PACKAGING OF THE MIT MICROENGINE

by
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Submitted to the Department of Aeronautics and Astronautics
on May 5, 2000 in partial fulfillment of the requirements for the
Degree of Master of Science in Aeronautics and Astronautics

Abstract

The MIT Microengine Project is an on-going effort to build a MEMS based gas turbine engine and
turbogenerator. This thesis covers the development and testing of the packaging needed for the
microengine and related devices. The packaging of the microengine includes the fluidic, electrical,
and sensor connections that interface the engine with the supplies and instruments necessary for
operation. Making connections to the device is difficult because each connection must be gas-
tight, able to bond to a device made entirely of silicon, and capable of withstanding temperatures
from 25°C to 900°C. The method chosen for connection was glass sealing, which has been used
extensively for metal-to-glass seals in electronics packaging but is a novel approach for making
metal-to-silicon connections.

The three strength-limiting factors found in metal-to-glass-to-silicon seals were the existence of
voids in the glass, the lack of wetting of the glass on the silicon, and the residual stress in the
silicon due to thermal expansion mismatch with the glass. Sessile drop tests were conducted to
determine which process parameters affect these strength-limiting factors. Voids were found to
depend on the type of glass used, specifically the manufacturing process of the preforms, and
wetting was found to depend on both the atmosphere used in the furnace while making the bond
and the type of glass being used. Test samples were made using both the baseline process and the
improved process recommended by the results of the sessile drop tests. Mechanical testing of the
samples confirmed that using the improved process increased the strength and reliability of the
joints.

Macro connections are the interface between the micro tubes and wires exiting a device and the
macro supplies and instruments in the lab. Several different options were explored for making
these connections, three of which were tried on different devices. A fixed plate method is
recommended for making macro connections due to its support of the tubes, protection of the
device, and ease of assembly. Inlet, exhaust, and electrical connections were also considered and
several different options for each are presented.

The results of this thesis are an improved process for making metal-to-glass-to-silicon seals, a
roadmap for packaging future devices, and recommendations for interfacing the microengine with
the micro aerial vehicle. Future work in packaging should include a more detailed study of
wetting, the manufacturing process of preforms, and the possibility of wafer level packaging.

Thesis Supervisor: Professor S. Mark Spearing
Title: Esther and Harold E. Edgerton Associate Professor of Aeronautics and Astronautics
Acknowledgements

This thesis would not have been possible without the help, support, and hard work of many others. Prof. S. Mark Spearing was instrumental in guiding my work and providing insight and knowledge where I previously had none. A special thanks goes to Greg Simpson and everyone at Thunderline Z for their expertise and extra attention, without which I (and several others) may have never finished our degrees.

I would like to thank my parents, Beth and Phil Harrison, and my sister, Ruth Harrison, for their support, love, and encouragement over the years. I would not be the person I am today without them.

Most of all I would like to thank my wife, Beth. For the past five years she has given me friendship, advice, encouragement, hope, humility, love, and patience. For all those times you cheered me up, calmed me down, shared my excitement, or just listened to me talk about really boring stuff—this thesis is dedicated to you, Beth.

This work was supported by ARO Grant # DABT63-98-C-0004, Technical Monitor Larry Carter.
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Chapter 1: Introduction

1.1. Overview of Microengine and Microrocket

1.1.1. Introduction to the Microengine Project

The MIT Microengine Project, funded by the Army Research Office and the Defense Advanced Research Projects Agency, is an on-going effort to build the world’s smallest gas turbine engine. The baseline engine is approximately 2.1 cm square, 0.37 cm thick, and weighs only 2 grams. It is built entirely from silicon by etching wafers and bonding them together to form a three-dimensional structure. A similar project is underway to build a microrocket motor using the same fabrication methods. The microrocket will operate at a higher temperature and pressure than the microengine.

The potential applications of the microengine and microrocket range from being a lightweight electrical power source to a miniature propulsion system. As an electrical power source, the microengine coupled with a micro-generator could be an attractive alternative to conventional batteries. Some estimates predict an order of magnitude increase in energy density for the microengine using hydrocarbon fuels over conventional batteries. A microengine/generator battery the size of a standard US Army BA-5590 (12.7cm X 11.2cm X 6.2cm) could potentially provide 25 watts of power for 88 hours without refueling. Another proposed application is to build a micro aerial vehicle (MAV) having all dimensions smaller than 15 cm and weighing less than 100 grams that uses the microengine for propulsion. A single microengine could provide up to 11 grams of thrust,
yielding a MAV that could travel at over 35m/s and carry a video camera or other sensors. Similarly, the microrocket could be used on a micro launch vehicle or as an on-board propulsion system for satellites. However, many advances in current micro-fabrication technologies will have to be made before the performance necessary for these microengine and microrocket applications can be achieved.

Intermediary devices were constructed to demonstrate engine components independently because the underlying micro-fabrication technology was already being pushed to its limits. A micro-bearing rig was built to test the operation of the gas bearing in the engine. A combustor rig was constructed and tested to study how fuels burn in small volumes, as in the microengine. An experimental recuperator was built to explore one option for increasing the fuel efficiency of the engine. The first engine with all of the components incorporated is the demonstration engine (Demo Engine), which is made to run on the test bench using hydrogen fuel only. Future generations of the microengine will be built that incorporate advances in materials, structures, combustion, and other areas as a result of micro device research.

This thesis covers the packaging of the MIT Microengine and related devices. The term packaging, as it is used in this context, refers to all of the connections that are external to the silicon device and are necessary for its operation and testing. These include fluidic connections for air, fuel, and pressure taps; electrical connections for igniters; macro connections; and inlet and exhaust ports. The following pages give a brief overview of
how these devices are made, the requirements for their packaging, the different options explored for making connections to silicon, and the motivation for the following chapters.

1.1.2. Micro-Fabrication

Capabilities of Micro-Fabrication

The enabling technology behind the Microengine Project is deep reactive ion plasma etching of silicon, which makes it possible to etch features with in-plane dimensions on the order of microns through silicon wafers with great precision. Both sides of a wafer can be etched with single or nested masks, and wafers can be bonded together to form complex three-dimensional structures.5 The microengine design uses a six-wafer stack and a total of 25 etches. This method of fabrication also lends itself easily to batch production because multiple devices can be made on a single wafer. Each four-inch diameter wafer holds ten microengines, and increasing the wafer size and scale of production can yield tremendous economies of scale. If the operation is expanded in the future, microengines could be produced in the same volume and for similar prices as computer processor chips.

Micro-Fabrication Limitations

While micro-fabrication has many advantages, there are some limitations to the types of devices than can be made. A major limitation to this method of fabrication is that only two-dimensional “extruded” geometries can be made on a wafer. The etching process also leaves some roughness and contamination behind on surfaces, which can affect the strength of components in the engine and the ability to make connections to the outer surfaces.
An important measure of any micro-fabrication process is the yield, or the percent of operational devices that are produced from the manufacturing process. Because six layers of wafers are used to build the engine, the yields of each wafer can be separated to a certain extent. In other words, if wafer two is destroyed during etching, it does not affect the other wafers. However, once the wafers are bonded together the yields multiply because a mistake in one wafer affects all of the other wafers. The later the step in the process, the more important its yield becomes because it can ruin all the work that has been done up to that point. Because packaging is the last step in the process of making a microengine, its yield is the most important.

1.1.3. Microengine Design

The microengine is a centrifugal flow gas turbine engine. It is assembled from silicon wafers that are etched on both sides and then bonded together to form a single structure. A cross-section of the engine is shown in Figure 1-1. In the center is a rotor, which is free from the rest of the structure and spins at approximately 2 million revolutions per minute during operation. The Demo Engine has starter air connections, fuel inputs, pressure taps, and an inlet on the top, as well as an exhaust tube and an igniter on the bottom side.

![Diagram of the microengine](image_url)

*Figure 1-1: Diagram of the microengine, courtesy Jon Protz and Diana Park*
1.1.4. **Microrocket Design**

The microrocket design is shown in Figure 1-2. It is made from six wafers bonded together and can burn a variety of fuels to produce up to 20 N thrust. The microrocket is designed to operate at a pressure of 12.6 MPa (125 atmospheres) and an outer structural temperature of 900°C.

![Figure 1-2: Layout of the microrocket, courtesy Adam London](image)

1.2. **Packaging Requirements**

The packaging of the microengine, microrocket, and intermediary devices includes making fluidic connections, electrical connections, inlet and exhaust ports, and macro connections. Making these connections is complicated because the temperature of the surface of the engine varies from room temperature to over 600°C and the engine is made of silicon, which has a coefficient of expansion that is lower than most metals. The connections must maintain a gas-tight seal throughout this range of temperatures and at pressures of 2 atm in the microengine and up to 125 atm in the microrocket. The requirements for each type of packaging connection for both the microengine and microrocket are discussed below.
These requirements stem from the operating conditions of the test devices that are currently being built and do not include requirements that may eventually be necessary for the application of these devices.

### 1.2.1. Microengine Packaging Requirements

**Fluidic Connections**

The microengine requires several fluidic connections for fuel, starter air, and pressure taps. All of these connections must be able to hold a pressure of up to 5 atm, although the operating pressure of the engine will be under 2 atm. The locations of the holes in the die are shown in Figure 1-3. The hole sizes are 2 mm diameter for the corner holes (starter air) and 0.8 mm diameter for the other holes along the sides (fuel and pressure taps). The key requirement is that the connections must remain gas-tight from room temperature to 600°C. The tubes connecting to these holes will pass only cold fluids, such as air, hydrogen, or hydrocarbon fuels.

![Figure 1-3: Hole configuration for the Demo Engine](image)
Electrical Connections

Electrical connections are needed to power the igniter, whether it is an implanted resistor, a spark gap between two wires, or a resistive loop of wire. The primary requirement is that a conductive connection is made with a resistance that is relatively low compared to the resistor or the gap. This connection must be able to withstand temperatures from room temperature to over 600°C (since it is directly connected to the combustor) and be gas-tight.

Inlet and Exhaust Ports

The inlet and exhaust ports exist to guide the air into and out of the engine without disrupting the flow or overheating the structure. The inlet was designed to guide the air through its first 90° turn as it enters the compressor, as is shown in Figure 1-1. The inlet must be made with tight tolerances so that the desired shape is achieved. It should be able to slide into the silicon structure easily but not move during operation. The inlet must also be attached to the silicon, and experience temperatures ranging from room temperature to 600°C.

