Fabrication and assembly of an array of micro fuel injector nozzles for a trapped-vortex combustor

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FABRICATION AND ASSEMBLY OF AN ARRAY OF MICRO FUEL INJECTOR NOZZLES FOR A TRAPPED-VORTEX COMBUSTOR

A Thesis

Submitted to the Graduate Faculty of the Louisiana State University Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering

in

The Department of Mechanical Engineering

by

Tracy Ettel Morris
B.S., Louisiana State University, 1996
May 2004
To my husband
Chad

And my children
Brooke, Sidney, and Cole
Acknowledgements

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Abstract

An array of four fuel injector nozzles, each with micrometer-scale swirlers and a microhole, is created on a single coupon. The nozzles are intended to atomize fuel to blend with swirling air before ejecting into a trapped-vortex combustion chamber. A first-generation metal prototype is fabricated in order to form the micro features, supply the fuel and gas to the nozzles, and guide the mixture to the combustion chamber.

Microfabrication methods and precision machining are used to create a multiple plate laminate assembly. The central plate contains the four microholes, which atomize the fuel, and facilitate the swirler structures needed to induce vorticity in the fuel and air. Fabrication of the swirlers requires electrodeposited nickel microstructures on both sides of the plate in order to segregate the high-pressurize air and fuel flows. Two other plates seal the air and fuel channels resulting in separate reservoirs for supply to the microstructures. A fourth plate defines a low-pressure complementary air reservoir needed for stoichiometric conditions.

Guided with alignment pins, the plates fasten together to seal the reservoirs and guide the injecting mixture along an axis with a total alignment error of less than 50 µm. Precision machining is needed to drill the holes required for alignment, fluid flow, and fasteners. Plunge EDM drilling through a thin plate creates the conically-shaped holes 75 µm -125 µm in diameter.

The project resulted in fully reproducible microfabrication methods, which when used in coordination with precision machining techniques produces smooth, level microstructures on both sides of the drilled nickel plate. The components were assembled and tested for leakage. Since the prototype leaked below atmospheric
pressure, the warped central plate could not be salvaged for patterned flow testing purposes. Recommendations for the next generation prototype are presented.

Preliminary investigation of a ceramic prototype is conducted with particular interest in sealing solutions involving cofired test samples. Viable testing methods and specimen geometry are investigated. Future ceramic prototype development requires shear joint testing as the initial step in realizing a self-sealing ceramic fuel injector prototype.
1 Introduction

1.1 Focus

The objective of this thesis was to fabricate and assemble a prototype array of fuel injector nozzles for subsequent study in high temperature flow-pattern analysis. Micrometer scaled atomizing nozzles were needed to generate reduced droplet sizes in order to improve combustion efficiency and reduce emissions. Swirler geometry was used to generate a swirling mixture of atomized fuel and air for immediate injection into the combustion chamber. A metal prototype assembly was the predecessor for a ceramic version, which may assist in sealing and perform better in the high temperature environment.

The long-term goal of this project was to produce many tiny fuel injector nozzles for use in a trapped-vortex aircraft combustor to improve combustion efficiency while meeting requirements for emissions control.

1.2 Motivation

Modern combustor designers balance two traditionally competing areas: efficiency and emissions. Emissions legislation has led to increased research in the reduction of pollutants found in exhaust. Past efforts to improve combustion efficiency resulted in elevated combustion temperatures, which increased the release of toxic products and violated today’s standards for emissions. Alternative approaches to improve combustion efficient within emissions guidelines include eliminating unstable combustion, or ‘flame-out’.
Flame stability directly affects efficiency since stability improvement results in more complete combustion, which increases the power delivered for thrust and enhances combustion uniformity. Combustion homogeneity leads to a more uniform temperature profile, which reduces resulting harmful emissions. Through elimination of cooler spots during combustion, partially combusted harmful products, such as carbon monoxide (CO) and unburned hydrocarbons (UHC), are reduced. Similarly, reduction of hot areas reduces the formation of oxides of nitrogen (NOx) in the exhaust. Thus, through enhanced flame stability during combustion, both fuel efficiency and emission quantities can be improved.

Improved flame stability enhances the performance of aircraft combustors as they operate through a wide range of environmental and operational conditions. Atmospheric conditions change rapidly during descent and climb. The engine must not flame-out or generate excessive flame temperatures while adjusting to continuously varying fuel flow associated with acceleration. Additionally, reduction of pressure, temperature, and density of the incoming air with increasing altitude requires flexibility in combustor operations. Combustion must be stable over a wide range of chamber pressures due to pressure differentials with change in altitude and forward speed. The air/fuel ratio conventionally varies from 60:1 to 120:1 for simple cycle gas turbines in aircraft (Cohen, 1996). Compared to the combustive stoichiometric ratio of 15:1, high dilution is required in order to maintain temperatures below those required for acceptable metallurgical conditions of the turbine. Additionally, poor combustion can set up aerodynamic vibration, which reduces the life of the chamber and causes blade vibration problems (Harmon, 1981).
A solution that improves both fuel efficiency and emissions through increased flame stability in the primary combustion chamber is to generate recirculation zones and fuel atomization within a secondary combustion chamber. Atomization has the potential to address flame instability and fuel efficiency through promotion of complete combustion by vaporizing the liquid fuel into finer droplets. The decreased droplet size increases total surface area, resulting in reduced vaporization time. This process effectively increases residence time of the prepared combustive reactants within the combustion chamber, increasing combustion and fuel efficiency (Harmon, 1981).

Atomization improvements have been achieved through creative fuel injector nozzle design. Conventionally, fuel injector nozzles are comprised of a small hole through which fluid is axially forced at elevated pressures. The resulting spray geometry is usually conical as it enters the combustion chamber. The use of modified nozzle geometries and micrometer-scaled atomizing orifices has improved nozzle design (Simmons & Harvey, 1995).

Recirculation zones are conventionally used in combustors to increase flame stabilization by sending the burning air/fuel mixture into a swirling flow pattern. Increasing residence time within the chamber so that there is sufficient time for complete combustion generates a self-piloting flame in the air stream. Directing a portion of the burning air/fuel mixture back onto the incoming fuel and air creates these recirculation zones.

There are many unique and creative solutions for producing recirculation zones (Harmon, 1981). One of these methods is referred to as a trapped-vortex combustor, since a secondary combustor is specifically sized to create recirculation zones to trap a
flame for subsonic speed flows. Continual injection of air and fuel into the vortex maintains a stable flame for reliable ignition of combustion gases in the primary combustion chamber.

1.3 Device Requirements

Specifications for fuel injector design were created in order to obtain a predetermined droplet size for the atomized fuel and to generate vorticity in the fuel/air mixture flow pattern as it exits the fuel injector. These values were determined from experience and results collected through research on gas turbine combustors and fluid dynamics within combustors. The primary function of the fuel injector was to atomize the fuel as it passed through a microhole. The microhole was required to have a conical geometry with 50 µm fuel inlet diameter and 100 µm exit diameter. This orifice was expected to vaporize the fuel into droplet sizes of approximately 10 micrometers.

A swirler was created to induce vorticity in the fluid mixture as it was injected subsonically into the combustion chamber. The swirlers were produced on both ends of the micro-hole in order to combine the swirling, atomized fuel with swirling air. A rendering of the specified swirler is shown in Figure 1.3(a). A swirler was composed of a number of channels converging tangentially into a swirl chamber. The fluid moved into the channels, achieving fully-developed flow before entering the swirl chamber. Within the swirl chamber the fluid moved tangentially about the circular cavity. The fuel then entered the micro-hole where it was atomized and developed a vortical profile induced by the swirling motion in the swirl chamber. Upon exiting the hole, the vaporized fuel blended to flow in the same direction with swirling air that was moving through a second swirl chamber. The mixture then exited into the combustion chamber with the vortical
fluid profile. A nozzle, as referred to in this project, encompassed the microhole with fuel and air swirlers located at each end of the connecting port.

The fully-developed flow criterion was used to improve vorticity in the mixture flow pattern as it was ejected into the combustion chamber. Accurate alignment of the microhole and the swirl chamber was required in order to achieve a proper flow pattern upon injection. Minimization of the travel path length from the fuel swirler to the combustion chamber was also necessary. This passage distance was expected to affect flow patterns of the air/fuel mixture within the combustion chamber.

