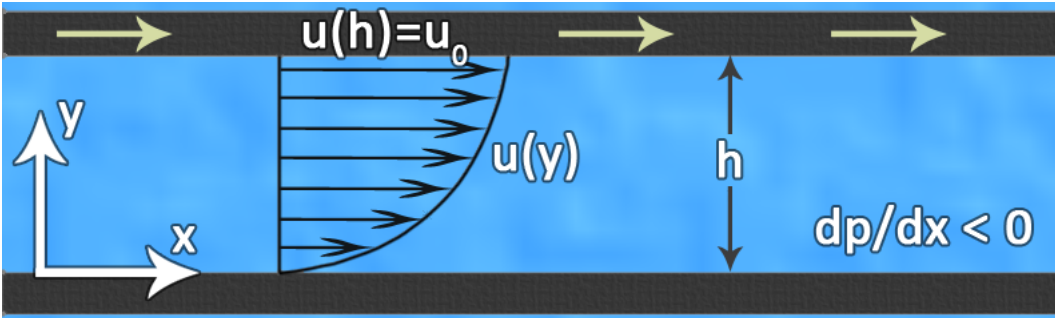


Figure 3.1: Coordinate system defining the fluid problem.

If the surfaces are parallel and h (the distance between them) small such that the Reynolds

number, defined by

$$Re \equiv \frac{\rho h u}{\mu} \quad (3.1)$$

is sufficiently low, a stable solution to the Navier-Stokes equations (known as Plane Poiseuille Flow) can be used to model this flow. This is a steady solution which assumes fully developed flow which occurs very rapidly when h is small since the boundary layers grow as $\delta \propto h/\sqrt{Re_x}$ (a result from dimensional analysis) [9].

$$u(y) = u_0 \frac{y}{h} + \frac{1}{2\mu} \left(\frac{dp}{dx} \right) (y^2 - hy) \quad (3.2)$$

The important aspects to be taken from this equation are:

- The flow velocity is nonlinear in y
- The flow rate is nonlinear with h
- The flow velocity is linear in pressure gradient dp/dx
- The flow is uniform in x
- The static pressure p is uniform in y

This formula can be used to calculate what the leakage rate would be, assuming the the dynamic seal conditions match the ideal geometry of the problem as above.

$$Q/W = \int_0^h u(y) dy = \frac{u_0 h}{2} - \left(\frac{dp}{dx} \right) \frac{h^3}{12\mu} \quad (3.3)$$

As a rough calculation, suppose that the gap between the seal material and the polymer is 10 micrometers and the seal is 1/3 meters (about 1 foot) long. Using the viscosity of water at room temperature, equation 3.3 predicts a leakage rate of approximately 6 mL of water per second for a polymer 1 meter wide. This number is very low as a pump can easily keep up with such a leakage, therefore the feasibility of this seal is favorable.

In practice, the condition of perfectly parallel planar surfaces is not met. The normal force on an area element of the seal surface is

$$\delta \mathbf{F} \cdot \mathbf{n} = p dS \quad (3.4)$$

The fluid can therefore deform the material upwards and create a fluid pocket in what will be referred to as “pooling”, shown in figure 3.2. This was the failure mode of the small prototype dynamic seal discussed in the previous chapter.

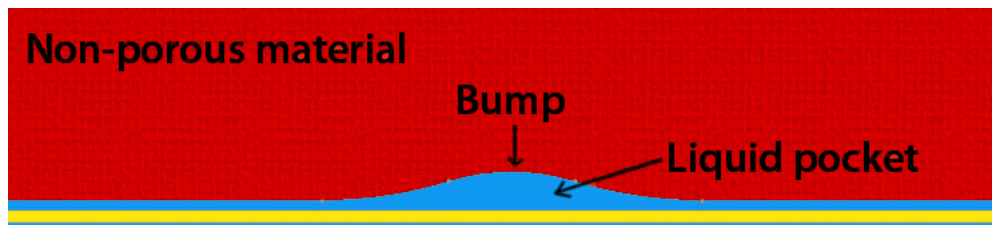


Figure 3.2: Liquid deforming the seal material to create a pocket. This large fluid volume has a much smaller pressure drop.

If this pocket extends to create a channel, the fluid will flow rapidly through this gap. This can be seen from the definition of viscosity

$$\tau \equiv \mu \left. \frac{du}{dy} \right|_{y=0} \quad (3.5)$$

and the definition of flow rate

$$Q = \bar{u}A \quad (3.6)$$

Since the wall shear τ must match the pressure drop (for steady flow), the leakage rate is increased by increasing the area A and by the average velocity \bar{u} increasing as well. This can also be seen qualitatively from the h^3 dependence on flow rate in equation 3.3.

The presence of a fluid layer between the polymer and the seal material creates a medium for the polymer to glide on without dry (Coulomb) friction as in an Air Caster. The only resistance, theoretically, is the fluid shear force (related to viscosity) given by

$$\delta F_x = \tau dA \quad (3.7)$$

3.1.2 Porous Materials

If the seal material has small interconnected pores, the liquid can flow inside the material. Since the pores are small, the Reynolds number is small also and the flow is laminar in the material. Flow through porous media is therefore often modeled by a linear relationship between pressure gradient and flow (known as Darcy's law)

$$\mathbf{q} = \frac{-k}{\mu} \nabla p \quad (3.8)$$

Since the net force per unit volume on a continuum particle is

$$\mathbf{F} = -\nabla p \parallel \mathbf{q} \quad (3.9)$$

it is apparent that the force on a porous material is parallel to the direction of flow.

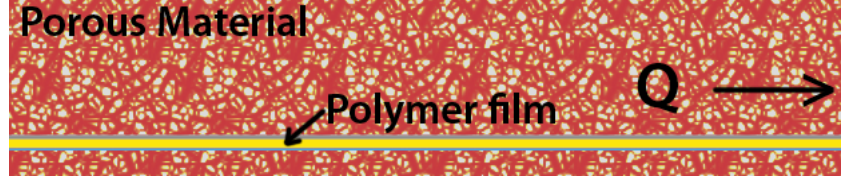


Figure 3.3: A porous seal material allows flow within. The fluid forces on this material are very different than those on the nonporous material.

Therefore, the fluid forces on the material are

$$F_y = \hat{\mathbf{y}} \cdot \nabla p \, dV = 0 \quad (3.10)$$

and

$$F_x = \hat{\mathbf{x}} \cdot \nabla p \, dV = |\nabla p| \, dV \quad (3.11)$$

meaning that “pooling” is not possible for a porous material and the net force is along the direction of the polymer and equal to the pressure gradient (for a unit volume).

Pressure Distribution With further analysis of equation 3.8, the pressure distribution in the material can be found. Since the fluid of interest is incompressible, we have by continuity

$$\nabla \cdot \mathbf{q} = 0 \quad (3.12)$$

Since k (permeability [m^2]) and μ (dynamic viscosity [$Pa \cdot s$]) are constants, the equation reduces to

$$\nabla \cdot \left(\frac{-k}{\mu} \nabla p \right) = \frac{-k}{\mu} \nabla^2 p \quad (3.13)$$

so the fluid pressure must satisfy Laplace’s equation everywhere in the porous material

$$\nabla^2 p = 0 \quad (3.14)$$

For 1D flow, which approximates the dynamic seal quite appropriately, the solution to equation 3.14 is a line $p(x) = ax + b$ which minimizes the gradient (and force).

3.1.3 Analysis

In both porous and nonporous materials, the ideal flow equations show that the pressure drop is linear with distance. For Poiseuille flow, equation 3.2 shows that dp/dx is a constant, while equation 3.14 has a line as the unique solution. It is important to note that a linear pressure drop offers the lowest maximum gradient for any curve that joins two points. This is a well known result from Calculus of Variations. Since by equation 3.9 the force is equal to the pressure gradient, it means that the maximum pressure force experienced by a volume element is also minimized. The pressure is predefined at the boundaries, $p = p_{vessel}$ at one end and $p = p_{atm}$ at the other. The pressure must be a continuous function of seal distance

and connect these two points. Therefore, a linear pressure drop requires the smallest seal length for a given maximum gradient. If the pressure distribution is non-linear, there must be a point where the gradient is larger than the average (mean-value theorem of calculus). Therefore, at this point the seal is outperforming the rest of the seal since the flow rate is constant throughout by conservation of mass, meaning that the seal is not optimized. This is why a linear pressure drop is seen as a key objective in the rest of the work on the dynamic seal.

3.2 Experiments

To test the applicability of the model to the porous materials available for the project, experiments with several open-cell foams purchased from McMaster-Carr company were conducted. The objective was to verify that the mathematical model can accurately predict the flow behavior and thus allow the model to be confidently used when moving forward with the project.

3.2.1 Experimental Methods

A circular test apparatus was constructed as shown in figure 3.4.

Water pressurized using CO_2 tanks and control valves was directed to the center orifice of the jig, through the porous seal material radially, and out to the atmosphere. The water was then collected for a fixed amount of time (typically 2 minutes) at each pressure to generate a plot of mass flow rate versus pressure.

To understand what Darcy's law would predict for this experiment, equation 3.14 was solved in cylindrical coordinates with the boundary conditions to match the experimental setup sketched in figure 3.6.

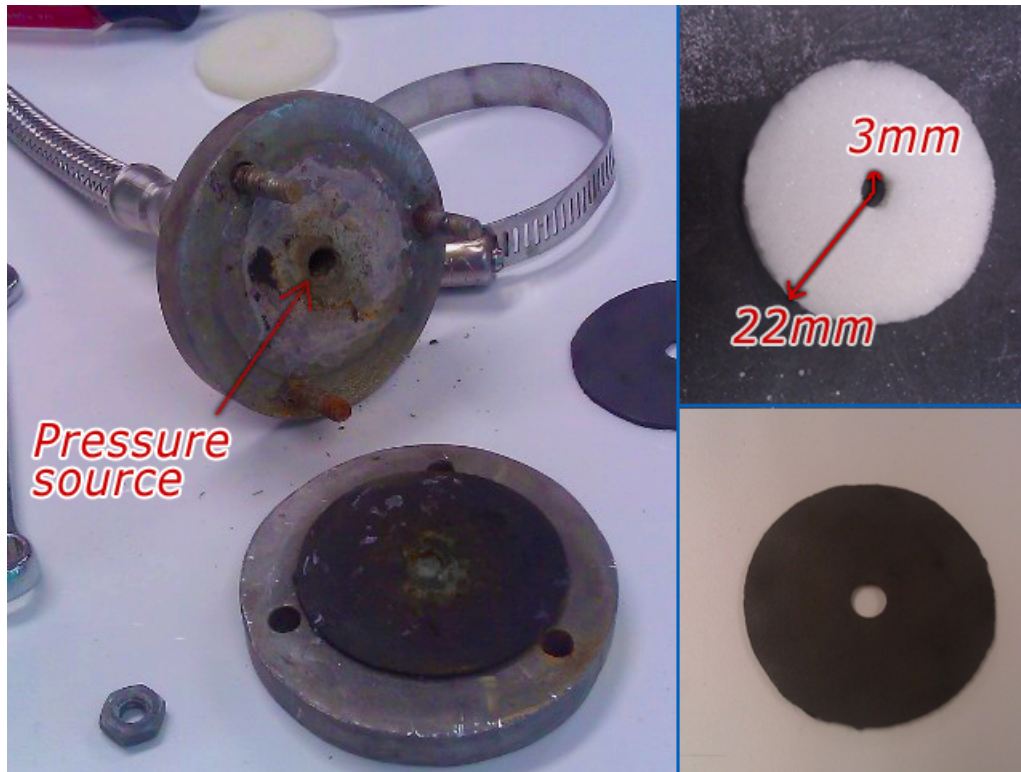


Figure 3.4: Aluminum experimental jig. Fluid is pushed by a controlled pressure through the small orifice at the center and flows through the porous disk to the outside edge exposed to the atmosphere. The porous material is adhered to the aluminum by contact cement on both faces. The distance between the plates was controlled by adjusting the nuts.

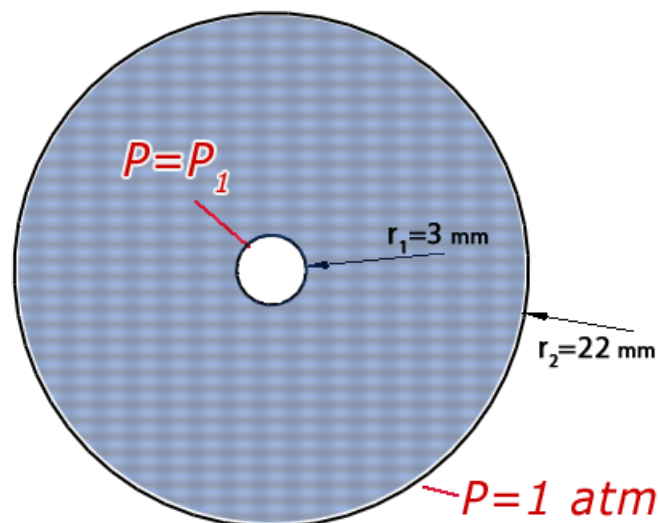


Figure 3.6: Problem definition. The domain is a disk with inner radius $r_1 = 3$ mm and outer radius $r_2 = 22$ mm. The inner boundary has an applied pressure p_1 and the outer boundary is at p_2 taken as 1 atmosphere.

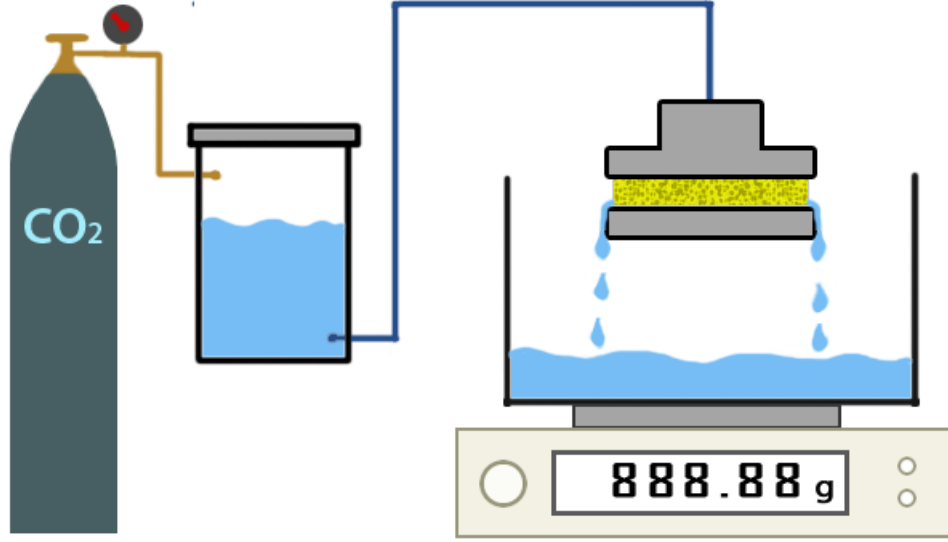


Figure 3.5: Experimental setup diagram. Pressurized water flows radially through the porous material and is collected and weighted using a digital scale.

Expressing the Laplacian in cylindrical coordinates

$$\nabla^2 f(r, \theta, z) = \frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial f}{\partial r} \right) + \frac{1}{r^2} \frac{\partial^2 f}{\partial \theta^2} + \frac{\partial^2 f}{\partial z^2} \quad (3.15)$$

transforms equation 3.14 into

$$\frac{1}{r} \frac{\partial}{\partial r} \left(r \frac{\partial p}{\partial r} \right) = \frac{1}{r} \left(\frac{\partial p}{\partial r} + r \frac{\partial^2 p}{\partial r^2} \right) = \frac{1}{r} \frac{dp}{dr} + \frac{d^2 p}{dr^2} = 0, \quad r_1 \leq r \leq r_2 \quad (3.16)$$

since the experimental setup is axisymmetric and uniform in z . This 1D problem has boundary conditions $p(r_1) = p_1$, $p(r_2) = p_2$. The general solution to the ODE (3.16) is of the form

$$p(r) = c_2 + c_1 \log(r) \quad (3.17)$$

Solving for constants c_1 and c_2 using the boundary conditions gives the unique solution to the problem

$$p(r) = \frac{p_2 \log(r_1) - p_1 \log(r_2) + (p_1 - p_2) \log(r)}{\log(r_1) - \log(r_2)} \quad (3.18)$$

The pressure gradient can be computed using the polar form of the gradient operator

$$\nabla f = \frac{\partial f}{\partial r} \hat{\mathbf{r}} + \frac{1}{r} \frac{\partial f}{\partial \theta} \hat{\boldsymbol{\theta}} \quad (3.19)$$

The gradient then becomes the ordinary derivative of p with respect to r

$$|\mathbf{q}| \frac{\mu}{k} = -\frac{dp}{dr} = \frac{p_2 - p_1}{r \log(r_1/r_2)} \quad (3.20)$$

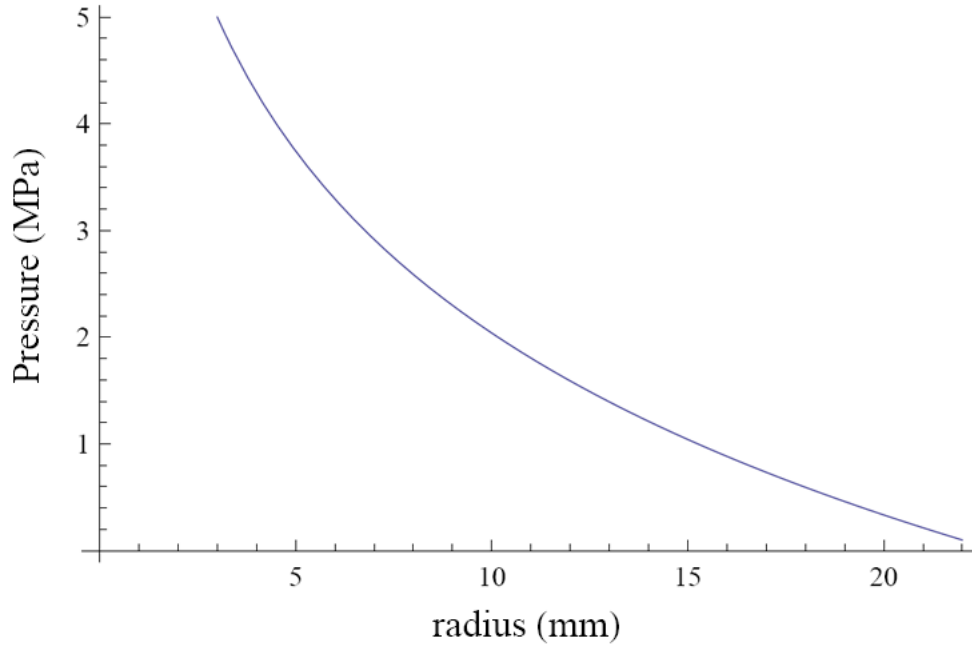


Figure 3.7: Plot of predicted pressure versus radius in the porous disk. The gradient is steepest at $r = r_1$

Thus, equation 3.20 shows that the flow rate is linear with $\Delta p \equiv p_2 - p_1$ and is 0 when $\Delta p = 0$. Plotting $|\mathbf{q}|$ vs. p would yield a straight line.

3.2.2 Results

The experimental results for each material are shown below in plot form. Raw data tables are in the appendix.

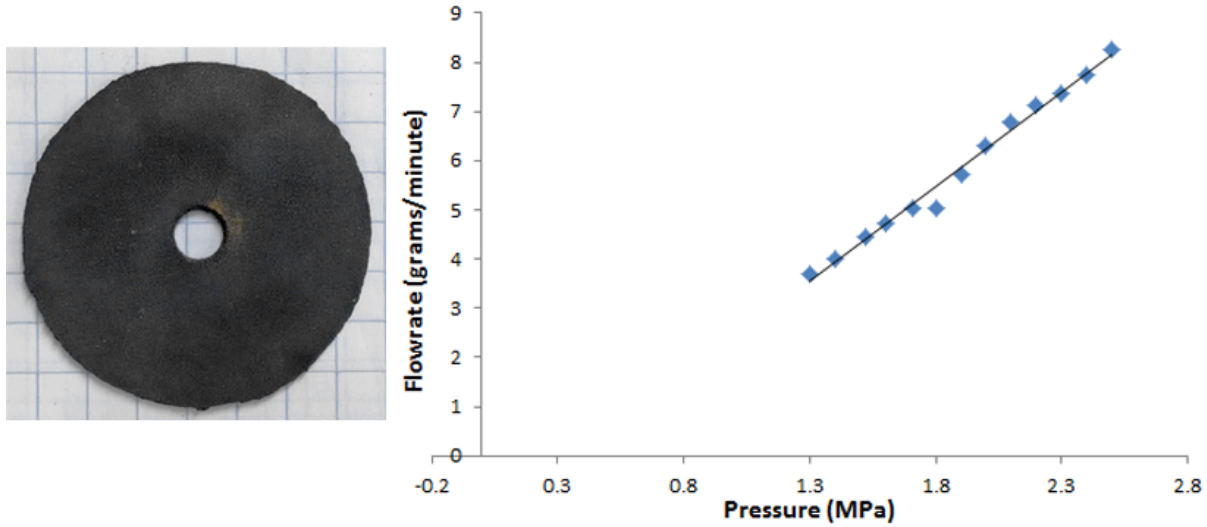


Figure 3.8: Polyurethane open cell foam of 1/16 inch (1.59 mm) thickness, 20 lb/ft³ density. The sample was compressed to 1.0 mm and the water was allowed to leak for 2.0 minutes at each pressure. $R^2=0.989$

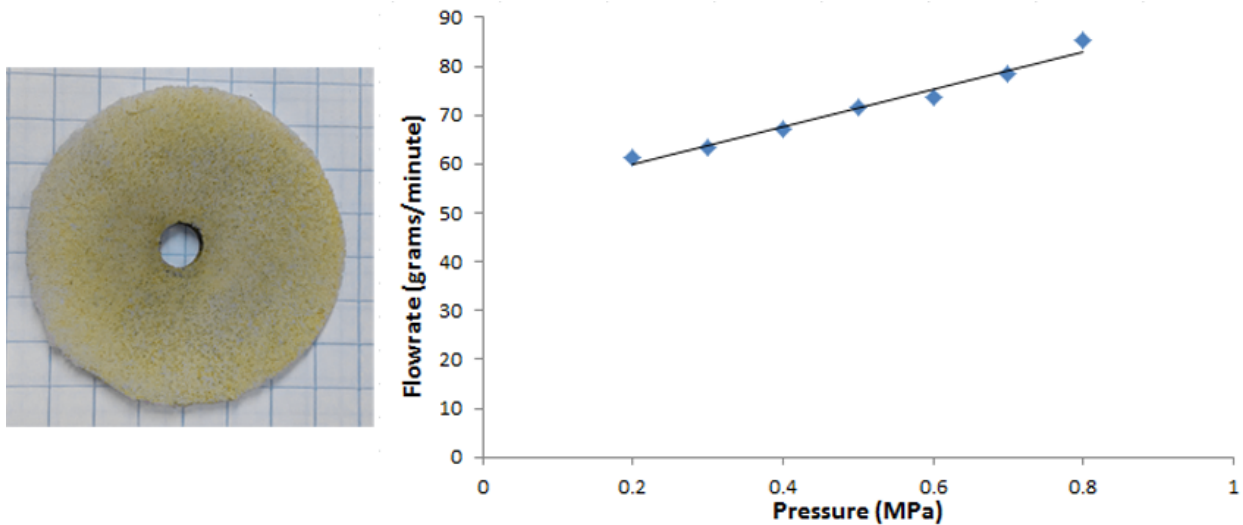


Figure 3.9: Silicone open cell foam of 1/8 inch (3.18 mm) thickness, 12 lb/ft³ density. The sample was compressed to 1.4 mm and the water was allowed to leak for 3.0 minutes at each pressure. $R^2=0.974$

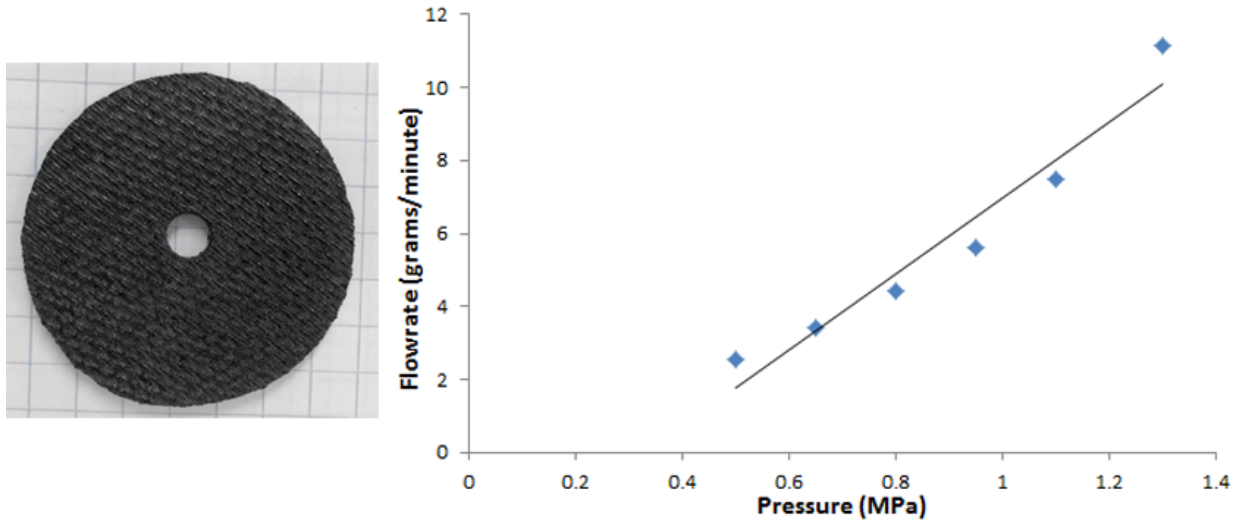


Figure 3.10: Natural Gum rubber open cell foam of 1/8 inch (3.18 mm) thickness, 30 lb/ft³ density. The sample was compressed to 1.0 mm and the water was allowed to leak for 5.0 minutes at each pressure. $R^2=0.943$

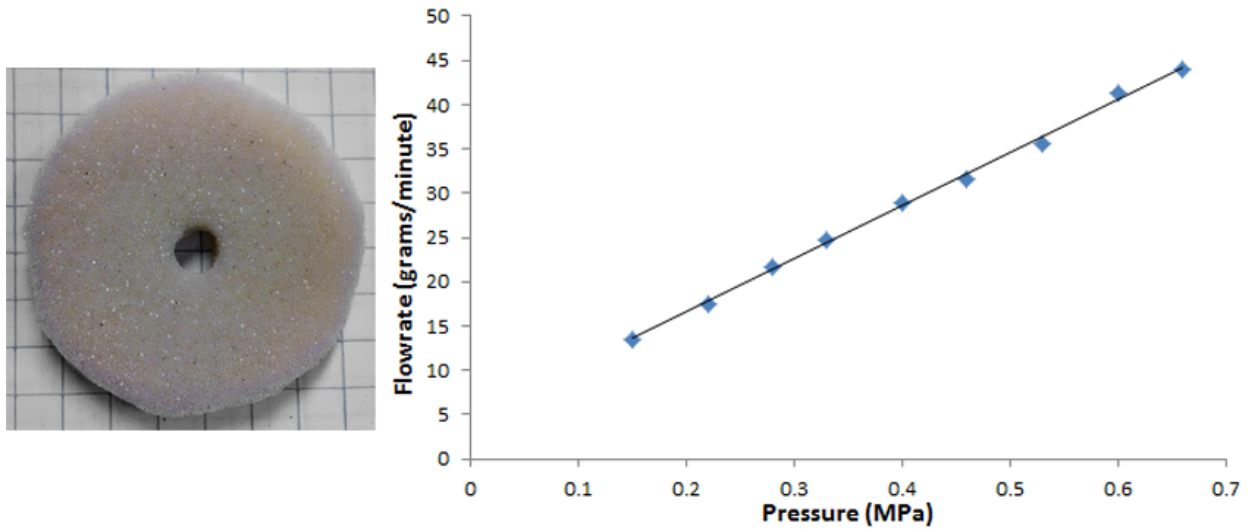


Figure 3.11: Polyurethane open cell foam of 1/4 inch (6.35 mm) thickness, 1.8 lb/ft³ density. The sample was compressed to 2.0 mm and the water was allowed to leak for 1.0 minute at each pressure. $R^2=0.997$

3.2.3 Conclusions

Each material was tested to failure of the material by tearing along the radial direction. This happened when the pressure was enough to burst the material explosively which let out large amounts of water. At this point, the experiment was terminated. It is important to note that

although the pressures seem small, it was already shown and stated multiple times in the previous sections that the fluid forces are proportional to the gradient of pressure and not to pressure. Since the geometry here is small (3 mm inner radius and 22 mm outer radius) the gradients are large even for seemingly small pressure differences. Using equation 3.20, the gradient can be calculated for a Δp of 0.65 MPa which is the lowest of all experiments (and occurred for 1.8 lb/ft³ density polyurethane foam)

$$|\nabla p| \Big|_{r=r_1} = \frac{0.65 \times 10^6 - 0}{0.003 \times \log(0.003/0.022)} = -1.09 \times 10^8 \text{ Pa/m} \quad (3.21)$$

A gradient of this magnitude would drop 5 MPa in only 4.6 cm of dynamic seal length. It is anticipated that the dynamic seal can be built much larger than 4.6 cm so these gradients are very large in comparison to what they would be in a dynamic seal to be built.

The results show linearity between pressure and flow rate with $R^2 \approx 1$ for all materials, however this is only for a subset of pressures. If the line is extended, the point (0,0) would not lie on the graph which is required by the model. Non-idealities such as surface tension and compressibility of the material were not considered. However, the experiments do confirm that for the pressure gradients expected in the dynamic seal, the behavior is linear as predicted by the model. The materials with smaller pores such as the 20 lb/ft³ polyurethane and the 30 lb/ft³ natural gum showed much slower flow rates due to their low permeability k in equation 3.8. Therefore, when choosing a material for the dynamic seal it is important to note this parameter as well as the stiffness modulus and coefficient of friction between it and the polymer.

3.2.4 Experiment with Fishing Line

After the above experiments were completed, the question arose out of curiosity as to what would happen if a fishing line (0.4 mm nylon filament) was threaded between the porous materials in the test jig, through the center hole, and into the pressure vessel.

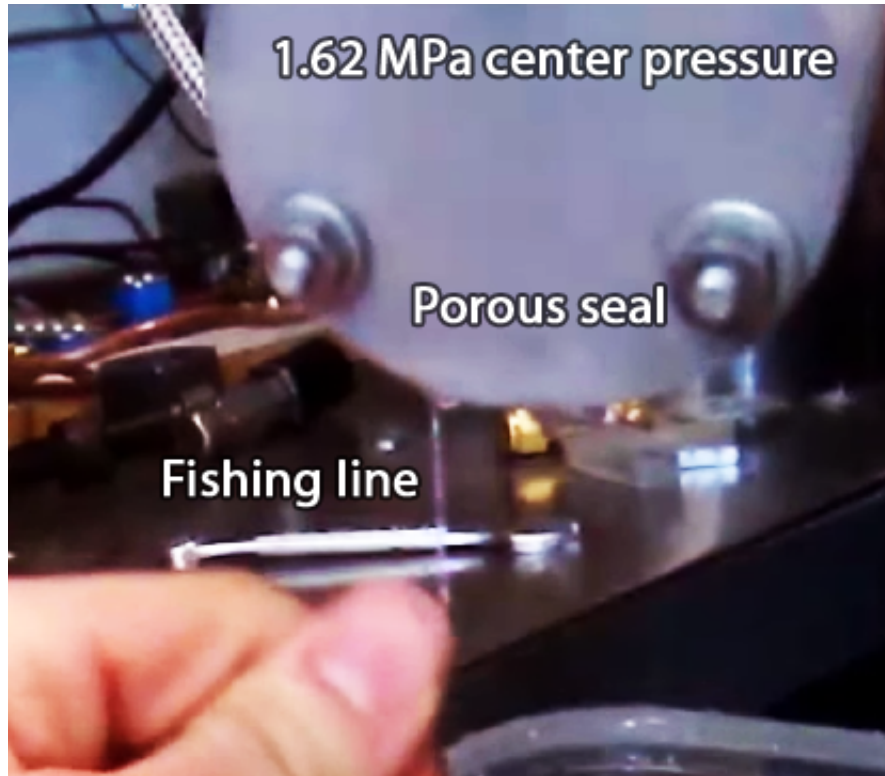


Figure 3.12: A 0.4 mm nylon fishing line was threaded through the center hole and out between the porous seal material (1.8 lb/ft^3 polyurethane foam). A pressure of 1.62 MPa was maintained at the center of the seal and the filament was drawn by hand easily with only a few water drops of leakage.

As can be seen in figure 3.12 the success of this test gave merit to the idea of using porous materials in the dynamic seal. The next task, therefore, was to build a dynamic seal for a polymer film with an entering pressure of 5 MPa.

Chapter 4

Design of a Pressure Vessel - Dynamic Seal System

With the information acquired through the experiments discussed previously, a lab scale dynamic seal design was planned. The purpose was to test these theories and observe how the design handles larger pressures and longer seal lengths. Emphasis was placed on strength and stiffness such that deformations would not be significant enough to change the geometry far away from the ideal design shape.

4.1 Vessel Design

A larger scale vessel capable of containing 6 MPa gas pressure was designed to test the dynamic seal materials on a full scale. For machining convenience, a 0.1 x 4.0 inch slit was cut on the bottom of a cylindrical vessel and was used to allow the polymer both in and out through the same opening. Two parallel plates bolted together provide the clamps that sandwich the seal material and polymer to create the dynamic seal. Figure 4.1 shows the idea.

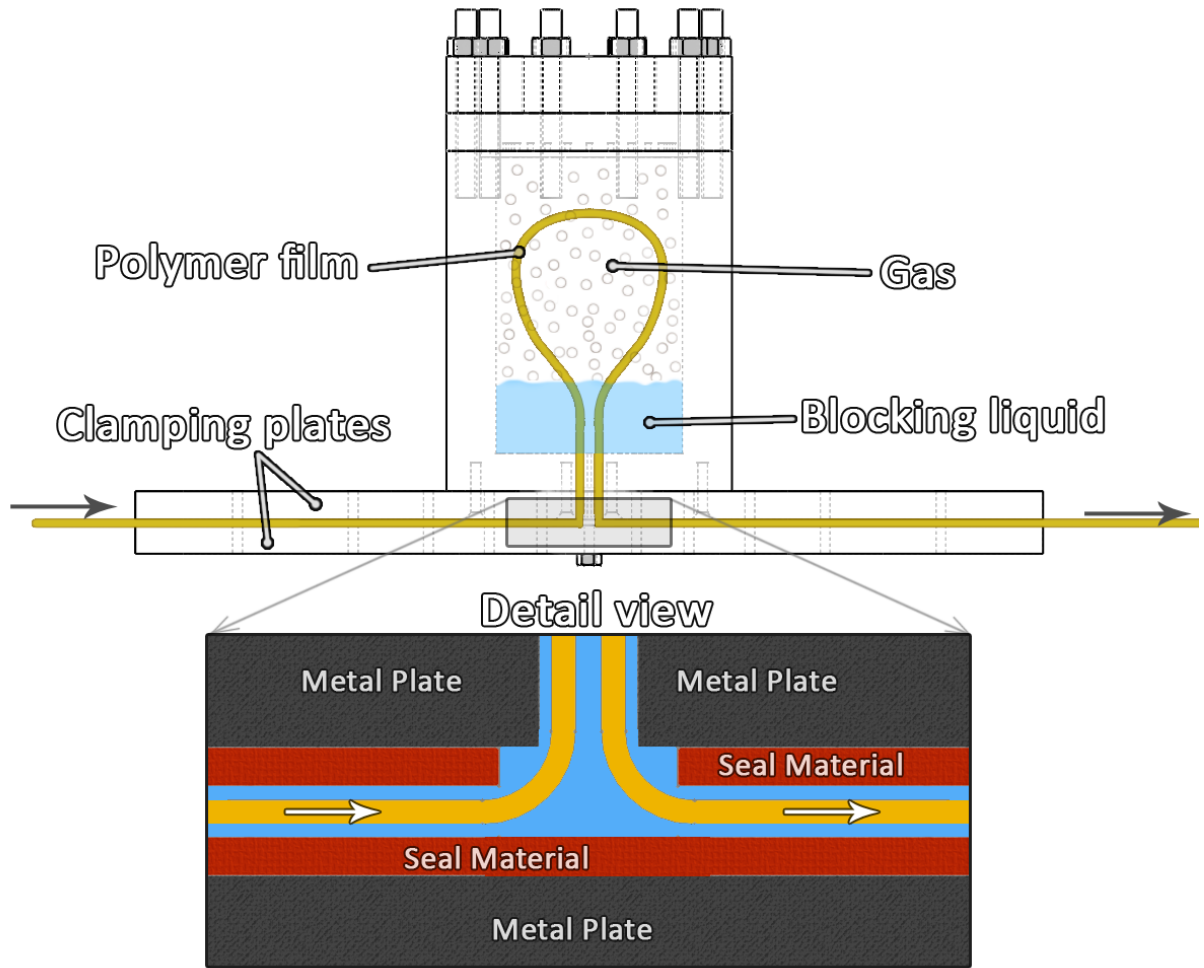


Figure 4.1: Top: CAD representation of the full size prototype vessel. The polymer (yellow) enters through the bottom seals, goes up through a slit in the bottom of the vessel, then returns through the same slit and exits on the opposite side. Bottom: Detail view of the polymer entering in and out of the slit, all between the seal material (red).