There is a natural tendency for the exhaust to travel parallel to the bottom of the engine when it exits the turbine because it has swirl. The exhaust temperature is above 1000°C and will overheat the silicon if this is allowed to happen. In addition, the exhaust must exit the engine perpendicular to the bottom surface to produce thrust. For this reason, an exhaust port must be added to direct the flow in the correct direction. The exhaust connection must survive temperatures above 600°C because it comes into direct contact with the exhaust gases, but some leaks can be tolerated.
Macro Connections

Once fluidic, electrical, and inlet and exhaust connections have been made to the engine, macro connections need to be made to connect the tubes coming out of the engine to their respective supplies and instruments. For bench-top tests of the engine, many different methods exist for making these connections. The key requirements are that the handling loads and thermal stresses induced as the device heats up do not cause any connection to break or leak. The macro connections must also be arranged in a manner that allows other sensors to access the device, such as an optical probe to measure the speed of the rotor.

1.2.2. **Microrocket Packaging Requirements**

**Fluidic Connections**

The requirements for the fluidic connections to the microrocket are more stringent than those for the microengine. There are eleven connections that need to be made to the microrocket. Each hole is 0.8 mm in diameter; the configuration is shown in Figure 1-4. Fuel, oxidizer, and coolant must pass through these connections into the motor at pressures...
of up to 12.6 MPa (125 atm). The temperature on the surface of the silicon where these connections are made may reach as high as 800°C.

**Igniters**

The igniters for the microrocket must create a spark that is hot enough to ignite the fuel/oxidizer mixture as it passes through the combustion chamber. The connection made to provide the current must survive both the temperature and pressure of the rocket, but the igniter does not need to be reusable.

**Macro Connections**

The requirements of the macro connections for the microrocket are essentially the same as for the microengine. The microrocket does differ slightly because 20 N of thrust is generated perpendicular to the direction of the connections and the sensors used to monitor the rocket’s operation are different.

1.3. **Making Connections to Silicon**

Several options were explored for making connections to silicon. These included using solder, epoxy, braze, and glass. Each of these options has advantages and disadvantages, which are discussed below, but only glass sealing proved to be viable. The process of making a glass-to-metal seal is well understood and has been used in electronics packaging for many years. However, a metal-to-glass-to-silicon seal is essentially new and untested, and several problem areas are identified for further discussion.

Kovar, also known as Nickel Alloy 29-17, was chosen as the material for the tubes because its coefficient of thermal expansion is a close match to silicon at temperatures
below $500^\circ C$. Figure 1-5 shows that above that temperature, Kovar departs from silicon but remains closer than other metals. This alloy was specifically designed to match the expansion of certain Pyrex glasses so that matched, hermetic seals could be formed in electrical packages.

![Graph showing the relative thermal expansion of Kovar, Steel, and Silicon](image)

*Figure 1-5: Relative thermal expansion of Kovar, Steel, and Silicon* 

1.3.1. **Soldering, Epoxy, Brazing, and Glass Sealing**

**Soldering**

Soldering was quickly dismissed as an option because it is performed at a temperature well below the operating temperature of the device. While it might be possible to make a connection between Kovar and silicon using a solder and metal plating of the silicon, it would not withstand the operating conditions.
**Epoxy**

Epoxy was considered as a method to make connections to silicon because it is easy to use, can be applied at room temperature, and does not require precision in its application. Most epoxies, however, are not made to withstand temperatures up to 600°C. In preliminary packaging efforts for the micro-combustor, a ceramic, high-temperature epoxy was used to make fluidic connections. While the connections did survive at higher temperatures, they were prone to leaks. Figure 1-6 shows how mounds of epoxy collected around the tubes as more adhesive was applied in attempts to seal leaks. The ceramic epoxy is porous and water soluble, which makes it nearly impossible to use for gas-tight seals. Intense handling of the delicate device was also necessary to apply the epoxy and frequently caused damage to the fluidic connections and the device itself.

![Epoxy CONNECTIONS](image)

*Figure 1-6: Attempts to use epoxy on the micro-combustor, courtesy Amit Mehra*

**Brazing**

A copper-silver braze was also considered because it has a eutectic temperature of 780°C, which would allow for a connection to be maintained at 600°C. However, the braze
material will not adhere directly to silicon, so a thin layer of nickel must first be deposited on the surface of the silicon around the hole. Several silicon pieces were prepared in this manner, and Kovar tubes were brazed to the nickel-plated silicon using braze performs. In each case the copper-silver braze material fractured the silicon beneath it as it cooled. The coefficient of thermal expansion for the braze material is significantly higher than that of silicon. Therefore, the stress induced by cooling the joint back to room temperature from temperatures in excess of 780°C was sufficient to fracture the silicon.8

**Glass Sealing**

Glass sealing was explored in order to bond a Kovar tube to silicon. The glass used was Corning-7052, a borosilicate glass which is ground into a powdered frit and pressed into performs along with a binding agent. Many similar glasses exist that are made for forming matched, hermetic seals with Kovar. This method proved successful, but not without some difficulties. While the borosilicate glass does form a strong bond with the silicon and Kovar, the baseline process for making a seal is not reliable and the joint is limited in strength. Glass sealing to Kovar is well understood and has been widely used for decades, but glass sealing a Kovar tube to silicon for fluidic connections is a novel approach and previously had not been thoroughly tested.

1.3.2. **Process of Making a Glass Seal**

Glass seals are typically used to make hermetic seals for pins in electronic packages. There are two types of seals commonly used: a compression seal and a matched seal. A compression seal is formed when a pin is passed through a hole in metal and sealed with glass so that the metal will contract more during cooling and leave the glass with a residual
compressive stress. This type of seal does not require any chemical bond between the metal and the glass and allows for many different metals to be used, including steel. The disadvantage is that the geometry of the connection is limited because the glass must be inside the hole. This type of bond is not possible for microengine applications because silicon has a lower coefficient of thermal expansion than the glass and would induce a tensile residual stress in the glass, possibly causing brittle fracture.

A matched seal, in contrast, relies on the glass and metal having matched expansion and a chemical bond. Kovar was specifically developed to match the expansion of a family of Pyrex glasses. The process of making a matched seal begins by growing a layer of oxide on the metal in a belt furnace using a reducing atmosphere. The oxide must be grown slowly and remain thin so that it does not separate from the metal or become porous. The glass is melted by the furnace and forms a bond with the oxide layer on the metal. The strength and reliability of the matched seal is enhanced by the incorporation of oxide material into the glass, which forms a true hermetic seal. The advantages of this method are that bonds can be formed in many different configurations and the seal will remain gastight over a wide range of temperatures. The chief disadvantage is that the materials that can be used are quite limited.

The baseline process for making metal-to-glass-to-silicon bonds in the present work is derived from the methods used to make matched metal-to-glass bonds. A fixture is made from carbon that holds the Kovar tubes in place over the silicon die. The carbon must be machined carefully to allow room for the glass to flow and to slide off easily over the tubes.
after the bond is formed. The silicon die is placed on a flat section of carbon and the glass preforms are carefully put in position over their respective holes. The carbon fixture is held in place over the preforms while the tubes are lowered through the fixture and preforms into the die. Once all of the tubes are in place, the fixture is lowered until it rests on the preforms, as shown in Figure 1-7. The entire assembly is run through a furnace using an exothermic, propane-rich, reducing atmosphere at 1038°C for 15 minutes. The assembly then passes through cooling chambers for two hours before it is returned to room temperature.

1.4. Motivation and Outline of Thesis

The motivation of this research is that strong and reliable packaging is needed for all microengine and microrocket devices. The microrocket places significant loads on the fluidic connections during operation, and all packaged devices need strength for robustness to handling loads. Reliable packaging is also critical because it is the last step in the fabrication process and affects the yield more than any other part of the process. Glass sealing proved to be the method of choice for making fluidic connections to silicon, but there are several factors that limit the strength and reliability of glass seals. Fracture of the joints is observed to be either through the glass or through the silicon, but not along the
bond between the glass and silicon. Because it is a brittle material, the strength of the glass is limited by the size of the largest flaw within it. Fracture in the silicon appears to be initiated at the surface either due to the obtuse wetting angle (which is similar to a crack), the residual stress from thermal expansion mismatch, or a combination of the two. Glass-to-silicon bonds and the processing factors that control them must be better understood to develop packaging that is both strong and reliable for the microrocket and microengine.

This thesis explores how the MIT Microengine and related devices are packaged and what steps can be taken to improve the strength and reliability of the packaging. Chapter Two begins by detailing why voids, wetting angle, and residual stress limit the strength of glass-to-silicon seals. This chapter also presents the results of experiments conducted to determine how processing parameters affect these strength-limiting factors. Chapter Three details the direct testing of metal-to-glass-to-silicon joints to determine their strength and reliability. The results of joints formed using the baseline process are compared to results of joints formed using the improved process suggested in the previous chapter. Chapter Four discusses the options available for making connections to macro-scale components on the test bench and other packaging items, including inlets and exhaust ports. Chapter Five is a discussion of all the results and the adaptation of microengine packaging for applications. Chapter Six is devoted to conclusions and recommendations for future work.
Chapter 2: Bond Strength Limitations

Failure in the glass joints is observed to occur either by fracture through the glass or through the silicon, but not along the bond line between the glass and the silicon. The glass has good adhesion to the silicon, so the strength of the joint is limited only by the strength of the two materials involved: single crystal silicon and borosilicate glass. This chapter presents the evidence of failure from voids, wetting angle, and residual stress, as well as the sources of these problems and experimental results suggesting which processing parameters affect them. Section 2.1 discusses the contribution of voids to fracture through the glass. The contributions of the wetting angle and residual stress to fracture through the silicon are examined in sections 2.2 and 2.3 respectively. Section 2.4 presents the results of an experiment to determine which processing parameters affect voids and wetting, and how the strength of joints can be improved by adjusting these parameters.

2.1. Voids

The glass used in making metal-to-glass-to-silicon bonds is a brittle material, and its strength is therefore limited by two factors: the fracture toughness of the glass, and the size of the largest flaw in the glass. The fracture toughness of the material is intrinsic and cannot be varied by changing process parameters in the furnace. Therefore, the limiting factor in the strength of the glass is the size of the largest flaw, which can be controlled.
2.1.1. Evidence of Strength Limited by Voids

In joints that fractured through the glass, spherical voids were observed to be present along the fracture surfaces in every case. Strength tests were conducted in bending and in tension on sample joints over a range of temperatures. Figure 2-1a shows the fracture surface of a joint that failed through the glass. In general, joints that fractured higher in the glass (farther from the silicon surface) were stronger. Joints that failed in the glass near the silicon surface were weaker, as Figure 2-3 (at the end of this section) demonstrates.