The combustor must withstand elevated temperatures and pressures and contain gases emanating as products of combustion. By-products of corrosion must not plug the microhole over the short life of the prototype, which was projected to be less than 100 hours. Design pressure in the combustion chamber was six atmospheres (607 kPa), which was the maximum expected pressure. Structural deformation and subsequent leakage at the design temperature were prohibited. The design temperature was originally limited to 1200°C. As the project progressed and compromises were required for fabrication purposes, the prototype design temperature was lowered to 600°C.
Combustion reactants in this gas turbine engine were oxygen and a high-performance liquid jet fuel. The anticipated fuels were kerosene (JP4) and ethanol. JP4 is a wide cut version of kerosene, which was developed for military purposes to yield the maximum volume percentage on crude oil (up to 40%). It has good relighting properties and reduced aromatic content for reduction of the smoke point. The high flash point (38°C) is safe for refueling and the low freezing point (-47°C) is adequate for high altitude flying and good water separation characteristics (Hobson, 1984).

Due to the difficulty in obtaining acceptable flow rates from a system in which the fuel passes through an orifice 50 µm in diameter, 0.13 mL/s (0.125 gal/hour) was established as the minimum fuel flow rate. Extraction of air flow rates for complete mixing of the two fluids and stoichiometric requirements for lean combustion were required. The amount of air passing through the air swirler chamber for mixing with the atomized fuel was not required to be stoichiometric. Alternatives were explored for injecting the remaining required air into the combustion chamber. Subsonic injection of the mixture into the combustion chamber was preferred.

1.4 Preview of Fuel Injector Design

A cross-sectional view of a single nozzle is shown in Figure 1.4(a). Fuel swirlers were located on the lower end of the passage, and air swirlers were located on the opposite end. Multiple plates were designed with reservoirs to contain the high pressure fuel and air separately before mixing. As shown in Figure 1.4(b) the reservoirs were sealed through a laminated plate assembly. The swirling air/fuel mixture was designed to bypass the remaining plates and inject into the combustion chamber. Stoichiometry was
controlled by low pressure air, which was contained in a third reservoir and injected into the combustion chamber adjacent to the mixture exit.

Each plate in the laminated plate assembly shown in Figure 1.4(b) had features required for proper function of the micro-nozzle array. Plate C contained the micro hole and interfaced with the swirlers. Plate B and Plate D also interfaced with the air and fuel swirlers, respectively. Plate A provided outlets for the air/fuel mixture and the complementary low-pressure air, along with containing Reservoir 1. Reservoirs 2 and 3 were located on the top and bottom sides of Plate C, respectively.

Figure 1.4(a) Cross-sectional view of swirlers mounted on both sides of connecting hole.

Figure 1.4(b) Cross-section of two fuel injector nozzles. Arrows indicate the direction of fluid movement.
1.5 Thesis Outline

The background is discussed to introduce previous and existing work on combustors, atomizers, microfabrication, and electroplating. Material selection is then reviewed for both metals and ceramics. The document then divides into two parts: metal prototype and ceramic testing. Calculations, modeling, component design, and fabrication methods are presented for the metal version. Ceramics are then discussed to indicate the direction of subsequent research on design for material properties and testing for bond strength. Finally, recommendations for future work on improving the fuel injector design and fabrication are discussed.
2 Background

2.1 Combustors

Trapped-vortex (TV) combustors have been the subject of recent study due to their potential for increased combustive efficiency through various design modifications. The concept of trapped-vortex as applied to combustors was originally investigated by Hsu, et al. (Hsu, 1995). This study described use of two discs placed in tandem and housed in a cylindrical shell with annular air flowing over the front disc. In this configuration, a vortex of annular gases was trapped in the cavity formed between the discs. The focus of this and a subsequent study (Hsu, 1998) included determining the dependence of the flame structure upon the optimal distance between the two discs for production of a stable vortex over a large operation range. Katta and Roquemore (1996) described a numerical investigation of vortex dynamics in TV cavities of various sizes in low temperatures andcombusting environments. Their follow up study (Katta and Roquemore, 1998) used a numerical simulation to investigate the vortex dynamics of a cavity into which fluid mass was directly injected. It was found that mass injection increased the optimum width-to-diameter size ratio of the cavity. Most recently, an experimental study on a TV combustor determined results of varying flow rates (injection and annular) and injection orientation (Mancilla, et al., 2001). This study confirmed reduced lean blow out for the combustor over a wide range of airflow rates, and improvement of mixing without disrupting the trapped vortex by tangential injection.

2.2 Atomizers

Atomization can be accomplished by forcing high-pressure fuel through a small orifice, resulting in a conical spray of fine droplets into the combustion chamber. A
Sauter mean diameter (SMD) of 50–100 µm is the typical size of atomization holes in fuel injector nozzles for most aircraft gas turbine combustors (Cohen, & Saravanamuttoo, 1996). As the droplet size decreases, evaporation time within the combustion chamber decreases, allowing more time for combustion reactions to occur with the vaporized fuel. This increased residence time provides more complete combustion than if the fuel were injected in larger droplets. Decreased amounts of unburned hydrocarbons (UHC) and carbon monoxide (CO) in the exhaust result from vaporized fuel.

Conventionally machined atomizers are expensive to manufacture due to the required precision and low repeatability of conventional methods. Microfabrication procedures provide the required dimensional precision for arrays of atomizers, resulting in reduced cost. A group at Case Western Reserve University, in collaboration with Parker-Hannifin, (Simmons et al, 1995) patented an atomizer formed using a deep reactive ion etching (DRIE) technique. UV lithography on a stainless steel substrate was used to form the double-sided mask pattern. The straight microhole diameters ranged from 50 µm to 2.5 mm with a 150 µm depth. The patent did not specify swirler channels size or a means to supply the injectors with fluids. Subsequent work by this group included optimization of atomizer geometry to produce minimal SMD values with maximum spray angles and flow rates (Singh, Mehregany, Phillips, Harvey, & Benjamin, 1996). These atomizers were similarly etched with a two step mask process on both sides of a 400 µm thick silicon substrate. Predicted corrosion problems led to the conclusion that silicon was not an acceptable material for gas turbine atomizers. Another study of this nozzle by members of the same group investigated the methods of coating the silicon atomizer with 3C-SiC ceramic layers by atmospheric pressure chemical vapor deposition
(APCVD) (Rajan, Zorman, Mehregany, DeAnna, & Harvey, 1997). In performance tests
the coated atomizers demonstrated superior performance and withstood corrosion better
than the uncoated silicon versions. The latest study used improved ceramic deposition
rates (25 to 50 µm/hr) to create solid SiC atomizers of the previously tested geometry
(Rajan, Mehregany, & Stefanescu, 1999). This study also compared the ceramic
atomizer to an electroless nickel atomizer formed in another etched silicon mold, a LIGA
nickel version, and another silicon atomizer. SiC and the nickel atomizers exhibited
similar flow performance to the silicon devices; however, the SiC had superior in
corrosive wear resistance.

2.3 Compressible Fluids

Compressible fluid dynamics were used to determine the dimensions of the air
swirler channels, supply pressures, and resulting flow rates of the compressed air. A
guideline to determining these parameters was the criterion of achieving fully-developed
flow of the compressed air before entering the swirl chamber. Definitions of model
features are presented in this section and the results are discussed in Section 4.1.

2.3.1 Hydraulic Diameter

Calculations for ducts of noncircular cross section may be performed by using the
hydraulic diameter. Hydraulic diameter is a simplification used for ducts of noncircular
geometry in order to use relations derived for circular cross sections. The hydraulic
diameter of a rectangular duct is calculated with Equation 2.3.1,

\[ D_h = \frac{2wh}{w + h} \]  

2.3.1

where \( D_h \) is the hydraulic diameter and \( w \) and \( h \) are the channel width and height,
respectively.
In order to correlate equations meant for circular cross sections with non-circular geometries, the error involved for the hydraulic diameter simplification must be determined experimentally. Calculations of Reynolds number with values extracted from this hydraulic diameter and duct surface roughness values from the Moody chart were compared to experimental results. The accuracies of these calculations were within 15% (Munson, 1990).

2.3.2 Reynolds Number

The Reynolds number (Re) is a dimensionless parameter reflecting the ratio of inertial forces to viscous forces. It is calculated with Equation 2.3.2,

\[ \text{Re} = \frac{\rho v D_h}{\nu} \]  

(2.3.2)

where \( \rho \) is fluid density, \( v \) is velocity of the developed fluid, and \( \nu \) is fluid viscosity. For compressible fluids, most of these parameters vary with pressure and temperature. These fluid properties may be solved through modeling of the flow through the ducts.

The Reynolds number was used to determine whether the flow was laminar or turbulent. The actual transition from laminar to turbulent flow within a pipe can occur at various Reynolds numbers. The precise point of transition can be difficult to predict since it depends on how much the flow is disturbed by pipe vibrations, surface conditions, and other phenomena. For general engineering purposes, the values in Table 2.3.1 indicate the range of Reynolds number for corresponding flow conditions.