The entire vessel (except the slit) was machined in the UW Mechanical Engineering Machine Shop during Summer 2013. A CAD drawing showing most of the important dimensions is shown in figure 4.2

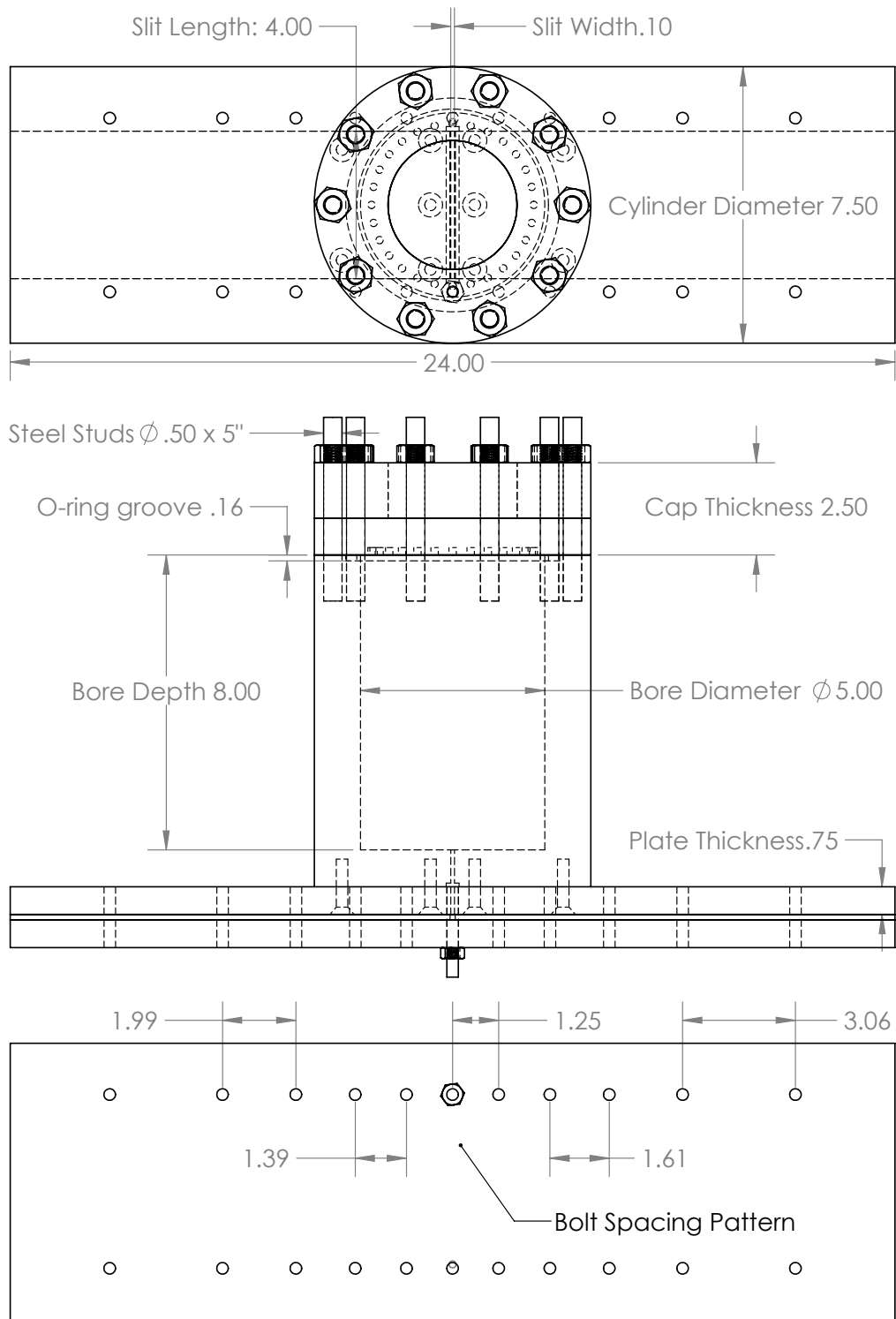


Figure 4.2: CAD sketch of the vessel body with dimensions in inches.

Opposite to the seal plates, the vessel contains a cap held in with ten 1/2-13 threaded

steel studs and nuts. The cap has a 3.5 inch diameter polycarbonate window through which the inside of the vessel can be seen.

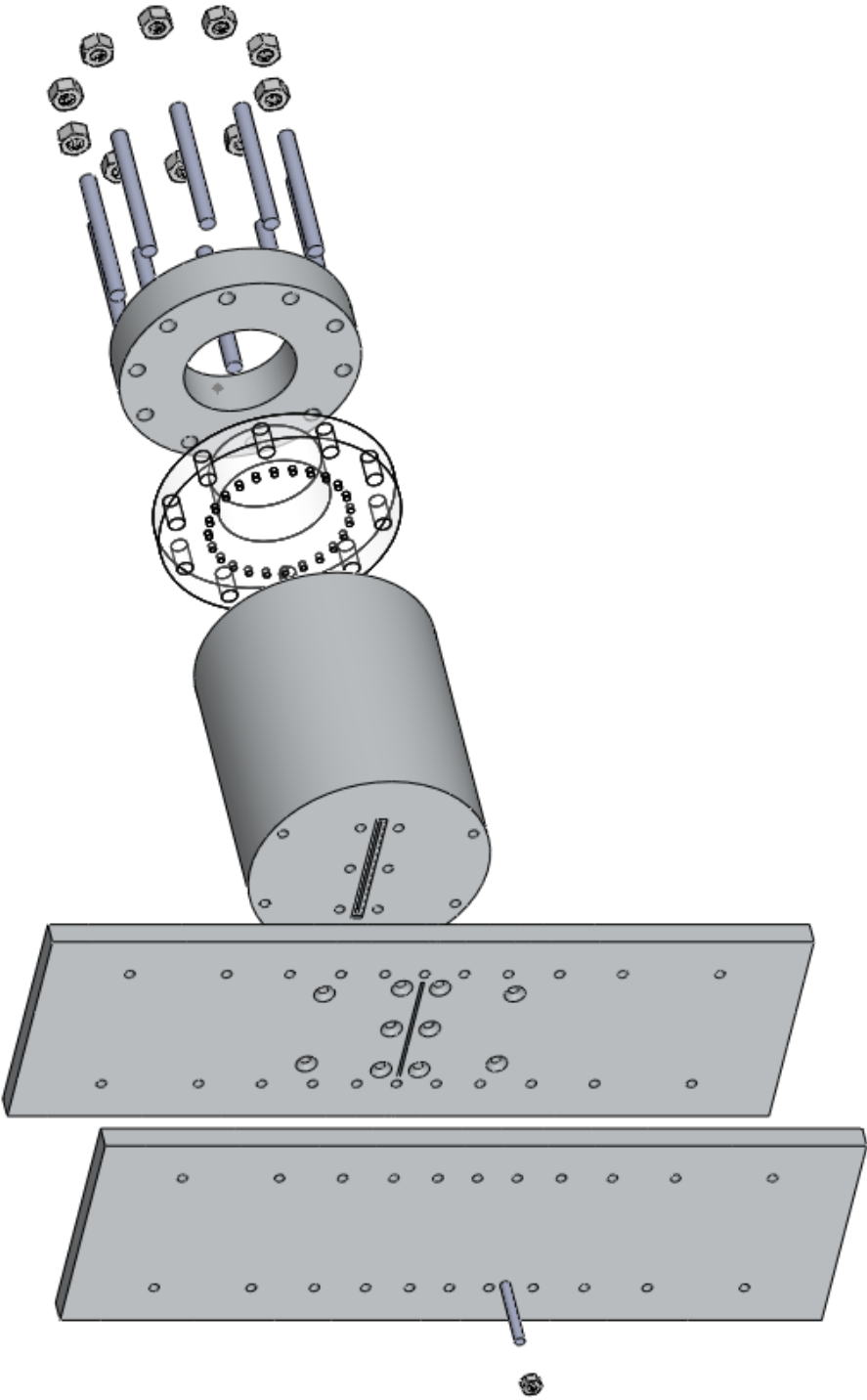


Figure 4.3: Exploded view of the pressure vessel CAD model.

A series of 30 concentric white LEDs inside the vessel cap provide illumination. The vessel itself is the electrical ground, and a positive voltage wire is threaded through a small hole in the cap and sealed with epoxy. Since the LEDs operate at 3.3 V and the available 6-cell lead-acid battery provides >12 V, the LEDs were broken into 3 series groups of 10 parallel, requiring a ~ 10 V power source. To step down from 12 V to 10 V, a LM317T adjustable voltage regulator was used.

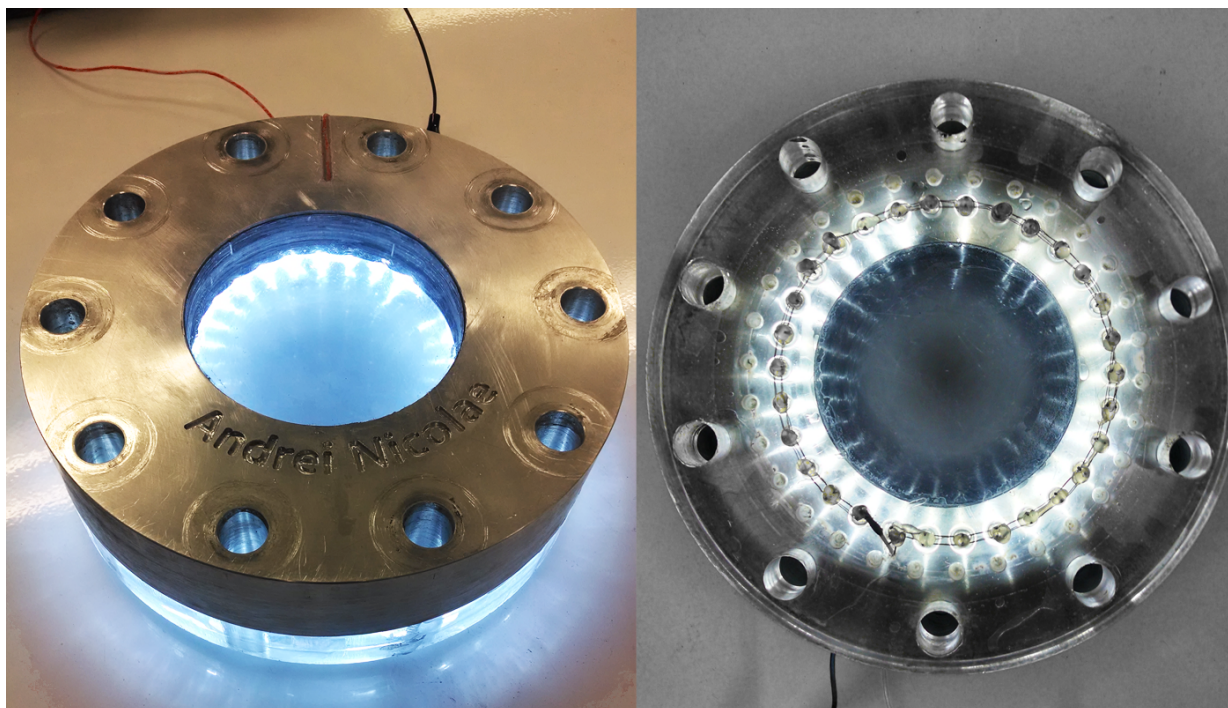


Figure 4.4: White LEDs in the cap illuminate the vessel so that the vessel interior can be seen through the clear window.

The vessel was bored on a Haas TL-3 CNC lathe using a boring bar. This operation required approximately 10 hours to complete. The slit was cut using a waterjet by the Seattle local company “Hydroslice Aerospace” for a fee.



Figure 4.5: View into the vessel showing the slit on the bottom through which the polymer can enter and exit the pressure vessel.

The vessel and plates are 6061-T6 Aluminum Alloy purchased from OnlineMetals, a local company in Seattle. The material is strong yet easily machinable with the High-speed steel (HSS) cutters available in the machine shop. The hardware items such as O-rings, nuts, studs, bolts, and washers were purchased from McMaster-Carr online. The total cost for materials was under \$700.

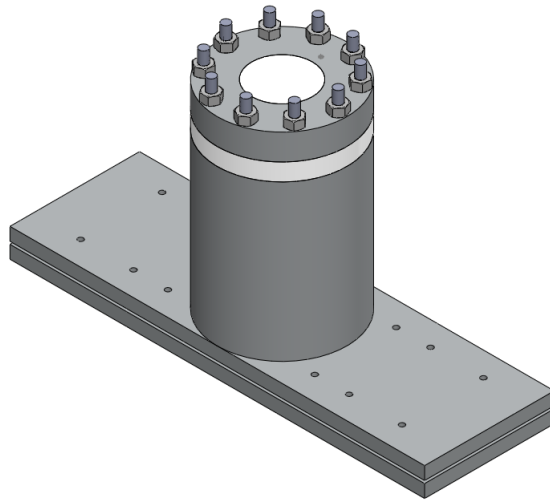


Figure 4.6: Isometric CAD view of the vessel to be built.

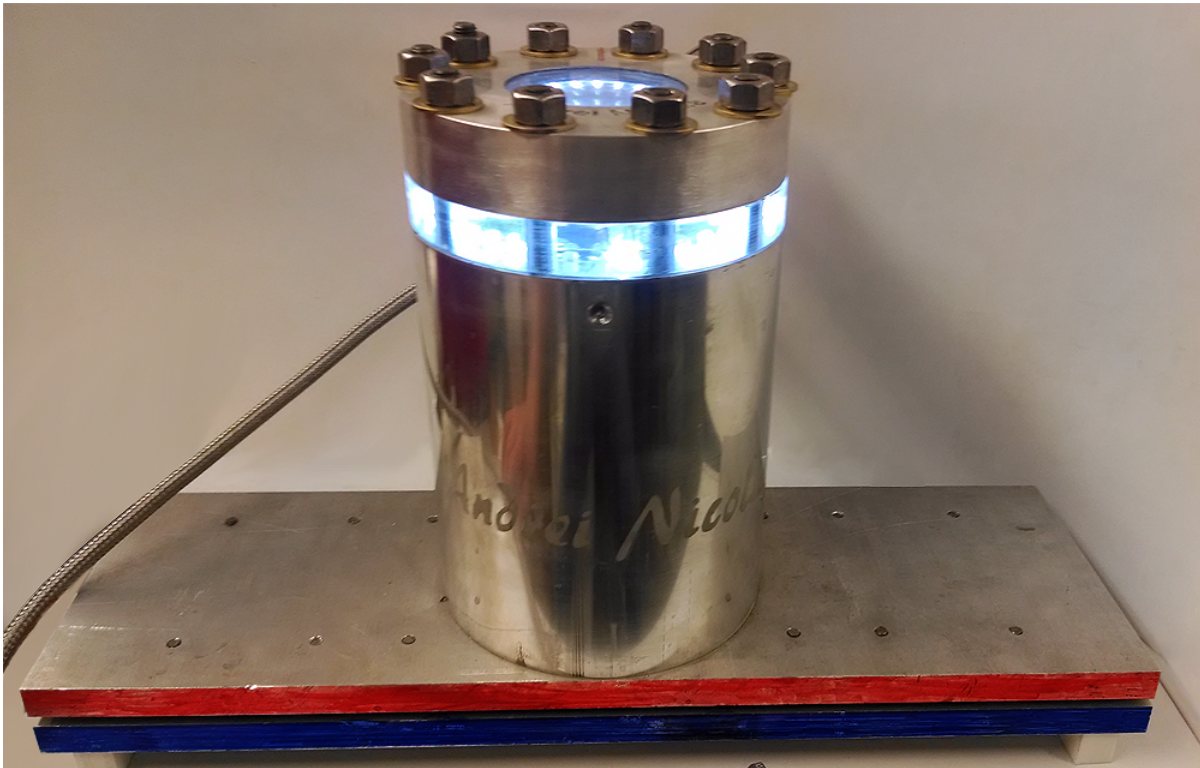


Figure 4.7: Side view photograph of the complete assembled vessel. It has been tested at the full CO_2 tank pressure of 5.17 MPa and no leaks were found. The gas was left pressurized overnight and no drop in pressure was observed.

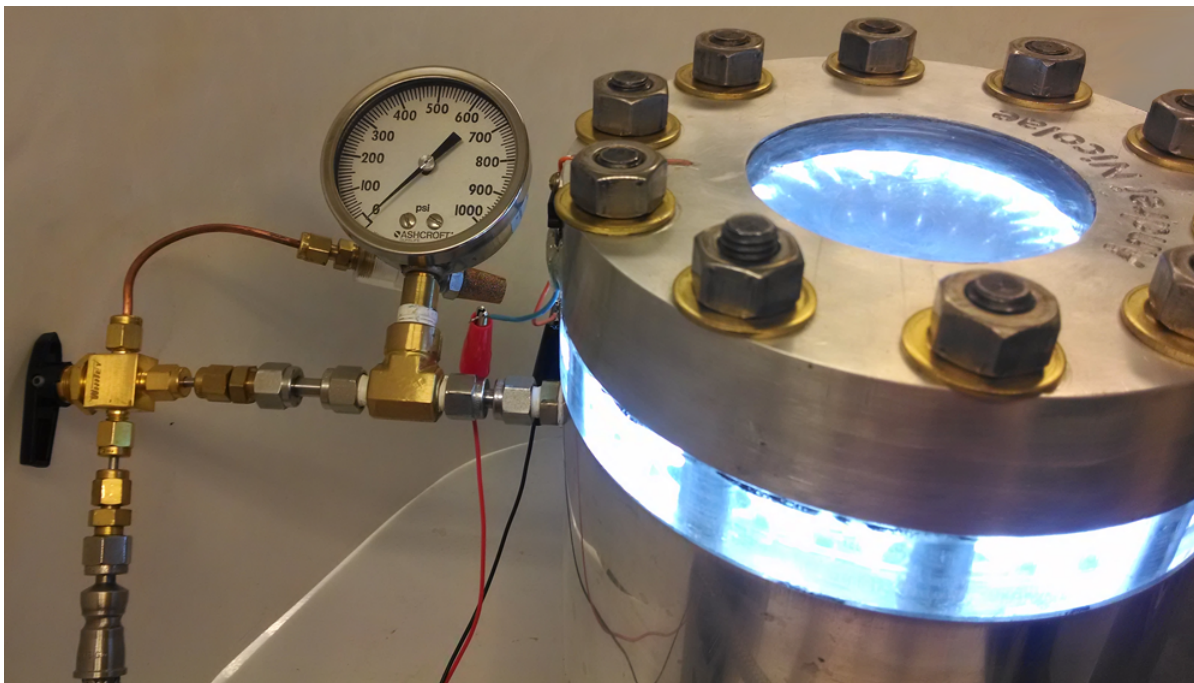


Figure 4.8: Inlet port to the vessel. The system is equipped with a mechanical pressure gauge, and a 3-way valve that allows venting through a porous brass plug (seen behind the gauge).

The fittings, valve, gauge, and plug were acquired from the stock in the Microcellular Plastics Lab so they were not purchased for this project specifically.

4.1.1 Machining Operations

The machining process was tedious as it required many operations. The long hours of labor, however, were valuable training and practice. It was also useful in ensuring that quality work was performed and the final product did not differ from the intended design. Since all the features on the vessel were 2.5D, a Trakk 2-axis CNC mill and a HAAS 2-axis CNC Lathe were used for the entirety of the machining process.

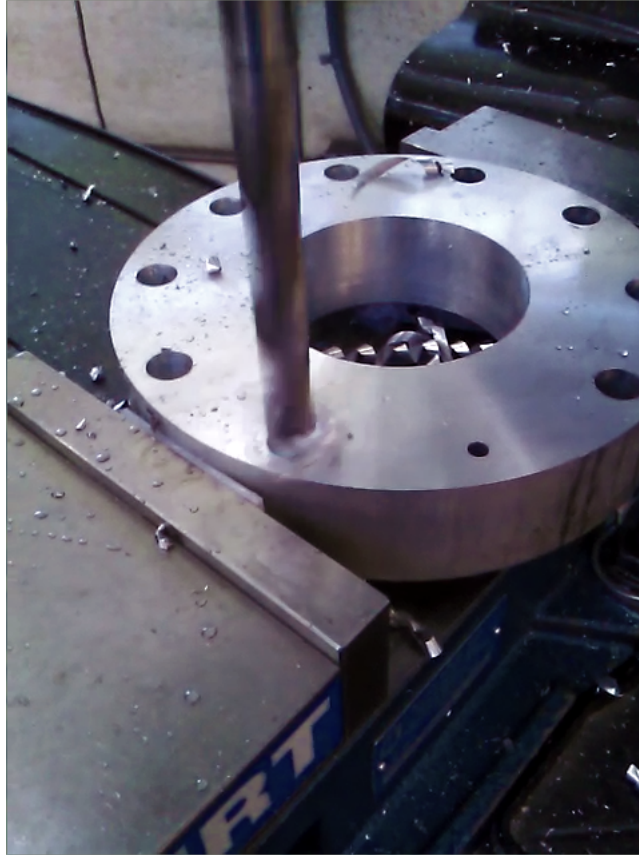


Figure 4.9: Machining of the vessel cap. A Trakk 2-axis mill was used; the Z-axis was controlled manually.

The vessel was initially center drilled on the lathe using progressively larger drill diameters, ranging from $3/8$ to 2 inch twist drills. Feeding the drill required manual moving of the heavy tail stock which demanded physical strength. After the 2 inch diameter hole was made, the boring bar could be fit inside and the machine was programmed to make small passes and bore out the inside. The boring operation performed on the lathe was the most time consuming since the feed rates and cutting depths were both very low. Cutting any faster would produce excessive chatter (vibration of the tools from intermittent contact) which would quickly damage the cutter tip and leave a rough finish.



Figure 4.10: Vessel boring setup on the HAAS TL-3 CNC lathe.



Figure 4.11: Inside view during the boring operation. The passes are very small (approximately 0.015") and the rotation speeds less than 80 RPM on average. The liquid seen is lubricant/coolant which also helps wash away the chips.

4.2 Stress Analysis

When working with large pressures like 6 MPa, safety is a concern because of the potential energy available in gas which can be released catastrophically in case of mechanical failure. For these reasons, engineering principles and tools were used to ensure that the stresses in the material at any point were safely below the material's yield strength. The yield strength of the 6061-T6 Aluminum alloy used is 276 MPa or 40,000 psi [10]. A minimum factor of safety for this design was taken to be 2.0, so any stresses above 20,000 psi or 138 MPa were considered excessive and therefore not tolerated.

4.2.1 Stress in a Thick-walled Vessel

The stress in a long, cylindrical pressure vessel wall can be found analytically [11].

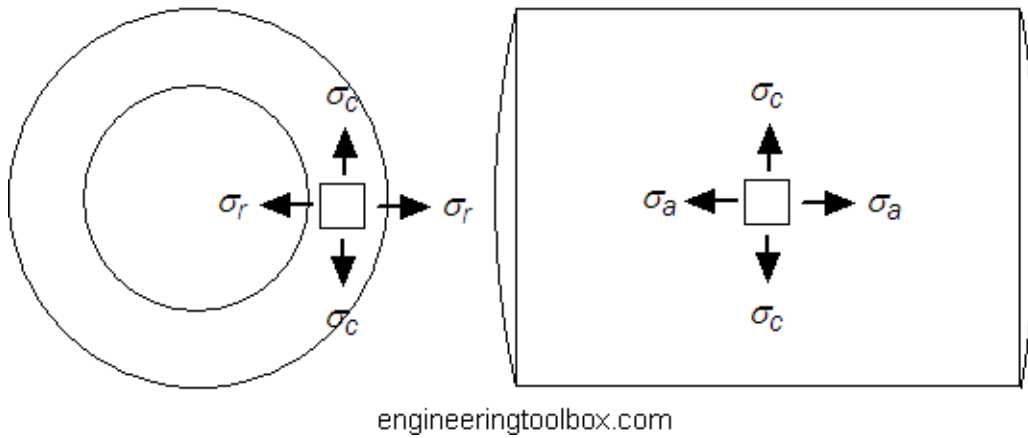


Figure 4.12: Diagram of the stress directions for the thick-walled pressure vessel equations. From www.engineeringtoolbox.com, used with permission.

For the axial direction:

$$\sigma_a = \frac{p_i r_i^2 - p_o r_o^2}{r_o^2 - r_i^2} \quad (4.1)$$

For the circumferential direction, the stress at a location r away from the center along the radius is:

$$\sigma_c(r) = \frac{p_i r_i^2 - p_o r_o^2}{r_o^2 - r_i^2} - \frac{r_i^2 r_o^2 (p_o - p_i)}{r^2 (r_o^2 - r_i^2)} \quad (4.2)$$

And finally, the stress in the radial direction is given by:

$$\sigma_r(r) = \frac{p_i r_i^2 - p_o r_o^2}{r_o^2 - r_i^2} + \frac{r_i^2 r_o^2 (p_o - p_i)}{r^2 (r_o^2 - r_i^2)} \quad (4.3)$$

By defining the stress directions in the manner above, there is no shear. Therefore, the three stresses above are the principal stresses (eigenvalues of σ_{ij}) and together with the defined directions as a basis for R^3 the stress tensor can be conveniently constructed.

Using these equations for the vessel built, the maximum axial, circumferential, and radial stresses are 1200 psi, 3900 psi, and -1500 psi respectively. This is well below the limit of 20,000 psi so the safety factor is large.

4.2.2 Cap Bolts

The largest stress occurs in the bolts that hold the cap connected to the pressure vessel. For this reason, Grade 8 High-Strength Alloy Steel threaded rods were used. This material has a tensile strength of 150,000 psi according to the supplier. The minor diameter of the 1/2-13 rod is 0.406 inch so each rod has a $150,000 \times 0.406^2 \times \pi/4 = 19,419$ lbs load carrying capacity. There are 10 such rods used for the vessel cap, so the total theoretical holding strength is 194,190 lbs. The pressure force on the cap is $PA = 800\text{psi} \times 5^2 \times \pi/4 = 15,708$ lbs therefore the safety factor for this design is 12.36.

4.2.3 Bolt Pattern for the Plates

Since the pressure distribution in the porous seal is non-uniform, the bolted connections of the plates cannot be evenly spaced if they are to be equally stressed. Given a linear load distribution of the form

$$P = a(1 - |x|/b), \quad |x| \in [0, b] \quad (4.4)$$

the problem was set up to find boundaries that split the area under P into equal elements. Then, a bolt is placed under the centroid of each area element.

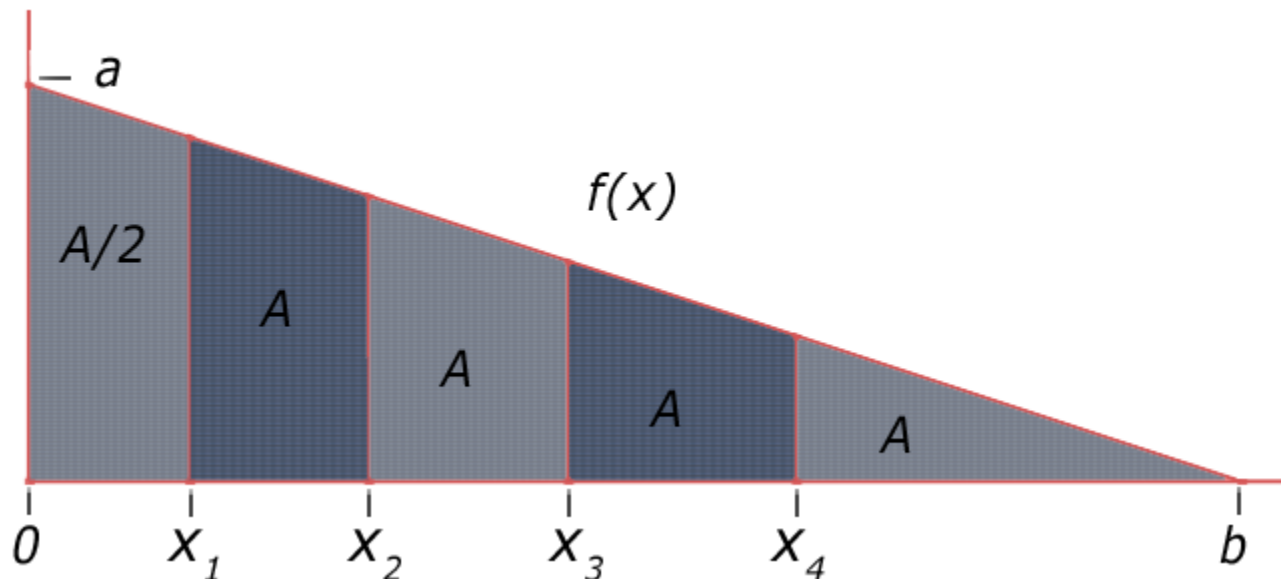


Figure 4.13: Problem definition and set-up, cut in half at $x = 0$ by symmetry. Each element has area A and boundary at x_i except for the first element.

Mathematical formulation

Let n denote the number of bolts to be used (including that at $x = 0$). This creates n unknowns (x_i and A) so a system of equations can be written for the area elements. For the interior nodes

$$\frac{f(x_i) + f(x_{i-1})}{2}(x_i - x_{i-1}) - A = 0, \quad 2 \leq i \leq n - 1 \quad (4.5)$$

and for the first and last area elements, respectively:

$$\frac{f(x_1) + a}{2}u_1 - A/2 = 0 \quad (4.6)$$

$$\frac{f(x_{n-1})}{2}(b - x_{n-1}) - A = 0 \quad (4.7)$$

Substituting $f(x) = a(1 - x/b)$, the above equations take the form

$$\frac{a}{2b}(x_{i-1} - x_i)(-2b + x_{i-1} + x_i) - A/2 = 0 \quad (4.8)$$

$$ax_1(1 - \frac{x_1}{2b}) - A = 0 \quad (4.9)$$

$$\frac{a}{2b}(b - x_{n-1})^2 - A = 0 \quad (4.10)$$

Once these equations are solved, the centroid of each area element can be computed from

$$\bar{x}_i = \frac{2}{f(x_i) + f(x_{i+1})} \int_{x_i}^{x_{i+1}} xf(x)dx = \frac{3b(x_i + x_{i+1}) - 2(x_i^2 + x_ix_{i+1} - x_{i+1}^2)}{6b - 3(x_i + x_{i+1})} \quad (4.11)$$

This system of nonlinear algebraic equations were solved numerically using a script ran by MATLAB. The code is copied in the appendix.

FEA verification

A solid-mechanics FEA model was created to verify the effectiveness of the proposed bolt pattern. A plate loaded by a linear pressure distribution was supported by identical pillars and the von Mises stress was surface plotted as seen in the figures.

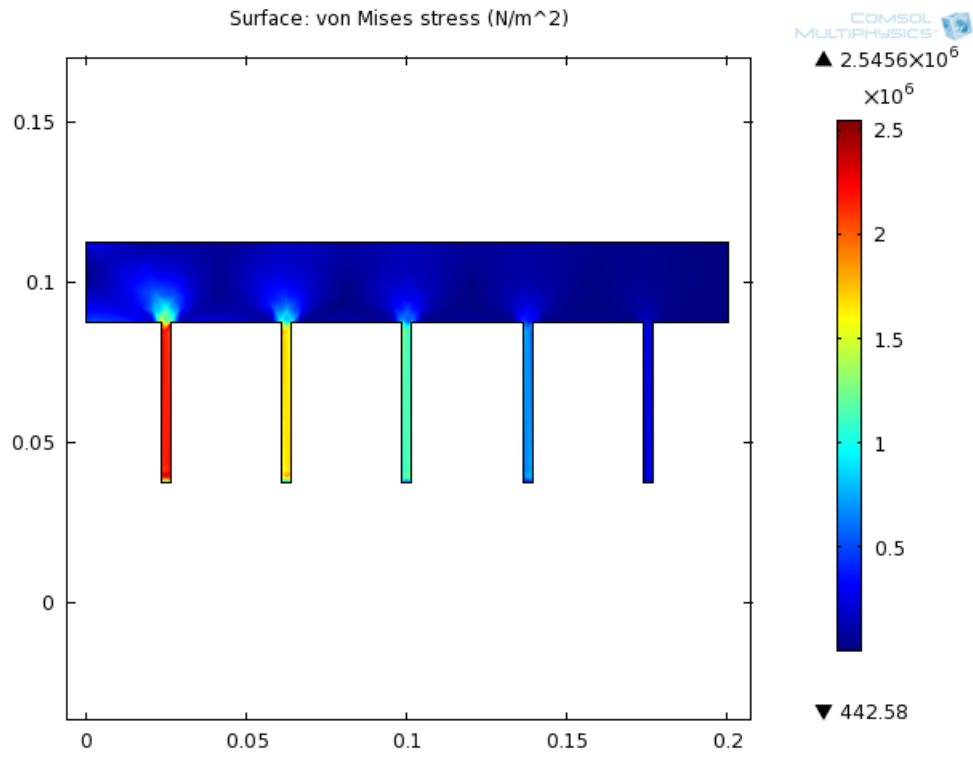


Figure 4.14: Equal spacing between supports leads to highly uneven load sharing.

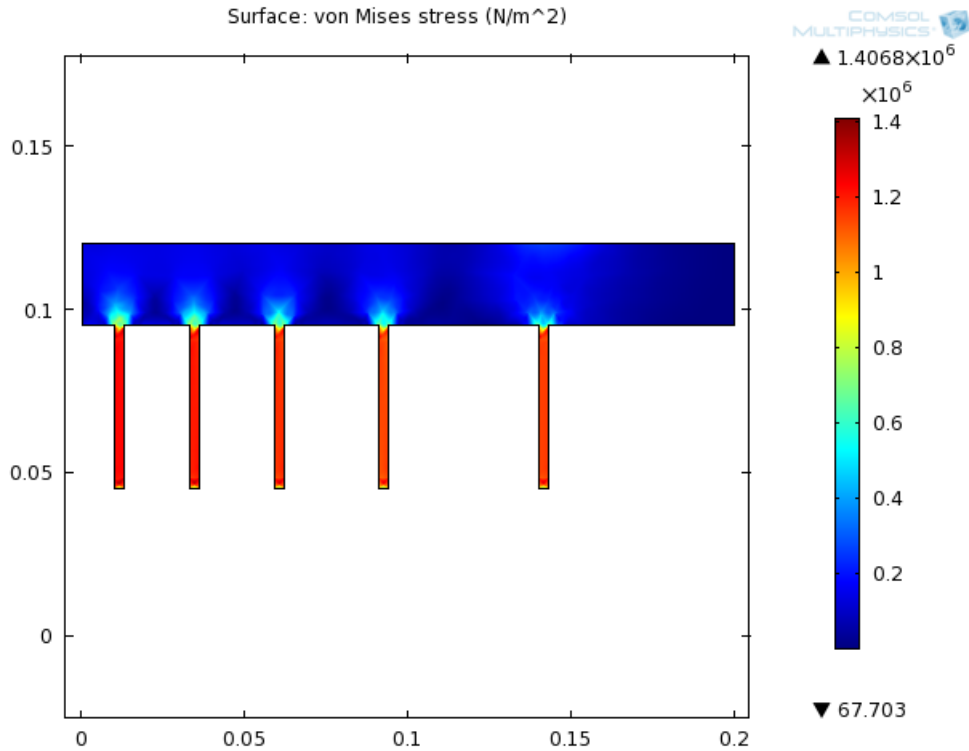


Figure 4.15: Support pattern as given by the MATLAB code solution. The stress is even in all members.

This validates method which was used to generate the dimensions for the bolt holes.

4.2.4 FEA

The aforementioned method was tested on the pressure vessel plate design with the exact dimensions that were to be machined. A linear pressure distribution (in psi) was used as the boundary condition for the plate in the form

$$p(x, y) = 750(1 - x) , x \in [0, 12] \quad (4.12)$$

as it would be in the real experiment. The equidistant bolt pattern was tested against the one generated by the MATLAB code for comparison. The model was cut by the symmetry plane to reduce computation time. The boundary condition on the symmetry face was “roller” in order to simulate the correct mechanics. The results are shown in the figures below.

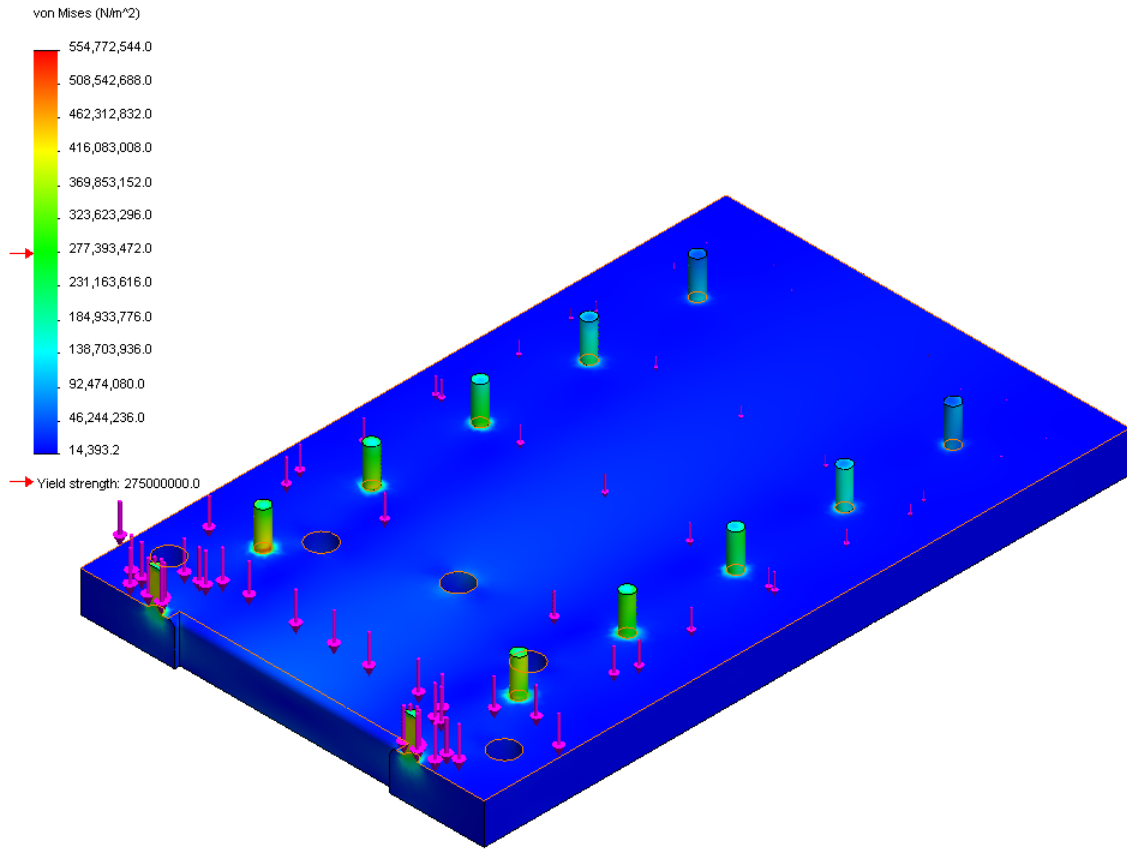


Figure 4.16: Equidistant bolt pattern. The bolts closest to the center (the slit in figure 4.1) take the most load.