![Figure 2-1: a) The fracture surface and b) cross-section of a glass joint](image)

Cross-sectioning several joints revealed that voids were present in the glass of every joint. (Appendix A details the process of cross-sectioning.) Figure 2-1b shows that these voids varied in size and location, some being as large as 20% of the preform thickness. The picture also reveals that most of the voids were concentrated along the bottom of the glass, near the silicon. This finding corresponds to the strength test results in Figure 2-3 and indicates that strength is limited due to the presence of voids.
2.1.2. **Source of Voids**

The voids are caused by the manufacturing process of the preforms. The preforms are made from a glass frit (powder), which is packed in a mold and pressed under heat. Several preforms were cross-sectioned prior to bonding, as shown in Figure 2-2, and revealed small voids uniformly distributed throughout the preform corresponding to the free volume in the packing of the glass particles. These small voids coalesce into larger voids in the furnace as the bond is being made. The voids take on a spherical shape in the molten glass to minimize their surface area.

![Figure 2-2: Cross-section of a glass preform before bonding](image)

*Figure 2-2: Cross-section of a glass preform before bonding*
2.2. Wetting Angle

The angle of contact, or wetting angle, between the glass and the silicon can affect the strength of a joint, especially when fracture occurs through the silicon. The wetting angle is measured as shown in Figure 2-4. An acute wetting angle is evidence of good wetting of the glass on the silicon, and an obtuse wetting angle indicates that the glass does not wet
the silicon. Good wetting is not necessary for the two materials to form an intimate bond, but it can increase the strength of the bond and the ease with which it is made. The baseline process for making a metal-to-glass-to-silicon bond consistently produces an obtuse wetting angle between the silicon and glass. This section explores the evidence for a reduction in strength due to large wetting angles and the causes of an obtuse wetting angle.

2.2.1. Wetting Angle as a Crack

An obtuse wetting angle, as shown on the right in Figure 2-4 and in the photograph in Figure 2-5, begins to look like a sharp crack as the angle $\theta$ becomes larger. When load is applied to the joint from handling or high pressure, the joint will be in tension at least on one side. This causes the circumferential “crack” formed by the poorly wetting glass to open in Mode I and results in a stress concentration around the edge of the joint in both the glass and the silicon. Mode II loading of the wetting angle crack occurs as the bond begins to cool after being formed and the glass contracts more than the silicon, leaving the two materials with a residual shear stress along the bond line. This will be discussed further in section 2.3. The wetting angle crack can also be loaded in Mode III by placing a torque on the joint. This can only occur in the microrocket due to the configuration of its macro connections, which will be presented in Chapter 4.

![Figure 2-4: Measurement of the wetting angle](image)
Mode I loading of the crack is the most important because it is observed to limit the strength of the joint. Figure 2-6 depicts an example of how fracture occurs through the silicon. In this example the joint was loaded in bending. The large wetting angle of the glass caused a stress concentration in the silicon at the edge of the bond, in addition to the residual stress already in the silicon. The fact that the crack grew through the silicon instead of along the bond line is evidence that the fracture toughness of the bond between the glass and the silicon is large relative to the fracture toughness of silicon. While this indicates that there is good adhesion between the glass and silicon, it is also proof that the wetting angle is a source of failure and reduced strength.
2.2.2. *Determination of Wetting Angle and Adhesion*

The wetting angle is determined by the surface energies between the different materials and the atmosphere in the furnace as the bond is made. The wetting angle is given by the equation:

\[ \cos \theta = \frac{\gamma_{sv} - \gamma_{sl}}{\gamma_{lv}} \]

where \( \gamma_{sv} \) is the surface energy between the silicon and the atmosphere, \( \gamma_{sl} \) is the surface energy between the silicon and the glass, and \( \gamma_{lv} \) is the surface energy between the glass and the atmosphere.\(^{11}\) For wetting to occur, \( \gamma_{sv} \) must be greater than \( \gamma_{sl} \), which results in an acute angle \( \theta \). Both a smaller \( \gamma_{lv} \) and a greater difference between \( \gamma_{sv} \) and \( \gamma_{sl} \) will result in improved wetting.

The adhesion of the glass on the silicon is governed by the equation:

\[ W_{sl} = \gamma_{sv} + \gamma_{lv} - \gamma_{sl} \]

where \( W_{sl} \) is the work of adhesion and a larger \( W_{sl} \) represents better adhesion.\(^{12}\) For the best adhesion, it is desirable to minimize \( \gamma_{sl} \) and maximize \( \gamma_{sv} \) and \( \gamma_{lv} \). This conflicts slightly with the criteria for improving wetting because a small \( \gamma_{lv} \) aids wetting and a large \( \gamma_{lv} \) aids adhesion.

The table below shows the six different possibilities for the relative magnitudes of surface energies. The baseline process produces a bond between the glass and silicon that is not
wetted but has adequate adhesion, corresponding to the fourth set of inequalities. Improved wetting can be achieved by raising $\gamma_{sv}$ and lowering $\gamma_{si}$.

Table 2-1: Wetting and adhesion due to surface energies

<table>
<thead>
<tr>
<th>Surface Energies</th>
<th>Wetting</th>
<th>Adhesion</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma_{sw}&gt;\gamma_{sv}&gt;\gamma_{sl}$</td>
<td>$0&lt;90^\circ$</td>
<td>Moderate</td>
</tr>
<tr>
<td>$\gamma_{sv}&gt;\gamma_{sv}&gt;\gamma_{sl}$</td>
<td>$0&lt;90^\circ$</td>
<td>Strong</td>
</tr>
<tr>
<td>$\gamma_{sv}&gt;\gamma_{sv}&gt;\gamma_{sl}$</td>
<td>$0&lt;90^\circ$</td>
<td>Strong</td>
</tr>
<tr>
<td>$\gamma_{sv}&gt;\gamma_{sv}&gt;\gamma_{sl}$</td>
<td>$0&gt;90^\circ$</td>
<td>Moderate</td>
</tr>
<tr>
<td>$\gamma_{sv}&gt;\gamma_{sv}&gt;\gamma_{sl}$</td>
<td>$0&gt;90^\circ$</td>
<td>Weak</td>
</tr>
</tbody>
</table>

Surface energy varies with several different factors that can be controlled. An increase in temperature during the bonding process will result in a decrease in surface energy for both the glass and the silicon, but not necessarily by the same amount. Changing the composition of the gases in the atmosphere can also affect the surface energy by adding or reducing contamination from adsorbed gases. For a crystalline material, such as silicon, bonding to a plane with a lower density of atoms results in a higher surface energy for the silicon.¹³

Three different crystal orientations are possible for silicon, depending on how the crystal was grown and how the wafers were cut from the boule. The ratios of the atomic density, number of dangling bonds, and bond density for each orientation are shown in the table below.¹⁴ The {100} orientation is preferred for making glass-to-silicon bonds because it has the lowest density of atoms, the highest number of dangling bonds, and the highest bond density—all of which contribute to a higher surface energy and better wetting.
Similarly, the \( \{110\} \) plane is the worst for glass-to-silicon bonds. The microengine and microrocket use \( \{100\} \) orientation wafers.

**Table 2-2: Properties of silicon in different orientations**

<table>
<thead>
<tr>
<th>Orientation</th>
<th>( {100} )</th>
<th>( {110} )</th>
<th>( {111} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Density</td>
<td>1</td>
<td>1.414</td>
<td>1.155</td>
</tr>
<tr>
<td>Number of Dangling Bonds</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Bond Density</td>
<td>1</td>
<td>0.707</td>
<td>0.577</td>
</tr>
</tbody>
</table>

### 2.3. Residual Stress

The contribution of residual stress to fracture through the silicon is evident by cracks that exist in the silicon beneath the surface of the glass before any load is applied. Figure 2-7 shows cross-sections of a glass preform bonded to a piece of silicon through two different planes. The visible cracks are due to the thermal stresses created in the silicon when the bond cools. The thermal expansion coefficients of the borosilicate glasses used are close to that of Kovar—which is higher than that of silicon. After the bond between the glass and the silicon is formed at 1038°C, the glass contracts more than the silicon as it returns to room temperature. This leaves a residual stress in the silicon near the edge of the glass, which is where cracks are visible.

*Figure 2-7: Stress cracks in the silicon beneath the glass-silicon interface*
Cracks form along the weaker \{111\} plane in the silicon, extending away from the glass-silicon interface at an angle of 54.7° and running at 90° angles as viewed from below. Some cracks travel parallel to the glass-silicon interface along the \{100\} plane, as shown in Figure 2-7, and could cause leaks in the joints before any significant load or pressure is applied. Using a lower expansion glass to bond to the silicon can reduce the residual stress in the joints. However, using a glass with a thermal expansion coefficient too close to silicon's might result in cracks in the glass near the glass-Kovar interface, creating an additional problem. Figure 2-8 shows the relative thermal expansions of silicon and of each of the glasses used. Other properties of the glasses used are listed in Appendix B.

![Figure 2-8: Expansion of glasses and silicon from 25°C](image)
2.4. Experimental Results

An experiment was conducted to determine which processing factors affect the size of voids and the wetting angle. Glass preforms with a 4 mm outer diameter were bonded directly to pieces of silicon in a sessile drop configuration, as shown in Figure 2-9. After bonding, each piece was potted in epoxy, cross-sectioned, and viewed under a microscope to determine the size of the largest voids and the wetting angle. The relative size of the voids in the glass was estimated from the voids visible in the cross-section plane and was not measured directly. Likewise, the wetting angle was not measured directly from the cross-section because any misalignment of the cross-section plane through the center of the test sample would alter the angle observed from the actual wetting angle. However, wetting or non-wetting of the glass on the silicon was determined from the cross-sectioned test samples due to the fact that if the observed angle is acute the actual wetting angle is acute and if the observed angle is obtuse the actual wetting angle is obtuse, regardless of the degree of misalignment of the cross-section plane. The justification for this conclusion is explained further in Appendix C.