<table>
<thead>
<tr>
<th>Re range</th>
<th>Flow condition</th>
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</thead>
<tbody>
<tr>
<td>Re &lt; 2100</td>
<td>Laminar</td>
</tr>
<tr>
<td>2100 &lt; Re &lt; 4000</td>
<td>Transitional</td>
</tr>
<tr>
<td>Re &gt; 4000</td>
<td>Turbulent</td>
</tr>
</tbody>
</table>

Table 2.3.1 Generalized flow conditions as estimated by Reynolds number (Munson, 1990).
2.3.3 Fully-Developed Flow and Entrance Region

Fully-developed flow refers to the condition of fluid flow in an enclosed pipe in which the velocity profile is a function only of the radial distance from the pipe center. The entrance region of a closed channel is the portion in which the fluid transitions between being independent of the pipe to being exclusively influenced by pipe geometry. The fluid is assumed to enter the channel with a uniform profile, as shown in Figure 2.3(a). A no-slip boundary between the channel walls and a thin layer of fluid along the channel walls is initialized at the pipe entrance and extends the length of the pipe. As the fluid travels through, the boundary layer thickness increases. If the pipe is long enough, the boundary layer extends radially, eventually filling the entire cross-sectional area of the pipe. At this location along the pipe length, the entrance region ends and hydrodynamic fully developed conditions are achieved.

The length of the entrance region was a guideline for determination of the channel length for the air swirlers. The design criterion requires fully developed flow of the compressed air exiting each swirler duct. The entrance length depends on fluid conditions, flow characteristics, and channel cross-sectional area. The entry length, $L_e$, can be calculated with Equation 2.3.3 for turbulent flow.

$$L_e = (4.4)D_h \frac{Re^{1/6}}{2.3.3}$$
2.3.4 Friction Factor

The friction factor is a parameter required for the Fanno flow analysis, which is presented in Section 2.3.9. Sidewall surface roughness was reflected in the friction factor, which was approximated by using the Moody chart (Munson, 1990). The Moody chart plots Reynolds number with surface roughness in order to extract the experimentally determined friction factor.

2.3.5 Ideal Gas Assumption

Ideal gas behavior can be assumed within an experimentally determined error. The error involved with the assumption of an ideal gas is dependent on the ratios of maximum pressure and temperature to critical point conditions \((P_{\text{crit}} = 33.5 \text{ atm}, T_{\text{crit}} = 126\text{K})\). With a maximum pressure ratio of 1.0 and all plotted temperature ratios ranging up to 12, the maximum error in the ideal gas assumption for nitrogen was less than 2% (Thompson, 1972). The ideal gas law is stated in Equation 2.3.4.

\[ P = \rho RT \quad 2.3.4 \]

2.3.6 Specific Heat Ratio

The specific heat ratio \((k)\) is assumed constant at 1.4 for an ideal gas. This parameter is dependent upon temperature. Using the maximum design temperature of 600°C, less than 4% error was incurred using a constant specific heat ratio (Thompson, 1972).

2.3.7 Viscosity

Viscosity \((\nu)\) is a function of temperature and can be extracted from experimental data. For compressible fluids, viscosity increases with increasing temperature (Thompson, 1972). According to experimental results obtained by Lennard-Jones
(Thompson, 1972), viscosity was approximately linear through temperatures between 25°C and 600°C. For air, linear interpolation of these temperatures resulted in viscosity values between 1.8E-5 kg/m·s and 3.8E-5 kg/m·s. Most analyses used the higher value to yield worst-case results.

2.3.8 Mach number

Mach number was required for compressible flow in order to determine fluid parameters such as temperature. Mach number (Ma) is composed of velocity in the numerator and speed of sound in the denominator, which is shown as a function of temperature in Equation 2.3.5,

\[
Ma = \frac{v}{\sqrt{kRT}}
\]  

2.3.5

where R is the gas constant, which is equal to 286.9 J/kg·K for air. Temperature and pressure must be calculated through relations derived through modeling assumptions. Fanno and Rayleigh flows are two well-defined models through which compressible flow through the swirler channels is simplified.

2.3.9 Fanno Flow

For a constant area duct flow with friction, Fanno flow assumptions may be used to determine fluid properties. The assumptions associated with this model are non-isentropic, adiabatic flow of an ideal gas through a constant cross-sectional area duct with friction. This model indicates the effect of friction on pipe flow. The dominant error involved with Fanno flow assumptions originated from the adiabatic assumption. The proximity of combustion to the fuel injector should generate high temperatures in Plate A. Propagation of this heat by conduction through the remaining plates could result in significant heat transferred to the fluids flowing through the fuel injector. Assuming
conduction through the plates and heat transfer by convection to the fluids, the temperature of the fluids should increase. The extent of this heat transfer was beyond the scope of this project. However, additional parametric modeling through Fanno flow assumptions for various fluid temperatures will yield information on flow rate characteristics for heated compressed air.

Temperature, pressure, and density were derived from the ideal gas law and the assumptions on entropy, heat transfer, and friction. These derivations can be reviewed in an undergraduate fluid dynamics text in the compressible flow chapter. Assuming isentropic, adiabatic flow through variable area ducts, the resulting equations for these ratios are dependent on Mach number and specific heat ratio. The parameters used stagnation properties (indicated by the subscript 0) for reference, according to Equations 2.3.6 – 2.3.8 (Munson, 1990):

\[
\frac{P}{P_0} = \left[1 + \frac{k}{2} \frac{Ma^2}{k-1} \right]^{\frac{k-1}{k}} 
\]

2.3.6

\[
\frac{T}{T_0} = \frac{1}{1 + \frac{k}{2} \frac{Ma^2}{k-1}} 
\]

2.3.7

\[
\frac{\rho}{\rho_0} = \frac{\frac{P}{T_0}}{\frac{P_0}{T}} 
\]

2.3.8

These equations are useful for determination of fluid properties through nozzles or diffusers. Another isentropic assumption used for determination of flow properties involved a ratio of critical area associated with Mach number (Equation 2.3.9).
The area ratio is a minimum for sonic (Ma = 1) conditions. Therefore, a given area ratio has two solutions for all conditions other than sonic: subsonic and supersonic. Care must be taken to understand the physical flow conditions in order to determine the proper solution. For example, if sonic conditions are obtained at the throat of a nozzle, then subsequent flow through a diffuser will generate supersonic flow. However, if subsonic conditions are maintained at the end of the nozzle or duct, then subsonic conditions will continue through the diffuser.

The Mach number can be extracted for Fanno flow through knowledge of a friction ratio channel length, duct diameter, and friction factor (f). Equation 2.3.10 relates Mach number to this friction ratio using the critical length (l*) as the reference. The critical state is indicated with the asterisk (*).

\[
\frac{A}{A^*} = \frac{1}{Ma} \left[ 1 + Ma^2 \frac{k-1}{2} \frac{k+1}{2(k-1)} \right]^{\frac{k+1}{2(k-1)}}
\]

\[2.3.9\]

\[
\frac{1}{k} \left( 1 - Ma^2 \right) + \frac{k+1}{2k} \ln \left( 1 + \frac{k-1}{2} Ma^2 \right) = f(l* - l) \frac{D_h}{D_h}
\]

\[2.3.10\]

To predict the effect of duct length on the Mach number from one value (state 1) to another (state 2), then Equation 2.3.11 can be used.

\[
\frac{f(l* - l_2)}{D_h} - \frac{f(l* - l_1)}{D_h} = \frac{f(l_1 - l_2)}{D_h}
\]

\[2.3.11\]

For these equations, the hydraulic diameter (Dh) of a square duct was used from Equation 2.3.1.

The pressure, temperature, and density ratios for Fanno flow use critical state parameters for reference, according to Equations 2.3.12 and 2.3.13.
Rayleigh Flow

Adiabatic assumptions may include elevated errors since a significant amount of heat could be transferred through the channel walls to the fluid. The amount of heat depended on the wall temperature of the duct, which was significant in the fuel injector. Diabatic refers to the condition in which heat is transferred from the walls to the fluid. Elevated temperatures originate from the reactions within the combustion chamber and propagate through the fuel injector plates by conduction. Within the scope of this project, modeling of this heat transfer was limited to channel flow only for the compressed air through the air swirlers. The maximum design temperature for the fuel injector of 600°C was considered the maximum temperature of the fluid. Rayleigh flow assumptions may be used to model this flow since they include diabatic flow of an ideal gas through a frictionless duct of constant cross section. The error involved with Rayleigh modeling primarily originates from the lack of friction. The surface roughness of sidewalls along with the duct size required high reservoir pressures for forcing fluid through the swirler channels.