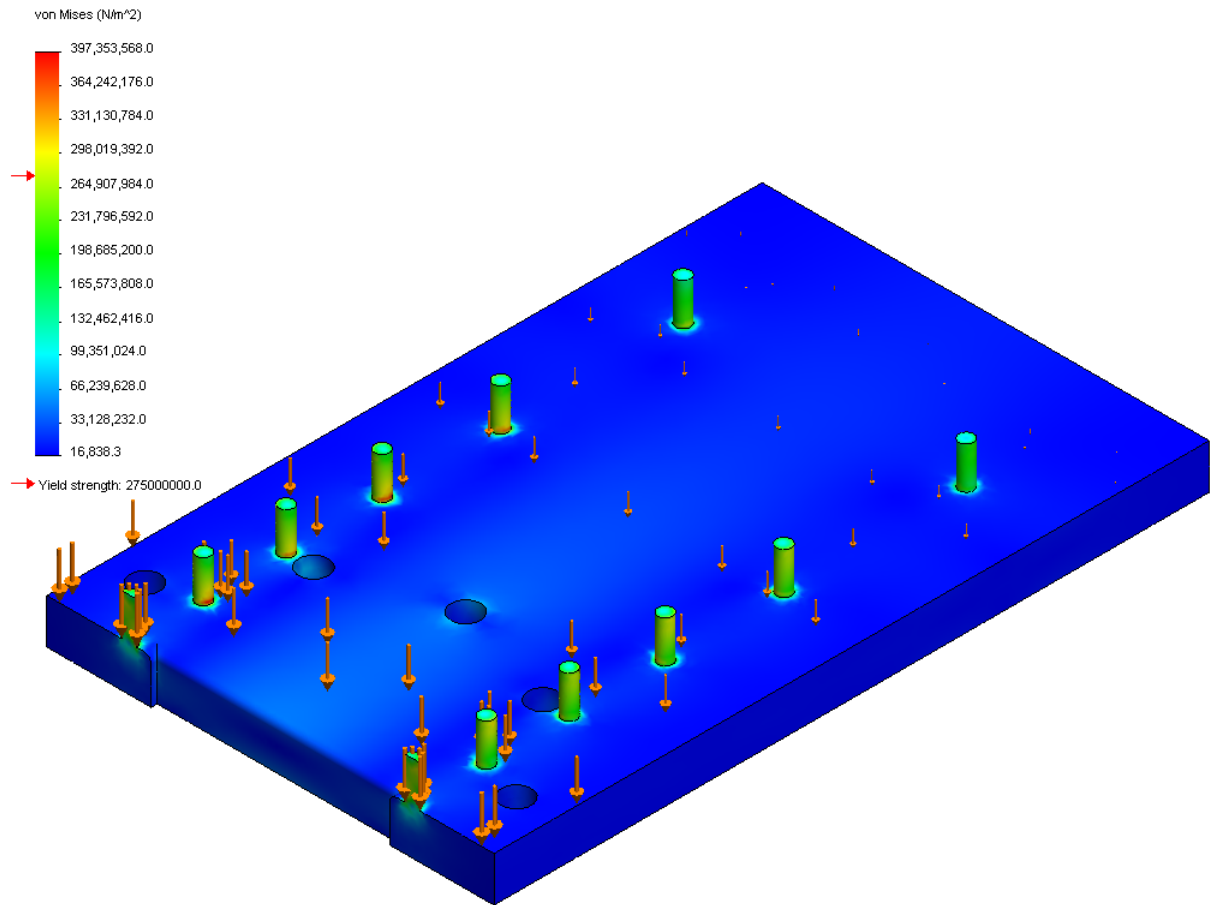


Figure 4.17: Bolt pattern generated by theory. The stress is much better shared between the bolts.

The improvement is obvious from the results. The stress concentration on the stud base is due to the modeling of the solid as one body made of the same material (Aluminum 6061 T6). In the real vessel, this problem will be eliminated since the studs are steel into aluminum threads: the much higher elastic modulus of steel will distribute the load along the plate depth and not concentrate at the surface. This analysis not only improved the safety of the prototype vessel but it is also a useful design tool for future implementations of the dynamic seal.

Chapter 5

Design of the Dynamic Seal

5.1 Porous Seals

The first seals made for the large prototype vessel consisted of thin ABS sheets on which were bonded the seal materials and side supports. These sheets were adhered to the aluminum plates of the vessel using 2-part epoxy (so that the fluid would not leak beneath them). The polymer film strip was sandwiched between these seals and entire assembly clamped together with the threaded studs. Figure 5.1 shows an example setup.



Figure 5.1: View of the bottom half of an example seal setup. The porous material (white) is the working seal, while the black rubber (neoprene) serves to block flow transversely.

A variety of designs were attempted in order to assess the effectiveness of each material and layout. These were built with the materials used in the experiments outlined in chapter 3. A good seal is one that allows for easy removal of the polymer film (low friction) and low leakage rate.

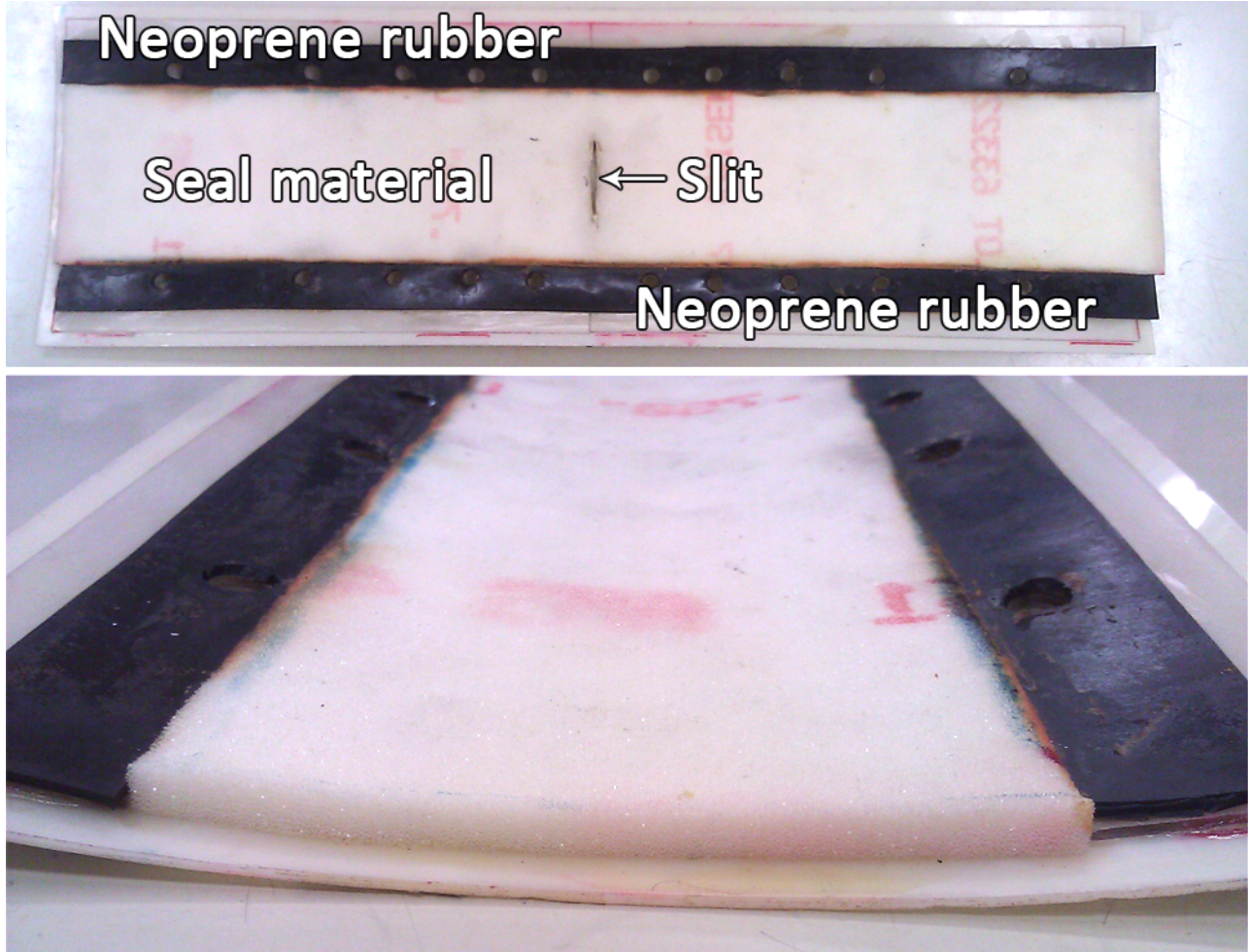


Figure 5.2: First seal design. The material used is 1/4 inch 1.8 lb/ft³ polyurethane open-celled foam. Along the sides, 0.75 inch strips of 1/16 inch thick neoprene rubber are attached for providing structural support and for sealing the flow transversely.

The design in figure 5.2 was the first attempted; the idea was to provide a low clamping force to the polymer film (for low friction) and stiff rubber sides that prevent the fluid from escaping in the transverse direction. The seal failed to adequately seal the pressure and resulted in excessive water leakage even at a pressure of 0.60 MPa. With this information in mind, a second design was conceived: the idea was to use a thinner material since

$$Q = \iint \mathbf{q} \cdot d\mathbf{A} \quad (5.1)$$

so a smaller cross section would result in lower flow rate. In addition, the thinner material available also had smaller pores which results in lower permeability k in the formula

$$\mathbf{q} = -\frac{k}{\mu}\nabla p \quad (5.2)$$



Figure 5.3: Seal material setup. The material used is 1/32 inch 20 lb/ft³ polyurethane open-celled foam. Along the sides, 1.5 inch strips of 1/32 inch thick neoprene rubber are attached for sealing the flow transversely.

When installed on the vessel plates, this seal performed poorly as well. At high clamping forces, the fluid was completely sealed even at 5.0 MPa. However, the friction was much too great to move the polymer film. As the clamping force was lessened, leakage occurred on the edges along the neoprene rubber sides; the leak rate became unacceptable before the friction was low enough to release the polymer.

It was then realized that a similar problem was occurring in the first seal shown in figure 5.2 the junction between the rubber sides and the porous film material crates a channel wherein the fluid can leak uncontrollably. This problem was examined next.

5.2 Edge Clamping Problems

From the experiments mentioned above, it became apparent that a new method of sealing the seal edges must be implemented. The problem lies in the mechanics of the solid materials. The soft seal material is pressed against a harder material (such as the neoprene rubber) and forced to conform to its shape. However, since the material has an elastic modulus greater than zero, it cannot be shaped into geometries of infinite curvature.

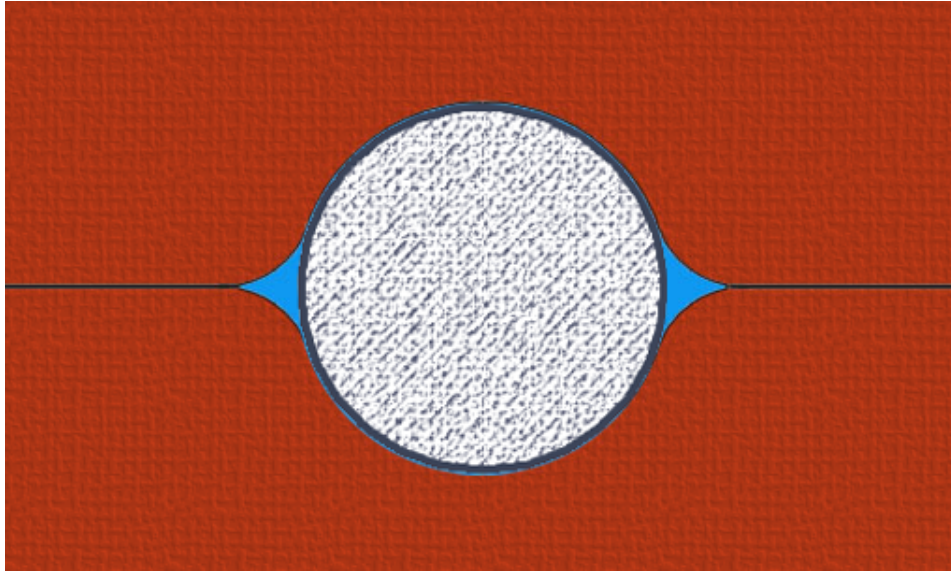


Figure 5.4: When a linear elastic solid (red) is pressed against a rigid body (circle) it will not conform exactly to its shape if the curvature is too large. Instead, gaps will be made through which fluid can leak (blue).

This can be seen from the relation between bending moment M , elastic modulus E , and curvature κ for an elastic beam [12]

$$|\kappa| = \frac{1}{\rho} = \frac{M}{EI} \quad (5.3)$$

A well known result from calculus relates the curvature to the first two derivatives of a plane curve $y = f(x)$

$$|\kappa| = \left| \frac{d\mathbf{T}}{ds} \right| = \frac{d^2y/dx^2}{[1 + (dy/dx)^2]^{3/2}} \quad (5.4)$$

To avoid this problem, a low-curvature geometry must be used as shown in figure 5.5. Circle arcs are used connected by tangent end conditions; this makes it easy to control the curvature whose magnitude in this case is $1/r$, the radius of the circle arc.

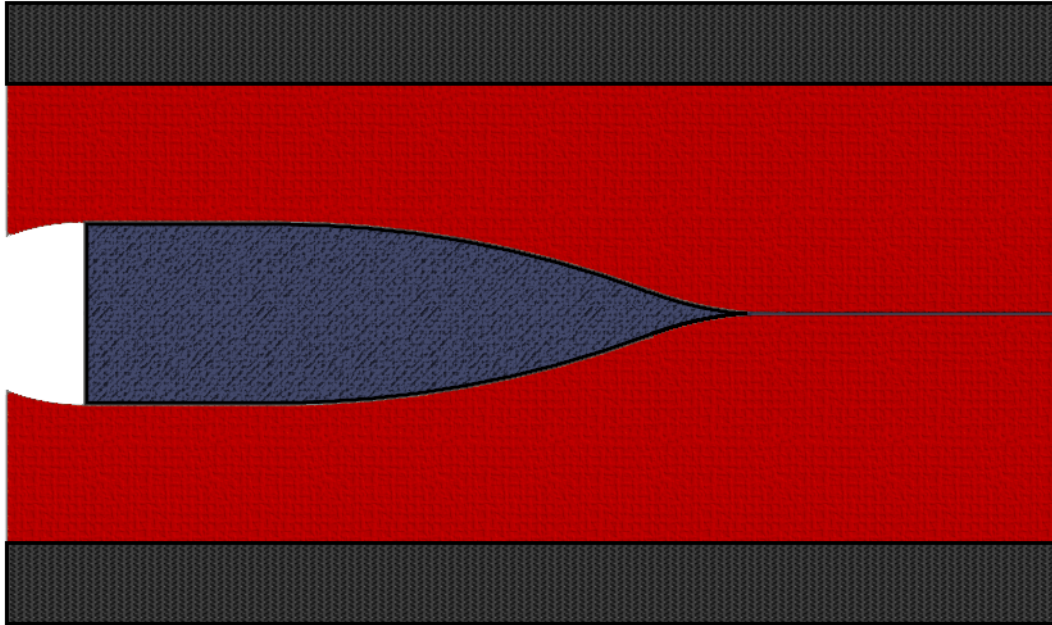


Figure 5.5: A low curvature wedge designed to seal the edges of the seal material.

A “rat-tail” structure was built as shown in figure 5.6 to serve as the edge barriers to flow. Although the sealing material is open cell porous, if the cell structure is sufficiently compressed it will close off and become non-permeable. This is specifically stated on the McMaster-Carr material information page and was verified in the lab as well.



Figure 5.6: Photo of the polycarbonate V-shaped piece used as a barrier for transverse flow. Since the edge does not have infinite curvature as does a rectangle corner, it was hypothesized that the seal material would conform to the shape and not produce any gaps.

This method was not highly successful, as the fluid still found a way to escape along the sides. The mechanism through which this happens is unknown since there is no visibility inside the seals; however, a hypothesis was that the high pressure present is able to create a channel and leak along the boundary of the side supports and the seal material.

An alternative method is to physically adhere the seals using glue or a deformable sticky putty (such as the sealing tape used for composite vacuum bags). This method was also attempted and is shown in figure 5.7.

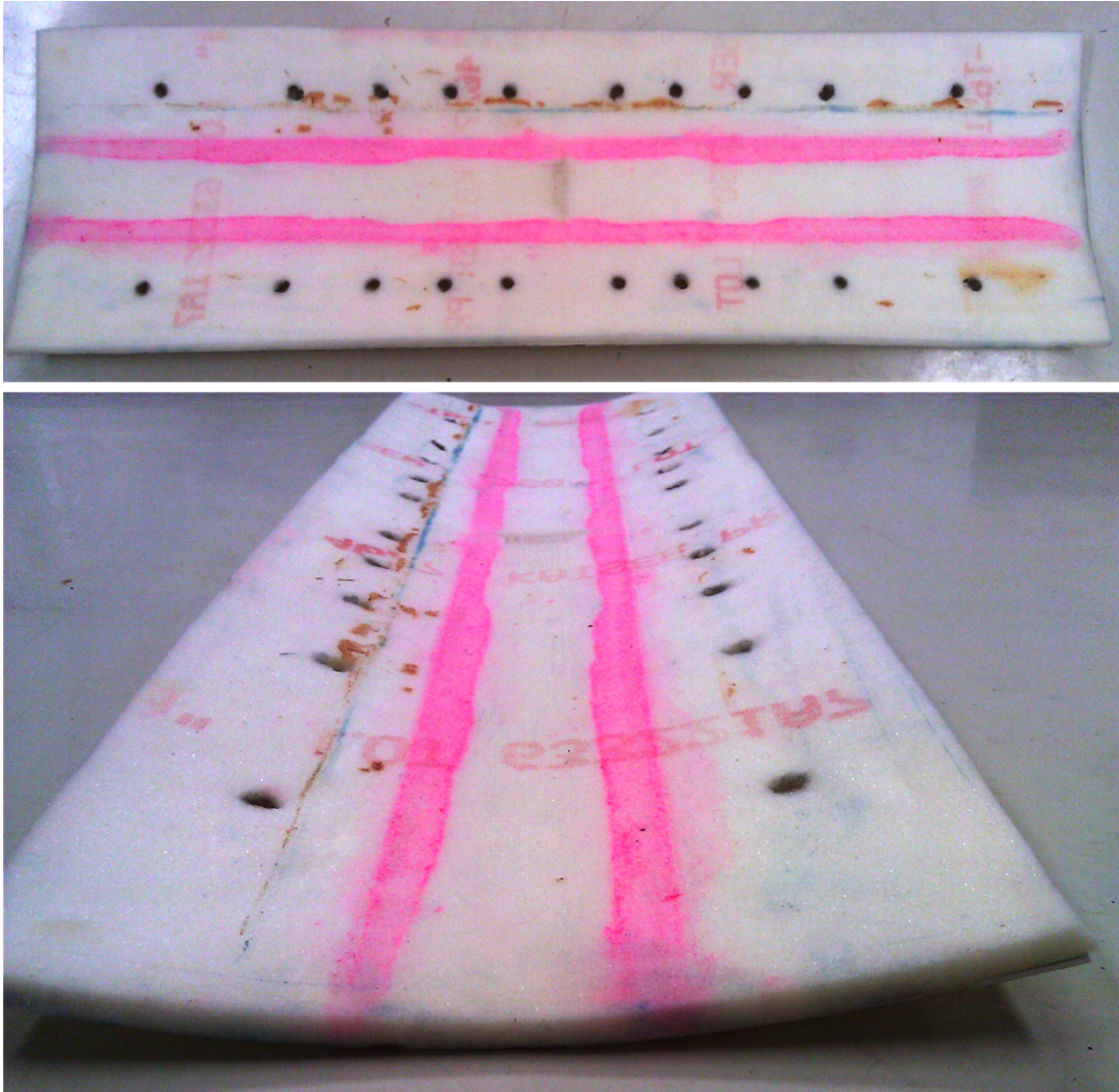


Figure 5.7: 1/4 inch 1.8 lb/ft³ polyurethane open-celled foam seal without side supports. Instead, adhesives were used to provide the barrier for transverse flow (seen as the pink leftover streaks).

The disadvantage of adhesives is that the seals cannot be simply taken apart to reset the polymer in between them. This makes it especially undesirable when performing tests. As a better alternative, the material can be clamped from the outside by a non-planar surface. This design idea is shown in figure 5.8 and was highly successful.

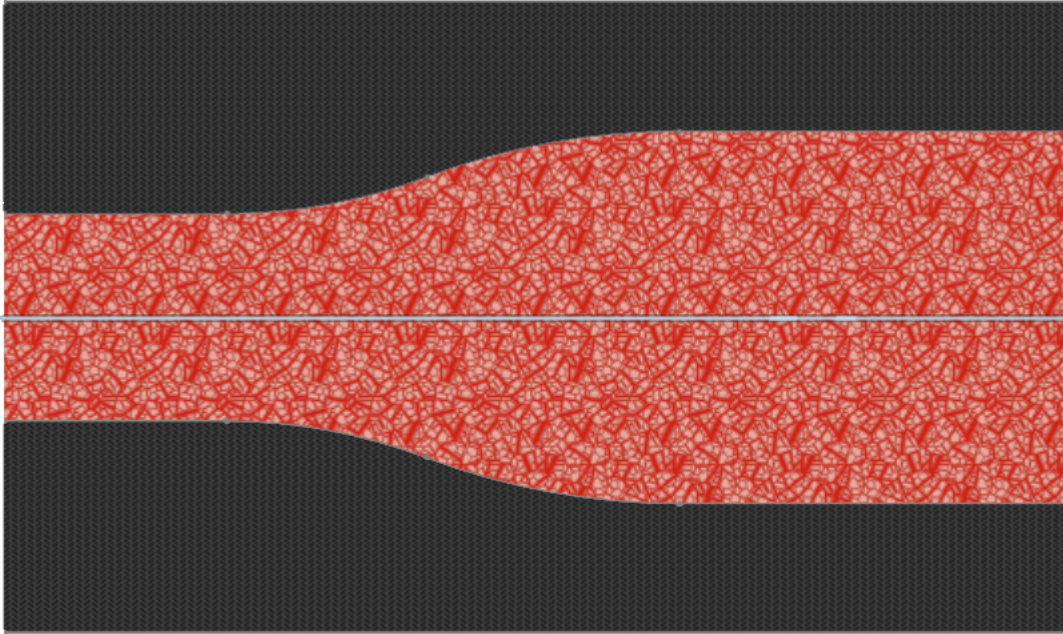


Figure 5.8: Instead of flat clamping plates, a smooth curve is machined such that the separation distance tapers down and squeezes the material sufficiently to block flow through that region.

5.3 Flow Visualization Through Porous Seals

The new design methods were implemented on a test prototype to better analyze the flow and understand the mechanics of the dynamic seal using porous materials. The purpose was to find the reason behind the failures of the previous two dynamic seal designs described in section 5.1. Clear polycarbonate plates were machined and used as clamping plates, with pressure delivered through a center hole. The material used was the 1.8 lb/ft^3 polyurethane as before. The following figures illustrate the prototype.



Figure 5.9: Curvature machined in the polycarbonate plate to clamp the seal material edges.

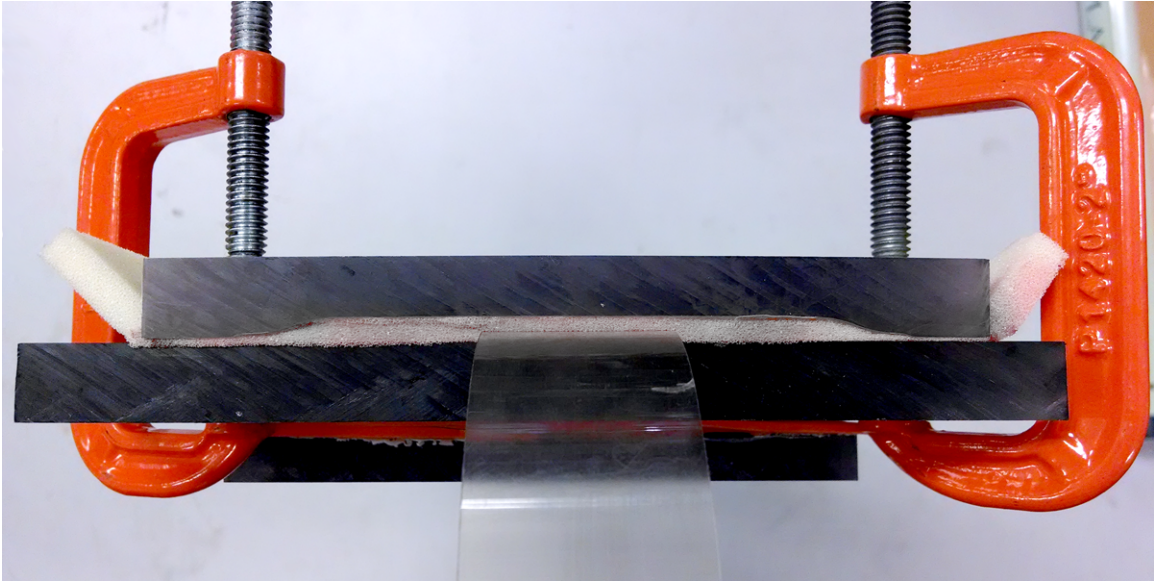


Figure 5.10: End view of the assembly. Note that the seal material (white) is highly compressed at the edges; this blocks transverse flow, i.e. seals the edges effectively.

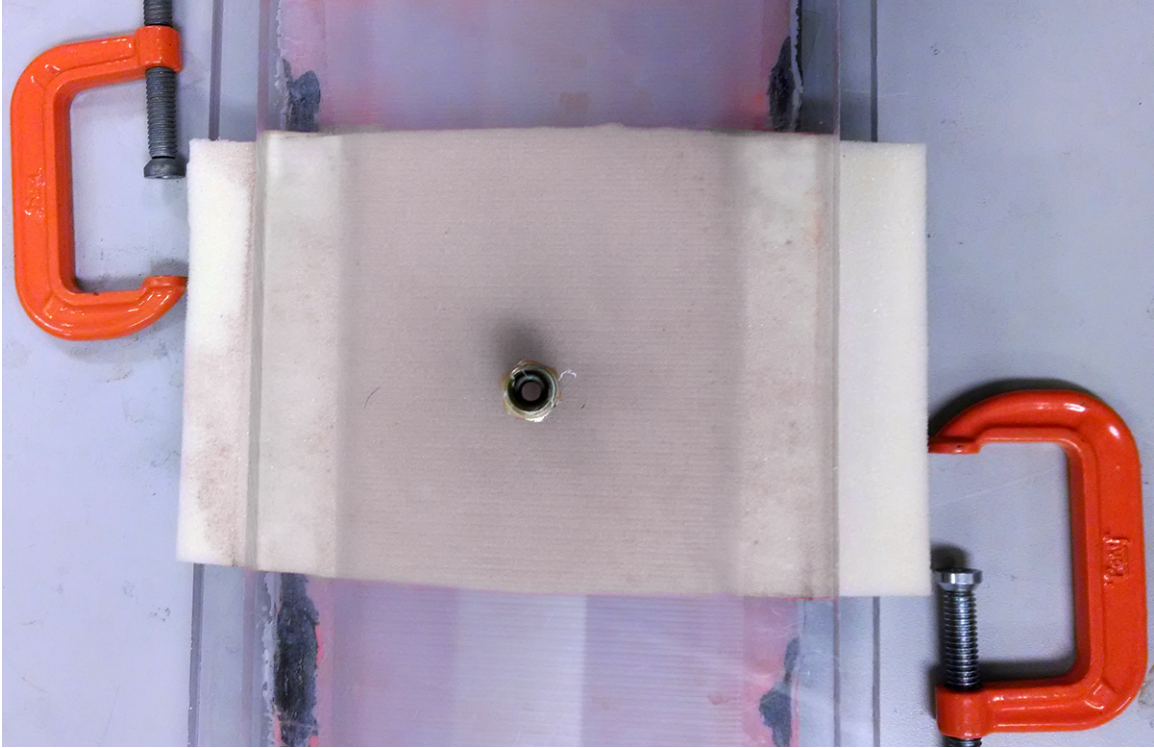


Figure 5.11: Top view of the assembly. The pressurized fluid comes in through the fitting in the center and then flows along the length of the seal. The polymer film sandwiched in the seal is 0.15 mm thick PEI.

Water with red dye (food color) was fed through this prototype using compressed shop air (which comes from the UW power plant at a maximum pressure of approximately 100 psi). The flow was thus visualized; images from a video are shown in the following figures.

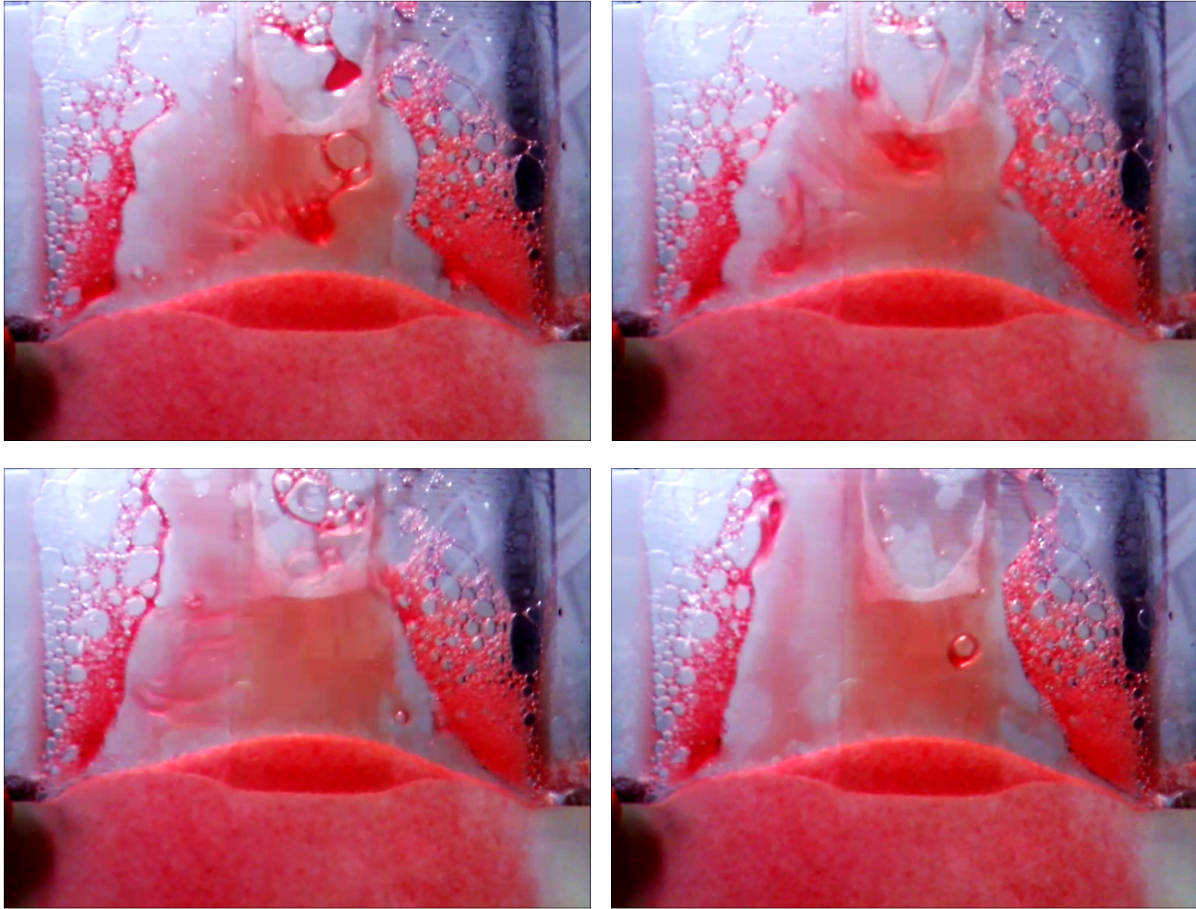


Figure 5.12: Flow visualization using red dyed water. The valve was only slightly open so that it restricted the flow rate.

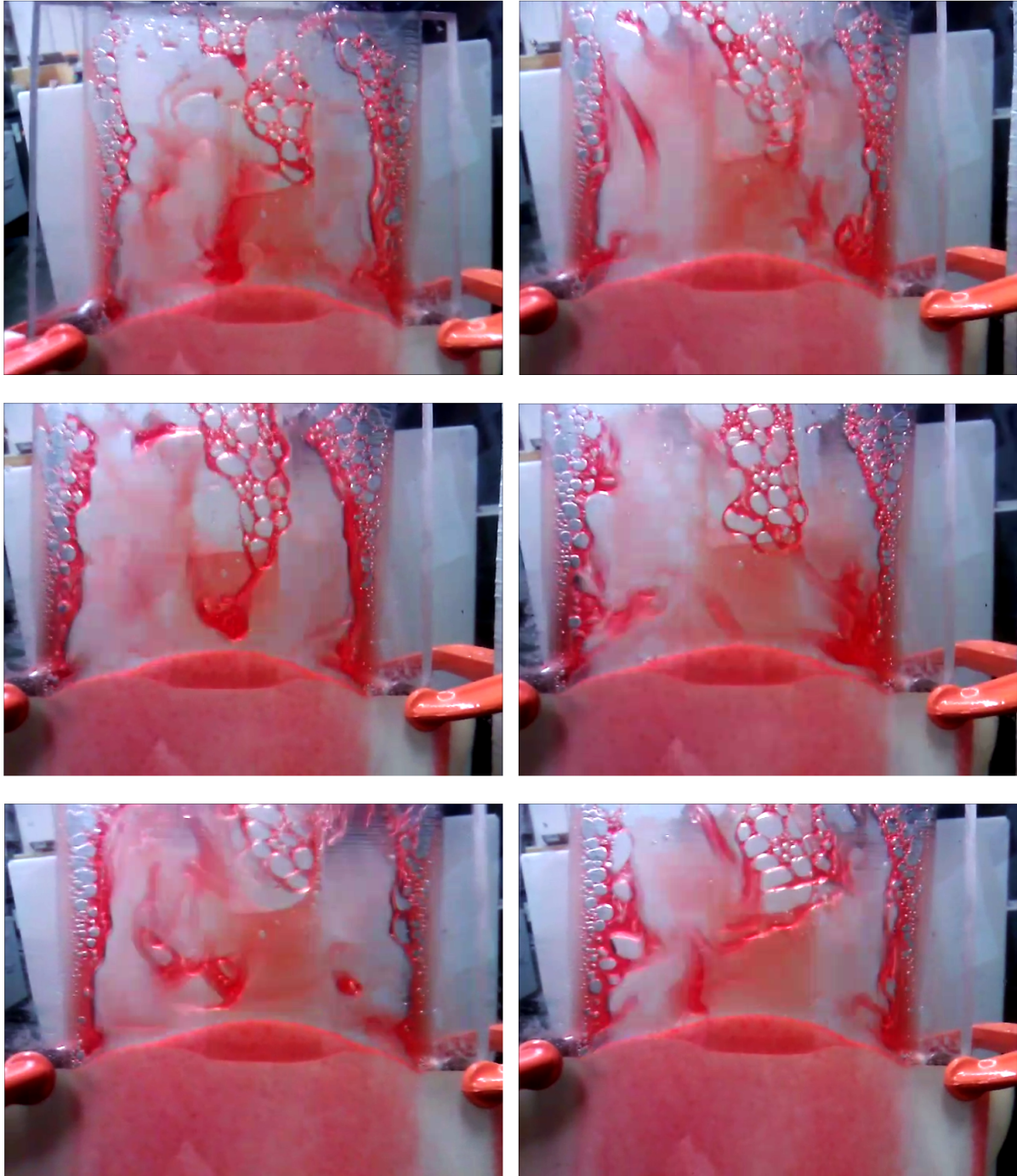


Figure 5.13: Identical setup except that the valve was opened further.

In the experiment depicted by figure 5.13, the pressure was further increased at the inlet and the flow exhibited turbulent behavior. It is apparent that the boundary layer is reduced and the fluid escapes at higher rates. It is evident that the fluid is flowing through the porous material and not between the porous material and the polymer film, as the friction force on

the film was not changed; it could not be pulled through the seal without deforming the seal material (white foam).

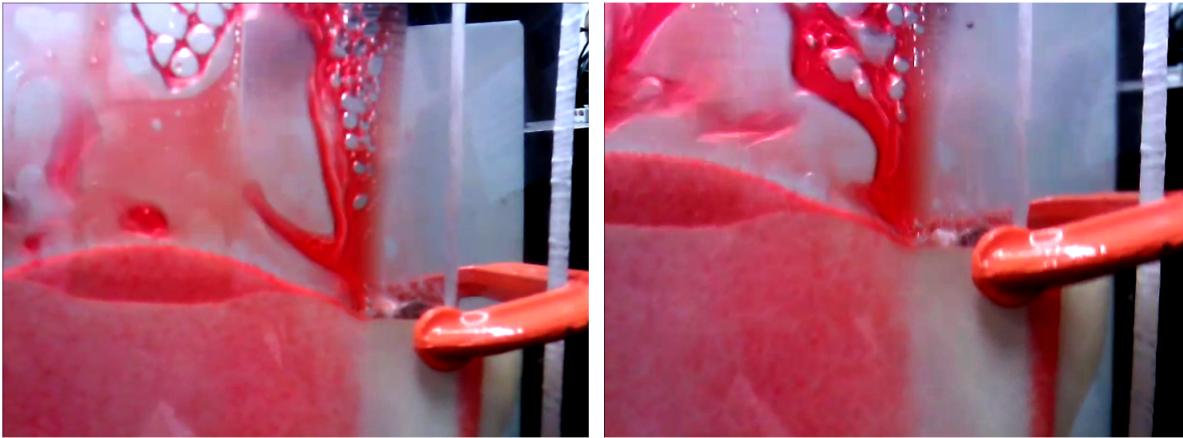


Figure 5.14: Close up of the flow around the seal edges. Note that the far edges are still white due to the clamping technique which kept the red fluid out of that region.

From this experiment, it became evident that the porous material behaves as expected in allowing the fluid to flow through in the longitudinal direction only. There are, however, edge effects as shown in figure 5.14 where the flow is slowed by

1. The seal material compression on the edges
2. The wall shear on the fluid

Although it is not shown in the figures, when the liquid was initially let into the seal it almost instantaneously penetrated all available space and the white material very quickly turned red from the dye, indicating liquid presence. An observation to note from the figures is that the seal material front edge is bulged into a concave-down shape due to the fluid forces from the pressure gradient as discussed earlier. It is similar to the shape taken by an elastic beam supported at the ends with a uniform force distribution along the length, so the behavior is totally expected. This problem is easily resolved by using adhesives to attach the seal material to the clamping plates. In this experiment, it was not done in order to preserve transparency.

It is also important to note the turbulence present in all tests. This is due to a high exit velocity, indicating high leakage. The friction force on the polymer was excessive, making it very difficult to slide through by hand. This led to an important conclusion: porous materials alone were not ideal for use in the dynamic seal. This is because there is no layer of fluid in between the polymer and the seal material to provide the necessary lubrication as discussed in chapter 3, and the seal material makes direct contact with the polymer film which leads to high friction.

5.4 Back to Closed Cell Seals

Even with the side gap problem fixed, the seals were still unsuccessful because the friction was far too great for the polymer film to move smoothly. The next step was to return to the closed cell seal materials which were proven to work in the first small prototype detailed in chapter 2. The setup from figure 5.10 was fitted with a non-porous seal (closed cell polyurethane) in order to control only one variable (the seal material) and leave the geometry and all other variables unchanged.