Figure 2-9: Sessile drop configuration with glass on silicon
Five parameters were varied in the experiment: the type of glass used, the surface condition of the silicon, and the atmosphere, time, and temperature in the furnace. Three glasses were used on three different surface conditions of silicon for each batch of test samples. Each batch was run with one variation from the baseline furnace conditions, resulting in a total of nine batches.

The three glasses used in the experiment were Corning 7052, Electroglass 3200 (EN-1), and Fusite K. GBC Inc. of Latrobe, Pennsylvania, manufactured the Corning and Fusite glass preforms. The EN-1 preforms were obtained through Thunderline Z and manufactured by Electro-Glass of Mammoth, Pennsylvania. The surface conditions of the silicon used were the polished side of a wafer, the unpolished side of a wafer, and an oxidized wafer.

Five different atmospheres, three temperatures, and three times were used in varying the furnace conditions. The exothermic, propane-based atmospheres used are labeled as 900/38, 900/42, and 900/48, which represent the volume ratio of air to propane. In addition to these atmospheres, both wet nitrogen and dry nitrogen reducing atmospheres were used. The three temperatures used were 1010°C, 1038°C, and 1066°C. The time spent in the furnace is controlled by the belt speed, which was set at 5.1 cm/min, 10.2 cm/min, and 15.2 cm/min, corresponding to 22.5 min, 15 min, and 7.5 min in the furnace respectively.
2.4.1. Factors That Affect Voids

The type of glass used was found to affect the size and number of voids within the glass more than any other factor. The table at the end of this section is a summary of the results that led to this conclusion. Because the voids present in the glass originate from the manufacturing process, it is not surprising that glass manufactured by different companies yields different numbers and sizes of voids. The Corning 7052 preforms made by GBC showed the least number and smallest voids overall. The Fusite K preforms, also made by GBC, exhibited the largest number and size of voids.

The pictures in Figure 2-10 show an example of the voids found in each type of glass, with Corning 7052 on the left, EN-1 in the middle, and Fusite K on the right. The EN-1 preforms revealed a microstructure within the glass that is not found in either of the other preforms. It is believed that this is due to the different processing used by Electro-Glass, namely a spray-drying tower that produces a more consistent raw material before the powder is pressed into preforms.

Figure 2-10 Cross-section of each type of glass showing voids
Table 2-3: Size of voids for given process parameters

<table>
<thead>
<tr>
<th>Glass</th>
<th>Surface</th>
<th>Baseline Process</th>
<th>Atmosphere (@ 10.2 cm/min &amp; 1038°C)</th>
<th>Time (@ 900/42 and 1038°C)</th>
<th>Temperature (@ 900/42 and 10.2 cm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-7052</td>
<td>Smooth</td>
<td>Small</td>
<td>Small</td>
<td>Small</td>
<td>Small</td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>Small</td>
<td>Small</td>
<td>Small</td>
<td>Small</td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>Small</td>
<td>Medium</td>
<td>Small</td>
<td>Small</td>
</tr>
<tr>
<td>EN-1</td>
<td>Smooth</td>
<td>Medium</td>
<td>Small</td>
<td>Small</td>
<td>Medium</td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>Small</td>
<td>Small</td>
<td>Small</td>
<td>Medium</td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>Large</td>
<td>Small</td>
<td>Medium</td>
<td>Medium</td>
</tr>
<tr>
<td>Fusite K</td>
<td>Smooth</td>
<td>Small</td>
<td>Large</td>
<td>Medium</td>
<td>Large</td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>Small</td>
<td>Medium</td>
<td>Large</td>
<td>Small</td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>Small</td>
<td>Small</td>
<td>Medium</td>
<td>Small</td>
</tr>
</tbody>
</table>

2.4.2. Factors That Affect Wetting Angle

Wetting was found to depend on the type of glass used and the atmosphere in the furnace. The table at the end of this section shows all of the results for wetting obtained through the experiment. Of the three types of glasses tested, Fusite K was found to wet silicon more readily than any other. EN-1 also wets the silicon under some conditions, and Corning 7052 only wet the silicon in one observed instance.

The most important factor found to affect wetting was the atmosphere in the furnace. While a reducing atmosphere is necessary for the formation of the bond between the glass and the Kovar and the glass and the silicon, a less reducing atmosphere than the baseline
process was found to be more conducive to wetting. The 900/38 and wet nitrogen atmospheres in particular performed well because they are less reducing and have more water vapor in them, which encourages more oxidation. The disadvantage to using these atmospheres is that if a brazing operation is occurring simultaneously, the braze material will flow less.

Table 2-4: Wetting for given process parameters

<table>
<thead>
<tr>
<th>Glass</th>
<th>Surface</th>
<th>Baseline Process</th>
<th>Atmosphere (@ 10.2 cm/min &amp; 1038°C)</th>
<th>Time (@ 900/42 and 1038°C)</th>
<th>Temperature (@ 900/42 and 10.2 cm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>900/42 10.2 cm/min 1038°C</td>
<td>900/48 900/38 Wet N Dry N 5.1 cm/min 15.2 cm/min 1010°C 1066°C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C-7052</td>
<td>Smooth</td>
<td>Yes No No No</td>
<td>No</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>No No No No</td>
<td>No No No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>No No No No</td>
<td>No No No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td>EN-1</td>
<td>Smooth</td>
<td>No Yes No No</td>
<td>No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>No No Yes No</td>
<td>No No No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>No No Yes No</td>
<td>No No No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fusite K</td>
<td>Smooth</td>
<td>Yes No No No</td>
<td>No No</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rough</td>
<td>No No No Yes No</td>
<td>No Yes</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Oxidized</td>
<td>No No Yes Yes Yes No No</td>
<td>No No</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.5. Summary

Voids, wetting angle, and residual stress limit the strength of glass joints by initiating cracks and creating stress concentrations in both the silicon and the glass. Voids were found to be dependent only on the type of glass used and the manufacturing process of the preform. Wetting, however, was found to depend on both the glass and atmosphere used.
Other factors, such as the belt speed and temperature of the furnace and the surface condition of the silicon, were not found to have a significant impact on voids and wetting.
Chapter 3: Strength Testing

3.1. Strength Testing Procedure

The strength of metal-to-glass-to-silicon bonds was tested directly to determine both the strength of the joints in bending and tension and the reliability of the process. These tests were conducted over temperatures ranging from 300K to 1000K. Single tube samples were made for testing purposes. The silicon substrate used for the single tube samples was 1 cm by 1 cm and consisted of a 450 micron thick layer of silicon, with a 900 micron diameter hole etched through the center, bonded to a second 450 micron layer of silicon with no hole. The silicon die were then bonded to straight, 3 cm long pieces of Kovar tube using a glass preform, as shown in Figure 3-1.

![Figure 3-1: Single tube test sample](image)
3.1.1. **Bending Tests**

**Experimental Setup**

Bending tests simulate the loads a joint is likely to experience from handling, thrust (in the case of the microrocket), and the thermal expansion and contraction of the macro connection assembly. The experimental apparatus used for conducting the bending tests on single tube samples is shown in Figure 3-2a. An MTS mechanical testing machine was used because it has a furnace capable of the temperatures needed and position control to an accuracy of 0.1 mm. A 1100 N (250 pound) load cell was installed that was capable of measuring forces as small as 0.05 N.

*Figure 3-2: a) Testing machine and furnace  b) clamping fixture and loading rod*
The test specimen was clamped to a steel block and held so that the tube protruded horizontally, as shown in a close-up photograph of the test setup in Figure 3-2b. A steel retaining plate with a hole in the center was used to clamp the silicon die to the steel block and allow the tube of the test specimen to pass through. A loading rod, with a sharp tip, was then lowered into contact with the protruding tube and applied a measured force until the joint failed. The moment was then calculated by multiplying the force applied by the distance along the tube from the joint to the tip of the force rod, which was 2.15 cm.

Limitations of Test Results

This method of testing was limited primarily by the accuracy of the load cell and the modes of failure of the joint. The signal received from the load cell contained noise that was significant compared to the level of accuracy desired. Figure 3-3 shows a graph of the load measured over time when no load was applied. The load cell reading has noise on the order of 0.05 N, which is 10% of the average load cell reading from joints tested in bending.

Figure 3-3: Noise in load measurement
Bending tests were also limited by the mode of failure of the joints. Because both glass and silicon are brittle substances, their strengths can vary considerably. In some instances the joint failed because of fracture through the glass or the silicon. In other instances, particularly at higher temperatures, the Kovar tube or glass yielded and prevented any higher load from being applied. Therefore, yielding was defined to be a failure for the purposes of strength testing. Wherever possible the manner by which a joint failed is noted.

3.1.2. Tension Tests

Experimental Setup

Tensions tests were conducted to simulate the loads experienced from high pressures in the tube and from handling. The same basic setup was used for tension tests as for bending tests, except a wedge gripping device was used in place of the loading rod. The wedge grips were used to apply a tension load to the tube without crushing it or applying a moment. The design of the wedge grips is shown in Figure 3-4. The same steel block was used to hold the silicon end of the single tube samples, and was positioned so that the tube was vertical instead of horizontal. Alignment was checked before each run and adjusted by moving the test sample on the steel block. The hinge above the grip also assisted alignment by correcting for tilt in the installation of the grip and/or steel block.
Limitations of Test Results

Tension tests do not exactly replicate the loads experienced from high pressure because the boundary conditions and failure modes are not identical. The packaging is considered to fail in a device when it leaks, but in this experiment leaks could not be measured. Instead, failure was defined as the separation of the tube from the device. These tests did, however, provide a basis for comparison of the strength and reliability of different glass sealing fabrication methods.
3.1.3. **Definition of Baseline and Improved Processes**

For the purpose of comparison, two different processes were used to make the test samples used in this experiment. The process labeled the “baseline process” in this Chapter refers to the process employed by Olin-Aegis using a Corning 7052 glass preform and a dry Nitrogen atmosphere. The “improved process” refers to Thunderline Z’s method of making the same test samples using EN-1 glass and a Propane 900/42 atmosphere. This process was chosen based on the results of the sessile drop tests presented in Chapter 2. A 900/38 atmosphere would have been preferred for the improved process, but a 900/42 was used instead out of error. All other factors, such as the surface condition of the silicon and the time and temperature in the furnace, were identical for the two processes.