Increased fluid temperature increases the fluid flow rate. According to the Rayleigh assumptions, continually heating of subsonic, compressible fluids creates a situation where there exists a Mach number at which the maximum temperature is

\[
\frac{P}{P^*} = \frac{1}{Ma} \left[ \frac{k+1}{2} \frac{2}{1 + Ma^2 \frac{k-1}{2}} \right]^{1/2} \tag{2.3.12}
\]

\[
\frac{T}{T^*} = \frac{k+1}{1 + Ma^2 \frac{k-1}{2}} \tag{2.3.13}
\]
obtained. For ideal gases with $k=1.4$, this Mach number is 0.845. Upon continued heating, the fluid temperature decreases as the Mach number reaches sonic conditions.

A Mach number equal to one is referred to as state ‘a’, which is the reference state for the fluid properties. Many of the previously discussed properties for Fanno Flow may be calculated for Rayleigh flow. However, for this analysis, only pressure was required; the appropriate equation is indicated in Equation 2.3.14 (Munson, 1990).

$$\frac{P}{P_a} = \frac{1+k}{1+kMa^2}$$

2.3.14

2.4 Incompressible Fluids

Low velocity motion relative to the speed of sound in the fluid can be modeled as incompressible with negligible error (Munson, 1990). Calculation of Mach number for compressible fluids is the ratio of velocity and speed of sound, $c$, through the fluid, which is similar to compressible fluids. The speed of sound for incompressible fluids can be found using the bulk modulus, $\beta$, and density, according to Equation 2.4.1.

$$c = \sqrt{\frac{\beta}{\rho}}$$

2.4.1

The Mach number can then be determined for incompressible fluids using Equation 2.4.2.

$$Ma = \frac{v}{c}$$

2.4.2

Pressure gradients may be calculated from two sources: resistance due to viscosity and energy alterations due to velocity gradients. These are reflected in Equation 2.4.3 and 2.4.4, respectively,

$$\Delta P_\mu = 128 \frac{Q\mu l}{\pi D_h^4}$$

2.4.3

$$P_v + \frac{1}{2} \rho v^2 + \rho gh = c_i$$

2.4.4
where $Q$ is volume flow rate, $l$ is length of channel, $g$ is acceleration due to gravity, and $h$ is the vertical distance traveled. Equation 2.4.4 is the Bernoulli equation, which states that for incompressible fluids, the energy associated with the fluid remains constant, $c_1$. The potential energy term is negligible when the height of fluid travel is small.

2.5 Plate Deflection

In order to model plate deflection between fastener sites, a simplified model was used to approximate order of magnitude values for the uniform pressure load applied to each plate. A clamped-clamped beam was used to simulate the fastening force at two adjacent sites. The clamps were defined as devices that resisted a force in any direction and a moment, which represented the bolt connectors used to fasten the plates together. A uniformly distributed load, $q_o$, applied to the beam was used to simulate the differential pressure acting upon the plate, as indicated in Figure 2.5(a) (Shigley & Mischke, 1989).

The moment of inertia, $I$, is obtained by use of Equation 2.5.1,

$$I = \frac{bh^3}{12}$$  \hspace{1cm} 2.5.1

where $h$ is the plate thickness and $b$ is the width of the simulated beam. Equation 2.5.2 reflects the maximum deflection of a clamped-clamped beam with an evenly distributed load. This maximum deflection is located at the middle of the beam.

$$y_{\text{max}} = \frac{q_oL^4}{384EI}$$  \hspace{1cm} 2.5.2

Figure 2.5(a) Uniform distributed load applied to a clamped-clamped beam represents the pressure applied to the cross-section of the bolted plate. The distance between bolt centers is represented by $b$. 20
where \( q_o \) is the distributed load per unit width, \( L \) is the distance spanned between the two fasteners, \( E \) is modulus of elasticity, and \( I \) is moment of inertia.

Another model used to calculate the maximum deflection of a plate under a uniformly distributed pressure load, \( q_p \), is derived assuming a flat circular plate of constant thickness clamped along the plate perimeter (Young et al, 2002),

\[
y_{\text{max}} = - \frac{3q_p a^4 (1-\nu^2)}{16Et^3}
\]

where \( a \) is the circular radius, \( \nu \) is Poisson’s ratio, and \( t \) is the plate thickness. The resulting bending stress, \( \sigma \), is obtained with equation 2.5.4 (Young et al, 2002).

\[
\sigma = -\frac{3q_p a^2}{4t^2}
\]

2.6 Cantilever Deflection

Relations involving the constraining moment, \( M \), were used to compare the applied load and yield strength, according to Equations 2.5.1, 2.6.1 and 2.6.2 (Popov, 1990),

\[
M = \frac{q_o x^2}{2}
\]

\[
\sigma_{\text{app}} = \frac{M c}{I}
\]

where \( M \) is the moment at any point along the beam as measured from the constrained end, \( c \) is the location of the centroid on the cross-section of the beam, \( \sigma \) is applied stress, and \( q_o \) is the distributed load. Here, \( c \) is half the thickness, \( h \), of the beam. The maximum moment is located at \( x = L \).

2.7 UV-LIGA

LIGA is the German acronym for X-ray lithography (X-ray lithographie), electrodeposition (galvanoformung), and molding (abformtechnik). The process involves
patterning a thick layer of X-ray resist with high-energy X-ray radiation exposure through a mask in order to create a three-dimensional resist structure. Electrodeposition subsequently fills the resist mold with metal. After resist removal, a free-standing metal structure is revealed. The metal form may be the final product or it may be used as a mold for creating the inverse geometry. The method borrows lithography from the Integrated Circuit industry and electroplating and molding from classical manufacturing. The benefit of high aspect ratio and accurate lateral dimensions makes it useful for microstructure (micrometer and submicrometer dimensions) fabrication, packaging (millimeter and centimeter dimensions), and connectors to those packages to the macro-world. The advantages of LIGA are high resolution features (<0.2 µm), long depth of focus, and a cooperative resist (PMMA) (Madou, 1997). Typical values of LIGA features are summarized in Table 2.7.

Many devices do not require the aspect ratios possible with LIGA. Instead, geometry of the micro features may be more critical to the device performance, and can be readily formed using UV definable thick photoresist films. UV-LIGA is a modified version of LIGA in which UV-sensitive resists, such as polyimides, AZ-4000 series

<table>
<thead>
<tr>
<th>Parameter</th>
<th>LIGA value</th>
<th>Approximate UV-LIGA values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Structural Height</td>
<td>20 – 2000 µm</td>
<td>5 – 1000 µm</td>
</tr>
<tr>
<td>Minimum Dimension</td>
<td>1 - 2 µm</td>
<td>3 - 5 µm</td>
</tr>
<tr>
<td>Smallest x, y surface detail</td>
<td>0.25 – 0.5 µm</td>
<td>1 - 10 µm</td>
</tr>
<tr>
<td>Maximum x, y dimensions</td>
<td>20 x 60 mm</td>
<td>200 mm diameter</td>
</tr>
</tbody>
</table>
(Hoechst Celanese, Somerville, NJ), and SU-8 (MicroChem, Inc.), are contenders for several of the applications for which LIGA is being promoted. Ultra-violet (UV) radiation for microfabrication use is composed of wavelengths ranging from 200 nm – 400 nm. The fabrication costs of LIGA and the time-to-market factor have driven the demand for these UV alternatives in Microelectromechanical Systems (MEMS) fabrication. SU-8 is a negative, epoxy-type, near-UV photoresist based on EPON SU-8 epoxy resin that was originally developed by IBM. After spin coating, the polymer film is photochemically cross-linked by exposure to UV light. In the exposed regions, an insoluble gel is formed. SU-8 is well-suited for acting as a mold for electroplating due to the relatively high thermal stability of the cross-linked resist.

Current advantages UV-LIGA has over X-ray LIGA include improved accessibility to the radiation source and easier mask fabrication. Additionally, the UV exposure stations generally allow more choices in substrate type and geometry than the X-ray scanners due to the differences in substrate chuck versatility. Disadvantages associated with UV-LIGA are the decreased resolution and feature size, resist removal, low throughput, and resist height variation (Yu, Ganser, Gehoski, Mancini, Rhine, & Grodzinski, 1999).

MEMS and high aspect fluidic devices formed through UV-LIGA have progressed in recent years. Lorenz et al fabricated working gears 200 µm thick (Lorenz, Despont, Vettiger, & Renaud, 1998). Micro mold inserts using SU-8 were compared to dry film resists, which revealed increased sidewall roughness compared to the SU-8 (Yu et al, 1999).
2.8 Electrodeposition

Electroplating nickel from a nickel sulfamate solution is a common, reliable method. Surely, it is the most repeatable step in the microfabrication process outlined in this document. A typical electroplating cell consists of an anode, a cathode, an aqueous-metal solution, and a power supply. In the simplified example shown in Fig. 2.8(a), the sacrificial anode is made of nickel, the cathode is made of another conductive material, and the aqueous-metal solution consists of nickel (Ni\(^{2+}\)), hydrogen (H\(^+\)), and sulfate ions (SO\(_4^{2-}\)). When the power supply is turned on, the positive ions in the solution are attracted to the negatively biased cathode. The nickel ions that reach the cathode gain electrons. Nickel atoms are formed from the union of ions and electrons. The atoms are deposited, or plated, onto the surface of the cathode forming the electrodeposit. Simultaneously, nickel is electrochemically etched from the nickel anode, to produce ions for the aqueous solution and electrons for the power supply. Hydrogen ions that also gain electrons from the cathode form bubbles of hydrogen gas.