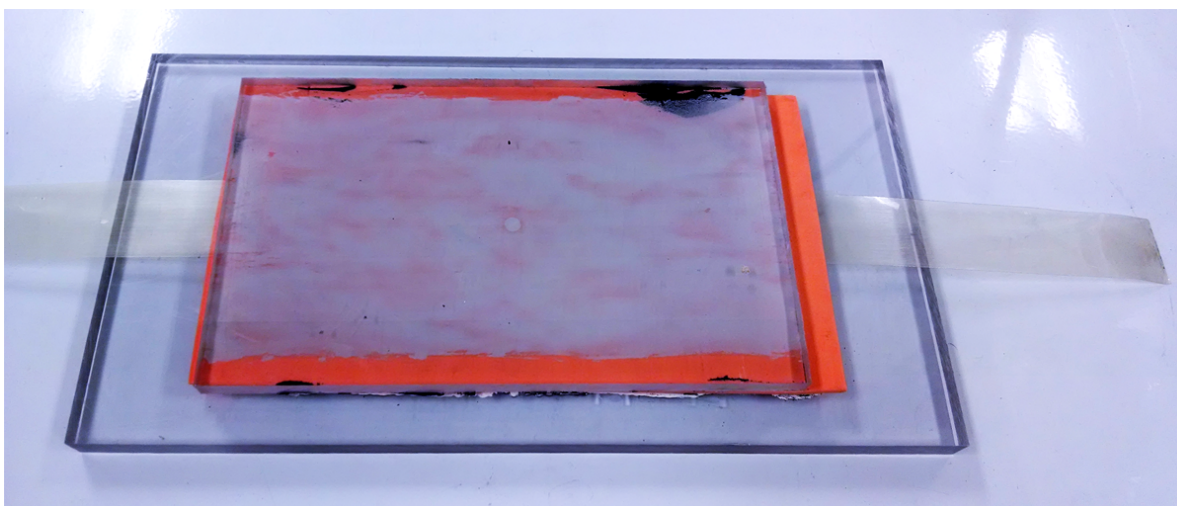


Figure 5.15: Porous open-cell seal material replaced with non-permeable closed-cell polyurethane. The friction was reduced significantly.

In order for the fluid to escape, it must create a gap between the two seal materials as it cannot escape through the material itself. This also creates a space for the polymer to pass through, resulting in very low friction. It is also simple to seal the edges when using a closed cell material because there is no worry about fluid permeating through the material thickness.

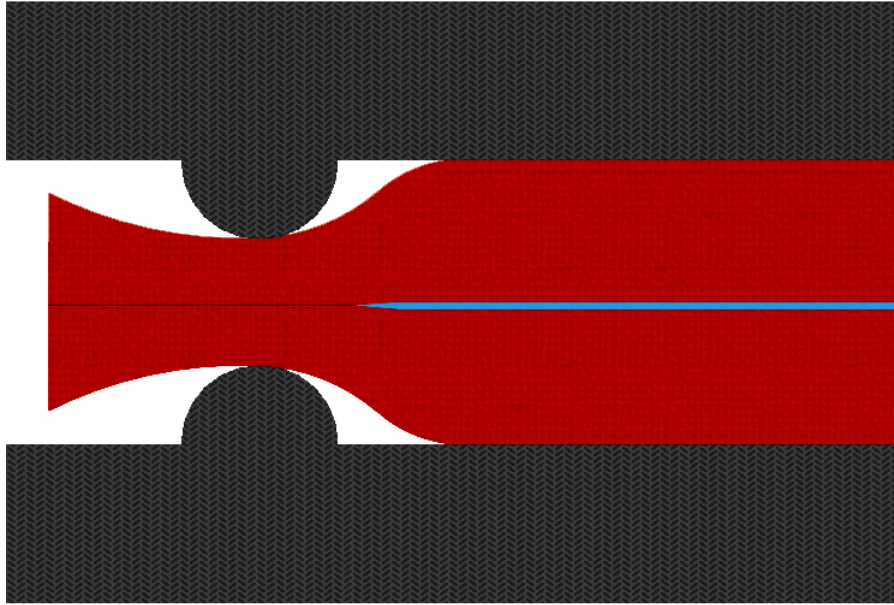


Figure 5.16: A gap occurring in a closed-cell seal material due to high curvature at the pinch zone is not a problem since the fluid cannot flow normal to the surface and is therefore sealed.

All that is necessary is a boss geometry to pinch off the material at the edges, as shown in figure 5.17.

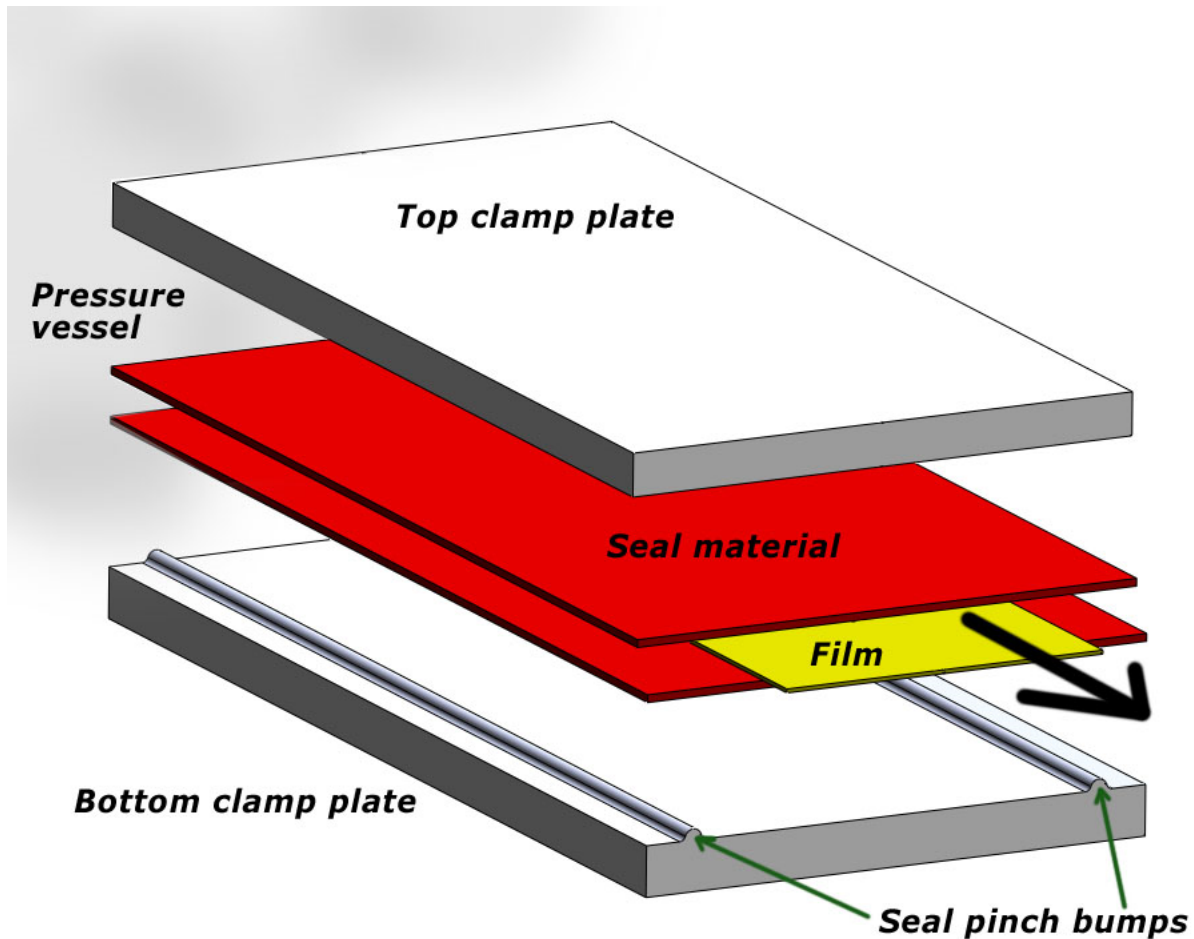


Figure 5.17: Exploded view of a closed-cell seal material implementation. The bumps on the clamping plates are sufficient in shutting off the transverse flow.

This has been successfully implemented in various prototypes and makes disassembly very quick.

5.4.1 Microcellular Sheet as Seal Material

Microcellular sheets, such as the PET panels produced at MicroGREEN Polymers, are closed cell foams that can be used as the sealing material. It was observed that the surface can be plastically deformed into a desired shape by press forming.



Figure 5.18: A US quarter was pressed into a PET microcellular sheet from MicroGREEN™ using a vise. The detail is permanently imprinted on the sheet.

It was therefore postulated that pressing the polymer film between two microcellular sheets using the seal clamping plates will create a channel with the dimensions of the polymer film and therefore surround the polymer film tightly in order to reduce leaks (by reducing interface gaps). Sheets were cut and adhered to aluminum plates to test out the hypothesis.

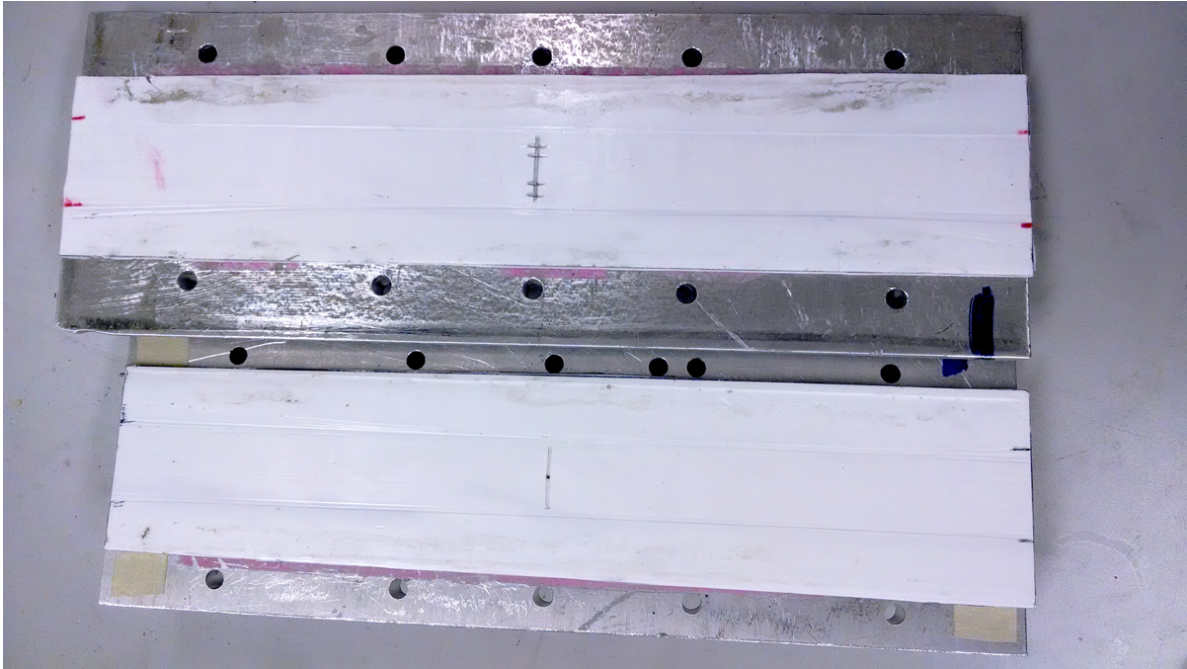


Figure 5.19: Photo of 2 mm microcellular PET sheets from MicroGREENTM used as seal material. A 1.0 inch wide by 0.0125 inch thick ABS film strip was fed through the center. The clamping pressure forced the microcellular material to conform to the shape of the film strip.

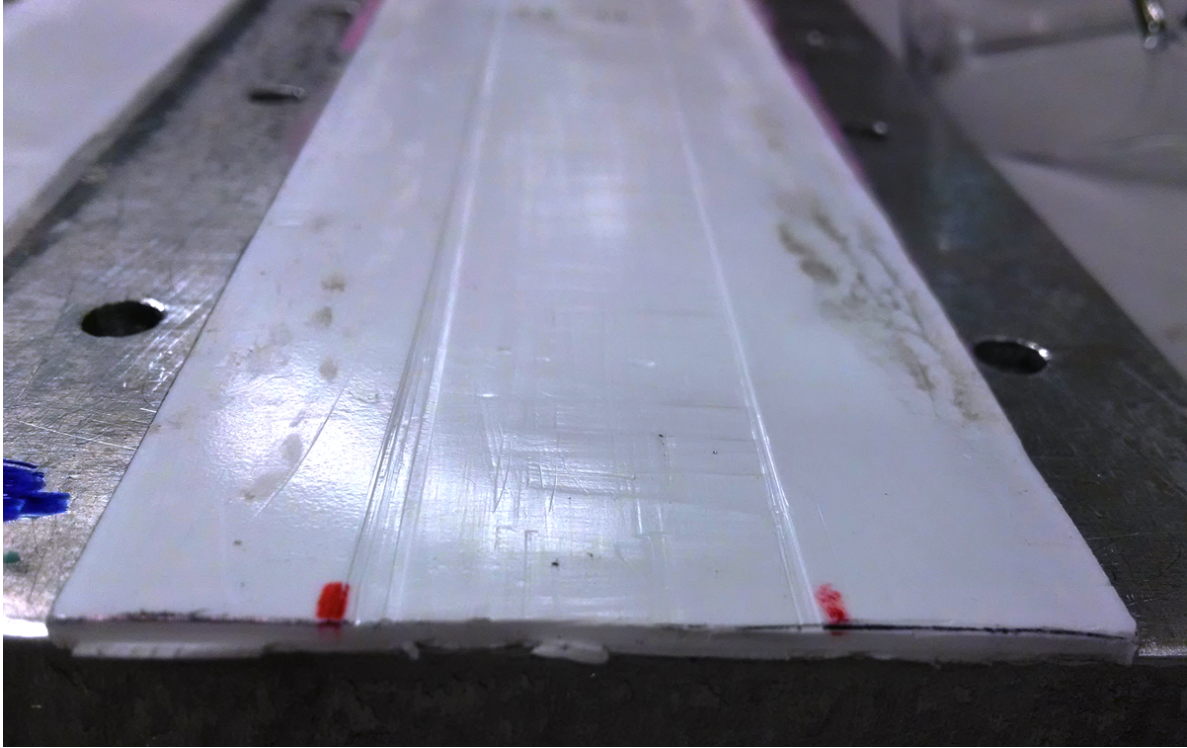


Figure 5.20: Close up of the microcellular seal material. The imprint on the surface is more visible.

From initial experiments, the *microcellular sheet proved successful as a sealing material*. The solid skin on the surface served as a low roughness surface for the polymer film to glide over. Due to time constraints, the material was not further analyzed so this is a future work task.

Regardless of the material used, the problem remains how to minimize the seal-polymer gap and reduce the flow at higher pressures while keeping the friction low.

Chapter 6

Pressure Measurements

After experimenting with the types of seals, a more in-depth analysis of the mechanics was called for. This led to a better understanding of the fluid behavior and fluid-solid interaction and led to new implementation ideas.

6.1 Pressure Inside the Seal

In order to improve the design, it was necessary to understand the fluid behavior between the closed cell seal materials. Specifically, the interest was in determining the pressure distribution along the longitudinal direction. If the flow is laminar and the boundaries are parallel planes (Plane Poiseuille flow) as discussed in chapter 3, the expected pressure is linear with distance. However, the boundary surfaces are soft materials (therefore not necessarily planar) and clamping not necessarily uniform, so the assumptions may not be valid. This required experimental verification.

6.1.1 Hardware

To do this, a test apparatus was designed and built in the image of the prototype vessel's seal plates as shown in figure 6.1.

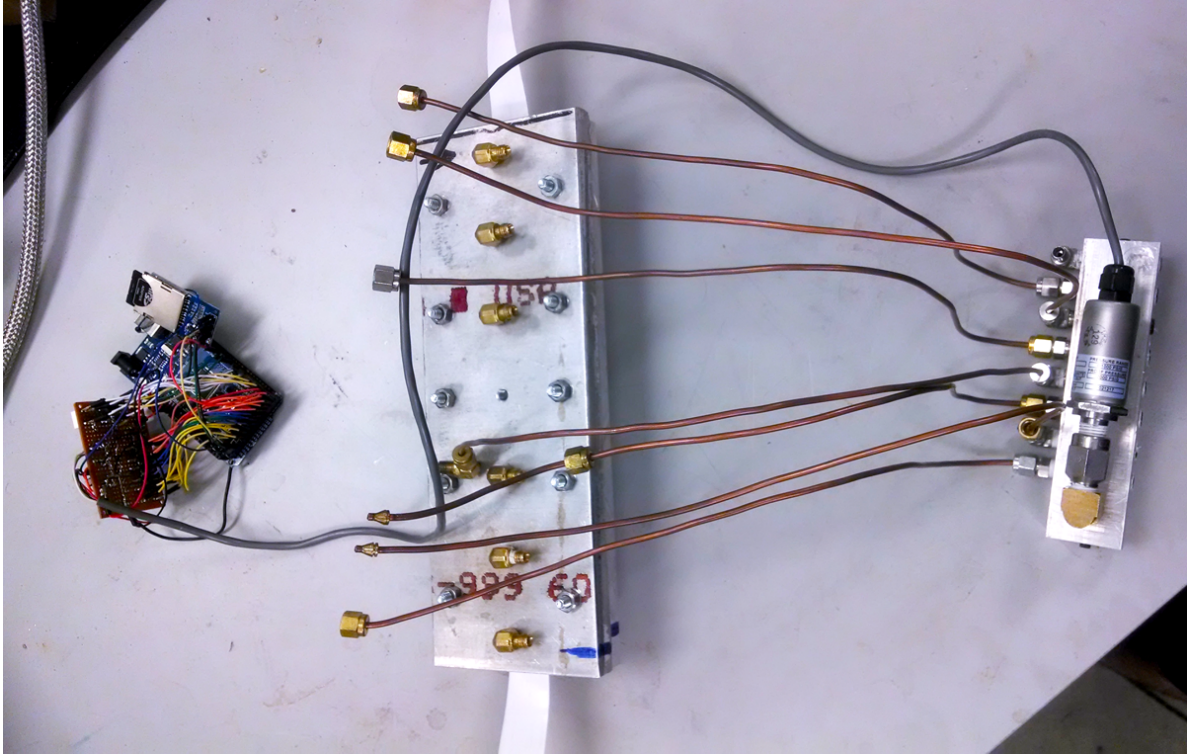


Figure 6.1: Broad view of the system built to capture pressure data inside the dynamic seal.

A series of small diameter holes were drilled through the top plate and seal material in order to probe fluid adjacent to the polymer film. The holes are equally spaced by 1.75 inch distances.

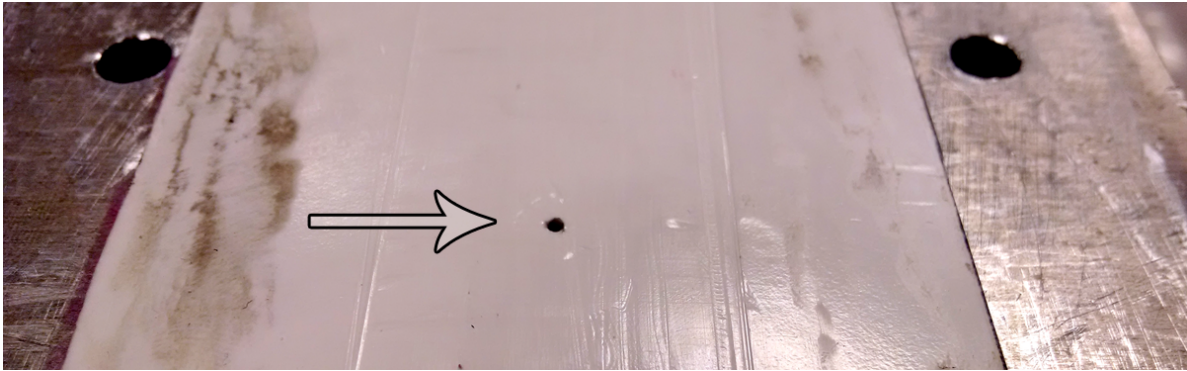


Figure 6.2: Photo showing a hole through the seal material. The fluid pressure at that location is thus measured.



Figure 6.3: The holes go all the way through the aluminum plates and connect to the compression fittings on the other side.

NPT 1/16 inch brass fittings were installed to transfer the pressure information to the sensor.

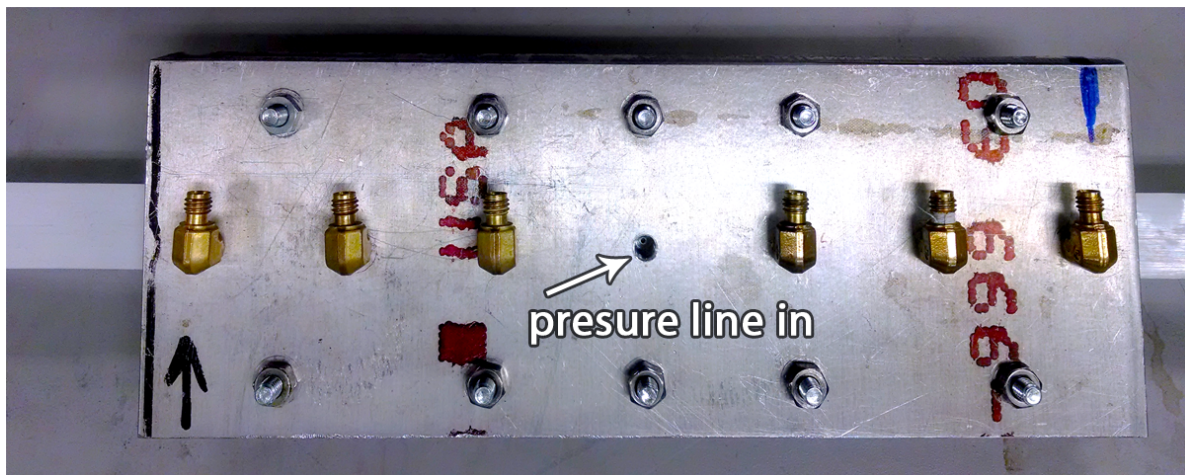


Figure 6.4: Fittings concentric to the small test holes. Copper tubes transfer the fluid pressure to the pressure sensor in order to be read.

The task left was to devise a way to selectively read one port at the time while keeping

all others closed. To do this, a special device was designed and built that allows the input of eight fittings whose paths to the sensor are opened/closed using a set screw. When in use, all ports are closed except for the one desired to be read. The figures better explain the device.

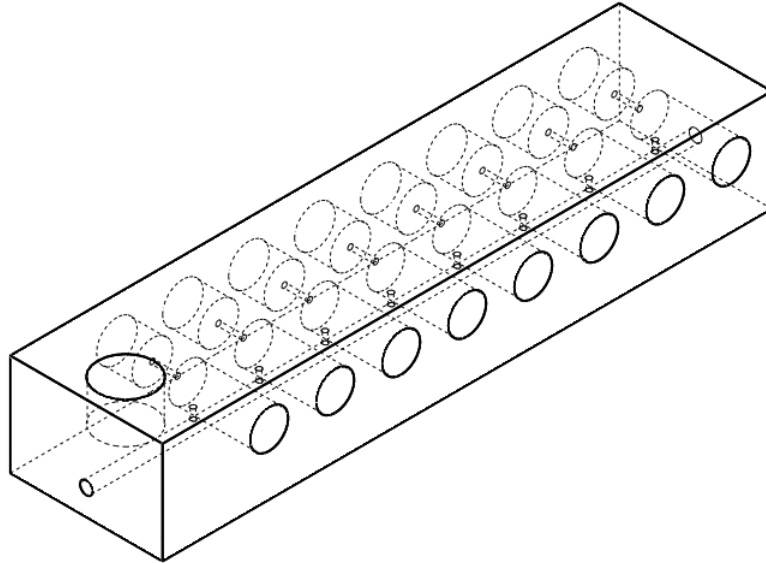


Figure 6.5: Isometric view of the line router device CAD model.

The sensor is fitted to the large hole on the top and connected to the long channel that runs along the length of the part. The ends of this through-hole are closed off with screws after drilling.

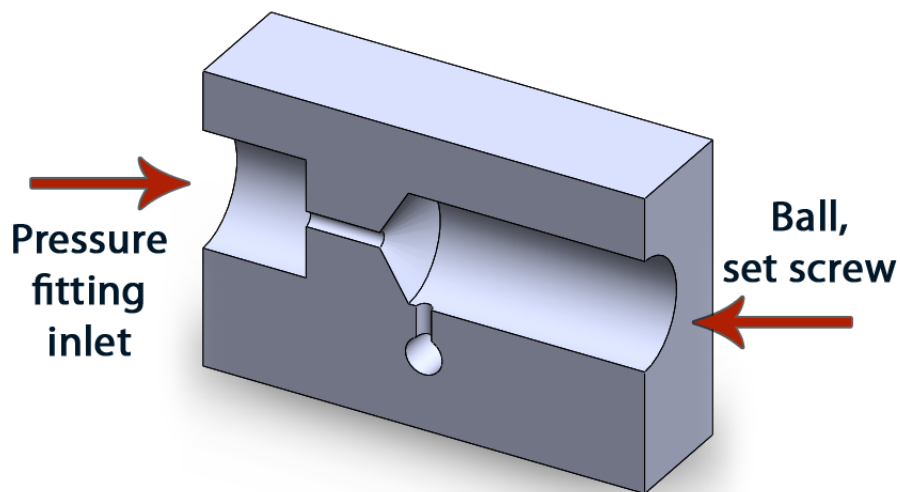


Figure 6.6: Section view showing the internal connections.

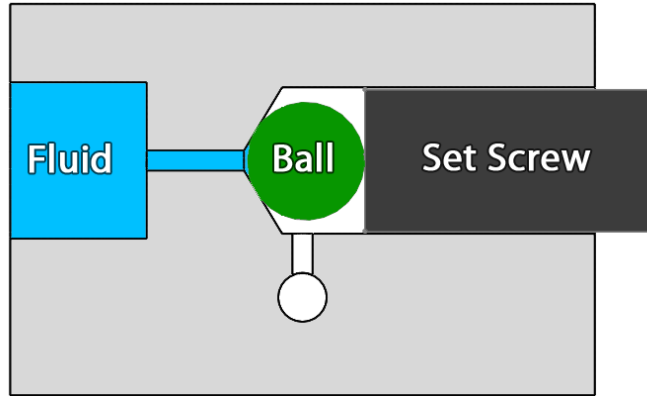


Figure 6.7: Diagram showing the working principle. A steel ball is pressed against the opening by a set screw to close off the fluid. When the fluid pressure is to be read, the set screw is backed off and the ball is pushed back by the fluid and can enter the channel (below) that routes to the sensor.

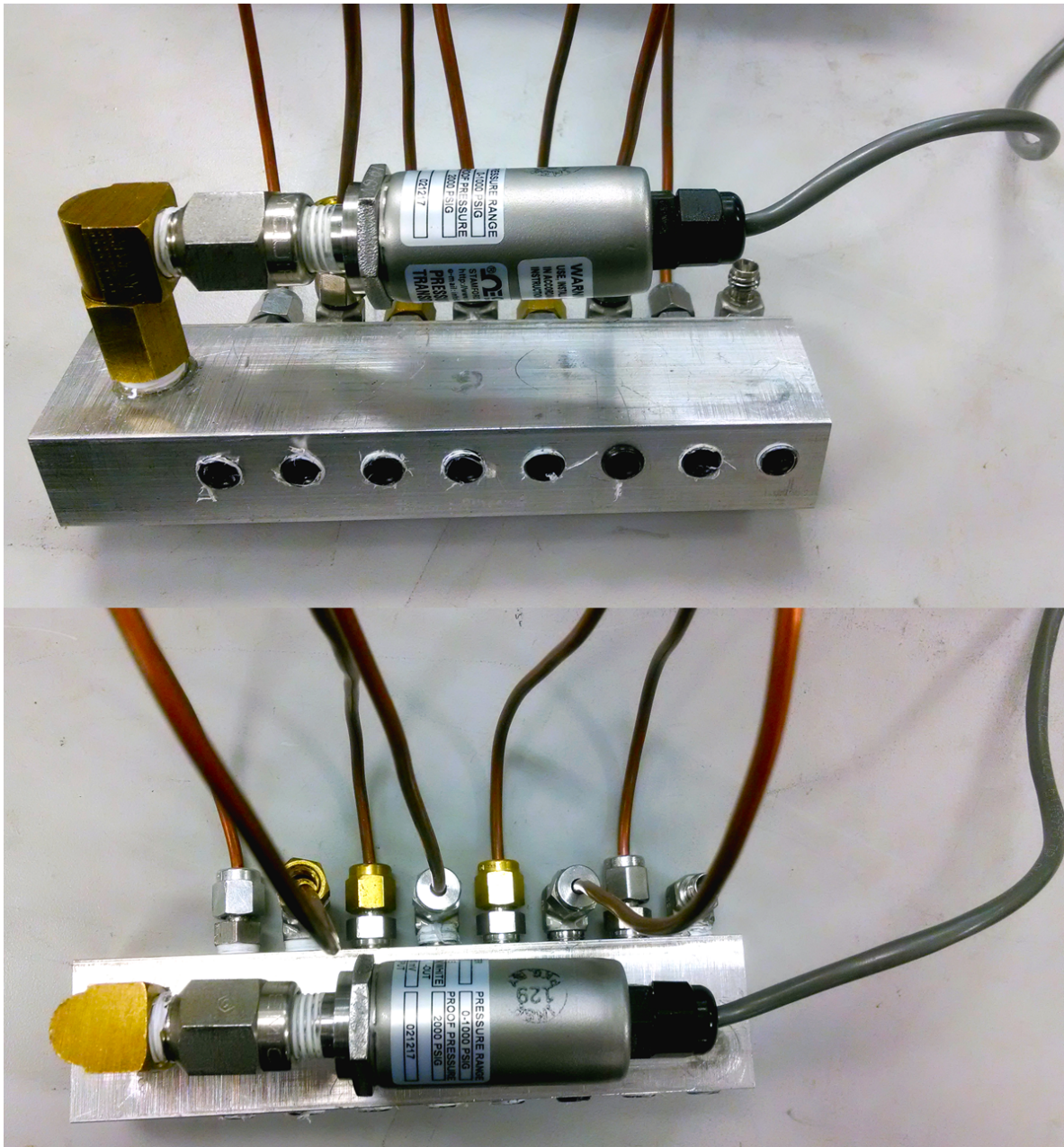


Figure 6.8: The finished product with sensor, screws, balls, and fittings assembled.

The device worked very effectively. This is because the body is made of aluminum and the sealing ball is steel whose elastic modulus is 2-3 times larger. This means that the ball can be pressed into the aluminum and create a ring “seat” which seals the opening. Upon release of the set screw, the higher pressure from the port pushes the ball away from the opening and allows the fluid through. When the fluid to be read is incompressible, there is very little flow necessary unless the system contains a trapped compressible fluid (such as air pockets). A quarter turn of the set screw was sufficient for the task.

6.1.2 Electronics

In order to read the electronic sensor and store the data, an electronic system was designed and built. It is centered around a microcontroller that reads the sensor's voltage and stores the data to an SD card as a .csv file. A small control panel was also constructed to allow input from the user and display the sensor reading in real-time.

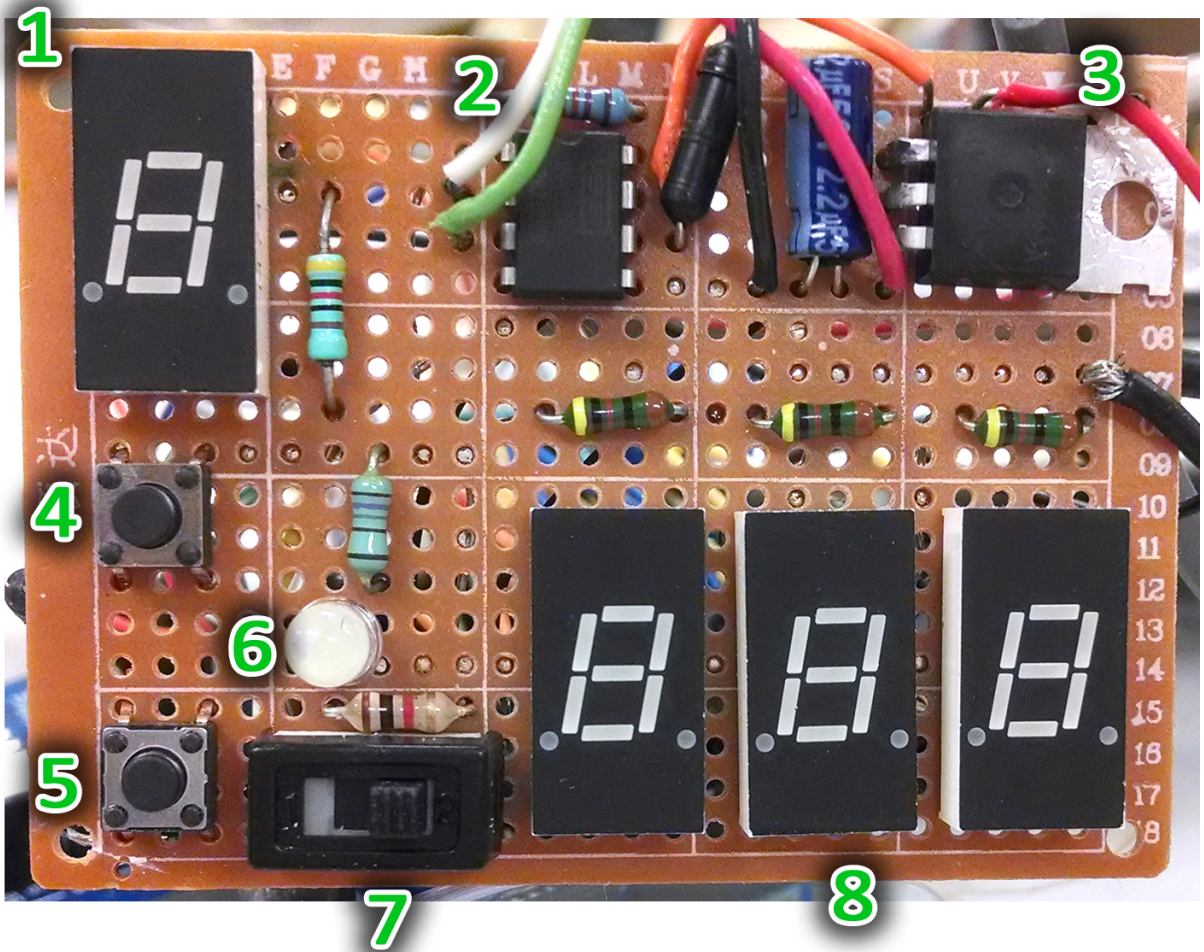


Figure 6.9: Display panel made on prototype board. This is how the user interacts with the data acquisition system.

The list of components labeled 1-8 in figure 6.9 are as follows:

1. SELECT Digit Display
2. Instrumentation Amplifier
3. 12 V Voltage Regulator

4. SELECT Button
5. START Button
6. BUSY LED
7. Gain Switch
8. Pressure Readout

All these circuit components are readily available “off the shelf” parts obtainable at the UW EE store (with the exception of the amplifier IC).

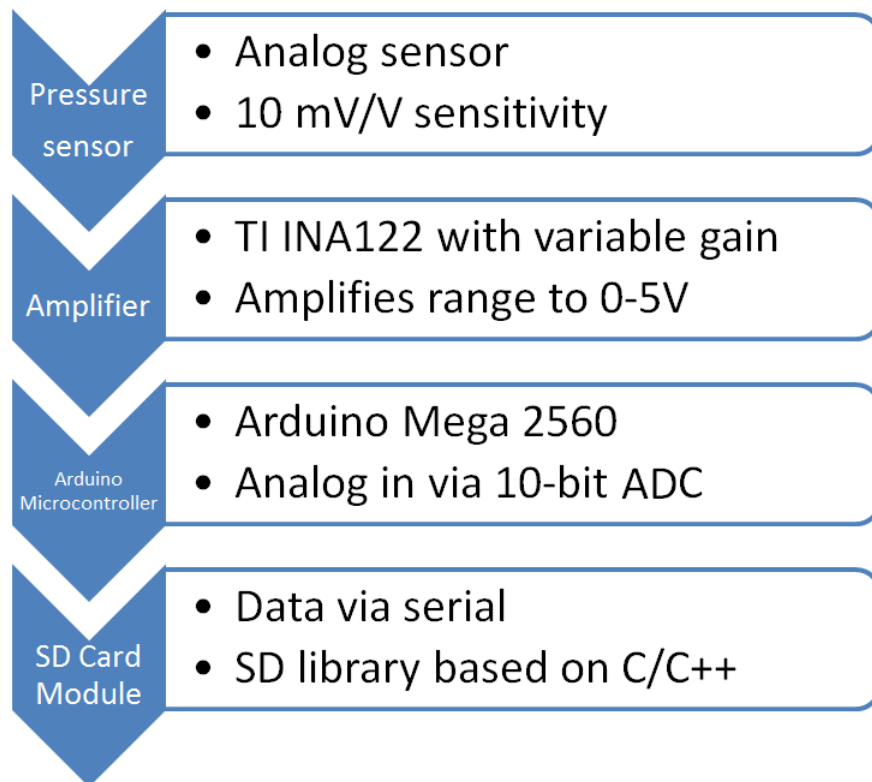


Figure 6.10: Diagram of the data acquisition process.

Data from the sensor is stored in the columns of an array. The array size is specified in the code (number of columns = number of experiments, number rows = number of data points in each experiment). The first row entry in each column is assigned a number that the user chooses using the SELECT button; this organization tool is useful in keeping track of what port the data is coming from.

	A	B	C	D	E	F	G	H
1	1	2	5	2	4	←	Column Label	
2	316	430	293	381	280	}		
3	315	429	292	380	279			
4	316	430	293	381	280			
5	316	430	293	381	280			
6	315	429	292	380	279			
7	315	429	292	380	279			Sensor reading
8	314	428	291	379	278			(digital voltage 0-1023)
9	314	428	291	379	278			
10	315	429	292	380	279			
11	315	429	292	380	279			
12								

Figure 6.11: Sample data file from the acquisition system. Each experiment collects N data points and stores them in a columns as CSV format. The first row entry of each column is chosen by the user before the data collection is begun.

A code was written using the Arduino programming environment (based on the language C) to perform the data acquisition and control the user control panel. The range of pressures to be measured is 0-800 psi. However, when measuring lower pressures it is desirable to obtain better accuracy by limiting the range. To do this, a switch on the board is used to change the amplifier gain by completing the circuit to an additional parallel resistor. The switch position 1 maps 0-250 psi to 0-5 V, while position 2 maps the full range 0-800 psi to 0-5 V. In order to display real time pressure rather than digital voltage, two sets of calibration values must be stored on the microcontroller. Since the sensor is linear with pressure (stated on the datasheet) each set has two calibration values. The operation works as follows:

1. After power-up, a mode is selected using the SELECT button. The three modes available are HIGH gain (0-250 psi), LOW gain (0-800 psi), and raw voltage (for calibration/debugging).
2. The START button is pressed and the microprocessor is ready for data acquisition.
3. The SELECT button is used to label the column (assign the first row entry value with a number from 0-9)
4. The START button is pressed and data acquisition begins to fill an entire column with pressure data. The data rate is set by the delay in the program loop. The white BUSY LED lights while this is happening.
5. After a column is written, the system is ready to repeat steps 3,4. When the maximum number of experiments is reached, the microprocessor writes all data to the SD card and blinks the BUSY LED 6 times to confirm that the data was saved on the SD

card. The file name of the data file is the number of milliseconds since microprocessor bootup. This helps keep track of experiment sequence.

The software is elaborate so the full code is shown in an appendix.

6.2 Preliminary Results

The device was used briefly to obtain some data for a closed-cell polyurethane seal. The pressure source was shop compressed air, and the film material was the same 1 inch wide ABS strip used previously. In this experiment, the film was aligned between the plates shown in figure 6.4 and clamped with the 10 bolts as shown. The pressure was applied through the center hole and the pressure at the other hole locations was measured with the sensor setup described above.