3.2. **Baseline Process Strength**

Single tube samples made using the baseline process were tested in bending and tension at a variety of temperatures. The bending tests revealed a large variation in the strength of the joints, as is evident in Figure 3-5. This plot shows the load applied versus the deflection of the tube for tests at room temperature. While most of the joints failed due to brittle fracture of the glass or silicon, the top line (7/7 #5) represents a joint that did not. The failure mode of this sample was yielding of the tube, which eventually prevented any more load from being applied.
As the temperature was increased and the yield stress of Kovar dropped, yielding occurred more often as the cause of failure. Figure 3-6a is a joint that failed due to yielding of the tube. At temperatures above 800K, yielding of the glass became a cause of failure, which is shown in Figure 3-6b. The chart in Figure 3-7 is a summary of the failure strength of each of the joints tested. The strength of the joints decreased as the temperature increased, primarily due to the limitation of the Kovar and the glass yielding. The mean strength in

![Figure 3-6: a) Tube yielding and b) glass yielding under bending load](image-url)
bending at room temperature was 0.0115 Nm with a standard deviation of 0.0057 Nm. This equates to a nominal stress in the Kovar tube of 304 MPa, which is close to the yield stress of Kovar (345 MPa).\(^\text{16}\)

![Figure 3-7: Baseline process bending test results as a function of temperature](image)

The tests in tension produced different results. In tension, the stress in the Kovar tube is minimal, and therefore failure occurs only through brittle fracture of the glass or the silicon. Figure 3-8 below is a summary of the strength of the joints in tension at various temperatures. No strong correlation is apparent between strength and temperature over the range of temperatures explored in this experiment, but it is expected that at temperatures above 800K the yield stress of the glass would become a limiting factor.
The scatter in the data is due to the brittle nature of the materials being tested and represents the unreliable nature of the joints produced using the baseline process. The strength varies from 1 N, which occurred several times at various temperatures, to 27 N, which occurred only once at 550K (this particular joint is pictured in Figure 2-3c). A total of twenty specimens made using the baseline process were tested at room temperature. The mean strength of the room temperature tests was 8.65 N, with 95% confidence limits of ±2.25 N. The standard deviation of this sample was 5.13 N. This has important consequences for packaging because a typical device has around a dozen connections, and the failure of only one joint causes the failure of the entire device.

![Figure 3-8: Baseline process tension test results](image-url)
While the tension tests do not perfectly represent high pressure loading of the joints, there is a rough correlation. One Newton of load applied in tension is equivalent to the tension load in the joint due to 15.6 atmospheres of pressure, calculated from the equation:

\[ F = P \pi r^2 \]

where \( F \) is the tensile force on the tube, \( P \) is the pressure inside the tube, and \( r \) is the inner radius of the tube. This means that the joints tested were capable of containing pressures from 15 atm to 440 atm, with an average of 134 atm and a standard deviation of 80 atm.

Figure 3-9 shows the cumulative distribution function for the sample data, a normal distribution, and the best-fit Weibull distribution. Assuming a normal distribution, which is justified in this case, it can be predicted that 33\% of the joints will be strong enough to handle the pressures of the microrocket and 86\% will be strong enough for the microengine, with 95\% confidence. Each device has multiple connections, and if one connection fails the entire device will fail—making the chance of all eleven connections on the microrocket being strong enough to withstand 125 atm of pressure extremely small, between 0.05\% and 0.0005\%. The chance of all sixteen connections working on the microengine using the baseline process is at least 9\%, which is still unacceptably low.
3.3. Improved Process Strength

Single tube test samples were fabricated using the improved process described in Chapter 2 and were tested in tension at room temperature to compare to the baseline process tests. The samples were made at Thunderline Z using EN-1 glass preforms and run through a furnace at 1038°C using an exothermic, propane-rich atmosphere with a mixture of 900/42. The silicon surface on the test samples was smooth and free of oxide, the same as the baseline process.

Twenty samples were tested in tension at room temperature using the same test setup as before. The results of these tests are shown in Figure 3-10 along with the room temperature results from the baseline process. The improved process has a mean strength
of 13.11 N with a 95% confidence interval of ±1.32 N and a standard deviation of 3.02 N. This represents a 52% increase in strength over the baseline process and a 41% improvement in the reliability of the process, as measured by the reduction in the standard deviation.

Figure 3-10: Improved process test results

Figure 3-11 shows the cumulative distribution function of the improved process sample compared to both a normal distribution and a Weibull distribution. Assuming a normal distribution, the probabilities of a joint being strong enough for the micro-rocket and the microengine are 86% and 99.9% respectively, with 95% confidence. Therefore, the probability of all eleven joints working on a micro-rocket is at least 19%, significantly higher than with the baseline process but still a limiting factor for the yield of operational devices. The chance of all connections working on the microengine is at least 98%.
3.4. Summary

Strength testing of single tube test samples revealed that the baseline process produces joints that are unreliable in strength for both the microrocket and microengine applications. Specimens made using an improved process were tested and showed a significant increase in strength and reliability. It is expected that both the mean strength and standard deviation would improve further using the correct atmosphere (900/38 instead of 900/42) for the improved process samples.
Chapter 4: Macro Connection Configuration

Macro connections form the interface between the tubes and wires exiting a device and the supplies and instruments in the lab. As part of the packaging, macro connections must be gas-tight, capable of the range of temperatures encountered during operation, reliable in manufacturing, and resilient to handling loads. The macro connections also serve to protect the device from handling. Many options exist for making macro connections, each with advantages and disadvantages. These options are examined in section 4.1. Several different devices have been successfully packaged and tested to date using different macro connection configurations, and the results of these experiences are discussed in section 4.2. Other packaging items not previously presented that can be considered part of the macro connection configuration, such as the inlet, exhaust, and electrical connections, are discussed in section 4.3.

4.1. Options For Making Macro Connections

4.1.1. Options Available

Many options are available for making macro connections on the test bench. These options are narrowed into five distinct categories for the purpose of this discussion—other methods exist that are variations on or combinations of the ones presented, but not all can be catalogued within the scope of this thesis. The five divisions of macro connection options are labeled fixed plate, piston-bore, U-tube, slip-fit tubing, and direct connections.
A fixed plate configuration is shown in Figure 4-1. Each of the tubes emerging from the device is brazed into through holes in a single metal plate. The metal plate is larger than the device and has either threaded or clearance holes in the corners for attachment to a manifold. Because the metal plate is thermally grounded to the manifold and the heat flux from the Kovar tubes is minimal, the metal plate will remain cool and o-rings can be used to seal each fluidic connection in the metal plate to its corresponding supply in the manifold.

![Figure 4-1: Fixed plate configuration](image)

The piston-bore configuration, shown in Figure 4-2, is designed to relieve the axial thermal expansion of the Kovar tubes. In this configuration each tube has a piston brazed to it. These pistons are then inserted into a large manifold with a bore and side supply hole for each piston. The pistons are sealed within the bore with grease and are free to slide in and out as the tubes expand and contract by different amounts. Therefore, the device does not see any loads due to the axial thermal expansion of the tubes.
Threaded Holes For Connections To Supplies & Instruments

Pistons Brazed To Kovar Tubes

Silicon Device

Bores For Pistons To Slide Freely In

Metal Plate With Through Holes

Figure 4-2: Piston-bore configuration

U-tubes can also be used for macro connections to allow thermal strain in multiple directions. U-shaped tubes are glass sealed onto the silicon device, as shown in Figure 4-3. The other ends of the u-shaped tubes are brazed into a metal plate, which attaches to a manifold similar to the one for the fixed plate configuration.

Figure 4-3: U-tube configuration
The slip-fit tubing configuration is the most basic form of macro connections. It does not require any sealing, brazing, or other processing beyond the glass sealing needed for basic packaging. Rubber tubing simply slips over the Kovar tubes emerging from the device. These rubber tubes connect directly to their respective supplies and instruments in the lab.

Direct connections are similar to slip-fit tubing because connections are made individually to each tube. In this configuration, a fitting is made for each connection. Each fitting is brazed to its respective tube and allows a supply tube to be attached directly to the Kovar tubes.

4.1.2. Comparisons Between Options

Each of the design options listed above has distinct advantages and disadvantages. Table 4-1 compares each of the options over ten different criteria that are important for the packaging of the microengine and microrocket. An explanation of the importance of each of these criteria is provided below:

- **Uniformity of the glass joint** is the ability to form a glass joint that is symmetric with uniform shaping from the carbon fixture.

- **The ease with which a carbon fixture can be made** determines the turn-around time needed when changes are made to the packaging of the device (e.g. another pressure tap is added or removed).

- **Resistance to handling loads** is the ability of the macro connection configuration to shield the packaging from large loads when the device is being transported or connected to instruments.
• **Compliance to thermal strains** is how well the macro connections allow the device to expand and contract without causing high stresses.

• **The resistance to leaks** is a measure of how many seals are needed and the reliability of each of the sealing processes.

• **The ease with which the parts for macro connections are manufactured and reused** determines the time needed to package each device and the turn-around time when changes are made.

• **Batch processing** for the packaging is the ability to make the packaging in large numbers simultaneously to be compatible with micro-fabrication.

• **Accessibility of the device** is necessary to monitor operation and connect certain instruments.

• **The ability to withstand high pressures** is only important for the microrocket since it will reach pressures many times greater than other devices.