If the metal thickness extends beyond resist height, the plating area slowly

2.8(a) Schematic of electrodeposition of nickel (Lowenstein, 1963).
expands, which is referred to as overplating. Overplating effectively reduces current density, which increases the amount of time required to obtain the desired plating thickness. Since the current density dictates resulting material properties (Lowenheim, 1963), inadvertent variations in material strength would result from overplating. Overplating can be prevented by spinning the resist at elevations higher than the design height of the electrodeposited structures. Subsequent leveling and polishing of the raw metal structures and resist yields the design height with the predicted material properties preserved.

Design with nickel microfabricated components requires mechanical property evaluation of electrodeposited structures. In recent years, many studies have indicated that the electrodeposition conditions dictate microstructure and, thus, mechanical properties. Sharpe et al developed a tensile test bed for ‘dog bone’ shaped structures. Depending on electroplating conditions, yield and ultimate strength values more than five times the bulk value were found (Sharpe & McAleavey, 1998).
3 Material Selection

The materials selected to address design issues for the array of fuel injectors must withstand elevated temperatures while maintaining adequate elastic modulus and yield strength. Additionally, resistance to corrosion in a high-temperature oxidizing environment for up to 100 hours was required. For sufficient fastening, high tensile strength was essential at elevated temperatures along with reduced high temperature creep. Fabrication processing that led to sealed reservoirs was also needed.

The materials selected for this project should provide inherent solutions for a robust design through maintenance of material properties at high temperatures, corrosion resistance, and reliable sealing. Ceramics have the potential to successfully address all three issues through a combination of material properties and monolithic bonding. Component design for ceramics required consideration of dimensional stability and consistency in component processing, as well as reliable data on material properties.

Metals that are commonly used in high temperature applications may also be used to construct the fuel injector. Machining methods are familiar and material property data for design is well established for the metal version. However, properties at elevated temperatures, sealing methods, and corrosion resistance, while worthy of a prototype, were inferior to the corresponding ceramic parameters.

This chapter reviews the material choices and their properties obtained for both the metal prototype and potential ceramic solutions.

3.1 Metals

For the metal application, creating metal plates to form the components of the fuel injector was feasible. Good strength and corrosion resistance could be found in specific
classes of metals. Accommodating plate clamping and sealing was necessary for this prototype design. Creating the micrometer-scaled features was possible through microfabrication techniques.

The metal prototype would require superalloys to meet the design requirements for the laminated metal plate assembly, presented in Figure 3.1(a). These metals are usually composed of nickel with traces of other materials and are commonly used in high-temperature applications. Different parameters were more important for some components than others. Plate A must resist the corrosive environment and withstand elevated temperatures since it would directly interface with the combustion chamber. Plate B must possess similar properties as Plate A, but with the addition of high modulus since the thin plate may be exposed to a large pressure differential between faces. Plate C primarily must be an adequate microfabrication substrate along with having high modulus. Micromachining substrates required high nickel content (>99%) in order to obtain adequate adhesion with electroplated structures. Plate D needed to have high strength and modulus since it may be subjected to a large pressure differential. Since Plate D housed the fluid connectors, it must also have high strength and corrosion resistance to the caustic fuel, and it must facilitate fluid connections.

![Figure 3.1(a) Cross-section of laminated plate assembly. Arrows indicate direction of fluid movement.](image-url)
Specific values were needed to adequately address the requirements for the properties required. The high pressures were on the order of 20 atmospheres. The maximum operating temperature was 600°C. The high modulus of elasticity was needed in order to minimize out-of-plane plate deflection. Finally, resistance to high temperature oxidation and the caustic effects of fuel exposure was required for prevention of accelerated corrosion of some of the components during use. The prototype was not designed for long life, so slight corrosion could be tolerated as long as the products of reaction did not obstruct the micro holes. There is some evidence that petroleum distillates, like fuel, are relatively pure and may be burned without severely corroding the turbomachinery (Richerson, 1992).

There are a number of factors that can lead to uncertainties in material property data for metals. These include variations in specimen fabrication procedures and partitioning methods (Callister, 1991). Subsequent fabrication steps such as surface modifications, annealing, and chemical exposure can alter the material properties. Since these modifications cannot be measured without experimental material testing, annealed material properties were used and the remaining effects of fabrication were considered negligible. The condition of the metal as-purchased was considered valid for use in calculations. Table 3.1(a) indicated material properties at the maximum design temperature for various metals that were considered for this application. Table 3.1(b) summarized a few of these choices from a corrosion perspective. The specified compositions of each material are indicated in Appendix B.
Table 3.1(b) Corrosion resistance properties of the nickel alloys (Callister, ASME, and Inco Alloys®).

<table>
<thead>
<tr>
<th>Material</th>
<th>Corrosion resistance parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni 200</td>
<td>Most useful in reducing environments. It can be used under oxidizing conditions that cause the development of a passive oxide film.</td>
</tr>
<tr>
<td>Ni 201</td>
<td>Preferred to Nickel 200 for applications involving exposure to temperatures above 315°C. Intergranular embrittlement resistance at temperatures above 600°C, except when exposed to carbonaceous and sulfur compounds. High temperature oxidation (HTO).</td>
</tr>
<tr>
<td>Inconel 600</td>
<td>A Ni-Cr alloy with good high temperature oxidation resistance and resistance to chloride-ion stress-corrosion cracking, corrosion by high-purity water, and caustic corrosion. Used for furnace components, chemical and food processing, nuclear engineering, and for sparking electrodes.</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>Similar to other Ni-Cr alloys, it is a function of its composition. Ni contributes resistance to corrosion by many organic, other than strongly oxidizing, compounds throughout wide ranges of acidity and alkalinity. Chromium imparts the ability to withstand attack by oxidizing media and sulfur compounds. Molybdenum contributes resistance to pitting in many media.</td>
</tr>
</tbody>
</table>

Other classes of metals that were briefly reviewed were the refractory metals and titanium. Refractory metals can be difficult to machine and expensive for a first-generation prototype. Titanium alloys have superior strength and melting point. Also, their corrosion resistance to petroleum and other chemicals makes these alloys attractive to aerospace and the petroleum industries. However, due to their high ductility, titanium
alloys have approximately half the elastic modulus of nickel alloys. Therefore, nickel alloys were considered more applicable to this project than the titanium alloys.

Inconel 600 was chosen as the high temperature metal for Plates A and B due to its maintenance of modulus of elasticity and tensile strength at temperatures above 600°C. Corrosion resistance was good in oxidizing environments and the protective oxide layer was an additional barrier to corrosion from the fuels used in the prototype. Additionally, these plates were machined with traditional tools, were compatible with specialty drilling, and were easily obtained. They were also dimensionally stable: the plates deflected out of plane less than 2 µm over the 100mm square area when they were originally ground to a thickness of 1.25 mm.

Plate C was composed of Nickel 201, for its high nickel content along with strength at elevated temperatures. The highest purity electrolytic nickel (99.999%) was the preferred nickel substrate; however, these materials did not maintain an adequate modulus at temperatures higher than 300°C. Nickel 201 with slightly lower content of nickel (99.9%) proved to be a sufficient substrate for microfabrication.

The composition of Plate D was stainless steel (SS) 316 since it welds easily to stainless steel fittings. SS 316 has high strength (240 MPa), but lower elastic modulus (193 GPa) at room temperature than the nickel alloys. These strength properties were of minor concern for Plate D since it was four times thicker than plates B or C. SS 316 is an austenitic steel, which is highly corrosion resistant due to the elevated chromium content and the nickel addition. Austenitic stainless steels can be used at elevated temperatures since they resist oxidation and maintain their mechanical integrity up to 1000°C. Fuels present negligible corrosion problems for these steels.
3.2 Ceramics

The material selection criteria for the ceramic version of the fuel injector were similar to those parameters of the metal version. Increased and reliable strengths were required for the elevated temperatures and pressures within the reservoirs. Sufficient corrosion resistances for the same degrading situations were needed. Sealing solutions and component size required reliable high strengths in tensile and shear.