It was observed that bolt tightness had a pronounced effect on the pressure distribution. Therefore, a wrench was used to vary the tightness of various bolts by less than 1/4 of a turn: when this was done it produced a completely different plot. Below are five such plots, showing how drastic the pressure distribution can be altered by slightly tightening or loosening a few bolts.

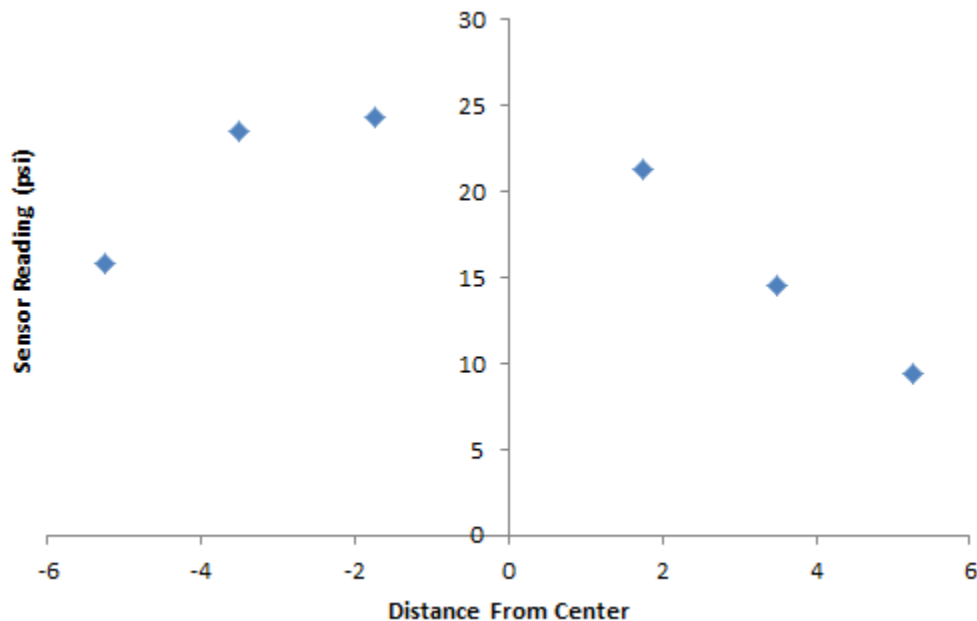


Figure 6.12: Pressure distribution experiment 1.

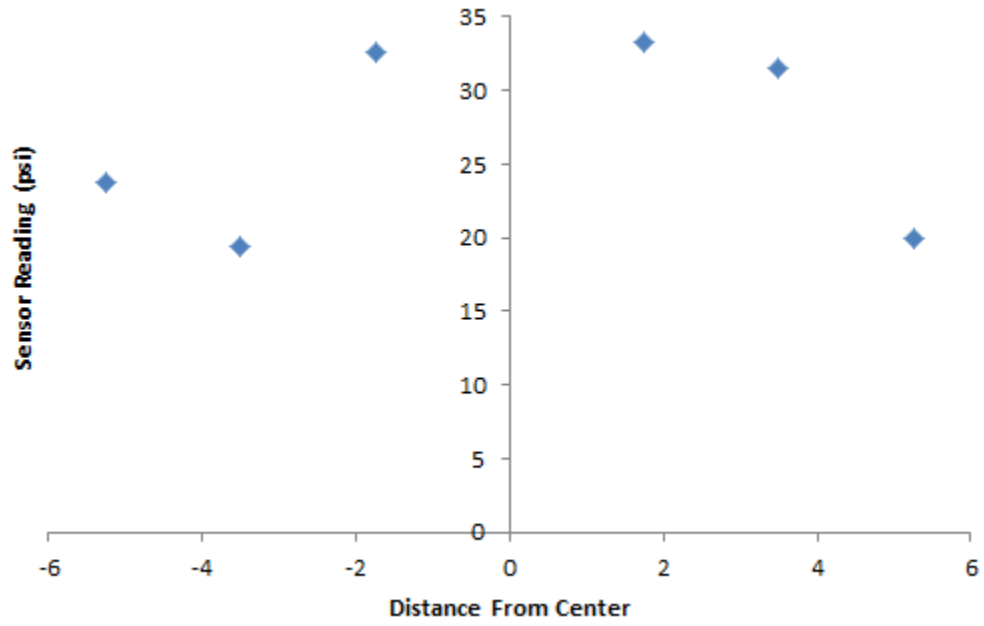


Figure 6.13: Pressure distribution experiment 2.

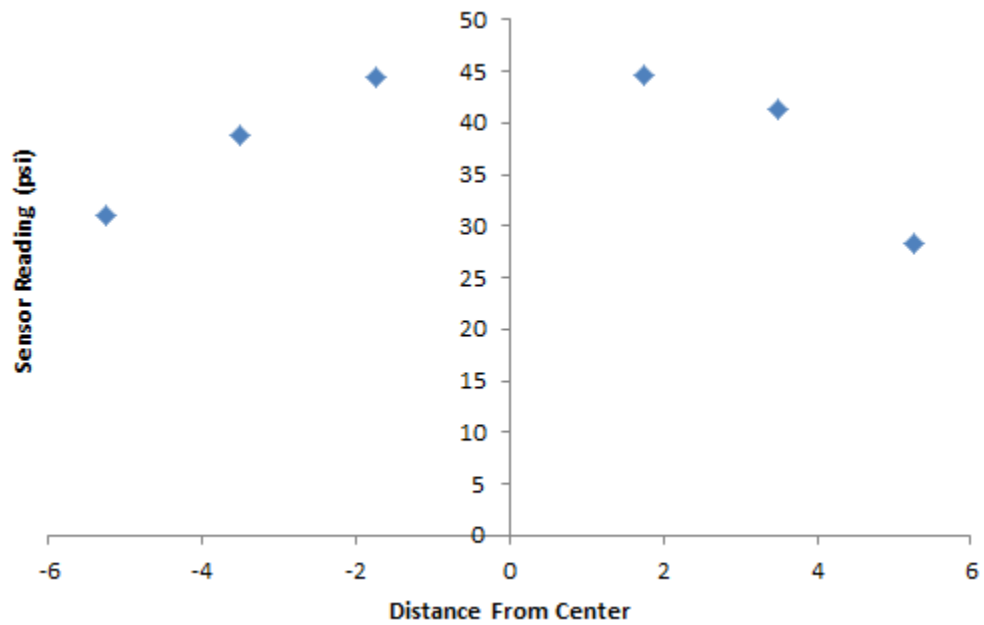


Figure 6.14: Pressure distribution experiment 3.

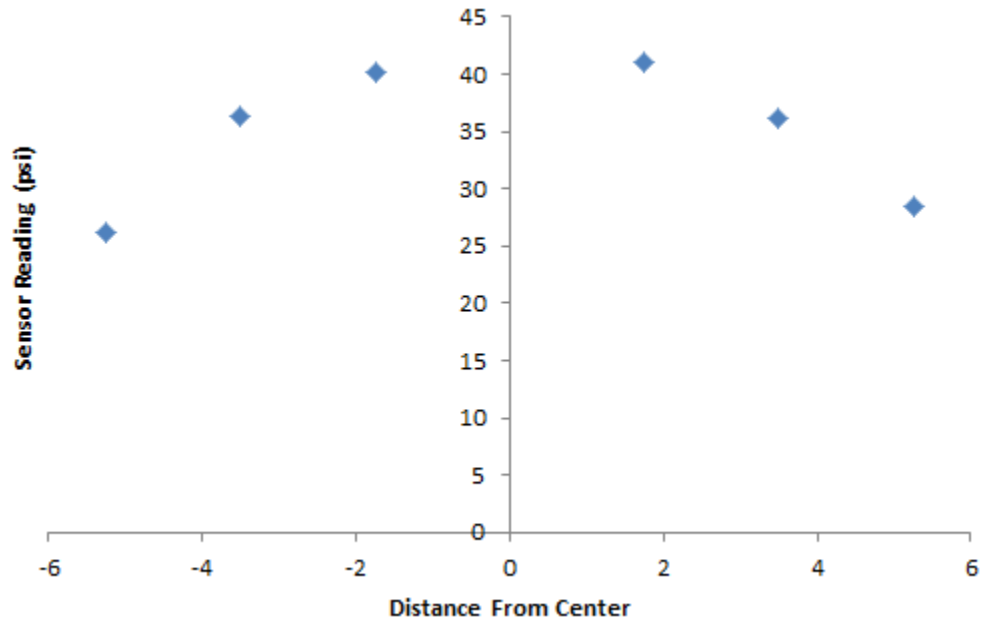


Figure 6.15: Pressure distribution experiment 4.

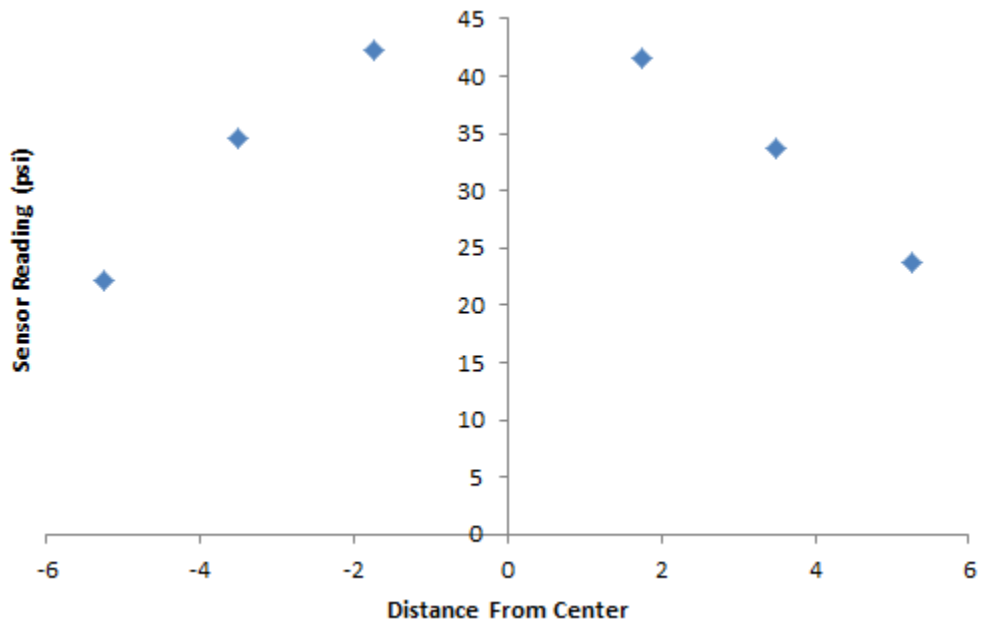


Figure 6.16: Pressure distribution experiment 5.

The results from these experiments were expected because the seal materials do not form perfectly planar, parallel, rigid surfaces for the fluid. Thus, the flow is unpredictable and

does not follow the Poiseuille-flow solution discussed in chapter 3 which states that dp/dx is constant, therefore the plots should be straight lines. This is because the uneven bolt tightness causes nonuniform flow regions inside the sea. This effect and possible solutions are discussed in the next chapter.

Chapter 7

New Ideas and Future Work

In light of the pressure measurements shown in the previous chapter, some thought was devoted to eliminating the problems of uneven pressure distribution with the goal of reducing leakage and friction. Due to time constraints, these have not been built but rather were developed purely from theory and things learned from past experiments and experiences with the behavior of the dynamic seal.

7.1 Cutting Channels

The experiments described in chapter 6 showed some evidence that uneven bolt clamping can close off areas of the seal and prevent fluid flow in those areas. This means that the polymer can come in direct contact with the seal material which increases friction and yields a nonlinear pressure distribution due to nonuniform velocities and fluid layer thickness.

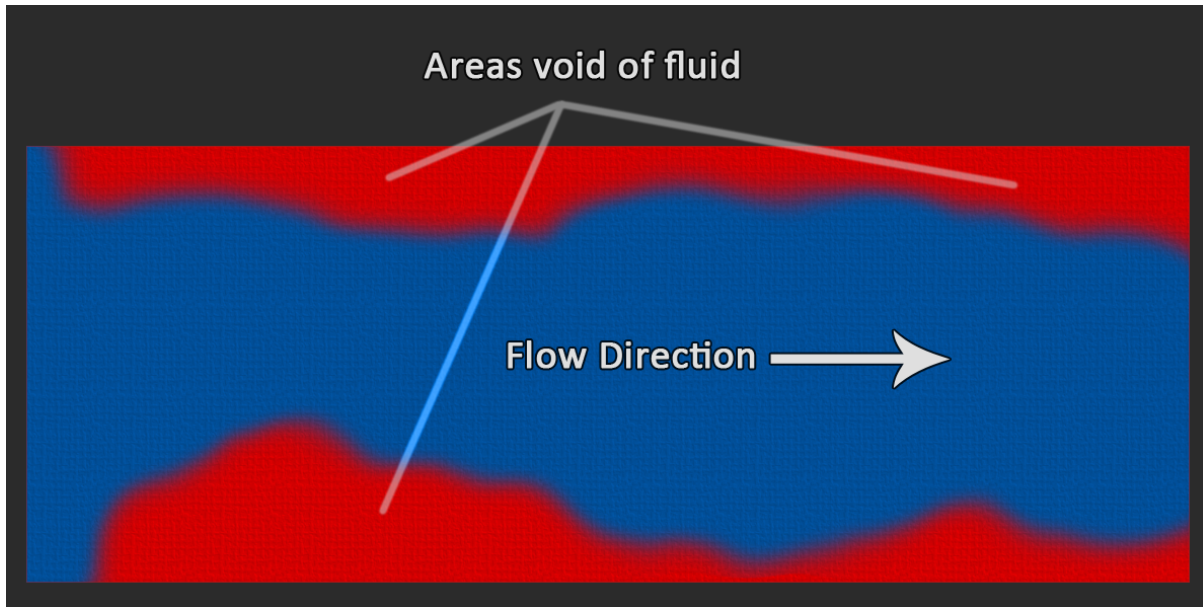


Figure 7.1: Due to uneven bolt clamping, the fluid may be squeezed out of certain areas which leads to non-uniformity in flow and pressure drops.

A concept to remedy this was developed. It involves cutting channels in the material in order to guide the fluid more uniformly across the seal material and ensure that it spans the width of the seal so that lubrication occurs at all points of the polymer film. These channels need be shallow to prevent excessive leaks. The following figures show a few implementations; there are many other possibilities as well.

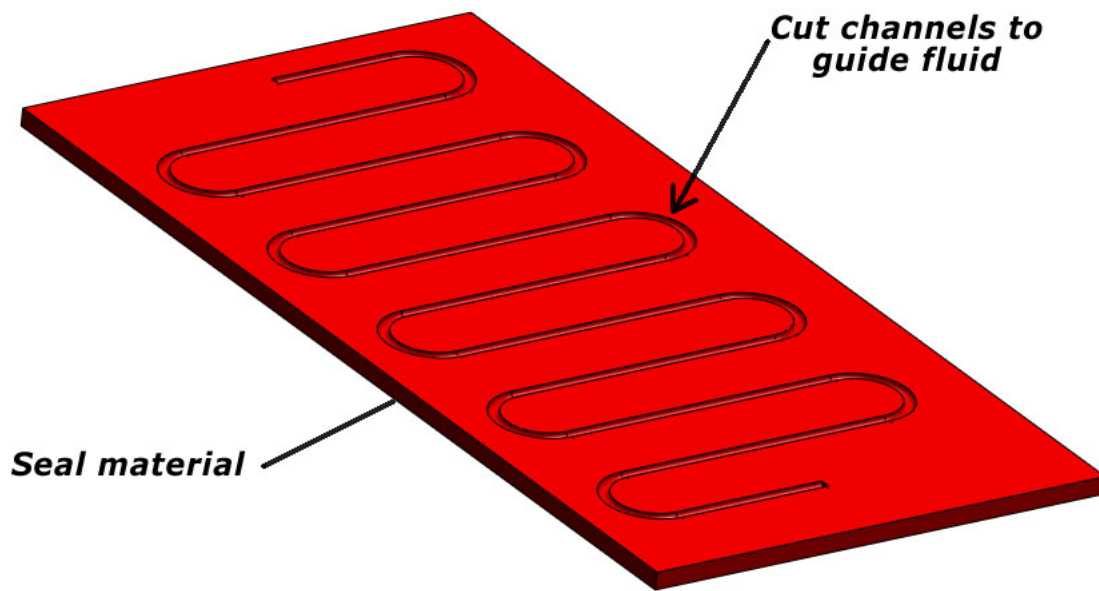


Figure 7.2: A serpentine style cut guides fluid along the width of the seal. Since the arc length of the curve is large, the resistance to fluid flow will be large and thus the leak minimized.

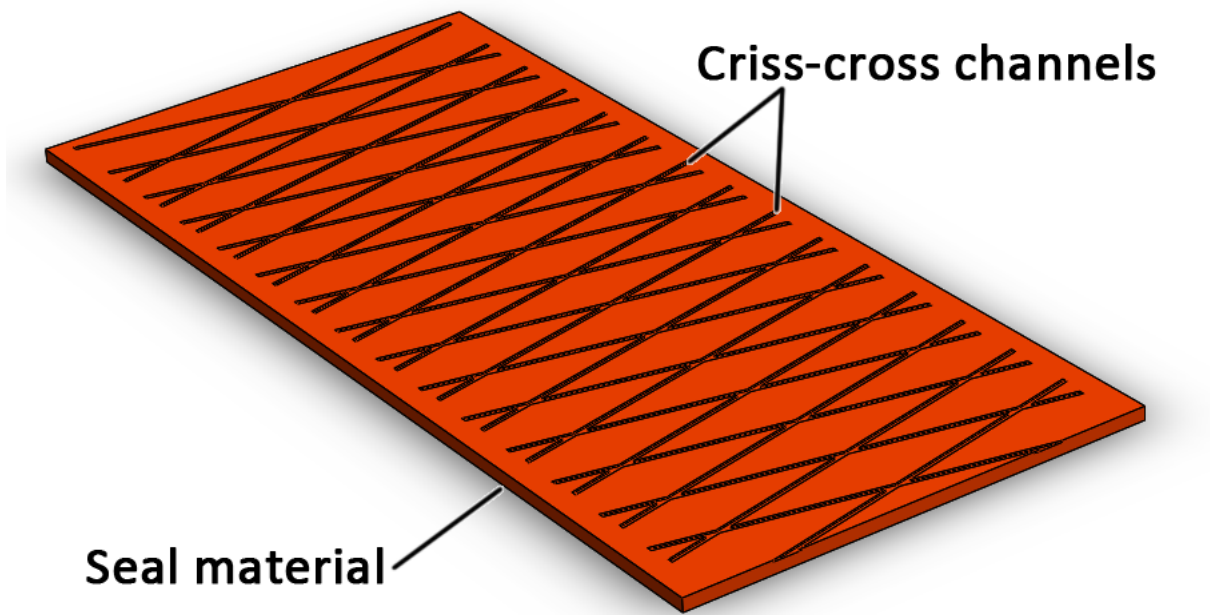


Figure 7.3: A criss-cross pattern style utilizing the same idea.

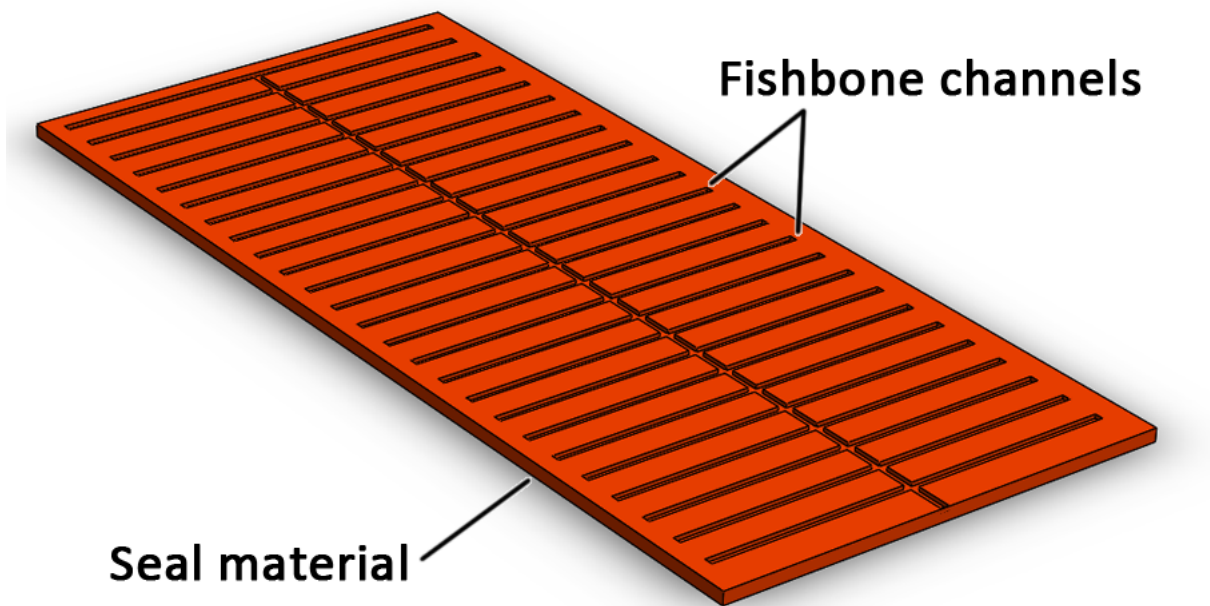


Figure 7.4: A fishbone style pattern.

In figure 7.4, the narrow longitudinal channel feeds fluid to the series of cuts along the

transverse direction. Since there is no flow in the transverse direction, the pressure is uniform. However, since there is flow in the longitudinal direction, each successive transverse cut will have a different pressure, decreasing discretely as it approaches the exit point.

7.2 Combining Porous and Nonporous Materials

It was shown in chapter 3 that porous materials offer a linear pressure distribution and that the flow rate through such material is proportional to the pressure gradient. However, these materials do not allow the formation of a lubricating thin fluid layer between the polymer film and the seal for the material to glide over as do non-porous materials. The question was asked whether the materials can be combined in an effective way and extract the benefits of each material type. Such a design is given in figure 7.5.

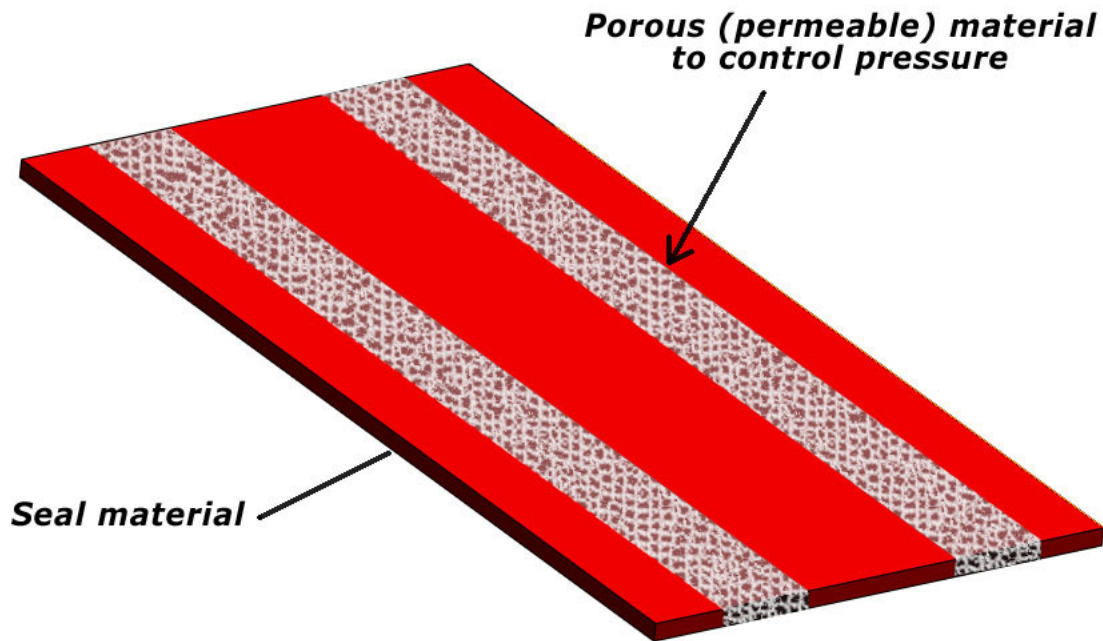


Figure 7.5: A possible seal design using both porous and nonporous material in order to obtain the benefits of both.

Strips of alternating porous/nonporous material run along the length of the dynamic seal. The majority of area should be covered by the nonporous material to allow for a larger fluid layer that reduces the friction. The idea is that the porous material enforces the linear pressure distribution by its inherent properties discussed earlier. Therefore, in theory, nonlinear pressure gradients can only exist along the nonporous material. However,

since these areas are narrow and their boundary conditions force a linear pressure drop, the non-linearity would be greatly reduced.

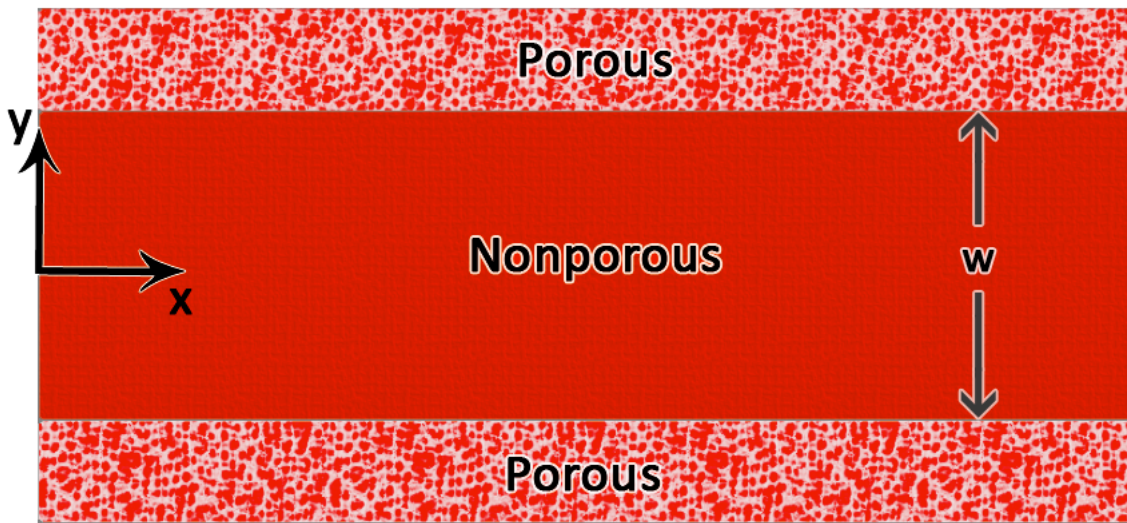


Figure 7.6: A nonporous material bounded by porous materials on either side. This seal material arrangement can make use of the advantages of both material behaviors.

Looking at figure 7.6 suppose that the polymer film is to be drawn along the x -axis. The pressure bounds are $P(x = 0) = P_v$, the vessel pressure, and $P(x = L) = P_{atm}$, the atmospheric pressure. Along the porous materials, the pressure will be linear with x as was shown in chapter 3.

$$P(x, y) = P_v(L - x) + P_{atm}(x/L) , \quad x \in [0, L] , \quad |y| > w/2 \quad (7.1)$$

Along the nonporous material (i.e. for $|y| < w/2$) imperfections can lead to unpredictable pressure distributions as seen in chapter 6. However, any pressure gradients in the y direction will cause flow along that direction. If w is small (i.e. $w/L \ll 1$), the gradients must be very large to have significant flow in the y direction; therefore, the pressure along y will be kept close to uniform. In effect, the porous materials are used to control the pressure distribution along the entire seal and bring it as close as possible to linear even in the nonporous regions.

The nonporous region, as discussed in chapter 2, will have a thin layer of fluid between the polymer and the seal material: this will serve as lubrication. The advantages of both porous and nonporous materials are therefore combined in this hybrid system.

7.3 High Viscosity Fluids/Gels

In chapter 3 it was shown that for a given pressure gradient, the flow is proportional to viscosity at low Reynolds numbers. Thus, if a fluid with very high viscosity is used, the flow will be substantially reduced.



Figure 7.7: A grease product tried as blocking fluid for the dynamic seal.

The grease product in figure 7.7 is a non-Newtonian fluid since it can retain its shape under nonzero stresses. However, the general relationship still holds and has been proven effective as a blocking fluid in a small test using the apparatus in figure 7.13; further work can be done to explore this idea and quantify the results.

7.4 A New Idea: Self-Sealing Design

While considering the dynamic seal design discussed thus far, the question arose as to what would happen if the seal material is improperly adhered to the metal clamping plates and becomes detached during operation. In this case, the vessel fluid will protrude into the void created by the detachment. The pressure of this fluid equals the vessel pressure and will exert a force on the seal material. It was then postulated that this fact could be used as a design tool and develop a seal embodiment wherein the fluid exerts the clamping force as opposed to the metal plates/bolts arrangement.

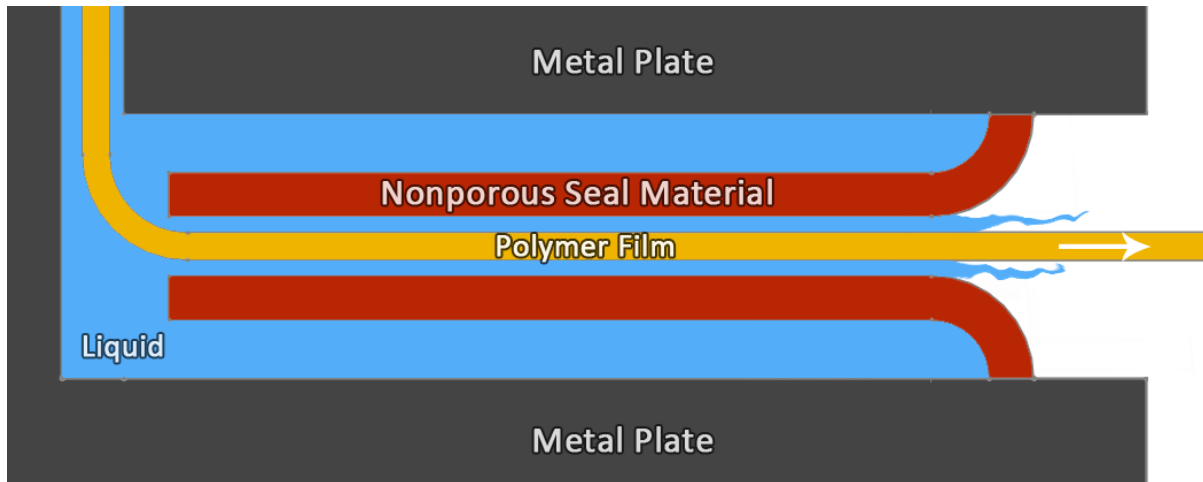


Figure 7.8: An example where the fluid from the vessel is allowed to enter the space between the clamping plates and the seal material. Thus, the seal material experiences forces from fluid pressure on either side.

By analyzing a free-body-diagram on the seal material, it becomes apparent that the stagnant fluid from the vessel will exert a net force on the seal material due to a pressure differential. Since the fluid leaks along the gap between the seal material and polymer, the wall shear τ necessitates a pressure drop. Looking the a free body diagram of a fluid element between the seal material and polymer it is clear that

$$p(x) - \tau\delta x \approx p(x + \delta x) \quad (7.2)$$

Since the fluid has nonzero viscosity,

$$\tau > 0 \quad \therefore p(x + \delta x) < p(x) \quad (7.3)$$

or equivalently

$$\frac{\partial p}{\partial x} < 0 \quad (7.4)$$

Therefore, since the pressure of the flowing fluid is continually decreasing, there is an increasing net force on the seal material that works to press it closer to the polymer film.

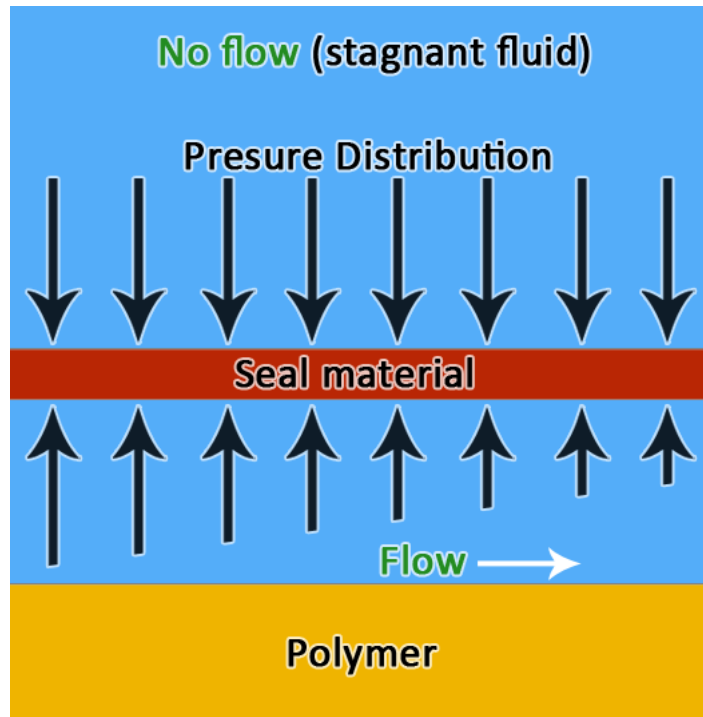


Figure 7.9: A look into the pressure distribution on the seal material if it was rigid and flat. Since the fluid between the seal material and polymer is flowing, the pressure drops due to wall shear (from viscosity).

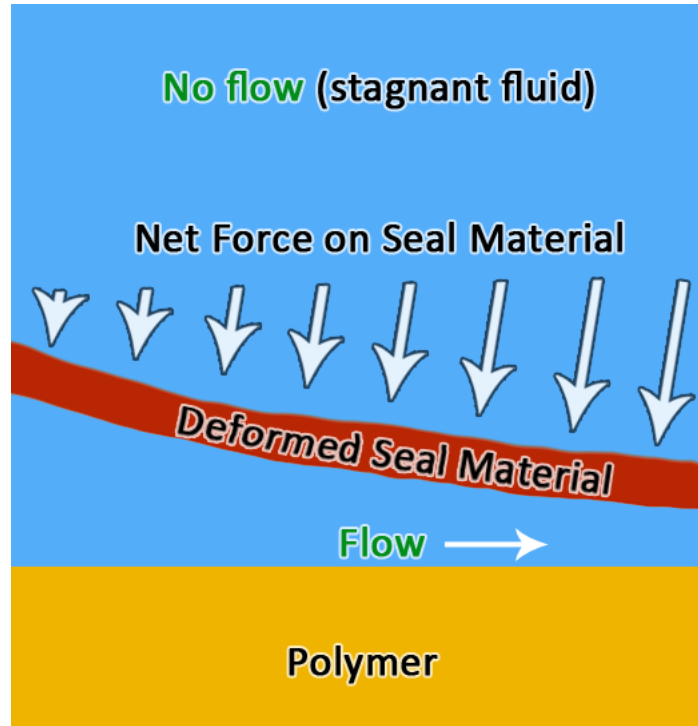


Figure 7.10: Since the seal material is deformable, the net pressure force presses it closer to the polymer film, decreasing the gap and choking the flow.

As shown in figure 7.10, the pressure imbalance acts to reduce the size of the channel through which the fluid is leaking (between the seal material and polymer film), effectively choking the flow. However, the flow cannot be stopped completely or else the wall shear vanishes and the pressure would then be equalized on both sides of the seal material. This creates a feedback control system where the gap is minimized however not eliminated, meaning that the leakage is minimized but there is still a layer of fluid for lubrication: the best possible scenario for the dynamic seal application.

7.4.1 Experimental Verification

The discussion above is essentially a paradox: the faster the fluid leaks, the higher the pressure differential and the tighter the gap becomes, reducing the flow. In reality, there must be an equilibrium solution to the problem. To test out the theory, an apparatus was built as shown in the following figures.

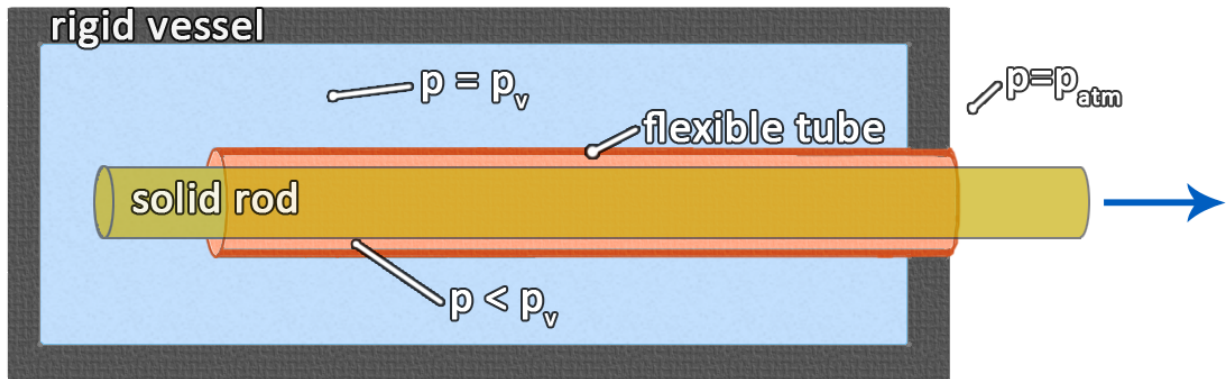


Figure 7.11: A solid rod is loosely fed through a soft tube which is adhered to a rigid pressure vessel. The only path for the vessel's fluid to escape is through the space between the solid rod and the inside of the flexible tube.

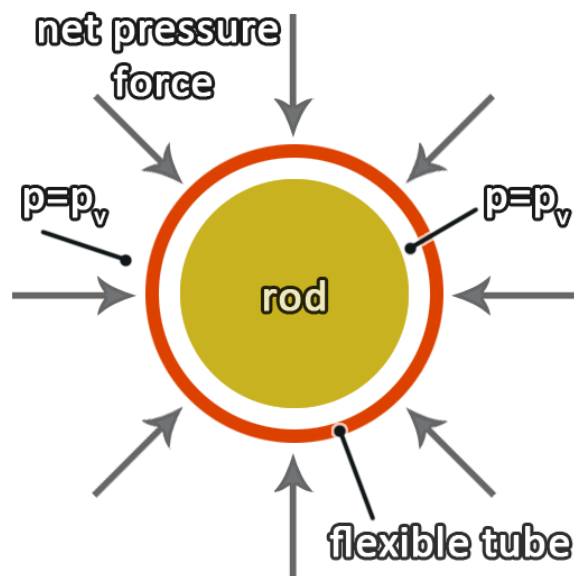


Figure 7.12: Net force diagram of the cylindrical setup. The pressure difference between the two sides of the tube work to collapse it onto the rod and reduce the flow.

The design in figure 7.12 sets up the same conditions as described in the previous discussion; it was built using readily available parts.