• **The capacity to carry load from thrust** is also unique to the microrocket since it will produce approximately 20 N of thrust perpendicular to the fluidic connections.
### Table 4-1: Macro connection design options

<table>
<thead>
<tr>
<th></th>
<th>Uniformity of Glass Joint</th>
<th>Easy To Make Fixture</th>
<th>Resistance To Handling Loads</th>
<th>Compliance To Thermal Strain</th>
<th>Resistance To Leaks</th>
<th>Easy To Manipulate and Reuse</th>
<th>Ability For Batch Processing</th>
<th>Accessability of Device</th>
<th>Used Carrying Capability</th>
<th>High Pressure Capability</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Fixed Plate</strong></td>
<td>Even pressure applied to glass preform.</td>
<td>Thin fixture will break away after glass sealing and brazing.</td>
<td>All tubes are brazed to a fixed metal base.</td>
<td>Does not allow for thermal strains in manufacturing or operation.</td>
<td>Braized joints are easy to make and won’t leak.</td>
<td>Needs many metal plates and one mandrel.</td>
<td>All bonds can be formed in one pass through the furnace.</td>
<td>Will have restricted access from the top.</td>
<td>Stronger glass seals due to macro connection configuration.</td>
<td>Will need a fixture to limit deflection of device.</td>
</tr>
<tr>
<td><strong>Piston-Bore</strong></td>
<td>Even pressure applied to glass preform.</td>
<td>Fixture can slip over tubes, but must be made to tight tolerances.</td>
<td>All tubes will go through a plate that helps resist bending moments.</td>
<td>Tubes expand and contract freely without introducing stresses.</td>
<td>Gap between piston and bore will leak, along with braised joints.</td>
<td>Bore and piston must be made to tight tolerances, many pistons needed.</td>
<td>Pistons will need to be attached individually.</td>
<td>Will have restricted access from the top.</td>
<td>At higher pressure the piston-bore gap will leak more.</td>
<td>Will need a fixture to limit deflection of device.</td>
</tr>
<tr>
<td><strong>U-Tube</strong></td>
<td>Univar pressure applied to glass preform due to complex fixture.</td>
<td>Must make a special fixture to accommodate bent tubes.</td>
<td>Fits U-tubes will not be protected.</td>
<td>Especially free to expand and contract with minimal stresses.</td>
<td>Braized joints are more difficult to make and may leak or drop.</td>
<td>Complex geometry with multiple fixtures made (set top).</td>
<td>Requires multiple passes through the furnace.</td>
<td>Will have restricted access from the bottom and top.</td>
<td>Weaker glass seals due to extra loads on glass seals from U-tube design.</td>
<td>Torque and bending moment introduced to the glass joint from thrust.</td>
</tr>
<tr>
<td><strong>Slip-fit Tubing</strong></td>
<td>Even pressure applied to glass preform.</td>
<td>Fixture can slip over tubes, but must be made to tight tolerances.</td>
<td>Process of attaching slip-fit tubing will introduce unknown loads.</td>
<td>Flexible tubing allows to freely expand and contract.</td>
<td>May leak at tube interface under high pressure.</td>
<td>Needs many metal plates and one set of tubing.</td>
<td>Slip-fit tubes must be secured individually.</td>
<td>Will have somewhat restricted access.</td>
<td>Will have somewhat restricted access.</td>
<td>Will have somewhat restricted access.</td>
</tr>
<tr>
<td><strong>Direct Fittings</strong></td>
<td>Even pressure applied to glass preform.</td>
<td>Fixture can slip over tubes, but must be made to tight tolerances.</td>
<td>Applied torque from tightening fitting must be counteracted effectively.</td>
<td>Somewhat more difficult to expand and contract with resistance from fittings.</td>
<td>Braized joints may be difficult to make and need many of them.</td>
<td>Small fittings may be difficult to make and need many of them.</td>
<td>Fittings will need to be attached individually.</td>
<td>Will have restricted access.</td>
<td>Will have restricted access.</td>
<td>Will have restricted access.</td>
</tr>
</tbody>
</table>

The shaded cells in the table represent unfavorable attributes, and the un-shaded cells represent favorable attributes. The fixed plate method emerged from this comparison as the best option available, with only one shaded cell. It provides protection for the device and packaging and is easy to make and use. The worst option that emerged from this comparison was the u-tube design, with all of its cells shaded except one. It requires considerable effort to design and manufacture the parts needed, and offers no protection for the device or the packaging. The u-tube design is a particularly poor choice because it
allows a twisting and bending moment to be applied to the glass seals, which results in unnecessary damage to the packaging from handling and operation.

4.1.3. **Impact On Glass Seals**

The macro connection configuration has a tremendous impact on the total strength of the packaging. The Kovar tubes emerging from the device create a long moment arm that causes even small loads to be transmitted to the glass joint as large bending moments. Packaging configurations that provide support for the free ends of the tubes, such as the fixed plate design, create a stiff structure that is more resistant to damage. Load applied to one tube is shared with its neighbors and the supporting structure. Configurations that allow tubes to remain free, such as the u-tube and direct fittings designs, are more prone to break from handling because each joint carries all of the load applied to it and is not helped by the strength of other joints.

Because the u-tube and direct fittings configurations allow torque to be transmitted through the tube to the joint, the apparent circumferential crack due to the obtuse wetting angle at the edge of the glass preform experiences Mode III loading. This adds an additional stress concentration to the stress fields already present in the silicon and the glass. The u-tube configuration is vulnerable to torsional loading when the device moves during handling or operation, as is the case with the microrocket when thrust is produced. The direct fittings configuration encounters torsional loading when a macro connection is being screwed into a fitting.
The macro connection configuration also determines what kind of carbon fixture can be used in the process of making the seal and how many times the device must pass through the furnace. The fixed plate design can be built in a single pass through the furnace with a thin, break-away carbon fixture. The brazing and glass sealing steps are accomplished at the same time, and the remaining carbon fixture left in-between is broken away without damaging the packaging. In contrast, the u-tube design needs two passes through the furnace, with a normal thickness carbon fixture. In the first pass the carbon fixture is put in place and straight Kovar tubes are glass sealed to the silicon device. Next, the fixture is removed and steel u-tubes are put in place to connect the Kovar tubes on the device to the metal plate. Finally, the device is run through the furnace a second time to braze the steel u-tubes to the Kovar tubes and metal plate. This process doubles the time and cost to package each device and can weaken the glass by allowing voids to further coalesce and the glass to re-flow.

4.2. Results From Intermediary Devices

After considering the advantages and disadvantages of each method for making macro connections, the turbocharger and micro-combustor groups elected to use the fixed plate design, while the microrocket group chose the u-tube design, and the recuperator group chose a direct connection design. These devices have been tested to various degrees and the results of the packaging are detailed below.

4.2.1. Micro-Combustor

The packaged micro-combustor is shown in Figure 4-4. A fixed plate configuration was used to connect the five tubes on the front and one tube on the back to the supplies and
instruments in the lab. Conventional o-ring seals connected the metal plate to a manifold, which hosted the final connections to fuel supplies, filtered air, and pressure transducers. All of the fluidic connections were made using 0.8 mm Kovar tubing, except one on the front that used 2 mm Kovar tubing. The carbon fixture was left in place between the metal plate and the device, providing an extra layer of insulation to increase the accuracy of temperature measurements. The exhaust tube was initially connected using epoxy, but later versions of the combustor used a glass-sealed exhaust tube. The combustor used a resistive loop of Kovar wire as an igniter, which was attached to the structure using a glass preform with two holes.

![Figure 4-4: Fully packaged micro-combustor, courtesy Amit Mehra](image)

Over a dozen micro-combustors have been successfully packaged and tested to date. Failures in the packaging have been limited and are primarily due to failures of the backside connections. The fluidic connection on the back is not supported in any way and is fragile during handling. A backside connection that needs to wrap around to braze into
the metal plate on the front, such as this one, creates additional complexity during packaging and should be avoided whenever possible. Attaching the exhaust tube with epoxy also proved difficult and caused the fracture of glass seals at times. Overall, however, the micro-combustor packaging proved to be reliable and robust during operation, even at high temperatures.

4.2.2. Microrocket

The microrocket was packaged using the u-tube configuration. Short Kovar tubes were glass sealed onto the device in the first pass through the furnace. In the second pass, steel u-tubes were brazed to the Kovar tubes and to a steel plate. A fully packaged microrocket is shown in Figure 4-5. A built-in igniter was not used in the demonstration rocket, but will eventually be needed.

![Fully packaged microrocket](image)

During the testing of the microrocket, the packaging encountered many difficulties, as expected. Braze material from the two braze joints flowed into the inside of some of the
tubes, restricting the flow and reducing the amount of cooling available for the motor. Some of the glass seals also leaked and fractured from handling and at high pressure. Solutions to these problems will be discussed in Chapter 5.

4.2.3. Turbocharger

A fixed plate configuration was used for the turbocharger, which has sixteen fluidic connections, an inlet on the front, and an exhaust tube and igniter on the back, as shown in Figure 4-6. Using a normal thickness fixture, as the micro-combustor used, proved difficult because the glass tended to stick to the carbon while trying to slide it off, usually resulting in one or more seals breaking from the excessive and uneven amount of force applied. A thin plate, in contrast, is much weaker and broke away without causing any damage. In addition, using a break-away fixture allowed both the braze and glass seals to be made in one pass through the furnace.

Figure 4-6: Fully packaged turbocharger dummy die
4.2.4. **Recuperator**

The recuperator used a direct fitting configuration. Six tubes were glass sealed onto the recuperator. Due to the larger size of the recuperator and the spacing between the tubes, two separate carbon fixtures were used. Each of the tubes then had a fitting individually brazed to it to connect to the supplies in the laboratory.\(^{17}\)

4.3. **Other Packaging Items**

4.3.1. **Inlet and Exhaust Ports**

The purpose of the inlet and exhaust connections on the microengine are to guide the air into the engine without disrupting the flow and to guide the hot exhaust gases away from the engine so that they do not overheat the structure. The inlet, shown below in Figure 4-7, was designed so that the boundary layer of the flow entering the engine would not separate while making the first 90° turn into the compressor. The inlet will slip into the inlet opening on the top of the engine, as was shown in Figure 1-1. The inlet will be made of Kovar and attached using a thin ring of glass.

*Figure 4-7: Isometric view of the inlet design*
The first generation microengine, however, will use a straight tube to connect to the inlet so that air can be forced into the engine. The other end of the inlet tube will be brazed into the metal plate along with the rest of the fluidic connections. This is necessary because the first generation engine will not close the thermodynamic cycle and thus will not be able to run without high-pressure air being forced into the compressor.

An inlet could also be made from glass by using a mold in the shape desired and melting glass in the mold. Glass frit could be heated and pressed inside the mold to form the ring shape and then cut in half to fit inside the engine. The inlet could then be bonded to the engine directly during the normal packaging process. This method would be more difficult to develop at first, but would eventually allow inlets to be produced and bonded at a much faster rate and at a lower cost.

The exhaust connection will use a straight Kovar tube glass sealed around the exhaust port on the rear of the engine. The other end of the exhaust tube will remain free, just as in the micro-combustor.

4.3.2. Electrical Connections

The only electrical connections required for the microengine, microrocket, and related devices are for igniters. The best method for making the electrical connections depends on what type of igniter is being used. Several different options have been explored for making igniters and electrical connections in the microengine and microrocket, including an on-chip resistor, a spark gap, and a resistive loop of wire. The only option that proved
feasible as an on-board igniter for the test devices was the resistive loop. Each option and the proposed methods for making electrical connections are described below.