Elastic modulus values for most ceramics are on the order of five times larger than those values for most metals. For example, silicon carbide (SiC) and aluminum alloys have elastic moduli of an average of 414 GPa and 69 GPa, respectively (Richerson, 1992). However, most ceramics under tensile loads behave elastically with no plastic deformation up to fracture. Brittle fracture is one of the most critical characteristics of a ceramic that must be considered in design for structural applications. Although the elastic modulus is high, great care must be taken to avoid brittle failure.

Ceramics show a broad range of resistance to corrosion in aqueous solutions, including strong acids and bases, for strongly bonded types like SiC. Application of SiC and silicon nitride (Si₃N₄) for gas turbine applications indicates minimal surface corrosion and absence of strength reducing effects (Richerson, 1992). For the reduced corrosion potential found in this project, ceramics such as SiC would remain inert up to the maximum temperature.

Ceramic tensile strength is commonly referred to as flexural strength (formerly, modulus of rupture). The flexural strength of a ceramic material is dependent on both its inherent resistance to fracture and the presence of defects. These defects can cause failure of the component well before the strength of the ceramic matrix is challenged.
Additionally, this parameter is unreliable since the defects are process dependent. Component preparation along with isotropy, defect population, component geometry, and surface conditions significantly alter strength measurements. A measurement result for flexural strength of a group of test specimen is influenced by several parameters associated with the test procedure. Such factors include the loading rate, test environment, specimen size and preparation, and test fixtures. Precautions must be made when applying material property data to component design. Due to process sensitivity, surface conditions, and testing methods, ceramic material properties extracted from property tables are of inadequate quality for design applications (Quinn & Morrell, 1991). Surface flaws are usually on the order of 5-200 μm in size and many strength limiting flaws are just below the surface, which makes detection difficult (Quinn et al, 1991). Thus, over-design of component size would be necessary to compensate for the undetectable strength-limiting flaws commonly found in the ceramic component.

There are many types of processes used to form ceramic components. Uniaxial and isostatic pressing, slip casting, extrusion, injection molding, tape forming, and green machining are among those available. Injection molding refers to a flow and press technique. This process can produce complex shapes that would normally require secondary operations. Generally the more complex the geometry, the more advantageous injection molding will be over other fabrication methods. Extremely tight tolerances, up to ±0.05%, can be achieved with this process (Coors Tek, Golden, CO). Injection molding also offers excellent reproducibility, batch processing, and automation, making this a low cost manufacturing technique for high volume throughput.
Fabrication of injection molded components started with mixing the ceramic powder with a thermoplastic binder, which wetted the ceramic powder. Mixing should destroy the agglomerations to create a homogeneous solution of up to 67% by volume of ceramic powder. The composition of the precursor mixture varies depending on feature size, aspect ratio, and processing parameters. Increased amounts of binder were needed to fill smaller and deeper crevices. For the injection molding step, many parameters must be monitored such as sufficient abrasion resistance, low compression along the screw, the risk of inhomogeneity due to variations of the cross-section along the flow path, and an even temperature distribution in the mold to minimize the residual stress. The binder was removed from the molded part in the debinding step, which requires temperatures up to 600ºC. Constant temperatures with low tolerances for fluctuation are required to avoid internal stresses, gas pockets, and/or cracks in the part. The condition of the part after debinding is called the ‘green’ state. The sintering stage was the final step to create the molded part. Maximum temperatures of 1800ºC caused the chemical reactions which increased the density of the ceramic and significantly shrunk the part to its final shape (Richerson, 1992).

Injection molding can be used in conjunction with LIGA microfabrication processes by using a micro mold. The advantages of micromachining were applied to ceramic formation plus batch processing from injection molding made the use of micro molds attractive. However, there were problems associated with micro molding. Use of a micro mold would require increased amounts of binder in the batch recipe to allow flow into the small crevices. Increased amounts of precursor can lead to increased dimensional reduction due to loses during dehydration and sintering. In cases where
micro components are molded on large pieces, the increased amount of precursor can lead to a weakened composite in the larger volumes.

For double-sided injection molding, two separate LIGA molds would be needed. Alignment of the facing plates with ±10 micrometer tolerance would be necessary while injection molding Plate C, as indicated in Figure 3.2(a). This tolerance was necessary in order to align the fuel swirler with the corresponding air swirler to ensure that the subsequently drilled microhole appears within the center of both swirl chambers.

A sealed joint may be realized by co-firing green ceramic components together. Co-firing is the process of forcing an assembly to contact throughout the sintering stage, resulting in a single monolithic component. Co-fire bond strength is process dependent and currently there are no strength data available. Considerable experimentation would

![Figure 3.2(a)](image.png) Cross-sectional schematic of double-sided injection molding (top) and electroplating (bottom).
be needed for characterization of this type of bonding and extraction of reliable mechanical properties for use in design for a ceramic fuel injector.

Though mechanical properties of ceramics depend heavily on processing, bulk data were used as preliminary criteria for material selection of test samples. Table 3.2(a) lists mechanical properties of injection molded materials in their specified states. The strength of nearly all ceramic materials decreases as the temperature increases.

Yttria-partially Stabilized Zirconia (YZTP) is a CoorsTek™ ceramic which was formed in previous ceramic studies. Samples of this material were used in early analysis of dimensional changes during processing, as discussed in Chapter 6. Properties of YZTP included high fracture toughness and wear resistance and excellent strength. Additionally, it is biocompatible for various medical and implant applications.

3.3 Conclusion

Material selection provided means for solving design issues, influencing design, expanding fabrication possibilities, and determining the materials to include in the metal prototype.

The materials used for the metal prototype were Nickel 201, as the high-

<table>
<thead>
<tr>
<th>Material</th>
<th>E (GPa)</th>
<th>Flexural Strength (MPa)</th>
<th>Fracture Toughness $K_{ic}$ (MPa (m)1/2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC (injection molded / sintered)$^a$</td>
<td>414</td>
<td>468-480</td>
<td>4 – 5</td>
</tr>
<tr>
<td>Silicon Nitride (Si$_3$N$_4$)</td>
<td>304</td>
<td>414-580</td>
<td>6</td>
</tr>
<tr>
<td>(sintered, 5% porosity)$^b$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>YTZP$^a$</td>
<td>100</td>
<td>900-1300</td>
<td>13</td>
</tr>
</tbody>
</table>

$^a$ Data provided by CoorsTek™.
$^b$ Data provided by Richerson, 1992.
temperature microfabrication substrate, Inconel 600 for corrosion resistance and strength at elevated temperatures, and stainless steel 316 for weldability and high temperature use.

Experimentation with ceramics would be required for more accuracy in strength data in order to design components. Since only in-situ sample testing with component size, geometry, and processing replication provide reliable strength results, either component over-design or iteration of component testing with design would be necessary. Additional testing for co-fired joint strength would be required in order to use this method for sealing and fastening components with separating pressure applied. SiC and YTZP were good materials to use in preliminary tests.

The remainder of this document is split into two portions: metal fuel injector design and fabrication, and ceramic component test specimen design. The next chapter begins with the calculations involved with the feasibility of the metal version. Subsequently, design details and then fabrication processes are included to complete the chapter.
4 Metal Fuel Injector Prototype

This chapter reviews development and fabrication of a prototype metal version of the fuel injector. Parametric modeling is initially presented for information on swirler scale and component design. The design section follows, which includes the functional requirements and resulting feature details involved in the design process. Fabrication of the metal prototype is then presented in two parts: precision machining and microfabrication.

4.1 Calculations

The modeling presented through these calculations was used to determine the feasibility of using metal plates for the components of the metal fuel injector. Order of magnitude parameters were derived for its design from the results obtained in this section. The features defined from these calculations were the supply pressure in the compressed air and fuel reservoirs, the swirler channel dimensions, and the distance between fasteners.

4.1.1 Compressible Fluid Modeling

Modeling through the use of Fanno flow and, separately, Rayleigh flow yielded information on the behavior of the compressible flow in the swirler channels. The resulting characteristics were used to extract dimensions of the swirlers and the supply pressures which were required to induce a Mach number of 0.95 at the air swirler channel exit.

Minimizing channel length was advantageous for increasing the number of swirlers in the fuel injector array and meeting space requirements for use in combustors.
The minimum size of each micrometer-scaled nozzle was determined by the length of the air swirler channels.

Minimization of supply pressure within the reservoir was important in order to prevent plate deflection. The supply pressure was required to overcome the pressure drop through the swirler channels, microhole, and combustion chamber, which was at 608 kPa. The pressure drop between the exit of the swirler channels and combustion chamber was negligible for compressed air. Therefore, the channel exit pressure was required to exceed the combustion chamber pressure by 100 kPa. In order to overcome the pressure drop in the swirler ducts, the supply pressure was increased using values derived from the models.