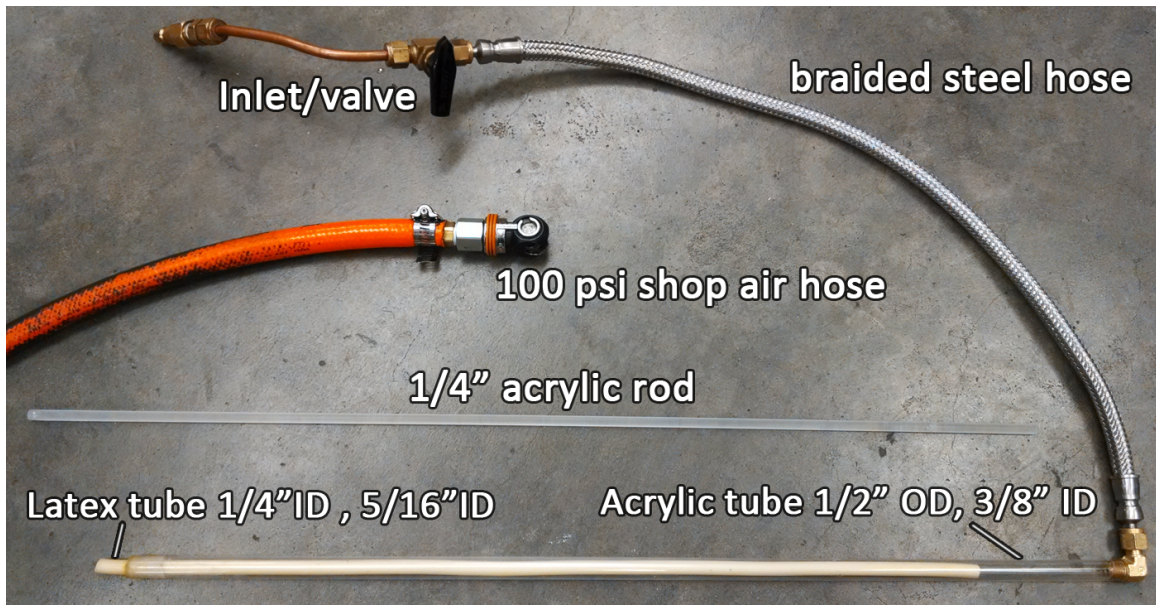


Figure 7.13: Photograph of the parts used in the experiment.

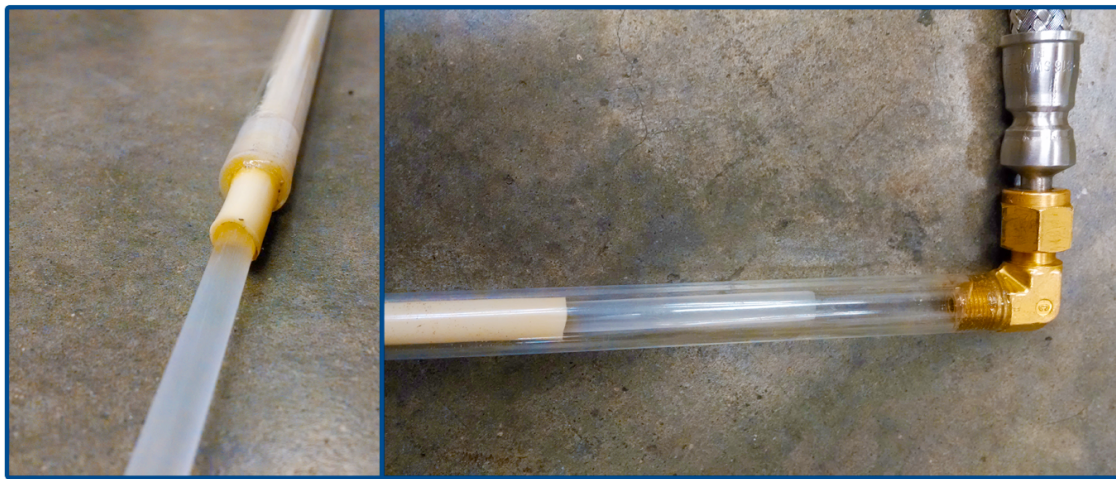


Figure 7.14: Closeup view of the experimental setup. Left: rubber tube is attached to the acrylic tube wall using epoxy and contact cement. Right: solid rod protruding all the way through the tube close to the brass fitting (pressure inlet).

An acrylic tube was fitted on one end with a brass fitting that connects to a quick-release compressed air hose. Thus, about 100 psi air is fed into the system. The soft latex tube is attached to the other end, and a rod is fed through its inside. The 1/4 inch solid acrylic rod was abraded with sandpaper in order to reduce the diameter to about 0.245 inch such that it would slide through unrestricted. The diameter difference between the solid rod and inside of the latex tube allows the high pressure fluid to flow towards the atmosphere.

When the system was pressurized using compressed shop air (at about 100 psi), the rubber tube collapsed onto the solid rod from the pressure difference so tightly that in one instance the rod actually became stuck. This is counterintuitive since the expectation would be that the solid rod will be pushed out like a piston by the high pressure gas. There was also very little gas leakage. The experiment was restarted with the solid rod only partially inserted into the tube (to reduce the grip area) and when the pressure was reapplied, there was a very small gas leak along the rod which was free to move easily by hand. This confirmed the success of the theory and design. Future work on this topic would be to model this system using solid-mechanics/fluid-mechanics combined theory and explore its behavior with non-ideal geometries.

7.5 Overall Conclusions

Designing a successful dynamic seal has been an unanswered challenge since the solid state microcellular process originated. The task is made difficult by the conflicting demands that the seal must block a gas but not a solid; usually, restraining a solid is much easier than a fluid. However, the present work has made progress into answering this request using engineering principles to design a system with the proper components for a working dynamic seal. The effectiveness was demonstrated in numerous experiments on both films, fibers, and even solid rods. In essence, designing such seals is almost an art, where the designer must strongly use intuition backed by engineering theory to make appropriate design decisions. This project has helped the writer develop that intuition, a very useful tool since modeling fluid behavior in a deformable solid medium is analytically challenging. The future work for this project is very exciting and promising, and the applicability of this research to a real industrial process is very likely. It was an excellent experience overall.

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Appendices

Appendix A

List of Programs Used

Many software packages were used to perform the work outlined in this thesis. These tools are valuable to the engineer for performing calculations, drafting designs, analysis, and for creating the contents in the thesis itself. The list is not exhaustive and acknowledges a subset of such programs.



Appendix B

MATLAB Code

The above equations were scripted in MATLAB. First, the system of equations was solved using the `fsolve` function which finds roots of vector functions $F(\mathbf{x}) = \mathbf{0}$. The centroids were trivially found following the solution.

Function file

```
function F=bolts(x); % n comes in length n+1
n=evalin('base','n');
a=evalin('base','a');
b=evalin('base','b');
F=zeros(n,1);
%area=evalin('base','area');
for i=2:n-1 % n is the number of unknowns (interior bolts, excluding 0 and b)
    F(i)=(.5*a/b)*(x(i-1)-x(i))*(-2*b+x(i-1)+x(i)) -x(n);
end
F(1)=(a*x(1)/2)*(2-x(1)/b) -x(n)/2;
F(n)=(.5*a/b)*(b-x(n-1))^2 -x(n);
```

Running script

```
clc
clear all
close all
n = 6; %n is number of bolts including first at 0 (middle one)
a=5;
b=13;
%area=a*b/(2*n+3);
area=a*b/(n+1)/2;
guessvector=ones(n,1);
%guessvector=[2,5,8,10]';
```

```

options=optimset('TolX',1e-10,'TolFun',1e-10);
[solution,extra,flag,out]=fsolve(@bolts,guessvector,options);
solution

U=[0;solution];
U(end)=b; %makes it length n+1
for k=1:n %find bolt location at centroid of each area element
    B(k,1)=(3*b*(U(k)+U(k+1))-2*(U(k)^2+U(k)*U(k+1)+U(k+1)^2))/(6*b-3*(U(k)+U(k+1)));
end
B
yb=zeros(length(B),1);
yu=zeros(length(U),1);
plot(B,yb,'o')
hold on
plot(U,yu,'k*')
hold on
plot([0,b],[1,0])

%a*b/2/n
%(a/b)*(2*b-solution(1))*solution(1)%first area, double block
%(.5*a/b)*(solution(3-1)-solution(3))*(-2*b+solution(3-1)+solution(3))
%(.5*a/b)*(solution(4-1)-solution(4))*(-2*b+solution(4-1)+solution(4))

```

Appendix C

Arduino Microcontroller Code

The code for programming the Arduino microcontroller was written using the “Arduino Programming Language” which is an open source environment based on the programming language C. The full code is shown below. Note that this will only run on Arduino MEGA boards as it uses more than 13 digital outputs.

```
#include <SD.h>
#define maxdata 100
#define maxcol 8
#define sensorpin 0 // analog in for pressure sensor
#define switchpin 2 // digital pin for clicker switch
#define gopin 3
#define busypin 4

int data[maxdata+1][maxcol];
int port=1;
int column=0;
int mode;
int A; // calibration constants
int B;

int segmentpin[4][8]={
{9,15,16,17,18,19,20,21}, // elements 0-7 as on sheet.
{9,22,23,24,25,26,27,28}, // pressure digit 1
{9,32,33,34,35,36,37,38}, // pressure digit 2
{9,42,43,44,45,46,47,48}, // pressure digit 3
};

int segmentsONOFF[11][8] = {
{0,1,1,1,0,1,1,1}, // number 0
{0,0,0,1,0,0,1,0}, // number 1
```

```

{0,1,0,1,1,1,0,1}, // number 2
{0,1,0,1,1,0,1,1}, // number 3
{0,0,1,1,1,0,1,0}, // number 4
{0,1,1,0,1,0,1,1}, // number 5
{0,1,1,0,1,1,1,1}, // number 6
{0,1,0,1,0,0,1,0}, // number 7
{0,1,1,1,1,1,1,1}, // number 8
{0,1,1,1,1,0,1,0}, // number 9
{1,0,1,1,1,1,1,0}, // X is 10
};

```

```

////////////////////////////////////
void fileRITE(int dataarray[maxdata+1][maxcol]) // writes array to SD card
{
//char filenamee[]="something.txt";
char time_name[25]; // first 4 digits of seconds since bootup
sprintf(time_name,"%d_pressureData",millis()/1000);
File dataFile = SD.open(time_name, FILE_WRITE);

// if the file is available, write to it:
if (dataFile)
{
for(int k=1;k<=maxdata;k++)
{
for(int j=1;j<=maxcol;k++)
{dataFile.print(dataarray[k][j]);
dataFile.print(",");
}
dataFile.print("\n");
}

dataFile.close();
}
}

```

```

////////////////////////////////////

void setup()

```

```

{
  pinMode(switchpin, INPUT);
  pinMode(gopin, INPUT);
  digitalWrite(gopin,HIGH); // enables internal pullup resistor
  digitalWrite(switchpin,HIGH);
  ledwrite(port,0);
  // switch on 7 segments
  for(int k=1;k<=4;k++)
  {
    for(int j=1;j<=8;k++)
    {pinMode(segmentpin[k][j],OUTPUT);
    }
  }

  // mode select
  int z=1;
  while(!digitalRead(gopin))
  {
    if(digitalRead(switchpin)) ++z;
    if(z>3) z=1;
    ledwrite(z,0);
    if(z=1) A=1, B=0;
    if(z=2) A=.5, B=0;
    if(z=3) A=2, B=0;
  }
}

////////////////////////////////////

void loop()
{
  int livepressure=givepressure();
  // select port
  if (digitalRead(!switchpin))
  {++port;
  delay(150);
  if(port>maxcol) port=1;
  ledwrite(port,0);
  }
}

//

```

```

if(digitalRead(!gopin))
{data[0][column]=port;
digitalWrite(busypin,HIGH);
// write pressure sensor data to array
for(int k=1;k<=maxdata;k++)
{data[k][column]=givepressure();
delay(50);
}
++column;

if(column=maxcol) // write the array to SD card and reset column
{fileRITE(data);
column=0;
for(int v=1;v<=6;v++) //flash busypin to indicate data was written to SD
{digitalWrite(busypin,LOW);
delay(100);
digitalWrite(busypin,HIGH);
delay(100);
}
}
digitalWrite(busypin,LOW);
}

////////////////////////////////////
void ledwrite(int x, int dig) //output number x to 7segment LED display "dig"
{
if(x>11 || x<0) return;
for(int k=1;k<9;k++) // start at 0 if want to include the dot
{digitalWrite(segmentpin[dig][k],segmentsONOFF[x][k]);
}
}

////////////////////////////////////
int givepressure()
{
int rawsensor=analogRead(sensorpin);
// now convert to MPa*100 integer

```

```
int p=(rawsensor-B)*A;
//int p=rawsensor;
int h=p/100;
int t=(p-(100*h))/10;
int o=p-h*100-t*10;
ledwrite(h,1);
ledwrite(t,2);
ledwrite(o,3);
}
```


Appendix D

Flow Experiments Data

The data collected during the experiments performed on the various porous materials discussed in chapter 3 is given below.

In this experiment, the leaked water was weighed at the given time intervals without taring the scale each time. Therefore columns 3 and 2 are subtracted to yield the total leakage in that time span.

Table D.1: Polyurethane (dark gray) open cell foam of 1/16" (1.59 mm) thickness, 20 lb/ft³ density.

Pressure (MPa)	Weight Start (g)	Weight End (g)	Time (min)	Flow Rate (g/min)
1.30	294.50	301.90	2.00	3.70
1.40	307.00	315.00	2.00	4.00
1.52	319.60	328.50	2.00	4.45
1.60	333.70	345.50	2.50	4.72
1.71	353.70	363.80	2.00	5.05
1.80	368.70	378.80	2.00	5.05
1.90	388.70	403.00	2.50	5.72
2.00	408.50	421.10	2.00	6.30
2.10	426.50	440.10	2.00	6.80
2.20	451.00	468.80	2.50	7.12
2.30	481.40	496.10	2.00	7.35
2.40	507.00	522.50	2.00	7.75
2.50	536.00	552.50	2.00	8.25

In the following three experiments, the scale was tared each time the clock was reset, and the "Leak" column represents the total water leaked in that time span.

Table D.2: Silicone open cell foam of 1/8" (3.18 mm) thickness, 12 lb/ft³ density.

Pressure (MPa)	Leak (g)	Time (min)	Flow Rate (g/min)
0.20	184.40	3.00	61.47
0.30	189.90	3.00	63.30
0.40	201.10	3.00	67.03
0.50	214.50	3.00	71.50
0.60	220.90	3.00	73.63
0.70	235.40	3.00	78.47
0.80	255.60	3.00	85.20
0.90	294.40	3.00	98.13

Table D.3: Natural Gum rubber open cell foam of 1/8" (3.18 mm) thickness, 30 lb/ft³ density.

Pressure (MPa)	Leak (g)	Time (min)	Flow Rate (g/min)
0.50	186.20	4.00	2.53
0.65	193.10	5.00	3.40
0.80	198.30	5.00	4.44
0.95	204.20	5.00	5.62
1.10	213.60	5.00	7.50
1.30	231.80	5.00	11.14

Table D.4: Polyurethane (white) open cell foam of 1/4" (6.35 mm) thickness, 1.8 lb/ft³ density.

Pressure (MPa)	Leak (g)	Time (min)	Flow Rate (g/min)
0.15	13.50	1.00	13.50
0.22	17.40	1.00	17.40
0.28	21.70	1.00	21.70
0.33	24.80	1.00	24.80
0.40	29.00	1.00	29.00
0.46	31.60	1.00	31.60
0.53	35.50	1.00	35.50
0.60	41.40	1.00	41.40
0.66	44.00	1.00	44.00

Appendix E

Patent Application

The dynamic seal ideas described in this thesis were disclosed to the University of Washington Center for Commercialization and subsequently a patent was filed to protect the intellectual property. The patent application in its entirety is attached below.

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference
(if desired) (12 characters maximum) UWOTL143613

Box No. I TITLE OF INVENTION	
DYNAMIC SEAL TO ENABLE MOVEMENT OF A FILM OR FIBER THROUGH A PRESSURIZED SPACE WHILE MAINTAINING A DESIRED PRESSURE	
Box No. II APPLICANT <input type="checkbox"/> This person is also inventor	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.) UNIVERSITY OF WASHINGTON THROUGH ITS CENTER FOR COMMERCIALIZATION 4311 11th Avenue Northeast, Suite 500 Seattle, Washington 98105-4608 US	Telephone No. n/a
	Facsimile No. n/a
	Applicant's registration No. with the Office n/a
E-mail authorization: Marking one of the check-boxes below authorizes the receiving Office, the International Searching Authority, the International Bureau and the International Preliminary Examining Authority to use the e-mail address indicated in this Box to send, notifications issued in respect of this international application to that e-mail address if those offices are willing to do so. <input type="checkbox"/> as advance copies followed by paper notifications; or <input type="checkbox"/> exclusively in electronic form (no paper notifications will be sent). E-mail address:	
State (that is, country) of nationality: US	State (that is, country) of residence: US
This person is applicant for the purposes of: <input checked="" type="checkbox"/> all designated States <input type="checkbox"/> the States indicated in the Supplemental Box	
Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)	
<input checked="" type="checkbox"/> Further applicants and/or (further) inventors are indicated on a continuation sheet.	
Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE	
The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: <input checked="" type="checkbox"/> agent <input type="checkbox"/> common representative	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Laura A. Cruz Christensen O'Connor Johnson Kindness PLLC 1420 Fifth Avenue, Suite 2800 Seattle, Washington 98101 US	Telephone No. 206-682-8100
	Facsimile No. 206-224-0779
	Agent's registration No. with the Office 46,649
E-mail authorization: Marking one of the check-boxes below authorizes the receiving Office, the International Searching Authority, the International Bureau and the International Preliminary Examining Authority to use the e-mail address indicated in this Box to send, notifications issued in respect of this international application to that e-mail address if those offices are willing to do so. <input type="checkbox"/> as advance copies followed by paper notifications; or <input checked="" type="checkbox"/> exclusively in electronic form (no paper notifications will be sent). E-mail address: patents@cojk.com	
<input type="checkbox"/> Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.	

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

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Applicant's registration No. with the Office	

State <i>(that is, country)</i> of nationality: US	State <i>(that is, country)</i> of residence: US
--	--

This person is applicant for the purposes of: <input type="checkbox"/> all designated States	<input type="checkbox"/> the States indicated in the Supplemental Box
--	---

Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> NICOLAE, Andrei c/o UW Center for Commercialization 4311 11th Avenue Northeast, Suite 500 Seattle, Washington 98105-4608 US	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input checked="" type="checkbox"/> inventor only <i>(If this check-box is marked, do not fill in below.)</i>
Applicant's registration No. with the Office	

State <i>(that is, country)</i> of nationality: US	State <i>(that is, country)</i> of residence: US
--	--

This person is applicant for the purposes of: <input type="checkbox"/> all designated States	<input type="checkbox"/> the States indicated in the Supplemental Box
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Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> (This section is currently blank)	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only <i>(If this check-box is marked, do not fill in below.)</i>
Applicant's registration No. with the Office	

State <i>(that is, country)</i> of nationality:	State <i>(that is, country)</i> of residence:
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This person is applicant for the purposes of: <input type="checkbox"/> all designated States	<input type="checkbox"/> the States indicated in the Supplemental Box
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Name and address: <i>(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</i> (This section is currently blank)	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only <i>(If this check-box is marked, do not fill in below.)</i>
Applicant's registration No. with the Office	

State <i>(that is, country)</i> of nationality:	State <i>(that is, country)</i> of residence:
---	---

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<input type="checkbox"/> Further applicants and/or (further) inventors are indicated on another continuation sheet.

Box No. V DESIGNATIONS				
<p>The filing of this request constitutes under Rule 4.9(a) the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.</p> <p>However,</p> <p><input type="checkbox"/> DE Germany is not designated for any kind of national protection</p> <p><input type="checkbox"/> JP Japan is not designated for any kind of national protection</p> <p><input type="checkbox"/> KR Republic of Korea is not designated for any kind of national protection</p> <p><i>(The check-boxes above may only be used to exclude (irrevocably) the designations concerned if, at the time of filing or subsequently under Rule 26bis.1, the international application contains in Box No. VI a priority claim to an earlier national application filed in the particular State concerned, in order to avoid the ceasing of the effect, under the national law, of this earlier national application.)</i></p>				
Box No. VI PRIORITY CLAIM AND DOCUMENT				
The priority of the following earlier application(s) is hereby claimed:				
Filing date of earlier application <i>(day/month/year)</i>	Number of earlier application	Where earlier application is:		
		national application: country or Member of WTO	regional application: regional Office	international application: receiving Office
item (1) 27 March 2013 (27.03.2013)	61/805,581	US		
item (2)				
item (3)				
<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.				
Furnishing the priority document(s):				
<input checked="" type="checkbox"/> The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) <i>(only if the earlier application(s) was filed with the receiving Office which, for the purposes of this international application, is the receiving Office)</i> identified above as:				
<input checked="" type="checkbox"/> all items <input type="checkbox"/> item (1) <input type="checkbox"/> item (2) <input type="checkbox"/> item (3) <input type="checkbox"/> other, see Supplemental Box				
<input type="checkbox"/> The International Bureau is requested to obtain from a digital library a certified copy of the earlier application(s) identified above, using, where applicable, the access code(s) indicated below <i>(if the earlier application(s) is available to it from a digital library)</i> :				
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Incorporation by reference: where an element of the international application referred to in Article 11(1)(iii)(d) or (e) or a part of the description, claims or drawings referred to in Rule 20.5(a) is not otherwise contained in this international application but is completely contained in an earlier application whose priority is claimed on the date on which one or more elements referred to in Article 11(1)(iii) were first received by the receiving Office, that element or part is, subject to confirmation under Rule 20.6, incorporated by reference in this international application for the purposes of Rule 20.6.				
Box No. VII INTERNATIONAL SEARCHING AUTHORITY				
Choice of International Searching Authority (ISA) <i>(if more than one International Searching Authority is competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):</i>				
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Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51 bis.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

in relation to this international application,

University of Washington through its Center for Commercialization is entitled to apply for and be granted a patent by virtue of an assignment of USSN 61/805,581 filed March 27, 2013 from Vipin Kumar and Andrei Nicolae to University of Washington through its Center for Commercialization dated February 5, 2014 and February 6, 2014, respectively.

This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".

Box No. VIII (iii) DECLARATION: ENTITLEMENT TO CLAIM PRIORITY

The declaration must conform to the standardized wording provided for in Section 213; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application specified below, where the applicant is not the applicant who filed the earlier application or where the applicant's name has changed since the filing of the earlier application (Rules 4.17(iii) and 51bis.1(a)(iii)):

in relation to this international application,

University of Washington through its Center for Commercialization is entitled to claim priority of earlier application USSN 61/805,581 filed March 27, 2013 by virtue of an assignment from Vipin Kumar, Andrei Nicolae to University of Washington through its Center for Commercialization dated February 5, 2014 and February 6, 2014, respectively.

This declaration is continued on the following sheet, "Continuation of Box No. VIII (iii)".

Box No. IX CHECK LIST for EFS-Web filings - this sheet is only to be used when filing an international application with RO/US via EFS-Web

This international application contains the following:	Number of sheets	This international application is accompanied by the following item(s) (<i>mark the applicable check-boxes below and indicate in right column the number of each item</i>):	Number of items
(a) request form PCT/RO/101 (including any declarations and supplemental sheets) :	6	1. <input checked="" type="checkbox"/> fee calculation sheet :	1
(b) description (excluding any sequence listing part of the description, see (f), below) :	24	2. <input type="checkbox"/> original separate power of attorney :	
(c) claims :	4	3. <input type="checkbox"/> original general power of attorney :	
(d) abstract :	1	4. <input type="checkbox"/> copy of general power of attorney; reference number: :	
(e) drawings (if any) <u>Figs. 1-18</u> :	10	5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s) :	
(f) sequence listing part of the description in the form of an image file (e.g. PDF) :		6. <input type="checkbox"/> Translation of international application into (<i>language</i>): :	
Total number of sheets (including the sequence listing part of the description if filed as an image file) :	45	7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material :	
(g) sequence listing part of the description		8. <input type="checkbox"/> (<i>only where item (f) is marked in the left column</i>) copy of the sequence listing in electronic form (Annex C/ST.25 text file) not forming part of the international application but furnished only for the purposes of international search under Rule 13ter. :	
<input type="checkbox"/> filed in the form of an Annex C/ST.25 text file		9. <input type="checkbox"/> (<i>only where item (f) is marked in the left column</i>) a statement confirming that "the information recorded in electronic form submitted under Rule 13ter is identical to the sequence listing as contained in the international application" as filed via EFS-Web: :	
<input type="checkbox"/> WILL BE filed separately on physical data carrier(s), on the same day and in the form of an Annex C/ST.25 text file		10. <input type="checkbox"/> copy of results of earlier search(es) (Rule 12bis.1(a)) :	
Indicate type and number of physical data carrier(s) :		11. <input type="checkbox"/> other (<i>specify</i>): :	

Figure of the drawings which should accompany the abstract: 2 **Language of filing** of the international application: English

Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

/Laura A. Cruz, Reg. No. 46,649/

 Laura A. Cruz, Reg. No. 46,649

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3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:	
4. Date of timely receipt of the required corrections under PCT Article 11(2):	
5. International Searching Authority (if two or more are competent): <u>ISA /</u>	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid

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Applicant
UW THROUGH ITS CENTER FOR COMMERCIALIZATION

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE 120 T

2. SEARCH FEE 1040 S

International search to be carried out by ISA/US
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Date: March 26, 2014

Name: Laura A. Cruz, Reg. No. #46,649

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Electronic Patent Application Fee Transmittal

Application Number:	
Filing Date:	
Title of Invention:	DYNAMIC SEAL TO ENABLE MOVEMENT OF A FILM OR FIBER THROUGH A PRESSURIZED SPACE WHILE MAINTAINING A DESIRED PRESSURE
First Named Inventor/Applicant Name:	UNIVERSITY OF WASHINGTON THROUGH ITS CENTER
Filer:	Laura Cruz/Vanessa Knowles
Attorney Docket Number:	UWOTL143613

Filed as Small Entity

International Application for filing in the US receiving office Filing Fees

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Basic Filing:				
Transmittal Fee	2601	1	120	120
PCT Search Fee- no prior US appl filed	2602	1	1040	1040
Intl Filing Fee (1st-30 Pgs.) PCT Easy	1701	1	1360	1360
Suppl. Intl Filing Fee (each page > 30)	1703	15	17	255

Pages:

Claims:

Miscellaneous-Filing:

Petition:

Description	Fee Code	Quantity	Amount	Sub-Total in USD(\$)
Patent-Appeals-and-Interference:				
Post-Allowance-and-Post-Issuance:				
Extension-of-Time:				
Miscellaneous:				
Total in USD (\$)				2775

Electronic Acknowledgement Receipt

EFS ID:	18593722
Application Number:	
International Application Number:	PCT/US14/31906
Confirmation Number:	8158
Title of Invention:	DYNAMIC SEAL TO ENABLE MOVEMENT OF A FILM OR FIBER THROUGH A PRESSURIZED SPACE WHILE MAINTAINING A DESIRED PRESSURE
First Named Inventor/Applicant Name:	UNIVERSITY OF WASHINGTON THROUGH ITS CENTER
Customer Number:	95093
Correspondence Address:	Laura A. Cruz Christensen O'Connor Johnson Kindness PLLC 1420 Fifth Avenue Suite2800 Seattle WA 98101 US 206.682.8100 patents@cojk.com
Filer:	Laura Cruz/Vanessa Knowles
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Attorney Docket Number:	UWOTL143613
Receipt Date:	26-MAR-2014
Filing Date:	
Time Stamp:	18:39:02
Application Type:	International Application for filing in the US receiving office
Patent Number:	

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Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1		43613PCT_Specification_Claims_Abstract_drawings.pdf	455494 dc2f0a8228d5f86bc93d7343966b99e0fc8a66e9	yes	39

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Document Description	Start	End
Specification	1	24
Claims	25	28
Abstract	29	29
Drawings-only black and white line drawings	30	39

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2	RO/101 - Request form for new IA - Conventional	UW43613_Request_FINAL.pdf	85769 02362503494ed8a0d25d40003e8db6dded18c4be	no	7
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3	Fee Worksheet (SB06)	fee-info.pdf	36803 bc33115aee8aa016488f765cc19fde93a58857e3	no	2
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DYNAMIC SEAL TO ENABLE MOVEMENT OF A FILM OR FIBER THROUGH A
PRESSURIZED SPACE WHILE MAINTAINING A DESIRED PRESSURE

CROSS-REFERENCE TO RELATED APPLICATION

5 This application claims the benefit of U.S. Provisional Application
No. 61/805,581, filed on March 27, 2013.

BACKGROUND

10 Thermoplastic polymers are capable of being formed into cellular materials by a
process generally known as solid-state foaming. Unlike the use of an extruder, wherein a
thermoplastic polymer is melted to a liquid state into which a blowing agent is injected,
the solid-state process does not melt the thermoplastic polymer via an extruder to inject
the blowing agent, but instead, the thermoplastic polymer remains solidified during the
process of absorbing a gas. Thereafter, the gas-saturated solid-state polymer is converted
into a foam by raising the temperature. A solid-state foaming process is illustrated in
15 FIGURE 1.

The solid-state foaming process illustrated in FIGURE 1, requires a solid-state
thermoplastic polymer that has been saturated with gas. The foaming occurs while the
polymer remains in the solid state either by a rapid decrease in pressure or through the
application of heat. This process differs from extrusion foaming processes because the
20 polymer is not required to be in a molten state when the gas is injected. Generally, at the
beginning of the solid-state foaming method, block 100, the polymer is in equilibrium
with the surrounding temperature and pressure so that the polymer is unsaturated. In
block 102, the thermoplastic polymer is treated at an elevated pressure with an inert gas
to cause the thermoplastic polymer to absorb the gas. Suitable gases may include
25 non-reacting gases, such as carbon dioxide or nitrogen. The treatment of the polymer in
block 102 may be carried out by placing the polymer in a pressure vessel which is sealed,
and then the polymer is exposed to the inert gas at a high pressure. The highly
pressurized gas will start to diffuse into the thermoplastic polymer over time, filling the
polymer's free intermolecular volume. The gas will continue to saturate the polymer until
30 a suitable gas concentration is reached or until equilibrium is reached. In block 104, the
fully saturated or partially saturated polymer is removed from the saturation pressure
vessel to an environment of lower pressure so that the polymer becomes
thermodynamically unstable, meaning that the polymer is supersaturated with gas that is

no longer at equilibrium with the surrounding environment. The polymer then desorbs gas from its surface into the surrounding environment. Desorption of some of the gas from the polymer's exterior surfaces will lead to, upon subsequent heating, the creation of a non-cellular skin at the exterior surfaces due to the lack of gas. Heating of the gas-saturated polymer in block 106 is carried out at a temperature below the melting temperature of the polymer. At a certain temperature, nucleation sites appear in the polymer-gas matrix, and further gas diffusion from the polymer-gas solution surrounding the sites causes the sites to form cells.

SUMMARY

10 This summary is provided to introduce a selection of concepts in a simplified form that are further described below in the Detailed Description. This summary is not intended to identify key features of the claimed subject matter, nor is it intended to be used as an aid in determining the scope of the claimed subject matter.

15 Some embodiments are related to a method of withdrawing a material from a pressurized vessel, wherein the method includes withdrawing a material from a gas-pressurized, liquid-filled vessel from a seal below a liquid level; applying a force with the seal on the material to limit a flow of liquid from the seal while creating a layer of liquid interposed between the material and the seal; and controlling a pressure within the vessel with a gas.

20 In some embodiments, the seal includes a first and second plate disposed generally parallel to each other, a first and second seal material juxtaposed on the interior sides of the first and second plates, and a film material is passed between the first and second seal materials while a liquid layer lubricates the sides of the film against the seal materials.

25 In some embodiments, the seal includes a chamber having a first larger diameter attached to the vessel, a second smaller diameter, and a transition that connects the larger diameter of the chamber to the smaller diameter of the chamber, and a seal material is placed inside the chamber, wherein the seal material is compressed against an inside wall of the transition, and a fiber material is passed within the seal material, while a liquid layer lubricates the sides of the fiber against the seal material.

30 In some or all embodiments, the gas can be carbon dioxide or nitrogen.

In some or all embodiment, the liquid can be water.

In some or all embodiments, the material can be a film.

In some or all embodiments, the material can be a gas-saturated film.

In some or all embodiments, the material can be a fiber.

In some or all embodiments, the material can be a gas-saturated fiber.

In some or all embodiments, the seal can extend axially.

5 In some or all embodiments, the seal material can be deformed to conform around the material to limit the flow of liquid out of the seal.

In some or all embodiments, the seal material can be polyurethane.

In some or all embodiments, the seal material can be permeable.

In some or all embodiments, the seal material can be non-permeable.

10 In some or all embodiments, the first and second plates can be parallel with respect to each other.

Some embodiments are related to a method of making a foam, wherein the method includes saturating a solid thermoplastic polymer with a gas within a gas-pressurized, liquid-filled vessel to produce a gas-saturated thermoplastic polymer; withdrawing the gas-saturated thermoplastic polymer from the vessel through a seal below a level of the liquid; applying a force with the seal on the gas-saturated thermoplastic polymer to limit a flow of liquid from the seal while creating a layer of liquid interposed between the gas-saturated material and the seal; and heating the gas-saturated thermoplastic polymer to a temperature to create cells without melting the thermoplastic polymer.

20

In some or all embodiments, the seal can be compressed around the gas-saturated thermoplastic polymer.

In some or all embodiments, the thermoplastic polymer can be a film or a fiber.

Some embodiments are related to a seal, wherein the seal includes a first structural member; a second structural member, wherein a first surface of the first structural member faces a second surface of the second structural member; a first seal material is juxtaposed on the first surface; a second seal material is juxtaposed on the second surface, wherein the first and second structural members are configured to be compressed toward one another to compress the first and second seal materials toward one another; and a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure between the surfaces of the first and second seal materials.

30

In some or all embodiments, the first and second structural members can be parallel.

In some or all embodiments, the first and second seal materials can be non-permeable polymeric materials.

In some or all embodiments, the first and second seal materials can be permeable polymeric materials.

5 In some or all embodiments, the first and second seal materials can include alternating layers of permeable and non-permeable polymeric materials aligned axially.

In some or all embodiments, the first and second seal materials can include channels on surfaces that face one another.

10 In some or all embodiments, the first and second seal materials can define an aperture into the vessel.

In some or all embodiments, a cavity can be provided adjacent to each of the first and second seal materials, and each cavity is filled with pressurized liquid that applies a constant pressure along one side of each of the seal materials.

15 Some embodiments are related to a seal, wherein the seal includes a structural member having a wedge-shaped chamber, wherein the chamber has a larger diameter that tapers toward a smaller diameter; a seal material within the chamber that assumes the shape of the wedge-shaped chamber, wherein the seal material includes a hole axially traversing the seal material; and a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure on the seal material and compresses the seal material in the wedge-shaped chamber.

In some embodiments, the seal may include a cavity provided between the structural member and the seal material, wherein the cavity is filled with pressurized liquid that applies a constant pressure along one side of the seal material.

25 DESCRIPTION OF THE DRAWINGS

The foregoing aspects and many of the attendant advantages of this invention will become more readily appreciated as the same become better understood by reference to the following detailed description, when taken in conjunction with the accompanying drawings, wherein:

30 FIGURE 1 is a flow diagram of a method according to a known solid-state foaming process;

FIGURE 2 is a flow diagram of a method according to one embodiment of the invention;

FIGURE 3 is a diagrammatical illustration of a pressure vessel and seal according to one embodiment of the invention;

FIGURE 4 is a diagrammatical illustration of a frontal cross section of a seal when the polymer is a film or sheet;

5 FIGURE 5 is a diagrammatical illustration of a frontal cross section of a seal when the polymer is a fiber;

FIGURE 6 is a diagrammatical illustration of a wedge-shaped seal according to one embodiment of the invention;

10 FIGURE 7A is a diagrammatical illustration of a seal material according to one embodiment of the invention;

FIGURE 7B is a diagrammatical illustration of the seal material of FIGURE 7A;

FIGURE 8 is a diagrammatical illustration of a cross section of seal according to one embodiment of the invention;

15 FIGURE 9 is a diagrammatical illustration of a wedge-shaped seal according to one embodiment of the invention;

FIGURE 10 is a diagrammatical illustration of a seal material according to one embodiment of the invention;

FIGURE 11 is a diagrammatical illustration of a seal material according to one embodiment of the invention;

20 FIGURE 12 is diagrammatical illustration of a pressure vessel, seal, and heater according to one embodiment of the invention;

FIGURE 13 is a diagrammatical illustration of a pressure vessel, seal, and heater according to one embodiment of the invention;

25 FIGURE 14 is a diagrammatical illustration of a pressure vessel and seal according to one embodiment of the invention;

FIGURE 15 is a diagrammatical illustration of pressure distribution along a seal material;

FIGURE 16 is a diagrammatical illustration of a pressure vessel and seal according to one embodiment of the invention;

30 FIGURE 17 is a graph depicting a pressure profile of a seal according to one embodiment of the invention; and

FIGURE 18 is a graph depicting a pressure profile of a seal according to one embodiment of the invention,

DETAILED DESCRIPTION

A problem of solid-state foaming arises when the material to be foamed is thin. The time after a thin gas-saturated polymer is removed from the pressure vessel and before being heated causes the thin material to lose enough gas that renders foaming unsuitable. Accordingly, thin materials, such as films, sheets, or fibers, tend to be difficult to foam using a solid-state foaming process. To address the foregoing problem, a method and apparatus is disclosed that will allow a thin gas-saturated material to be withdrawn from a pressure vessel in a manner that lessens the amount of time the gas-saturated material is subjected to ambient pressure before heating.

Part of the problem may be due to the fact that in a conventional process, the material, such as a roll of film, is placed into a pressure vessel, and the entire roll of material is removed from the pressure vessel all at once. Therefore, while the initial material coming off the roll may have a suitable gas concentration, by the time the end or middle of the roll is reached, the gas concentration may be unsuitable for foaming. The disclosed method and apparatus allows the passing of a gas-saturated material into and/or out of a gas-pressurized, liquid-filled vessel so as to avoid disparities in gas concentration throughout the length of the material, and to allow for the immediate heating of the gas-saturated material into a foam. A particular seal is disclosed that allows the passing of a material into or out of the pressure vessel, without the pressure vessel seeing a significant amount of pressure loss via the seal, either through the loss of gas or liquid. A significant loss is one that is not possible to replenish with make-up liquid or gas.

The particulars shown herein are by way of example and for purposes of illustrative discussion of the preferred embodiments of the present invention only and are presented in the cause of providing what is believed to be the most useful and readily understood description of the principles and conceptual aspects of various embodiments of the invention. In this regard, no attempt is made to show structural details of the invention in more detail than is necessary for the fundamental understanding of the invention, the description taken with the drawings, and/or examples, making apparent to those skilled in the art how the several forms of the invention may be embodied in practice.