**On-Chip Resistor**

The micro-combustor group built a test version of an on-chip resistor to serve as an igniter. The advantages of using an on-chip resistor are that it allows for reliable ignition and measurement of the temperature inside the engine once it is operating. The on-chip resistor that was built had two conductive pads for electrical connections, each isolated by a thin layer of oxide. These conductive pads were extremely fragile and continually broke when any pressure was applied to make a connection. While tests showed that the resistor did produce enough heat to ignite the flow, fabrication and structural difficulties prevented its incorporation into the micro-combustor or turbocharger.

![Figure 4-8: Electrical connections to an on-chip resistor](image)

The two proposed methods for making electrical connections to an on-chip resistor are shown in Figure 4-8. On the left is a Kovar wire bonded to the silicon pad using a conductive glass preform. A conductive glass preform in the right size, however, may be difficult to find. The method represented on the right is a spring connection to the
conductive pad. The device is lowered onto the spring a predetermined distance, and can be removed or reconnected later. The spring can be made of steel or a nickel alloy, and the spring constant can be adjusted to account for the weakness of the oxide holding in the conductive pad.

**Spark Gap**

Another method for making an igniter is to place two wires close together with a high voltage across them to create a spark gap. The maximum temperature created by the spark is not limited by the material properties of the wire or the device, as is the case with the on-chip resistor and the resistive loop. The problem with this method is that it is difficult to make the correct size spark gap in the proper location without shorting out through the silicon or the wires. This packaging step must be conducted blindly because the spark gap is inside the device where it cannot be seen, as displayed in Figure 4-9. The tight tolerances needed for a spark gap are not possible with current packaging methods.

![Figure 4-9: Two options for a spark gap igniter](image)

**Resistive Loop**

A resistive loop of Kovar wire is the simplest way to make an igniter. A resistive loop igniter can be made by using a glass preform with two holes and running a single piece of wire through one hole and back out through the other. Figure 4-10 shows how the preform and wire are bonded into the silicon device so that the loop extends into the combustion
region. The wires protruding from the device are then connected to a power source in the laboratory. When current flows through the wire, it heats up the loop to a high enough temperature to ignite the flow. This method is used in the micro-combustor and has proven to be reliable.

![Diagram of a resistive loop igniter](image)

**Figure 4-10: Diagram of a resistive loop igniter**

### 4.4. Summary

This research has determined that the best macro connection configuration is the fixed plate. It has proven to be successful in operation on the micro-combustor and turbocharger. An inlet can be machined from Kovar or can be made of glass using a mold and then sealed to the device. Likewise, the Kovar exhaust tube can be sealed to the device using a glass preform. Igniters and electrical connections have been made using several methods, the most successful of which has been the resistive loop of Kovar wire.
Chapter 5: Discussion

The work presented in this thesis focused on developing and refining the baseline process used to make connections to the microengine and microrocket and on packaging intermediary devices as they were fabricated. The result of this work, discussed in Section 5.1, is an improved packaging process and a better understanding of how process parameters affect metal-to-glass-to-silicon seals. Section 5.2 is a road map for packaging a device, detailing which device features and packaging options should be used to package future devices in order to reduce the turnaround time and to prevent mistakes from being repeated. The adaptation of the microengine packaging to a flight vehicle application is discussed in Section 5.3.

5.1. Improved Process

Five different process parameters were tested, as discussed in Chapter 2, to determine their affect on the strength and reliability of metal-to-glass-to-silicon bonds: the type of glass used, the surface condition of the silicon, and the atmosphere, time, and temperature in the furnace. Of these, only the type of glass preform used was found to affect the size of voids in the joints. The voids are a direct result of the manufacturing process of the preform, which varies from company to company. Thus, the manufacturing process of the preform is actually the controlling factor for the size of voids, not the specific chemical composition of the glass. Electroglass uses a spray drying method for making the frit used in the EN-1 preforms, which produces a finer powder and unique microstructure in the resulting preforms. Fusite K, made by GBC, uses a standard ground frit with larger particles, and has larger voids as a result.
The wetting of the glass on the silicon was found to depend on both the type of glass used and the atmosphere in the furnace. While Corning 7052 did not wet the silicon under most conditions, EN-i and Fusite K wet the silicon when the atmosphere was less reducing. The wet Nitrogen and Propane 900/38 atmospheres contain more free oxygen and cause \( \gamma_{sv} \), the surface energy of the silicon with respect to the atmosphere in the furnace, to increase due to the increased driving force for oxidation of the silicon. When \( \gamma_{sv} \) exceeds \( \gamma_{sil} \), the silicon is at a lower energy state in contact with the glass than with the atmosphere, and thus the glass wets the silicon. Increasing \( \gamma_{sv} \) further by using an even less reducing atmosphere will continue to improve the wetting until the contact angle \( \theta \) is small and spreading occurs, similar to brazing.

The improved process recommended from the results described in Chapter 2, namely to use EN-1 glass and a Propane 900/42 atmosphere in the furnace (900/38 would have been preferred), was then tested for strength and compared to the baseline process, using Corning 7052 glass and a dry Nitrogen atmosphere. The results presented in Chapter 3 revealed a noticeable improvement in the strength of the joints using the improved process. The standard deviation of the strength tests was also reduced using the improved process—meaning joints made using the improved process are more reliable. Therefore, the results in Chapter 3 support the conclusions of Chapter 2.

The improved processes recommended and tested in this thesis, however, are not necessarily optimal. They merely represent an improvement over the baseline process,
which was chosen based on the process used for metal-to-glass sealing. Further testing is necessary to determine the best furnace atmosphere and glass due to the fact that only a few options have been tested to date.

5.2. Road Map to Packaging a Device

The packaging of a device must begin long before the device is fabricated. Some parts of the device’s design should be driven by the needs of the packaging, such as the size, location, and depth of the holes and the inclusion of standoffs for fluidic connections. In addition, the parts necessary for performing the packaging, such as the proper size Kovar tubes and glass preforms, need to be ordered well in advance. Finally, the best macro connection configuration is the fixed plate, using a break-away carbon fixture and simultaneous brazing and glass sealing. By following the few simple guidelines outlined below, both the turnaround time and the reliability of the packaging can be greatly improved.

5.2.1. Device Features to Incorporate

Hole Sizes And Locations

The sizes and locations of the connection holes in a device have a significant impact on the speed with which packaging is developed. The sizes of the holes determine both the size of the Kovar tubes and the glass preforms to be used. Delays of over three months will result if a hole size requires Kovar tubing and glass preforms to be specially manufactured because they are not available in stock. It is easier to change the size of the hole than to order custom made tubes and preforms. Likewise, holes that are placed too close together
on the silicon may require either smaller preforms or a specially made preform so they will fit side-by-side.

While the micro-combustor has demonstrated that connections can be made on both sides of a device, this adds considerable complexity to the packaging process and should be avoided if possible. Backside fluidic connections that wrap around to the front are particularly precarious because they create a large, unprotected moment arm that will break the connection with only a slight jar. Backside connections will always be necessary for exhaust and igniters in the microengine, but avoiding additional backside connections is imperative. The optimal design is to space the holes evenly and symmetrically on one side of the device, using as few holes as possible and sizes that correspond to tubing and preforms already in stock.

**Hole Depth And Opening**

The depth of the holes and the openings beneath them also affect the time needed to package a device. The depth of the holes should be at least 500 microns to allow enough room for the tubes to rest inside the holes with adequate side support. A depth of 1 mm or greater is preferred, the measurement of which is shown in Figure 5-1. Another important consideration is that all of the hole depths should be the same on the device. Using different hole depths on a single device requires multiple tube lengths to be cut, which has proven to be time-consuming and tedious. Multiple tube lengths also complicate the assembly of the device by allowing errors in the lengths of cut tubes to cause misalignment with the macro connections.
Three different methods have been used for forming openings for the fluidic connections within the holes of the device. Each of these methods is shown in Figure 5-1. The slant cut tube, shown on the left, is made by cutting the end of the tube at an angle. While this is simple to do, even on very small tubes, the chief disadvantage is that it does not provide even distribution of the flow within the device. Even if uneven distribution is desired, it is difficult to accurately define the direction of the flow during the packaging process because it must be done blindly once the tube is inserted into the hole, and cannot be checked. The notched cut, shown in the center, provides a more even distribution of the flow. However, it is the most difficult to manufacture because of the small scale of the tubes and the precise cuts that are necessary. The notched cut method in particular adds a considerable amount of time to the packaging of each device because of the extra step in preparing the tubes. A built-in standoff, shown on the right, is the preferred method because it allows an even distribution of the fluid and does not require any extra tube preparation. Building this feature into the device will reduce the amount of time needed for packaging and increase the consistency of flow distribution within the device.

Figure 5-1: Three options for fluid distribution within a hole
5.2.2. **Macro Connections and Other Packaging Considerations**

Experience with packaging intermediary devices has shown that the preferred method for making macro connections is the fixed plate configuration. The metal plate and other components needed for the fixed plate configuration are simple to design and build. Having the tubes all connect to a single metal plate also protects the individual joints from bending loads and accidents due to handling. In contrast, the u-tube configuration has proven to be the worst choice due to its manufacturing and packaging difficulties, increased loading of the glass seals, and lack of protection for the device and packaging.

A fixed plate design also allows the packaging to be performed in only one pass through the furnace. Because the brazing operation used to bond the tube to the metal plate uses a similar atmosphere and temperature as the process used for glass sealing, the two seals can be formed simultaneously. This eliminates the need for multiple passes, which can weaken the glass seals, re-flow the glass, and promote the coalescence of larger voids.

The fixed plate configuration also yields advantages for the carbon fixturing of the device. When the metal plate is brazed to the tubes at the same time that the tubes are sealed to the device, the fixture is left in between the metal plate and the device. The carbon can then be slid to the side of the metal plate and used as an insulator, as in the case of the micro-combustor, or a thin carbon fixture can be used and broken away after the sealing process. In addition, having the metal plate in place to hold the tubes together as the carbon fixture is first lifted off the device after glass sealing is advantageous because the fixture tends to stick to the glass joints, causing the process engineer to apply more force and the fixture to
tilt as it is removed. This extra handling and tilting of the fixture has broken many joints in the past due to the large moment it places on them and can be avoided by using a thin fixture.