At increased supply pressures, deflection of each plate generated gaps for fluid leakage. By applying the results of supply pressure to deflection calculations, the distance between fasteners was determined to minimize plate deflection.

Duct length was determined by considering the development conditions required for fully developed flow. Since duct cross-section affects duct length, a balance of these two parameters was required to minimize duct length and supply pressure.

A broad channel was modeled according to the simplified geometry shown in Figure 4.1(a). The reservoir was assumed stagnant at State 0, where the stagnation pressure forced fluid through the channels. This pressure depended on duct geometry, surface roughness, and fluid viscosity.

An isentropic nozzle represented the micro channel entrance, and State 1 was located at the nozzle end. Isentropic refers to the situation in which compression of the fluid is frictionless and no heat is exchanged with the surroundings.
Fanno flow was used to model flow through the swirler channel, from State 1 to State 2, followed by a brief look at Rayleigh flow.

Specification of the fluid velocity at channel exit (State 2) was necessary to maintain subsonic conditions. If the conditions were to reach choked flow at State 2, then the fluid expansion at the swirler channel entrance would have behaved like flow through a diffuser and accelerated the air to supersonic speeds. Therefore, conditions were selected to avoid supersonic flow. In order to maximize flow yet maintain subsonic conditions within the channels, a Mach number of 0.95 was specified at State 2.

In order to extract a reservoir pressure, the pressure at State 2 was specified as 709 kPa. Additionally, the low-temperature viscosity value for air of 3.8E-5 kg/m·s was established as the conservative case.

The hydraulic diameter used frequently in these calculations is 92.3 µm, which reflects the final dimensions of each air channel of 75 µm high and 120 µm wide. These dimensions were selected after understanding flow characteristics along with microfabrication limitations.

For turbulent, compressible fluids, flow is nearly independent of the surface roughness. Figure 4.1(b) indicates the Reynolds number variation with Fanno friction factor for a constant area duct. The friction factor spanned the entire range of values.
indicated in the Moody Chart (Munson, 1990). The Reynolds number maintained relatively constant values of the friction factor range, with a variation of 2.5% for the full range of wall roughnesses.

Figure 4.1(c) indicates the effect of varying friction factor on hydrodynamic entrance length. A difference of 0.4% in the normalized entrance length was observed over the range of friction factor values that spanned the Moody Chart range. Therefore, a negligible error resulted when using estimated friction factor values in the calculations of entrance length and Reynolds number.

Due to the relative independence of the Reynolds number and entrance length with respect to wall surface roughness, no attenuation of friction factor values was required for turbulent flow. Additionally, since most of the calculations found in this document were worst-case assumptions, these errors were well within acceptable levels for convergence on a design and for subsequent analysis. An average sidewall roughness
properly scaled to micrometer features was chosen for analysis. A ratio of the surface roughness to hydraulic diameter of 0.01 was selected. Including an estimated turbulent Reynolds number of 15,500 along with the surface roughness, the Moody Chart indicated a value of 0.042 for the Fanno friction factor.

4.1.1.1 Flow Conditions

The entrance length for the developing flow within the channels needed to be calculated. For laminar flow, this parameter was highly dependent on geometry and surface roughness. For turbulent flows, the entrance length depended primarily on duct size. In order to determine if the flow was turbulent, Figure 4.1(d) was used to elucidate Reynolds number variation with hydraulic diameter. The Reynolds number values in the plot predicted turbulent flow at the exit of the micro channels.

The linear relation in Figure 4.1(d) was reflected in Equation 2.3.2. Since the Mach number was held constant, the remaining properties of Equation 2.3.5 were also
constant. This plot provided information on turbulence for determination of the correct assumptions to calculate entry length.

4.1.1.2 Fanno Flow Modeling Results

The effects of Reynolds number on the entry length are shown in Figure 4.1(e). Using Equation 2.3.3 and dividing by the hydraulic diameter normalized the entry length, which gave the number of diameters required for fully-developed hydrodynamic flow. The Reynolds numbers varied due to changes in hydraulic diameter. The shape of the curve in Figure 4.1(e) reflects the dependence of entry length on $Re^{1/6}$.

The designated constant properties at state 2 resulted in constant density, velocity, and temperature, according to Equations 2.3.4 and 2.3.5. The Reynolds number and volume flow rate depended only on the cross-sectional area of the duct at state 2.
Therefore, the relation between entrance length and diameter was almost linear ($D_h^{7/6}$), as indicated in Figure 4.1(f).

Figure 4.1(g) shows the variation of developed duct length, which was normalized

Figure 4.1(e) Plot of the effects of entrance length aspect ratio ($Le/D_h$) for various Reynolds numbers.
by hydraulic diameter, with increasing hydraulic diameter when using Fanno assumptions. As shown in the figure, the entry length was less than 20 diameters for smaller hydraulic diameters, so less entry length is required for smaller ducts.

The variation of supply pressure with duct hydraulic diameter was plotted in Figure 4.1(h). The curve was asymptotic to the supply pressure axis. Small cross-sectional areas require far higher pressures to force the fluids through the channel. For a hydraulic diameter of 92.3 µm, the supply pressure indicated is 21 atm (2126 kPa).

The plot in Figure 4.1(i) reflects the variation of combustion chamber pressure with reservoir pressure. For a given duct cross-sectional area, the required supply pressure increased with exit pressure. If chamber pressure exceeded the 607 kPa, the fuel injector would be unable to withstand an increase in supply pressure to prevent backflow.
Figure 4.1(h) Supply pressure for compressed air required to generate Mach of 0.95 at end of channel for duct height of 75 µm and duct widths varying from 5 µm to 255 µm.

Figure 4.1(i) Effects of combustion chamber pressure on supply pressure for a constant area duct of hydraulic diameter of 92.3 µm.

Fanno flow did not account for variations in fluid temperature from external sources due to the adiabatic assumption. In order to include the diabatic effects, fluid
temperature was varied for a single duct size, as plotted in Figure 4.1(j). Variations in volume flow rate and reservoir pressure with temperature were generated for a duct of 120 µm wide and 75µm high. The lower temperatures required an increased supply pressure to generate a specified volume flow rate. The duct exit pressure was 709 kPa (7 atm), which, in the absence of a pressure gradient, corresponded to zero flow in the figure. For a supply pressure of 21 atm (2126 kPa) and 14 atm (1417 kPa) at 600ºC, the corresponding flow rates are 4.7 ml/s and 3.5 ml/s, respectively.

As shown in Figures 4.1(f) and 4.1(h), minimization of entry length and supply pressure created conflicting design constraints. Entry length increased with increasing channel size due to the slower boundary layer development. For broader channels, the no-slip effect required longer lengths to become fully developed. Supply pressure increased dramatically with decreasing channel size due to the increase in surface

![Figure 4.1(j)](image)

**Figure 4.1(j)** Variation of exit volume flow rate from a duct of 5mm length with supply pressure (P₀) at entry temperatures ranging from room temperature to 600°C.
roughness to hydraulic diameter ratio. As this ratio increased, sensitivity to channel surfaces increased. Another important consideration was the requirement of sufficient volumetric flow rate in order to feed enough combustive gases into the secondary combustion chamber to create the proper atmosphere for flame stability. Figure 4.1(k) indicates the variation in volumetric flow rate as a function of channel size at elevated room temperature (325K).

The design conflict could be reduced significantly by prioritizing the parameters according to fabrication limitations. Fabrication constraints led to a reduced thickness for Plate C, which contained the air swirlers on the surface, within the confines of out-of-plane deflection limitations. The intolerance of the design to plate deflection was the primary limitation on reservoir pressure. This matter will be further analyzed in Section 4.1.3. Ranges of hydraulic diameters corresponding to required parameters were summarized in Table 4.1(a).

![Figure 4.1(k) Volume flow rate variation with channel size at channel exit at 325K.](image)
4.1.1.3 Rayleigh flow

To maximize heat transfer, the temperature \((T_0)\) of the fluid within the stagnation reservoir was assumed to be 25°C and the exit temperature was 600°C, which was the maximum design temperature of the prototype fuel injector. Using Equation 2.3.14, the corresponding reservoir pressure \((P_0)\) was extracted using these conditions. The resulting supply pressure was 1508 kPa (14.9 atm).

4.1.2 Fuel Supply Pressure

Simplification of the flow path and fluid properties was necessary for approximation of the fuel supply pressure. Figure 4.1(l) shows the predefined points for tracing pressure gradients in the fuel. In this section, the results of calculations using the finalized fuel cross-section are presented.

Two different fuels were useful for flow and injection experiments with this prototype: ethanol and kerosene. These fluids vary only slightly in their properties, as indicated in Table 4.1(b), so ethanol was selected for all the remaining fuel calculations.