The following definitions and explanations are meant and intended to be controlling in any future construction unless clearly and unambiguously modified in the following examples or when application of the meaning renders any construction

meaningless or essentially meaningless. In cases where the construction of the term would render it meaningless or essentially meaningless, the definition should be taken from Webster's Dictionary, 3rd Edition, or a dictionary known to those of skill in the art, such as the Oxford Dictionary of Biochemistry and Molecular Biology (Anthony Smith
5 (ed.), Oxford University Press, Oxford, 2004).

As used herein and unless otherwise indicated, the terms "a" and "an" are taken to mean "one," "at least one," or "one or more." Unless otherwise required by context, singular terms used herein shall include pluralities, and plural terms shall include the singular.

10 Unless the context clearly requires otherwise, throughout the description and the claims, the words "comprise," "comprising," and the like are to be construed in an inclusive sense as opposed to an exclusive or exhaustive sense; that is to say, in the sense of "including, but not limited to." Words using the singular or plural number also include the plural and singular number, respectively. Additionally, the words "herein," "above,"
15 and "below" and words of similar import, when used in this application, shall refer to this application as a whole and not to any particular portions of the application.

The description of embodiments of the disclosure is not intended to be exhaustive or to limit the disclosure to the precise form disclosed. While the specific embodiments of, and examples for, the disclosure are described herein for illustrative purposes, various
20 equivalent modifications are possible within the scope of the disclosure, as those skilled in the relevant art will recognize.

All of the references cited herein are incorporated by reference. Aspects of the disclosure can be modified, if necessary, to employ the systems, functions, and concepts of the above references and application to provide yet further embodiments of the
25 disclosure. These and other changes can be made to the disclosure in light of the detailed description.

Specific elements of any foregoing embodiments can be combined or substituted for elements in other embodiments. Furthermore, while advantages associated with certain embodiments of the disclosure have been described in the context of these
30 embodiments, other embodiments may also exhibit such advantages, and not all embodiments need necessarily exhibit such advantages to fall within the scope of the disclosure.

The present disclosure describes seals capable of continually and controllably passing a polymer film or fiber through a pressurized vessel, while maintaining a desired pressure inside the vessel and a desired liquid level. In certain embodiments, the seals are capable of limiting the flow of gas and/or liquid from the pressurized vessel to lower pressure environments.

Normally, if an opening is created in a pressurized vessel, the pressurized gas or air or other liquid will leak out. Disclosed is a sealing mechanism that allows for a film or a fiber to freely pass through an inlet and/or an outlet in the vessel while keeping the desired pressure inside the vessel.

The disclosed method and dynamic seal may be used to continuously process micro-cellular and nano-cellular thin films and fibers. For examples of other methods of processing micro- and nano-cellular films and fibers, see U.S. Patent Nos. 5,684,055; 7,807,260; 8,247,464; 8,168,114; 7,923,104; 8,241,741; and 8,357,319, which are incorporated herein by reference in their entirety. Since thin films quickly lose the absorbed gas when removed from a pressure vessel, the processing window is very small. Thus, if the film or fiber can be processed before gas desorbs, an industrially viable process can be envisioned for micro-cellular and nano-cellular films and fibers. A method for continuously feeding a gas-saturated thin polymer to a heater for foaming is disclosed.

In some embodiments, seals described herein use a liquid to provide a sealing function while the polymer film or fiber is moving through the seal. In some embodiments, the film or fiber are gas-saturated when exiting the vessel, however, in some embodiments, the film or fiber need not be gas-saturated within the vessel.

The short processing window has prevented the commercialization of the solid-state micro-cellular and nano-cellular processing of thin films and fibers. A solution to this problem is to continuously pass a polymer film or fiber through a pressurized environment and process it shortly after it exits the pressure vessel so that the gas has insufficient time to desorb out of the polymer.

Towards this end, devices are disclosed which are capable of continuously passing polymer films or fibers through a pressurized environment to continuously provide gas-saturated polymer films and fibers. In this way, not all of the gas-saturated fiber or film is exposed to atmospheric pressure. Instead the gas-saturated thin film or fiber is continuously fed to a heater, which allows the film or fiber to remain gas-saturated until it

passes from the pressure vessel directly to the heater without an intermittent waiting period.

In certain embodiments, the devices of the present disclosure include,

a pressurized vessel containing a pressurized gas and a liquid;

5 an inlet in the pressurized vessel capable of continuously accepting a polymer film;

an outlet in the pressurized vessel capable of continuously allowing the polymer films to exit the pressurized vessel while maintaining gas pressure in the pressurized vessel,

10 wherein the outlet is a seal comprising:

a gas-tight aperture configured to securely conform to the shape of the polymer film or fiber.

In certain embodiments, the outlet is made of a sponge which contains a slit or hole through which the polymer film or fiber is passed.

15 In certain embodiments, the liquid contained in the pressurized vessel is selected from at least one of water, aqueous solutions, and oils. In certain embodiments, the liquid contains a combination of two or more liquids. In certain further embodiments, the liquid is a silicon oil.

20 In certain embodiments, the inlet in the pressurized vessel also comprises a gas-tight aperture configured to securely conform to the shape of the polymer film or fiber.

In certain embodiments, the gas in the pressurized container is selected from CO, CO₂, O₂, N₂, He, Ne, Ar, H₂, NH₃, NO₂, CH₄, C₂H₆, C₃H₈, C₄H₁₀, and the like.

25 In certain embodiments, the level of the outlet is below the level of the liquid in the pressurized vessel such that the polymer film or fiber exits the pressurized vessel immersed in the liquid.

30 Some embodiments are related to a method of withdrawing a material from a pressurized vessel, wherein the method includes withdrawing a material from a gas-pressurized, liquid-filled vessel from a seal below a liquid level; applying a force with the seal on the material to limit a flow of liquid from the seal while creating a layer of liquid interposed between the material and the seal; and controlling a pressure within the vessel with a gas.

In some embodiments, the seal includes a first and second plate disposed generally parallel to each other, a first and second seal material juxtaposed on the interior sides of the first and second plates, and a film material is passed between the first and second seal materials while a liquid layer lubricates the sides of the film against the seal materials.

In some embodiments, the seal includes a chamber having a first larger diameter attached to the vessel, a second smaller diameter, and a transition that connects the larger diameter of the chamber to the smaller diameter of the chamber, and a seal material is placed inside the chamber, wherein the seal material is compressed against an inside wall of the transition, and a fiber material is passed within the seal material, while a liquid layer lubricates the sides of the fiber against the seal material.

In some or all embodiments, the gas can be carbon dioxide or nitrogen.

In some or all embodiment, the liquid can be water.

In some or all embodiments, the material can be a film.

In some or all embodiments, the material can be a gas-saturated film.

In some or all embodiments, the material can be a fiber.

In some or all embodiments, the material can be a gas-saturated fiber.

In some or all embodiments, the seal can extend axially.

In some or all embodiments, the seal material can be deformed to conform around the material to limit the flow of liquid out of the seal.

In some or all embodiments, the seal material can be polyurethane.

In some or all embodiments, the seal material can be permeable.

In some or all embodiments, the seal material can be non-permeable.

In some or all embodiments, the first and second plates can be parallel with respect to each other.

Some embodiments are related to a method of making a foam, wherein the method includes saturating a solid thermoplastic polymer with a gas within a gas-pressurized, liquid-filled vessel to produce a gas-saturated thermoplastic polymer; withdrawing the gas-saturated thermoplastic polymer from the vessel through a seal below a level of the liquid; applying a force with the seal on the gas-saturated thermoplastic polymer to limit a flow of liquid from the seal while creating a layer of liquid interposed between the gas-saturated material and the seal; and heating the gas-

saturated thermoplastic polymer to a temperature to create cells without melting the thermoplastic polymer.

In some or all embodiments, the seal can be compressed around the gas-saturated thermoplastic polymer.

5 In some or all embodiments, the thermoplastic polymer can be a film or a fiber.

Some embodiments are related to a seal, wherein the seal includes a first structural member; a second structural member, wherein a first surface of the first structural member faces a second surface of the second structural member; a first seal material is juxtaposed on the first surface; a second seal material is juxtaposed on the second surface,
10 wherein the first and second structural members are configured to be compressed toward one another to compress the first and second seal materials toward one another; and a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure between the surfaces of the first and second seal materials.

In some or all embodiments, the first and second structural members can be
15 parallel.

In some or all embodiments, the first and second seal materials can be non-permeable polymeric materials.

In some or all embodiments, the first and second seal materials can be permeable polymeric materials.

20 In some or all embodiments, the first and second seal materials can include alternating layers of permeable and non-permeable polymeric materials aligned axially.

In some or all embodiments, the first and second seal materials can include channels on surfaces that face one another.

In some or all embodiments, the first and second seal materials can define an
25 aperture into the vessel.

In some or all embodiments, a cavity can be provided adjacent to each of the first and second seal materials, and each cavity is filled with pressurized liquid that applies a constant pressure along one side of each of the seal materials.

30 Some embodiments are related to a seal, wherein the seal includes a structural member having a wedge-shaped chamber, wherein the chamber has a larger diameter that tapers toward a smaller diameter; a seal material within the chamber that assumes the shape of the wedge-shaped chamber, wherein the seal material includes a hole axially traversing the seal material; and a vessel connected to the seal, wherein the vessel

contains a liquid under pressure, wherein the liquid exerts pressure on the seal material and compresses the seal material in the wedge-shaped chamber.

In some embodiments, the seal may include a cavity provided between the structural member and the seal material, wherein the cavity is filled with pressurized liquid that applies a constant pressure along one side of the seal material.

While gas selection depends entirely on the process and its purpose, the seal design is indifferent to the gas chosen. In the context of making micro-cellular and nano-cellular foams, the gas is absorbed in the polymer until a certain gas concentration is achieved. The size of the pressure vessel is adjusted in this case to allow sufficient residence time for the polymer sheet, film, or fiber to absorb the desired amount of gas.

Small amounts of liquid will typically leak between the polymer film or fiber and the sealing material. The formation of a liquid film or layer juxtaposed next to the film or fiber on both sides is a desirable effect since the liquid layer will create a pressure gradient along the opening in the axial direction and also aids in lubricating the polymer film or fiber as it exits through the seal. The small amount of liquid leakage can be controlled and pumped back into the high-pressure chamber for reuse.

The working principle of the seal is to create a very high resistance to the high-pressure gas but a very low resistance to the polymer film/fiber.

The seal can have further applications other than the processing of polymer thin films or fiber. Any solid material that is to be continuously removed from a high-pressure environment with minimal leakage could make use of the dynamic seal described in this disclosure.

The choices of sealing material and liquid have an impact on the performance of the seal. Also, the viscosity of the liquid is directly related to the leakage rate, and its lubrication characteristics have an effect on friction between the polymer and the sealing material.

Referring to FIGURE 2, a method is illustrated for passing a material from a pressurized vessel. In block 110, representative solid-state thermoplastic polymer materials include, but, are not limited to thermoplastics, such as polystyrene, high impact polystyrene, polyethylene terephthalate, polycarbonate, polyvinylchloride, polylactic acid. The material can either be a film or fiber; however, the method is not thereby limited. The thermoplastic material is introduced into a gas-pressurized, liquid-filled container via a seal. The thermoplastic material is characterized as a thin material, either

in the thickness dimension if a film or sheet or a small diameter if a fiber. As used herein, "fiber" encompasses any material having a thin diameter. Representative thicknesses of films and diameters of fibers can be in the range of 0.01 mm to 1 mm, including 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45, 0.50, 0.55, 0.60, 0.65, 0.70, 0.75, 0.80, 0.85, 0.90, and 0.95, or any range derived therefrom.

Block 112 is for introducing a solid-state thermoplastic polymer material through an inlet seal into a vessel, while the vessel is already under pressure and has a liquid, the level of which is higher than the entrance of the inlet seal. Block 116 is for removing a solid-state thermoplastic polymer material through an outlet seal from the vessel, while the vessel is under pressure and contains a liquid, the level of which is higher than the outlet of the outlet seal.

For saturating the thermoplastic polymer material, a vessel may have both an inlet and outlet seal. In this case, the polymer material may be continuously introduced into and withdrawn from a gas-pressurized, liquid-filled vessel. The polymer material enters unsaturated through one seal and a gas-saturated polymer material exits through the second seal. The vessel has, on the inside, a retention device that allows the material to reside in the vessel within the gas space (above the liquid) for the time needed to saturate the polymer material. For example, a pressure vessel may include a spool that rolls the incoming unsaturated polymer material simultaneously while paying out gas-saturated polymer material. The amount of time that the polymer material resides on the spool is sufficient to saturate the material at the set gas pressure and temperature.

Alternatively, instead of an inlet and outlet seal, the vessel may have a single outlet seal for withdrawing the gas-saturated polymer material. In this case, the unsaturated polymer material, such as in a roll, can be placed into the liquid-filled vessel, and thereafter, the vessel may be sealed and pressurized once the polymer material is inside. The starting end of the polymer material is fed through the seal, and the seal may be clamped down against the polymer material. The vessel is then sealed and the gas is introduced. When determined to be saturated, the gas-saturated polymer material may be withdrawn from the vessel through the seal, without taking the entire roll out of the pressurized vessel and without having to de-pressurize the vessel.

Block 114 depicts the material being maintained at a pressure to reach a desired gas concentration within the pressure vessel, block 114. As mentioned above, unsaturated polymer material may be continuously fed to the vessel while simultaneously

gas-saturated polymer material is withdrawn, or alternatively, gas-saturated material is withdrawn from the vessel. In either case, the time needed to reach a certain gas concentration can be determined beforehand. For example, various experiments may be performed where the polymer material is tested at a number of different gas pressures and at a number of different time intervals. The temperature can be room temperature. After each experiment, the material may be weighed. The difference in weight between the polymer material after saturation as compared to before saturation is an indication of the gas concentration of the material for the given gas pressure and time. When the polymer material ceases to increase in weight, the material may be considered saturated. A polymer material will reach a certain gas concentration for any given temperature and pressure. The time versus gas concentration for any thermoplastic polymer material can be determined experimentally, or is already well known. Once the time needed for saturation is known, the vessel can be built to provide the appropriate residence time. After the allotted time for gas absorption has passed, the gas-saturated polymer material is withdrawn from the vessel via a seal, block 116.

Additionally, if the gas-saturated material is to be created into a foam, the material may undergo a foaming step, block 118. When the gas-saturated thermoplastic polymer material is withdrawn from the pressure vessel continuously, instead of all at once, the material may have sufficient gas-concentration for foaming, despite the thin nature of the material. Placing a heater in proximity to or directly after the gas-saturated thermoplastic material exits the pressure vessel allows the foaming to occur before the film or fiber gas-saturated material is desorbed of too much gas. It is known that the glass transition temperature of the polymer material is lowered because of the gas absorbed in the polymer material. In order to foam the material, the temperature of the gas-saturated polymer is raised to a point where the cracks appear in the polymer's internal structure and the polymer's molecules move past each other providing nucleation sites where the gas fills in and begins to create cells. The rise in temperature causes the gas to diffuse from the surrounding area into the nucleation site which causes the cells to grow. The foaming process uses a temperature that does not melt the polymer, however. It is known that the gas concentration, heating time, and heating temperature may influence the cell structure. For example, these parameters may be varied to affect whether the cells are micro-sized or nano-sized, whether the cells are closed or open, and for adjusting the density of the resulting foam.

In block 120, once the gas-saturated polymer material is made into a foam, the foam may be shaped, such as via a molding process, in order to create a product, such as, but not limited to, containers, food trays, cups, and the like. Because of the cells contained in the foam, the foams can provide insulating properties to the products.

5 The seals for including either in the inlet, outlet or both of a vessel will be described in more detail with reference to FIGURES. Some embodiments of a seal may include a first structural upper member, a second lower structural member, wherein a first surface of the first structural member faces a second surface of the second structural member, a first seal material juxtaposed on the first surface of the first structural member,
10 a second seal material juxtaposed on the second surface of the second structural member, wherein the first and second structural members are configured to be compressed toward one another to compress the first and second seal materials toward one another; and a vessel is connected to the seal, wherein the vessel contains a liquid under pressure, and the liquid exerts pressure between the surfaces of the first and second seal materials.
15 While the structural members are rigid, non-deforming materials, the seal materials are deformable, and pliable to conform to a cross-sectional shape of either a film or fiber. The one or more seal materials form the opening for the inlet to the pressure vessel and/or the outlet to the pressure vessel.

 Some embodiments of a seal may include a structural member having a wedge-
20 shaped internal chamber, wherein the chamber has a larger diameter that tapers toward a smaller diameter, a seal material within the chamber that assumes the shape of the wedge-shaped chamber, wherein the seal material includes a hole axially traversing the seal material, and a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure on the seal material and compresses the seal
25 material in the wedge-shaped chamber. While the structural member is rigid, non-deforming material, the seal material is deformable, and pliable to conform to a cross-sectional shape of either a film or fiber.

 Referring to FIGURE 3, a diagrammatical illustration of a seal is illustrated. The seal 200 provides an opening through which a material 222, such as a gas-saturated thin
30 polymer, may be passed into or out of the vessel 202 without the loss of significant pressure from the vessel 202 and without significant loss of liquid 208 from the vessel 202. The opening is adjusted to a size around the material to limit the pressure and liquid escaping from the seal. The vessel 202 is designed to withstand the anticipated

pressures for saturating thermoplastic materials described herein. For example, the pressure may be from 1 MPa to 25 MPa, or any narrower range inbetween. The literature contains ranges of pressures suitable for saturating a variety of polymer materials. A non-reacting gas 206, such as nitrogen or carbon dioxide, is used to pressurize the vessel 202. A gas source may be connected to the vessel 202 such that a pressure control valve may open or close depending on the sensed pressure within the vessel 202. The vessel is also filled partially with a liquid 208. The height of the level of the liquid 208 is at least higher than the opening out of the vessel 202. A suitable liquid 208 is water or any aqueous solution compatible with both the gas 206 and the thermoplastic polymer material 222. The liquid should have low solubility in the polymer, and the solubility of the gas is significantly higher in the polymer than the solubility of the liquid in the polymer or in the gas so that the liquid does not interfere with the diffusion of the gas into the polymer. A liquid source with a control valve may be used to maintain the liquid in the vessel 202 based on a level sensor to replenish any liquid that is lost through the seal. Additionally, any liquid that escapes through the seal may be collected and returned to the vessel 202. Significant loss of pressure and liquid is to be avoided that could not be compensated for by the pressure source and the liquid source control valves.

The seal 200 extends axially. Here, axially means a direction along the direction of travel of the thermoplastic polymer material 222. In the illustrated embodiment of FIGURE 3, the seal is constructed from an upper structural plate 204 and a lower structural plate 210 that are connected to the vessel 202 in a pressure-tight manner. The upper structural plate 204 and the lower structural plate 210 can be parallel with respect to each other. Lining the respective upper plate 204 and lower plate 210 is an upper seal material 214 and a lower seal material 212. The upper seal material 214 and lower seal material 212 are juxtaposed and can be attached to the corresponding upper and lower plates 204, 210 so that the upper seal material 214 and the lower seal material 212 face each other. In some embodiments, the seal materials 212 and 214 are compressible to some extent so as to deform and assume the negative of the shape of the material 222 when compressed against the material 222. The upper plate and seal material 204, 214 along with the lower plate 210 and seal material 212 may be compressed toward each other to provide a sealing effect around the material 222. However, the upper plate and upper seal material and the lower plate and lower seal material are not compressed so much that the force to withdraw the material 222 is excessive to cause the material to

break. On the other hand, the compression pressure between the upper plate and upper seal material and the lower plate and lower seal material is not so loose as to allow significant quantities of liquid to be expelled therebetween. Ideally, the pressure in the vessel 202 forces a liquid layer 208 interposed both between the upper side of the material 222 and the upper seal material 214 and between the lower side of the material 222 and the bottom seal material 212 when the material 222 moves axially. While the vessel is pressurized, the pressure of the liquid can drop linearly as a function of the seal material length, so that the pressure of the liquid at the exterior of the seal is at ambient pressure. A linear pressure drop is thought to create a uniform liquid layer that aids in lubrication of the material 222 as it passes through the seal. In order to achieve a linear pressure drop, the upper plate 204 and lower plate 210 and corresponding seal materials 214, 212 have to be essentially parallel to exert a uniform pressure over the surface area of the material 222. However, less than uniform pressure distribution may also create a seal sufficient to allow the material to be easily withdrawn without the loss of pressure and liquid from the seal.

An aperture 213 is created between the upper seal material 214 and the lower seal material 212 through which a polymer material 222 is withdrawn and which allows liquid to leak through. Since the polymer material 222 may include films or fibers, the seal 200 can be shaped accordingly. For example, in FIGURE 4, a seal is shown for the case where the material 222 is a film. As seen in FIGURE 4, the material 222 is surrounded by a layer of liquid 208 directly and immediately in contact on the top and bottom sides of the material 222. The liquid layer 208 lubricates the material when moving through the aperture 213 created between the seal materials. It is to be appreciated that the figures are merely diagrammatical and are not drawn to any particular scale. The upper seal material 214 and the lower seal material 212 are shown surrounding the material 222 but are preferably not in contact with the material 222 during movement, as the aim is to create a liquid layer to allow lubrication as the material is passed through the seal. While the upper seal material 214 and the lower seal material 212 are shown as separate layers, the seal material can be formed of a single unitary polymer block with a slit cut into the block. The upper plate member 204 and the lower plate member 210 are generally rigid nondeflecting structural members to withstand the pressures reached inside the vessel and also to withstand the clamping force without deflection to apply a generally uniform pressure distributed on the material 222.. The upper plate member 204 and the lower

plate member 210 respectively press on the respective upper seal material 214 and lower seal material 212 such that the seal materials deflect into the shape of the material 222. The seal materials may be made from a solid (non-permeable), but deformable, polymer or a deformable permeable foam polymer with an open network of cells. A suitable material for the seal material is polyurethane, but others, may include, polyethylenes, polyamides, siloxanes, and the like. In order to seal the sides of the along the seal, a rigid rod 240, such as a wire, may be placed respectively along the axially-extending edges of the seal to prevent liquid from being expelled from the seal transversely. Alternatively, the upper and lower plates may include a ridge or bump extending axially that applies pressure against the seal materials to seal transversely.

Referring to FIGURE 5, a different seal configuration is shown in the case where the material 222 is a fiber 236. Similar to the seal described in association with FIGURE 4, the seal of FIGURE 5 includes a similar structural layering. However, the components are designed taking into consideration that the material 236 being withdrawn is a generally round material. The upper seal material 234 and the lower seal material 232 may be shaped as half cylinders. The upper plate member 216 and the lower plate member 218 may be similarly shaped as half cylindrical members. In either embodiment of FIGURE 4 or FIGURE 5, the upper plate and upper seal material may be compressed against the lower plate and seal material via various mechanisms. For example, hydraulics may be utilized in an automatically controlled manner. Alternatively, the upper and lower plates may be compressed via the use of manually tightened bolts, and the like. In another embodiment of a seal for a fiber, the seal material can be a cylinder with a hole bored through the axial center, and the cylinder is tightened around the circumference via the use of clamps.

Referring to FIGURE 6, another embodiment of a seal for use with a fiber is diagrammatically illustrated. In this embodiment, the structural member need not be divided into two halves. Instead, the structural member 242 includes a chamber having a cylinder with a first larger diameter connected to the pressure vessel (not shown). The larger diameter portion of the cylinder 242 is connected to a second smaller diameter portion 248 via a transition piece 246 that reduces in size from the first diameter to the second diameter. Thus, giving the cylinder a wedge-shape. The seal material 244 is shaped to have similar dimensions as the interior of the structural member and is fitted inside. The seal material 244 may have a central axial hole through which the

material 250, such as a fiber, is passed. In this instance, an axial force may be applied on the seal material 224 in the axial direction. Such force is then directed by the transition piece 246 downward such that the force compresses the seal material 244 around the material 250. The axial force F may be brought about by screwing the seal into a port on the vessel such that the vessel wall is compressed against the end of the seal material 244, or the pressure inside the vessel may apply the axial force that compresses the seal material 244.

Referring to FIGURES 7A and 7B, another embodiment of a first and second seal material is diagrammatically illustrated. Instead of a seal material that is deflected when compressed, in this case, the lower seal material 254 (or upper seal material) has been pre-shaped by having a permanent indentation 256 that matches the size of the material to be passed through the seal. The seal material 254 can be molded or machined to have the indentation 256. As seen in FIGURE 7B, the indentation 256 would have the same width and/or thickness of any film that is to be passed through the seal. The seal material 254 can be made from cellular foam that has been molded specifically for the material or the foam can be pressed with a hard tool to form the indentation 256. Alternatively, the indentation 256 can be machined by cutting or milling out the indentation 256.

FIGURE 8 is a diagrammatical cross-sectional illustration of another embodiment of a seal. The seal includes a first upper plate 260, a lower plate 262, a first seal material 264 juxtaposed on the interior side of the upper plate 260, and a second seal material 266 juxtaposed next to the interior side of the lower plate 262. A film 268 is shown between the first and second seal materials 264, 266. In the embodiment shown in FIGURE 8, the lower clamping plate 262 includes upward projecting bumps 270 on each transverse side of the plate 262. The upward projecting bumps 270 pinch the seal materials 266, 264 together and prevents liquid leakage through the transverse sides of the seal. Other alternatives to the bumps may include physical attachment of the seal materials 264 and 266 along the edges such as via glue or melting.

FIGURE 9 diagrammatically illustrates another embodiment of a seal. In the embodiment of FIGURE 9, the seal includes a rigid metal housing 272 as the structural member shaped in the form of a cylinder, and the interior of the metal housing includes a wedge shaped chamber. As used herein, wedge simply denotes a shape that has a larger diameter facing the high-pressure side of the vessel which tapers to a smaller diameter facing the outboard end of the seal. The tapered sides can have straight or smooth curves,

or have both. A seal material 274 in the form of a wedge is placed within the chamber. The high pressure of the liquid 278 within the vessel (not shown) pushes the seal material 274 against the tapered sides of the wedge causing the seal material 274 to constrict around the film 276 (or a fiber). The tapered geometry of the wedge uses the high-pressure liquid 278 inside the vessel as the force to squeeze the seal material 274 onto the film 276 (or fiber) providing the sealing effect.

Referring to FIGURE 10, one embodiment of a flat seal material 280 is diagrammatically illustrated. In this embodiment, the seal material 280 may have features added to help distribute the liquid between the seal material 280 and the film or fiber. In the embodiment illustrated in FIGURE 10, a serpentine channel 282 is cut or formed on the surface facing the film. The serpentine channel acts to guide the liquid more evenly along the surface of the film. While the illustration shows a serpentine channel, the channels can be any shape including straight vertical, straight horizontal, diagonals, or a zigzag or X pattern.

Referring to FIGURE 11, another embodiment of a seal material is diagrammatically illustrated. In the embodiment of FIGURE 11, the seal material is formed from a plurality of non-permeable layers 284, 286, and 288 alternating with permeable layers 290 and 292. The combination of a non-permeable seal material 284, 286, and 288 and permeable seal materials 290 and 292 regulate the pressure inside the seal to force a linear pressure drop from the entrance to the exit. The non-permeable materials may offer low friction, while the permeable materials may offer better control over the pressure drop.

The seal materials of FIGURES 10 and 11 may be used in any of the seals described herein.

Referring to FIGURE 12, a diagrammatical illustration of a device 301 to saturate a roll of film, pass the gas-saturated film through a seal, and foam the gas-saturated film, is illustrated. In the case of saturating films with a gas, the films can be provided in rolls, for example. A pressure vessel 300 may be configured to hold the rolls 308 in the gas-filled space 306 of the vessel 300. An end of the film 304 may be threaded through roller 310 and out via the seal 312. Once the roll is placed within the vessel 300, the vessel may be closed, the liquid is then introduced to a level above the seal 312 opening, and the gas is introduced and raised to pressure determined previously. Then, the rolled polymer is allowed to absorb gas for the predetermined time desired to reach the final gas

concentration. Once the gas concentration is determined to be suitable, the gas-saturated film is passed through the seal 312 and immediately heated by upper and lower ovens 316, 318 which produce a foamed film 320. Since the entire roll is not removed from the vessel, the roll remaining within the vessel will stay gas-saturated until ready to be foamed immediately after exiting the pressurized vessel. Seal 312 can be any one of the seals described herein.

Referring to FIGURE 13, a device 401 is diagrammatically illustrated for the introduction of a nonsaturated fiber polymer material into a pressurized vessel 400 simultaneously with withdrawing the gas-saturated fiber, and heating ovens for the foaming of a fiber. In the embodiment of FIGURE 13, the vessel 400 includes a first seal 422 for the material 404 inlet and a second seal 412 for the material outlet. The seals 422 and 412 can be any one of the seals described herein. The vessel 400 may include a revolving drum 402 with a helical groove which winds and unwinds the continuous fiber 404 at a pace that allows the fiber 404 to become saturated to the desired gas concentration by the time spent rolling and unwinding along the drum 402. In this case, the fiber is threaded through the inlet seal 422, the inlet roller 426, the drum 402, the outlet roller 410, and the outlet seal 412 in preparation for use. The vessel is then closed, filled with liquid and gas, and pressurized to the desired pressure. When the starting portion of the fiber is determined to be saturated, the drum may begin revolving to continuously provide gas-saturated fiber for foaming. Further, additional fiber length can be added as the fiber nears its end by welding multiple fiber lengths together to avoid stoppage. Such welding can take place prior to the fiber entering the pressure vessel. A similar technique can be used to add length to films. Any adhesive (e.g., tape) may also be used for connecting films and fibers of finite length into one continuous length that can avoid stoppage of production of saturated films and fibers. The drum 402 passes the fiber into the gas-pressurized, liquid-filled vessel 400 while the gas-saturated fiber is simultaneously withdrawn from the vessel 400 using another drum (not shown), for example. Immediately following the outlet seal 412, the gas-saturated fiber is passed through an upper and lower oven 416, 418 to create a foamed fiber 420.

Referring to FIGURE 14, another embodiment of a seal is diagrammatically illustrated. A pressure vessel 510 includes a gas space 512 and a liquid space 516. The seal is a dual purpose seal that serves both as an inlet and outlet from the pressure vessel. The seal includes an upper structural plate 502 and a lower structural plate 504. The

upper structural plate 502 and the lower structural plate 504 can be parallel with respect to each other. The lower end of the vessel 510 rests on the upper surface of the upper structural plate 502. The seal includes an upper seal material 506 and a lower seal material 508. The seal provides an aperture 524 formed between the upper seal material 506 and the lower seal material 508 for the introduction of a material 514 from one direction simultaneously with the withdrawal of the material 514 in another direction, after the material 514 has resided in the vessel 510 for a desired length of time. The aperture 534 is only large enough to allow the passage of the material 514 and for a thin layer of liquid to form between the material 514 and the seal materials 506 and 508, without the loss of a significant amount of liquid.

The upper seal material 506 and the lower seal material 508 may be constructed to have a cavity 518 and 528, respectively, between the surface of the respective structural plate and the seal material. In FIGURE 14, the cavities 518, 528 are shown containing an optional permeable support scaffold. The cavity 518 of the upper seal material 506 is connected to the liquid space 516 via aperture 522 in the upper plate 502. The aperture 522 allows liquid from the vessel 510 into the cavity 518 of the upper seal material 506. The same aperture 522 that allows liquid into the cavity 518 of seal material 506 may also serve as the path for the material 514 into the vessel 510. An aperture 526 in the upper seal material 506 is aligned with the aperture 524 in the upper plate 502 to permit the passage of the material 514 therethrough. Similarly, the cavity 528 in the lower seal material 508 is connected to the liquid space of the vessel 510 through an aperture 520. The aperture 520 may be connected to the liquid space through, for example, a channel (not shown) that connects the aperture 520 to the aperture 522 in the upper plate 502. In this manner, the aperture 522 can feed both cavities 518, 528 in the upper and lower seal materials 502, 504, respectively.

The cavities 518, 528 of the upper seal material 506 and lower seal material 508 being connected to the liquid space 516 allows the pressure from the liquid to be experienced on the high pressure side of the seal material, while the low pressure side (the side open to atmosphere) sees a decreasing pressure. As shown in FIGURE 15, a representative seal material, such as 506 and 508, will experience a constant pressure (represented by arrows of similar length) on the side where the seal material cavity is connected to the liquid space 516. However, the pressure of the liquid that is leaking out along the polymer film/fiber 514 decreases along the length of the seal material from the

high pressure of the vessel to atmospheric pressure. The higher pressure within the cavities will push against the respective seal materials to tighten the seal materials around the polymer film/fiber. However, the pressure does not completely shut off the liquid out of the seal, but can provide a self-sealing, self-regulating seal that minimizes leakage.

5 The seal of FIGURE 14 may also eliminate problems with non-parallel plates or uneven clamping of plates, because the liquid pressure on the sealing material compensates for any irregularity caused by non-parallel plates or an uneven clamping force.

Referring to FIGURE 16, another embodiment of a seal is illustrated. The vessel 610 includes a gas space 612 under pressure, a liquid space 616 below the gas space also under pressure. The vessel 610 may have a cylinder shape. The end of the vessel 610 has a dome shape 602 with an aperture 622 in the center. The interior of the vessel 610 defines a wedge-shaped chamber. The aperture 622 allows liquid 616 to slowly leak out from the vessel, and also serves to withdraw the film or fiber 614 from the vessel 610. A wedge-shaped seal material 606 is placed within the dome shaped end 602 in the wedge-shaped chamber. The aperture 622 extends axially through the wedge-shaped seal material 606 into the vessel 610. A cavity 620 in the shape of a cylinder is made axially in the wedge-shaped seal material 606 so as to leave a layer of seal material surrounding the aperture 622. In one embodiment, the cavity 620 does not include a support scaffold. In another embodiment, the cavity 620 can include a permeable support scaffold. In either case, the cavity 620 is filled with liquid from the liquid space 616. The pressurized liquid within the cavity 620 applies pressure against the sides of seal material surrounding the aperture 622. While the pressure on the high-pressure side of this layer of seal material is substantially constant, the pressure on the exterior side (along the aperture 622) of the seal material gradually decreases from a high pressure to atmospheric pressure as the liquid exits the aperture 622.

EXAMPLES

Two aluminum plates were held in a generally parallel orientation with respect to each other. A porous (permeable) polymer sealing material is placed on the inside surface of each aluminum plate. The aluminum plates are held with a series of bolts along the transverse sides of the plates. One aluminum plate (the upper plate) has a port in the center to accept a pressurized liquid. The port extends through the upper sealing material. The top aluminum plate includes ports in both directions extending from the center port. A total of three ports are drilled on each side of the center port extending

axially. The six ports were coupled to sensors to read pressure. A film was captured between the seal materials, and the pressurized liquid was turned on so that the liquid was distributed between the upper and lower surfaces of the film and the seal materials.

5 FIGURES 17 and 18 show plots of voltage (mV) representing pressure versus distance as expressed by port number from the central pressure port (no. 4 on the horizontal axis). While the FIGURES generally show a linear relationship, the pressure profile can be altered with the clamping force of the bolts. Tightening any one of the bolts may severely alter the pressure at a particular sensor port.

10 Nevertheless, the prototype seal described above allowed the film to move through very easily when liquid pressure is applied, otherwise, the clamping force keeps the film locked in place.

The liquid leakage is very small, and the force required to pull the polymer film/fiber through the seal is also small. Liquid pressures of up to 5.17 Mega Pascal (750 psi) have been successfully sealed by a lab prototype while allowing a fiber to be
15 drawn out by hand.

While illustrative embodiments have been illustrated and described, it will be appreciated that various changes can be made therein without departing from the spirit and scope of the invention.

CLAIMS

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of withdrawing a material from a pressurized vessel, comprising:

withdrawing a material from a gas-pressurized, liquid-filled vessel from a seal below a liquid level;

applying a force with the seal on the material to limit a flow of liquid from the seal while creating a layer of liquid interposed between the material and the seal; and

controlling a pressure within the vessel with a gas.

2. The method of Claim 1, wherein the seal comprises a first and second plate disposed generally parallel to each other, a first and second seal material juxtaposed on the interior sides of the first and second plates, and a film material is passed between the first and second seal materials while a liquid layer lubricates the sides of the film against the seal materials.

3. The method of Claim 1, wherein the seal comprises a chamber having a first larger diameter attached to the vessel, a second smaller diameter, and a transition that connects the larger diameter of the chamber to the smaller diameter of the chamber, and a seal material is placed inside the chamber, wherein the seal material is compressed against an inside wall of the transition, and a fiber material is passed within the seal material, while a liquid layer lubricates the sides of the fiber against the seal material.

4. The method of anyone of Claims 1-3, wherein the gas is carbon dioxide or nitrogen.

5. The method of anyone of Claims 1-3, wherein the liquid is water.

6. The method of anyone of Claims 1-3, wherein the material is a film.

7. The method of anyone of Claims 1-3, wherein the material is a gas-saturated film.

8. The method of anyone of Claims 1-3, wherein the material is a fiber.

9. The method of anyone of Claims 1-3, wherein the material is a gas-saturated fiber.

10. The method of anyone of Claims 1-3, wherein the seal extends axially.

11. The method of anyone of Claims 2-3, wherein the seal material is deformed to conform around the material to limit the flow of liquid out of the seal.

12. The method of anyone of Claims 2-3, wherein the seal material is polyurethane.

13. The method of anyone of Claims 2-3, wherein the seal material is permeable.

14. The method of anyone of Claims 2-3, wherein the seal material is non-permeable.

15. A method of making a foam, comprising:
saturating a solid thermoplastic polymer with a gas within a gas-pressurized, liquid-filled vessel to produce a gas-saturated thermoplastic polymer;
withdrawing the gas-saturated thermoplastic polymer from the vessel through a seal below a level of the liquid;
applying a force with the seal on the gas-saturated thermoplastic polymer to limit a flow of liquid from the seal while creating a layer of liquid interposed between the gas-saturated material and the seal; and
heating the gas-saturated thermoplastic polymer to a temperature to create cells without melting the thermoplastic polymer.

16. The method of Claim 15, wherein the seal is compressed around the gas-saturated thermoplastic polymer.

17. The method of Claim 15, wherein the thermoplastic polymer is a film or a fiber.

18. A seal, comprising:
a first structural member;

a second structural member, wherein a first surface of the first structural member faces a second surface of the second structural member;

a first seal material is juxtaposed on the first surface;

a second seal material is juxtaposed on the second surface, wherein the first and second structural members are configured to be compressed toward one another to compress the first and second seal materials toward one another; and

a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure between the surfaces of the first and second seal materials.

19. The seal of Claim 18, wherein the first and second structural members are parallel.

20. The seal of Claim 18, wherein the first and second seal materials are non-permeable polymeric materials.

21. The seal of Claim 18 wherein the first and second seal materials are permeable polymeric materials.

22. The seal of Claim 18, wherein the first and second seal materials include alternating layers of permeable and non-permeable polymeric materials aligned axially.

23. The seal of Claim 18, wherein the first and second seal materials include channels on surfaces that face one another.

24. The seal of Claim 18, wherein the first and second seal materials define an aperture into the vessel.

25. The seal of Claim 18, wherein a cavity is provided adjacent to each of the first and second seal materials, and each cavity is filled with pressurized liquid that applies a constant pressure along one side of each of the seal materials.