5.3. Adaptation to Flight Engine

One of the most demanding of the eventual applications of the MIT Microengine and its packaging is the Micro Aerial Vehicle (MAV). A flight version of the microengine will need to be packaged and attached to the aircraft as a propulsion system. The packaging will need to be light, robust, and compact. Figure 5-2 shows the design of the JMAV 2A, a MAV which is under consideration by DARPA to use the microengine.

Fluidic and electrical connections could be made to the flight engine using the same processes explored in this thesis. However, the flight engine will have several new issues that will need to be considered, namely the fragility of the rotor during transport and handling, the weight of the packaging materials, and the installation of an air filter in the inlet. To date, a microengine with a rotor has not been subjected to the conditions of the packaging process. These include transport by hand to and from the packaging house, over 50 miles away, handling by multiple personnel in a non-cleanroom environment, and passing through a furnace at high temperature with a reducing atmosphere. Because the engine will operate in an open-air environment, an air filter will also need to be installed in the inlet to prevent dust particles from entering the engine and damaging small features, such as the bearing gap.
The largest loads the packaging of the flight engine will encounter will be from landing and handling before and after flight. The strength of the glass seals is sufficient to carry the thrust and inertial loads of the engine during operation, but their strength is not necessarily adequate to carry the loads from an operator’s handling of the aircraft. To avoid mishaps on the ground, a protective shroud should encase the rear of the aircraft until just before flight.

The macro connections on the flight engine will be the interface between the tubes and wires on the engine and the fuel tank, batteries, and avionics on the aircraft. The design in Figure 5-2 shows the engine connected to the airframe only by the tubes needed for fuel and starter air. The other end of these tubes will need to connect directly to either valves or to the airframe. A fixed plate configuration cannot work because the metal plate used is

![Diagram of the JMAV 2A Design](image)

*Figure 5-2: Diagram of the JMAV 2A Design*
too heavy. Since the tubes must connect directly to the other components in the aircraft, and due to the low temperature materials with which the aircraft will be made, the macro connections cannot be made simultaneously with the glass seals. Packaging techniques presently used for the fixed plate configuration will not be applicable to the flight engine.

The electrical, inlet, and exhaust connections in the flight engine will also differ from those currently used. To conserve weight, electrical connections may need to be made through the Kovar tubes wherever possible. The inlet will be of the design shown previously in Figure 4-7, and will need to be rigidly attached to the engine surface. Both the inlet and exhaust connections will include an external augmenter structure, shown in Figure 5-2, which will be critical to the performance of the aircraft.20
Chapter 6: Conclusions

6.1. Summary of Experimental Results and Conclusions

This thesis has examined the strength limitations of glass seals to silicon, the testing of the strength of joints made from different processes, and the options available for making macro connections to a packaged device. Sessile drop tests were used to identify the influence of process parameters on the formation of voids within the glass and wetting of the glass on the silicon. Bending and tension tests were used to directly measure the strength of single tube test samples and compare those results to samples made using an improved process defined by the Sessile drop tests. Three different macro connection schemes were used on different devices and their results were employed to determine the best configuration.

This work in developing and refining the packaging process for the MIT Microengine and related devices led to the following conclusions:

- The volume fraction of voids within the glass can be reduced by using glass preforms made with a finer powder, such as EN-1 which is made using a spray-dried powder.
- Wetting depends on the combination of atmosphere in the furnace and the glass being used — less reducing atmospheres, such as 900/38 and wet Nitrogen, can raise the surface energy of the silicon relative to the atmosphere sufficiently to achieve wetting of the glass on the silicon.
• Residual stress depends on the type of glass being used and can be minimized by finding a glass with a coefficient of thermal expansion closer to silicon.

• Reducing the number and size of voids in the glass using the improved process implemented by Thunderline Z improves both the strength and reliability of the joints. Further improvements could be made by wetting the glass on the silicon.

• The fixed plate method of making macro connections is preferred for its simplicity, reliability, and robustness.

• Adjusting the type of glass and furnace atmosphere used to achieve better wetting and more closely matched expansion with silicon must be balanced with the need to wet the glass on Kovar and match the expansion of the glass with Kovar.

6.2. Future Work

Future work in packaging should include a more detailed study of how the furnace atmosphere affects wetting, what preform manufacturing processes are available that might reduce voids, other types of glasses that could be used, and the feasibility of wafer level packaging. A better understanding of the mechanism by which wetting is achieved is necessary to create a stable, reliable, and optimized process. This thesis only examined five different atmospheres and how they affected the wetting of three different glasses. Future work should include measurements of the surface energies and how the atmosphere and type of glass used affects them.

There are dozens of glasses made that form hermetic seals with Kovar, and only three of these have been used so far to seal Kovar to silicon. Different manufacturing processes for the preforms should be examined to find which methods reduce voids. Wafer glass in
particular should be considered because it is void free. Preforms could be drilled or etched out of the wafer glass and then used in the existing process. Another potential application of wafer glass is in wafer level packaging, which is discussed below.

A long-term goal of the MIT Microengine project is to mass produce microengines for a variety of applications. In support of this goal, packaging processes must be developed that have a high yield and are conducive to batch processing—a chief advantage of the micro-fabrication technology used to make the microengine. Wafer-level packaging technologies could be developed that would allow some of the processing steps for making the connections to be achieved at the wafer level, thereby reducing the amount of individual assembly needed for each device.

![Diagram of the proposed wafer level packaging process](image)

Figure 6-1: Diagram of the proposed wafer level packaging process
Wafer level packaging would begin when a wafer of devices leaves fabrication. A wafer of borosilicate glass (the same as used in the preforms) with holes drilled or etched through in the appropriate places would be joined to the wafer of devices using an anodic bond. The glass/silicon stack would then be die sawed just as before. The individual devices would then have tubes and wires dropped into place through a carbon fixture and passed through a furnace, as shown in Figure 6-1. This would reduce the assemble time needed with preforms, increase the glass/silicon bonded area, eliminate the possibility of voids in the glass by utilizing wafer glass, and allow entire wafers of devices to be packaged at a time.
Appendix A

Cross-sectioning of samples was performed in the Center for Material Science and Engineering (CMSE) specimen preparation room. The process of cross-sectioning samples for microscopy is as follows:

1) Prepare samples so that they can fit into 1” diameter rubber pots. Trim excess tubing as necessary.
2) Place samples horizontally in pot so that the plane through which the cross-section is desired is parallel to the bottom of the pot. Use a metal clip if needed to hold sample in position.
3) Mix the appropriate combination of epoxy resin and hardener. Slowly fill the pot with the epoxy, making sure that samples do not move.
4) Place the pot in a vacuum for approximately two hours to remove any air bubbles that may have formed in the epoxy.
5) Remove pot from vacuum and allow epoxy to set for 24 hours.
6) Remove solid epoxy block from the rubber pot and clean the edges as necessary.
7) Grind down the block using polishing wheels and low grit sandpaper until the level through which the cross-section is desired is reached.
8) Gradually use higher grit paper, up to 4000, to polish the sample until no lines or scratches are visible to the naked eye. Be careful not to grind too far.
9) Rinse the sample well under water and then polish further using a 0.3 micron slurry. Continue polishing until a mirror-like finish is achieved.
10) View sample under microscope. Repeat steps 7-9 if the cross-section plane is not deep enough into the sample.
### Appendix B

<table>
<thead>
<tr>
<th>Glass</th>
<th>Thermal Expansion Coef. (0-300°C)</th>
<th>Working Point</th>
<th>Softening Point</th>
<th>Annealing Point</th>
<th>Strain Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corning 7052</td>
<td>51 x 10^{-7}/°C</td>
<td>1058°C</td>
<td>718°C</td>
<td>512°C</td>
<td>472°C</td>
</tr>
<tr>
<td>EN-1</td>
<td>47 x 10^{-7}/°C</td>
<td>1115°C</td>
<td>716°C</td>
<td>482°C</td>
<td></td>
</tr>
<tr>
<td>Fusite K</td>
<td>45 x 10^{-7}/°C</td>
<td>1050°C</td>
<td>720°C</td>
<td>500°C</td>
<td>440°C</td>
</tr>
</tbody>
</table>
Appendix C

The angle measured when viewing the cross-section of test samples is not necessarily the wetting angle. This is due to the fact that the plane through which the sample is cross-sectioned can be offset by some tilt angle $\beta$ and depth angle $\alpha$. The diagrams below show how an arbitrary cross-section plane is taken through a test sample with actual wetting angle $\theta$. The wire frame represents the cross-section plane and the center plane is shaded in gray. The tilt angle, $\beta$, is the angle between the cross-section plane and the center plane as viewed from the side. The depth angle, $\alpha$, is the angle between a line extending from the center of the preform to the point at which the glass and silicon meet on the cross-section plane and a line extending along the center plane. The tilt angle results from either the sample not being placed perfectly perpendicular to the bottom of the epoxy mold or from polishing down at an angle. The depth angle is caused by not polishing all the way to the center of the preform, or from polishing too far (which would result in a negative $\alpha$).
The angle observed when viewing the cross-section, $\phi$, is the angle of the projection of the wetting vector on the cross-section plane. The projection in each dimension of the plane adds a cosine term to the original projection term, giving Equation 1. Rearranging this equation to solve for the actual wetting angle yields Equation 2.

\begin{equation}
V \cos \phi = V \cos \theta \cos \alpha \cos \beta 
\end{equation}

\begin{equation}
\theta = \arccos \left( \frac{\cos \phi}{\cos \alpha \cos \beta} \right)
\end{equation}

It is impractical to measure $\alpha$ and $\beta$ to precisely determine the wetting angle from the observed angle. However, it is possible to determine whether or not the wetting angle is obtuse or acute from the observed angle. Since $\alpha$ and $\beta$ only vary from $-90^\circ$ to $+90^\circ$, the cosine of each of these angles will be positive regardless of orientation of the cross-section plane. The sign of the cosine of the observed angle will determine the sign of the value within the parentheses, being positive if the observed angle is acute and negative if obtuse. Because the inverse cosine of a positive number will always be an acute angle and the inverse cosine of a negative number will always be an obtuse angle, the wetting angle will always be the same (either acute or obtuse) as the observed angle. Therefore, if the observed angle is acute the wetting angle is acute, and if the observed angle is obtuse the wetting angle is obtuse—independent of the orientation of the cross-section plane.
References


8 Research performed by Adam London.

9 Linde, Union Carbide. *Glass-To-Metal Sealing: Improved Yields And Quality Using Nitrogen-Based Atmospheres.*


17 Research performed by Shaun Sullivan.