26. A seal, comprising:

a structural member having a wedge-shaped chamber, wherein the chamber has a larger diameter that tapers toward a smaller diameter;

a seal material within the chamber that assumes the shape of the wedge-shaped chamber, wherein the seal material includes a hole axially traversing the seal material; and

a vessel connected to the seal, wherein the vessel contains a liquid under pressure, wherein the liquid exerts pressure on the seal material and compresses the seal material in the wedge-shaped chamber.

27. The seal of Claim 26, further comprising a cavity provided between the structural member and the seal material, wherein the cavity is filled with pressurized liquid that applies a constant pressure along one side of the seal material.

ABSTRACT

The present disclosure describes devices capable of continually and controllably passing a polymer film or fiber through a pressurized vessel, while maintaining a desired pressure inside the vessel. In certain embodiments, the devices of the present disclosure
5 include dynamic seals capable of limiting the gas flow from the pressurized vessel to lower pressure environments.

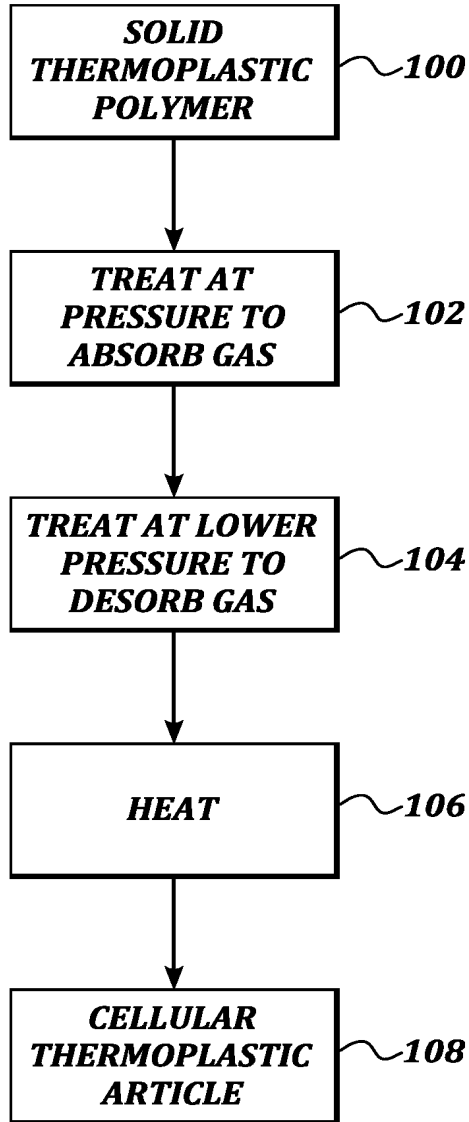


FIG. 1
(PRIOR ART)

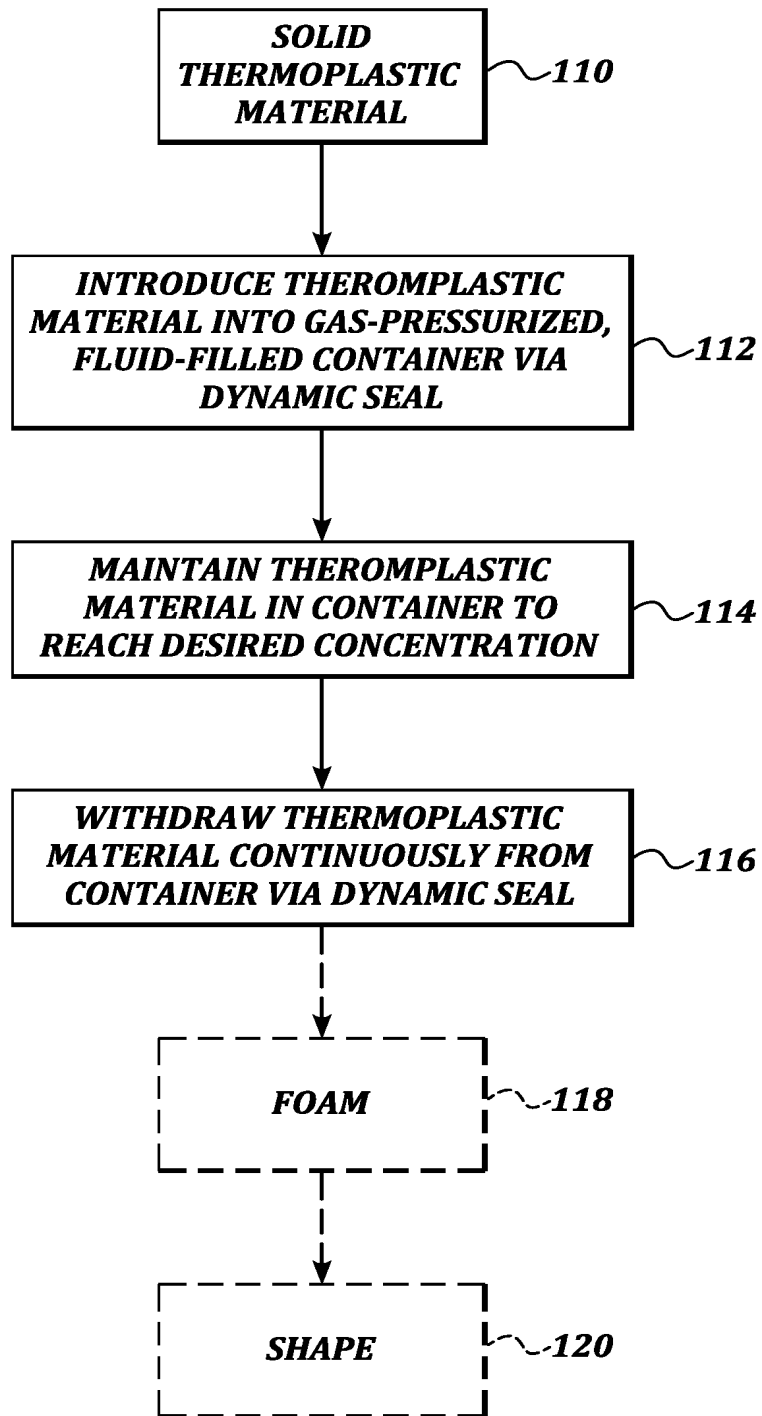


FIG. 2

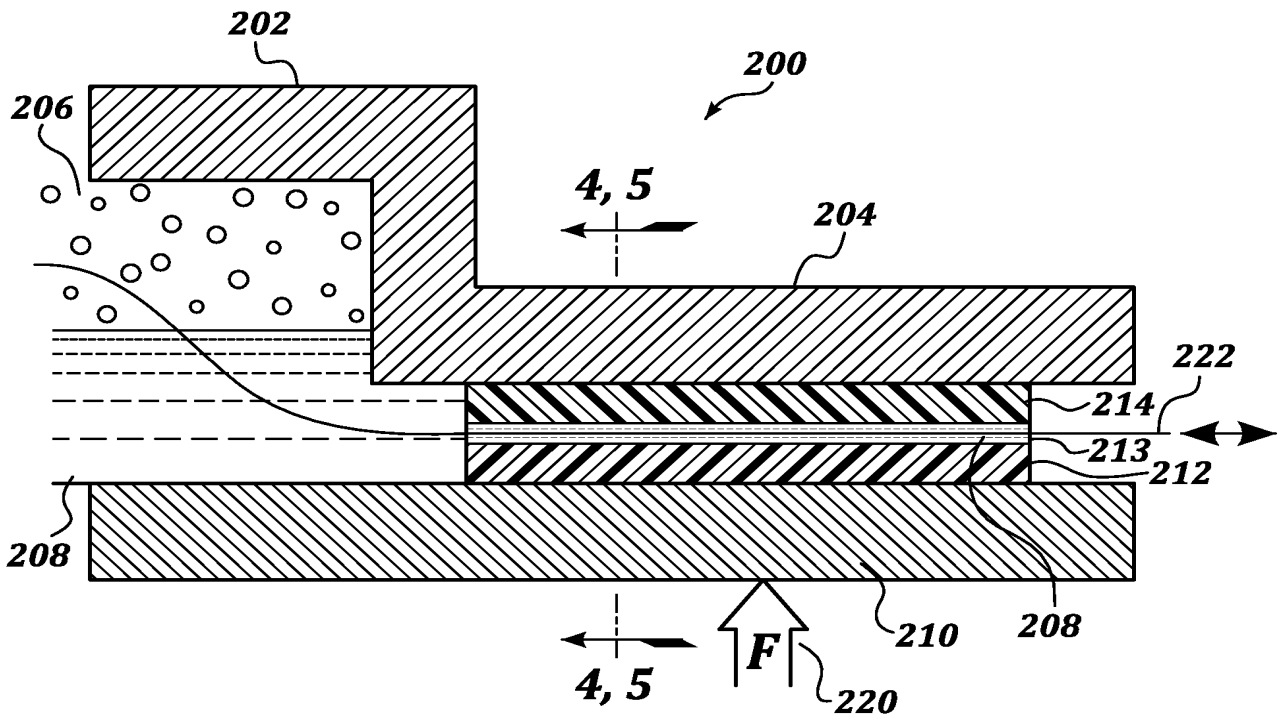


FIG. 3

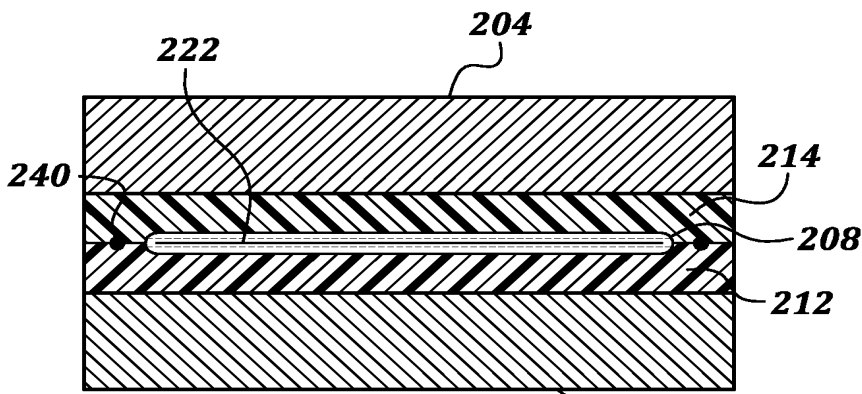


FIG. 4

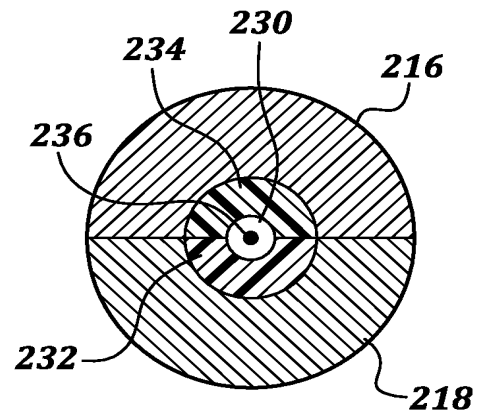


FIG. 5

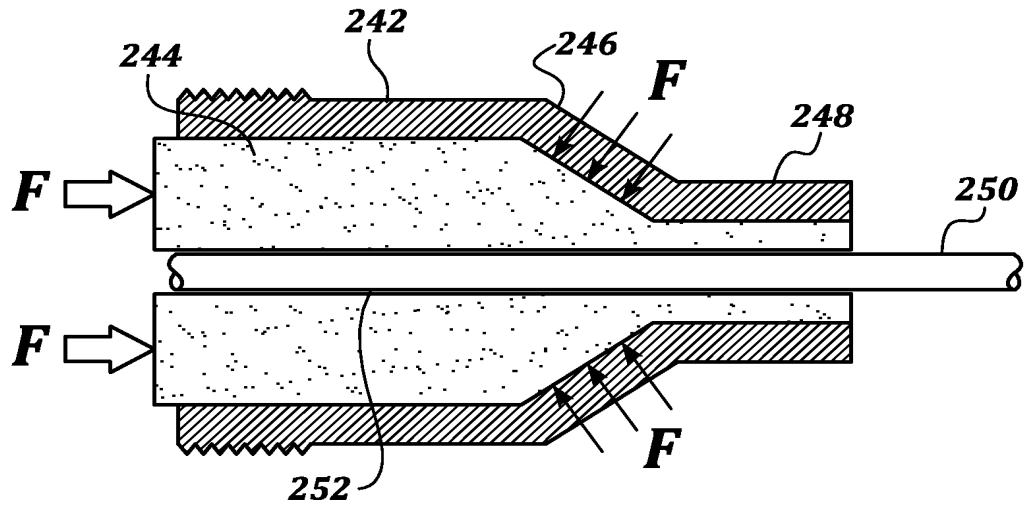


FIG. 6

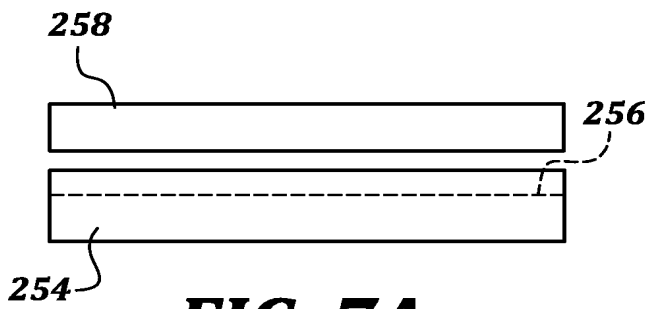


FIG. 7A

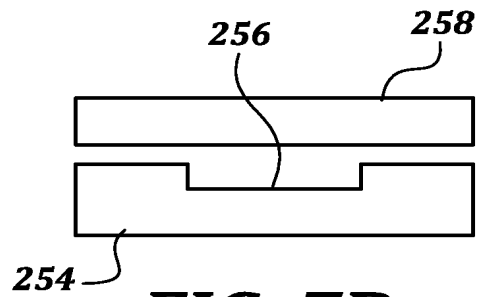


FIG. 7B

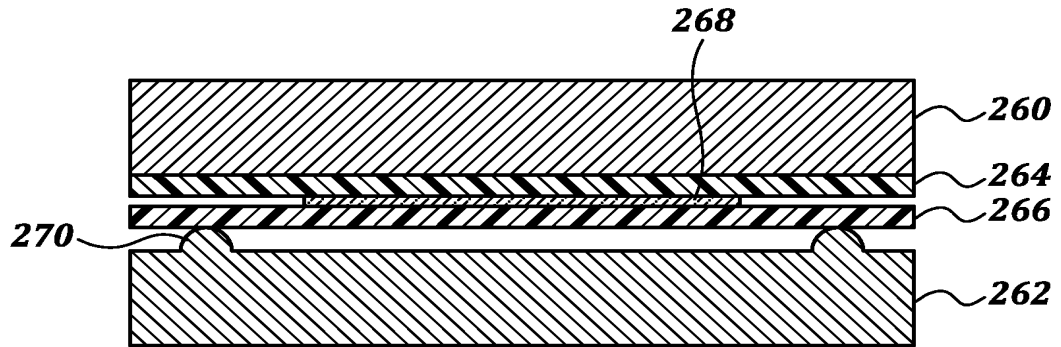


FIG. 8

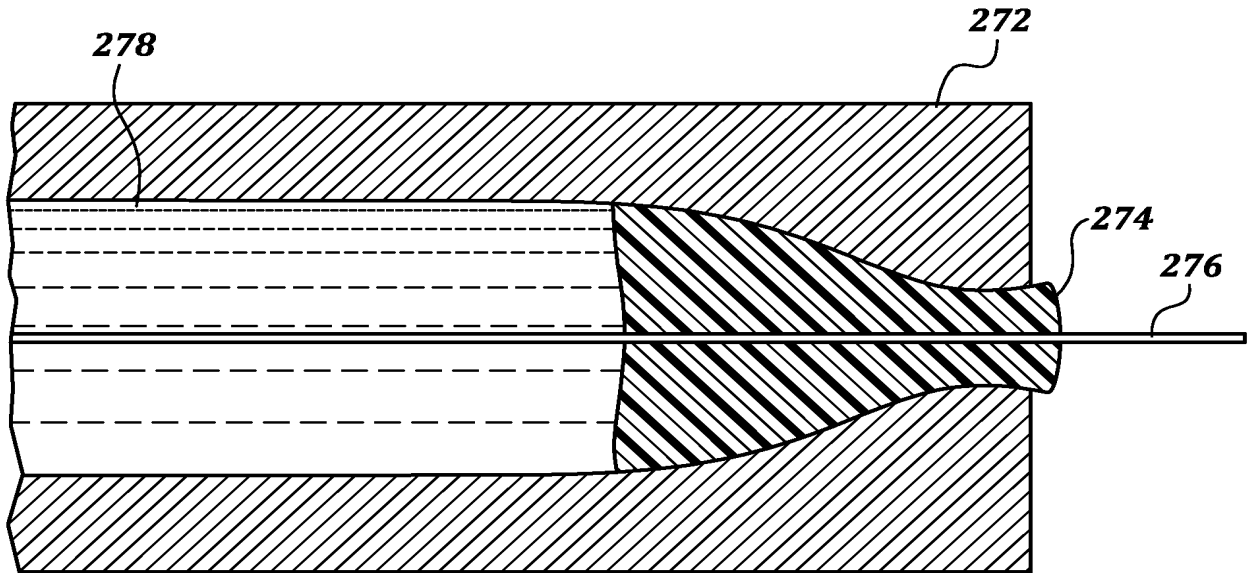


FIG. 9

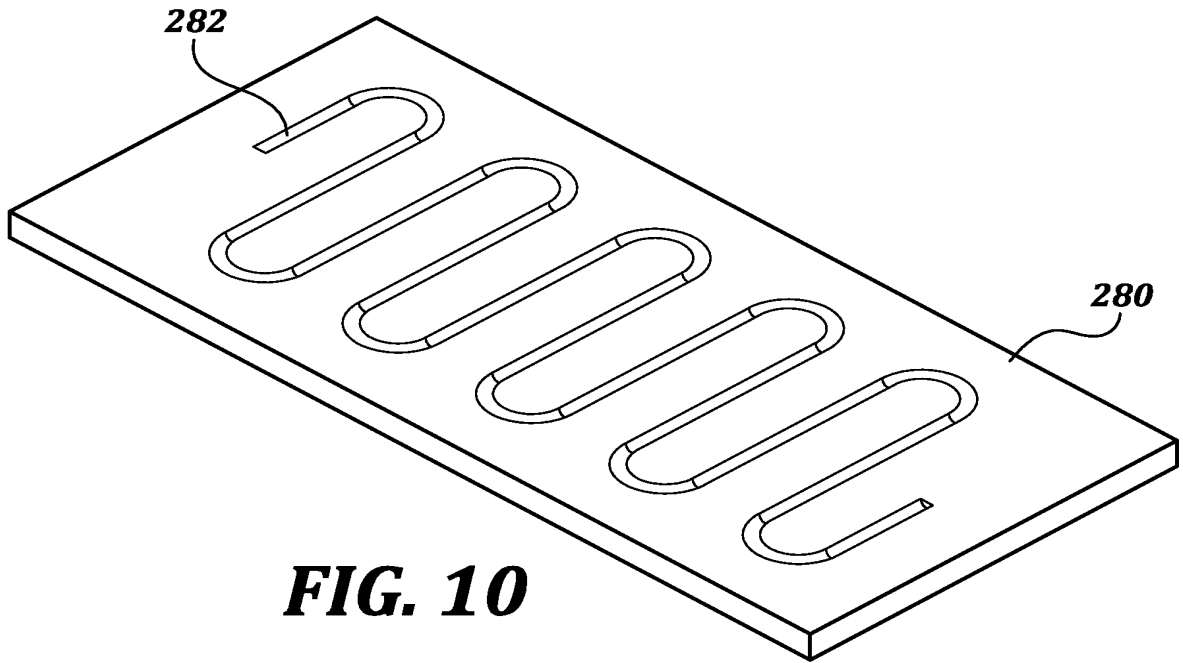


FIG. 10

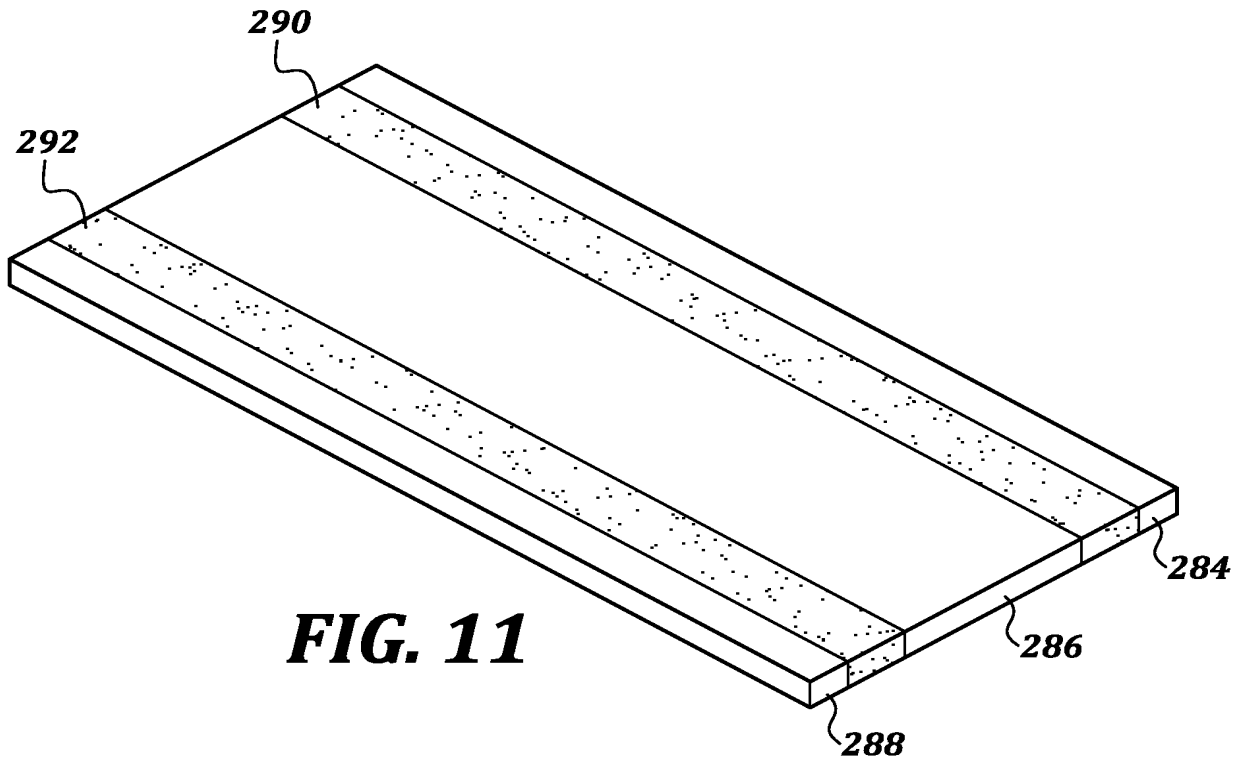
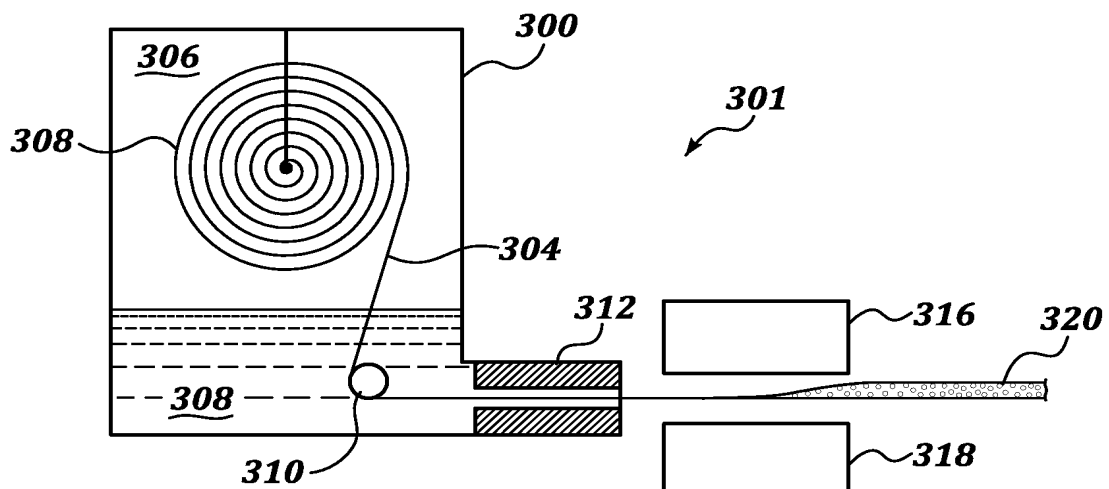
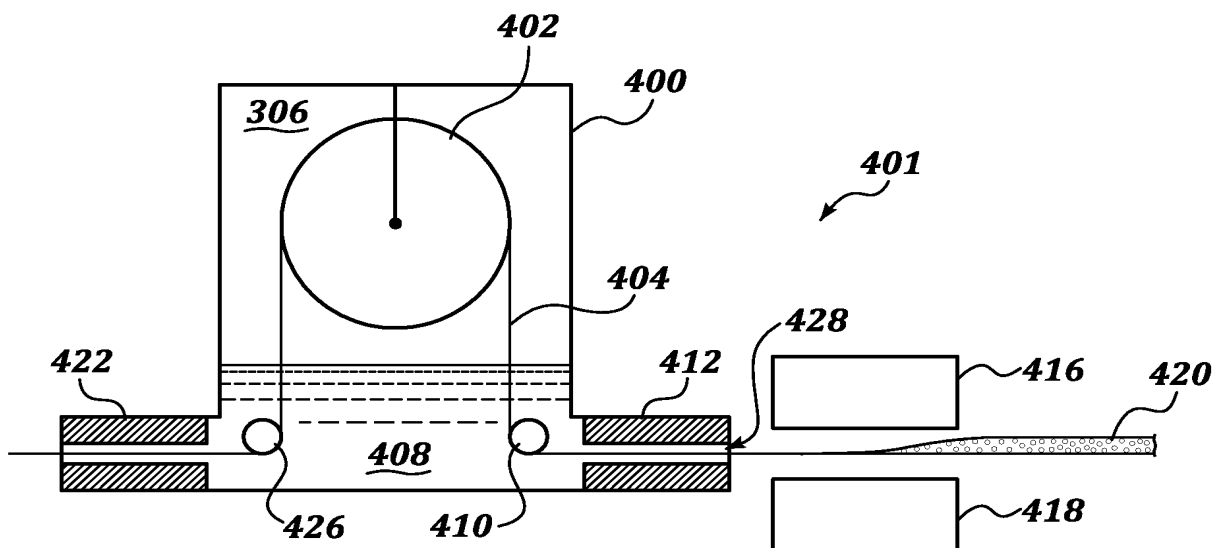


FIG. 11

**FIG. 12****FIG. 13**

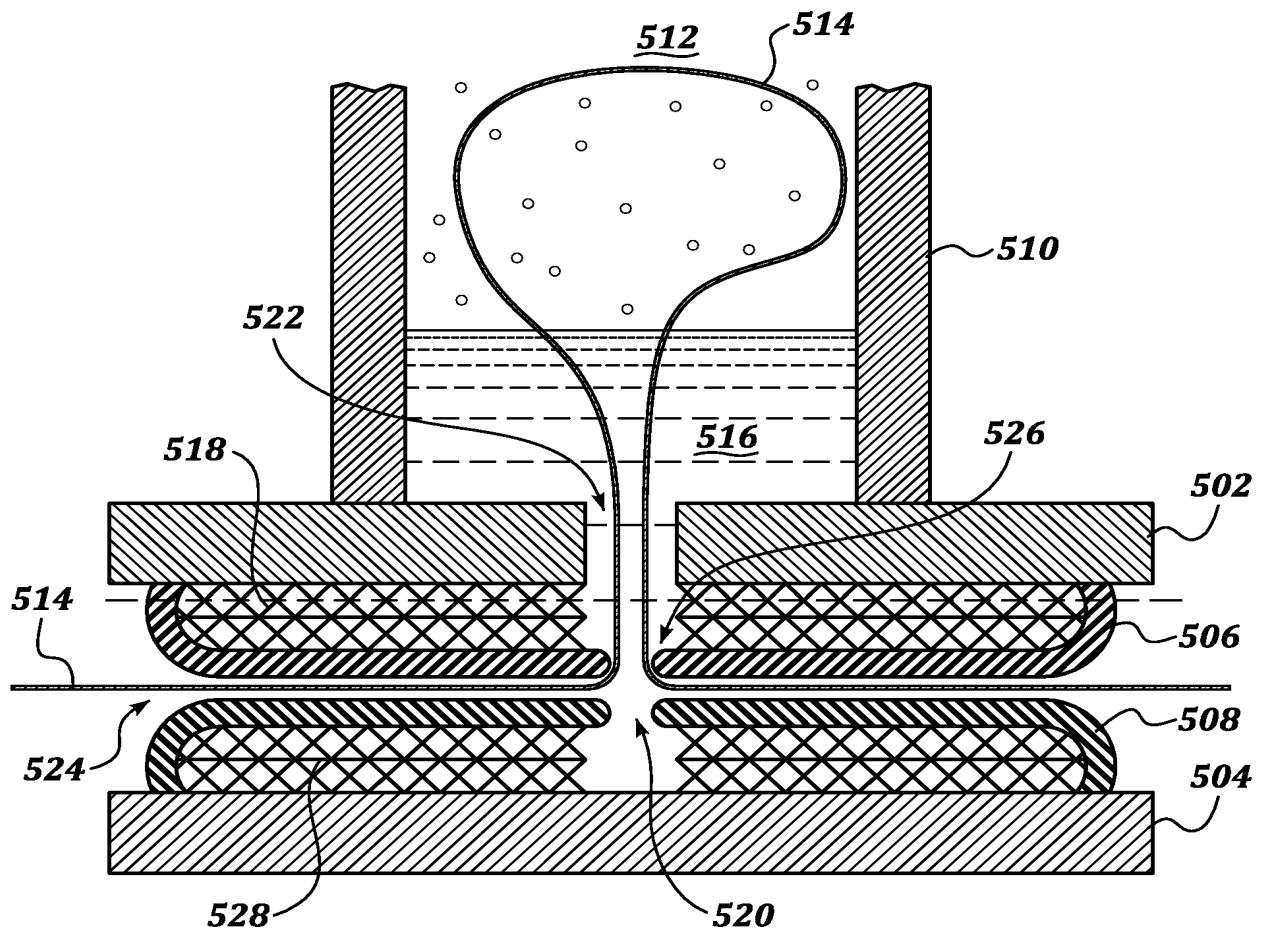


FIG. 14

9/10

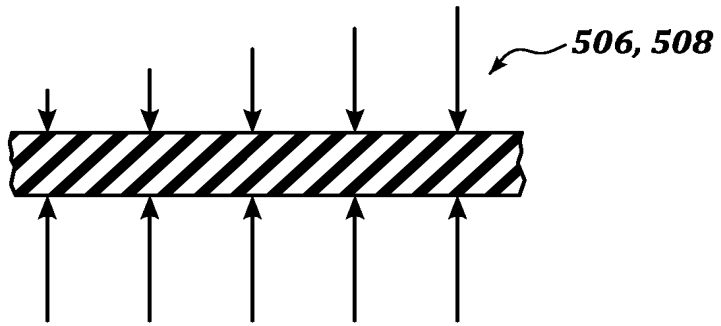


FIG. 15

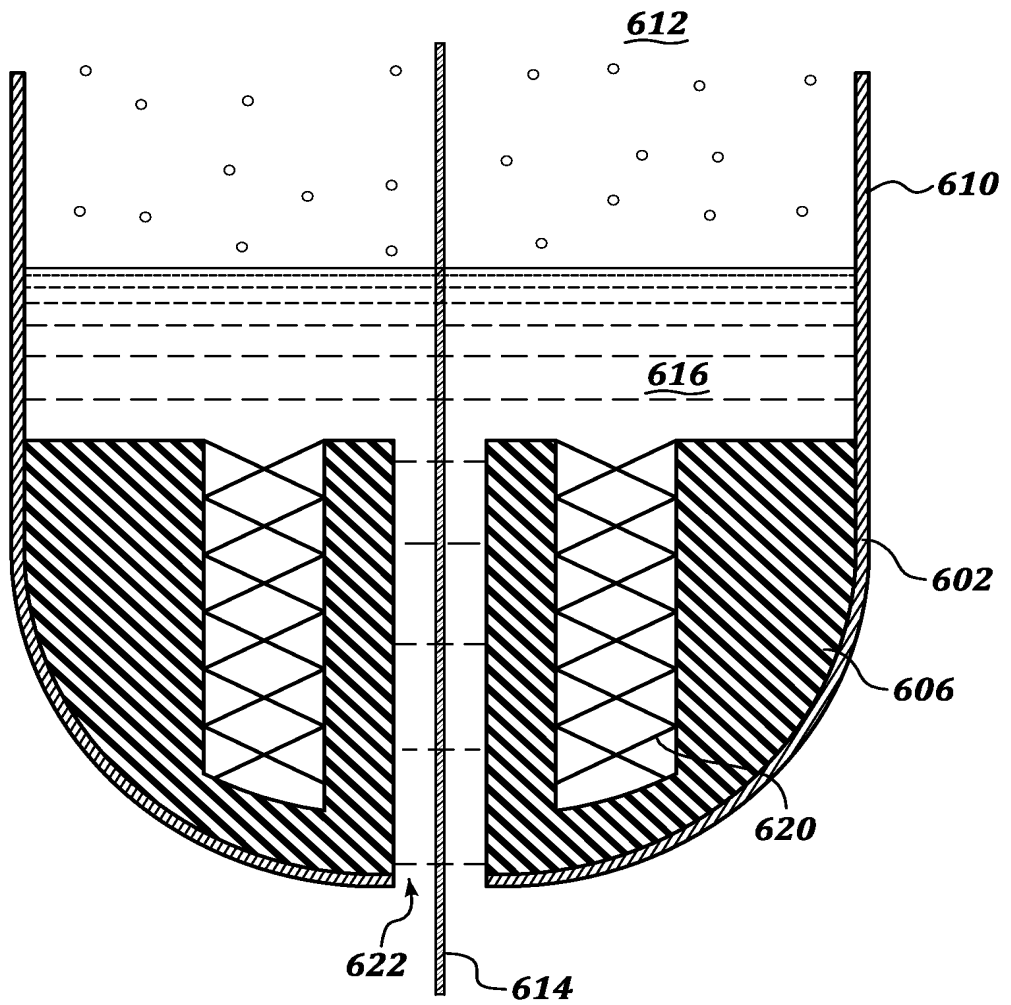


FIG. 16

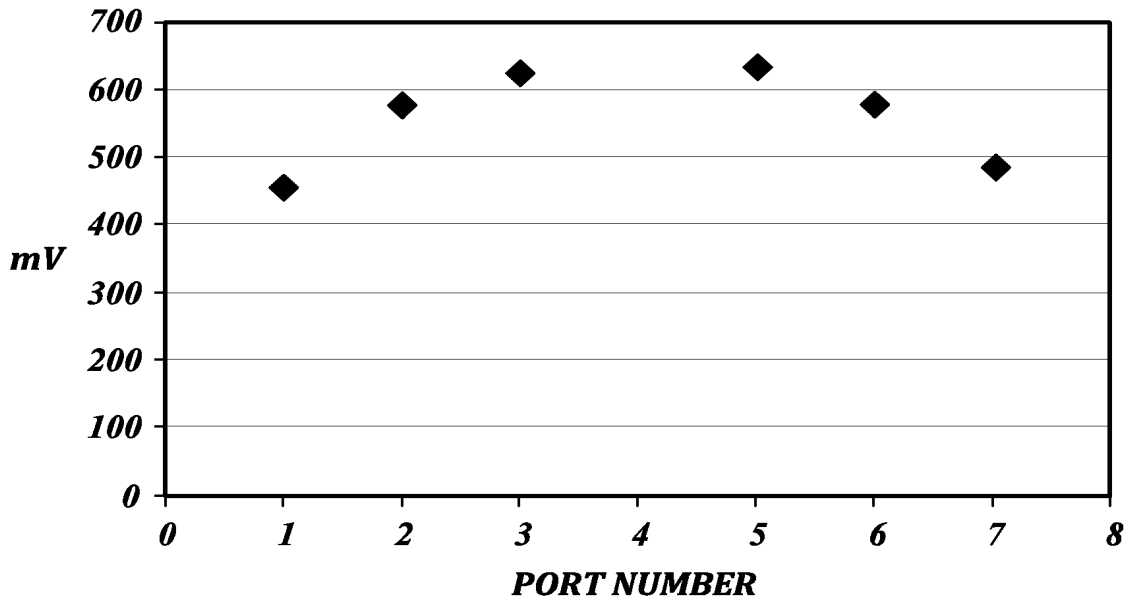


FIG. 17

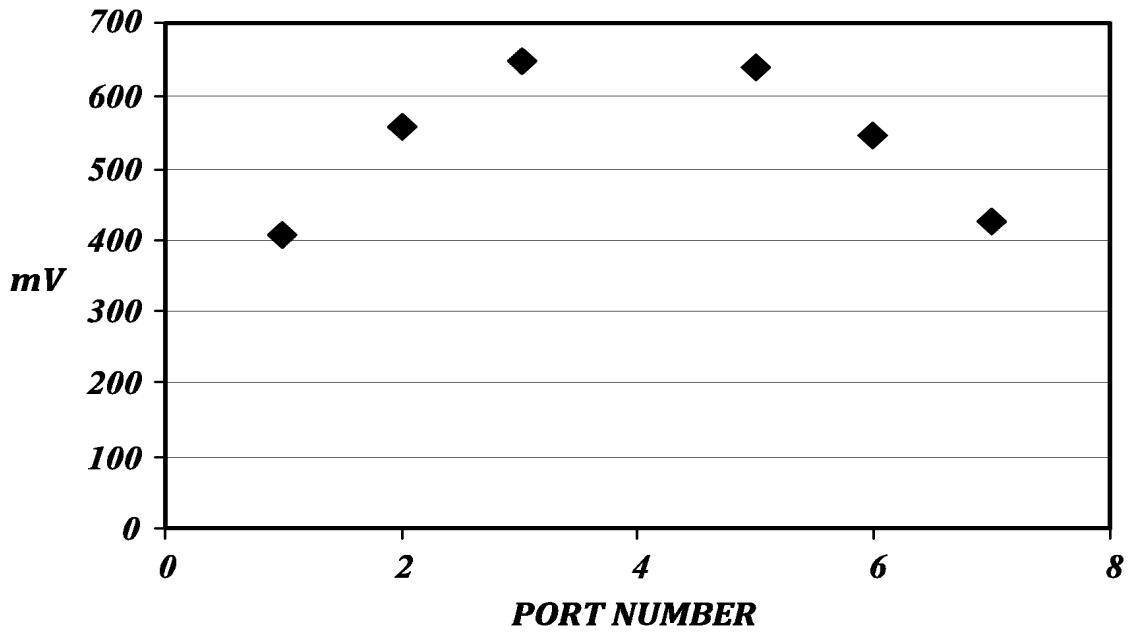


FIG. 18