<table>
<thead>
<tr>
<th>(D_h ) (mm)</th>
<th>(L_e &lt; 200)</th>
<th>(P_0 &gt; 80)</th>
<th>(P_0 &lt; 2025 \text{ kPa (20 atm)})</th>
<th>Air flow rate</th>
<th>Fuel flow rate = 0.13 mL/s With 60:1 air dilution</th>
</tr>
</thead>
<tbody>
<tr>
<td>(L_e &lt; 5.0 \text{ mm})</td>
<td>(P_0 &lt; 2025 \text{ kPa (20 atm)})</td>
<td>Air flow rate</td>
<td>Fuel flow rate = 0.13 mL/s With 60:1 air dilution</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Since the fuel was atomized through the micro hole between States 2 and 3, it was necessary to determine whether compressible or incompressible models were to be used for fuel flow calculations. It is widely accepted that a compressible fluid with a Mach number less than 0.3 may be modeled as incompressible (Munson, 1990). To determine if fuel velocity was low enough to be considered incompressible, Equations 2.4.1 and 2.4.2 were used to determine the Mach number of the ethanol. The design fuel flow rate at the exit of each nozzle (State 3) was 0.13 ml/s. The Mach numbers of the fuel at States 2 and 3 were 0.03 and 0.01, respectively. These values were the maximum fuel velocities generated in the fuel injector in a channel length sized with the following dimensions: length of 1 mm, height of 75 µm, and width of 50 µm. Since these Mach values were less than 0.3, the incompressible fluid assumption was valid.

Table 4.1(b) Summary of fuel fluid properties.

<table>
<thead>
<tr>
<th>Fluid Properties</th>
<th>Ethanol</th>
<th>Kerosene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>789</td>
<td>800</td>
</tr>
<tr>
<td>Viscosity (Pa·s)</td>
<td>$1.2 \times 10^{-3}$</td>
<td>$1.6 \times 10^{-3}$</td>
</tr>
<tr>
<td>Bulk modulus (GPa)</td>
<td>0.902</td>
<td>-</td>
</tr>
</tbody>
</table>
Conservation of mass with constant density was used to estimate changes in flow rate with variations in orifice size. Equations 2.4.3 and 2.4.4 were used to calculate the pressure drop due to viscosity ($\Delta P_\mu$) and energy effects ($\Delta P_v$), respectively. Table 4.1(c) summarizes both types of contributions. The total pressure drop was 671 kPa, which indicates the pressure required to accelerate the fluid from stagnation to the air swirl chamber at the prescribed flow rate. Overestimation of the required pressure was made to include safety factors where possible since these calculations were so simplified. Thus, the negative values obtained for pressure changes from state 2 to 1 were neglected. Since the injection pressure into the combustion chamber was assumed 101 kPa over the combustion chamber pressure, the fuel reservoir pressure summed to 1381 kPa (200 psi). Some intermediate parameters in the fuel nozzle are indicated in Table 4.1(d).

### 4.1.3 Fastener Separation Distance

Table 4.1(c) Pressure contributions at the paths between the locations indicated in Figure 4.1(l).

<table>
<thead>
<tr>
<th>Paths</th>
<th>$\Delta P_\mu$ (kPa)</th>
<th>$\Delta P_v$ (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3→2</td>
<td>251</td>
<td>267</td>
</tr>
<tr>
<td>2→1</td>
<td>Negative</td>
<td>Negative</td>
</tr>
<tr>
<td>1→0</td>
<td>123</td>
<td>30</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Paths</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Total pressure loss (3→0)</td>
<td>671 kPa</td>
<td></td>
</tr>
<tr>
<td>Pressure required for entrance into combustion chamber</td>
<td>710 kPa</td>
<td></td>
</tr>
<tr>
<td>Total Fuel Reservoir pressure</td>
<td>1381 kPa</td>
<td></td>
</tr>
</tbody>
</table>

Table 4.1(d) Summary of flow conditions at the corresponding sites indicated in Figure 4.1(l).

<table>
<thead>
<tr>
<th>Location</th>
<th>Geometry</th>
<th>Velocity (m/s)</th>
<th>Flowrate (E-6 m³/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>$\phi_{\text{hole}} = 110 \mu$m</td>
<td>13.7</td>
<td>0.13</td>
</tr>
<tr>
<td>2</td>
<td>$\phi_{\text{hole}} = 75 \mu$m</td>
<td>29.4</td>
<td>0.13</td>
</tr>
<tr>
<td>1</td>
<td>Four 50µm x 75µm channels</td>
<td>8.7 per channel</td>
<td>0.03 per channel</td>
</tr>
<tr>
<td>0</td>
<td>Reservoir</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>
Eliminating out-of-plane deflection of the assembled plates by strategically locating fasteners would assist in sealing the reservoirs. The distances between fasteners were determined by initially considering the maximum distance between two fasteners that would maintain acceptable deflections. The distances were then mapped onto the nozzle pattern for orientation. Table 4.1(e) summarizes the material and physical properties that were used for the calculations for each of the plates.

The distributed load, in force per unit length, was based on values that were derived in the last two sections. The distributed load was the result of the pressure differential across the plate. It was calculated by multiplying the pressure differential and the width of the applied load. The width could have been chosen as a variety of different values for modeling purposes. The most conservative case was the one that generated larger loads. For these calculations, the chosen width was the working area of the circular reservoir, which was 76.2 cm². For each plate, the distributed load was determined by finding the pressure differential between the sides of the plate and multiplying that value times the working area. These values are indicated in the drawing in Figure 4.1(m).

The reservoir and combustion chamber pressures in Figure 4.1(m) show that the potential for back-flow of compressed air through the microhole existed. The maximum pressures on Plate C were predicted to be higher on the air (top) side than the fuel.

<table>
<thead>
<tr>
<th>Material</th>
<th>E (GPa)</th>
<th>Thickness (mm)</th>
<th>Poisson’s Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate A</td>
<td>Inconel 600</td>
<td>180</td>
<td>1.0</td>
</tr>
<tr>
<td>Plate B</td>
<td>Inconel 600</td>
<td>180</td>
<td>1.3</td>
</tr>
<tr>
<td>Plate C</td>
<td>Nickel 201</td>
<td>170</td>
<td>1.3</td>
</tr>
<tr>
<td>Plate D</td>
<td>Stainless Steel 316</td>
<td>193</td>
<td>5.2</td>
</tr>
</tbody>
</table>
Adjustment of the compressed air reservoir pressure to that of the fuel reservoir, 1417 kPa, prevented the opportunity for back-flow while maximizing the air flow rate.

Equations 2.5.1 and 2.5.2 were used to determine the maximum distance between fasteners.

\[
I = \frac{bh^3}{12} \quad 2.5.1
\]

\[
y_{\text{max}} = \frac{q_o L^4}{384EI} \quad 2.5.2
\]

The resulting fastener distance, L, determined the width shown in Figure 4.1(o). Assuming a square plate portion, L is equal to b in Equation 2.5.1. Therefore combining Equations 2.5.1 and 2.5.2 with the distributed loads indicated in Figure 4.1(n) and solving for L resulted in the fastener distance for a given maximum deflection. Figure 4.1(o)
Figure 4.1(n) Uniform distributed load applied to a clamped-clamped beam represents the pressure applied to the cross-section of the bolted plate. The distance between bolt centers is represented by L.

indicates the distance between fasteners for each plate as a function deflection. The final separation distance of 20 mm is indicated on the figure in this section.

Figure 4.1(o) indicates the minimal deflection of Plate D; however, the remaining plates created unacceptable gaps under their loads, due primarily to plate thicknesses. More realistic prediction of gaps in Figure 4.1(o) could be found by combining plates.

The assembled fuel injector with the plates laminated reinforced each plate to produce

Figure 4.1(o) Resulting fastener distances for incremental deflection values for each plate individually.
reduced deflections. As observed when comparing reservoir pressures in Figure 4.1.n, Plates A, B, and C would deflect to form a three-dimensional convex geometry and, similarly, Plate D would form a 3-D concave geometry. Plates A, B, and C were combined as a single Plate_{ABC} for modeling purposes. Plate_{ABC} was considered 3.25 mm thick with material properties of Inconel 600. This laminated single plate was a realistic simplification since the plates were designed to be tightly clamped together at many points. The pressure differentials between the fuel reservoir and the two external pressures located outside of Plates A and D as shown in Figure 4.1(m) were used. The resulting relation between the deflection of the two plates and the bolt distance are shown in Figure 4.1(p). The maximum deflection values indicated at the two points of intersection between the curves and 20mm fastener distance are 1 µm and 2.3µm for Plate D and for Plate ABC, respectively.

Figure 4.1(p)  Deflection values of assembly using air reservoir limited to 14 atm and the thin plates reinforced with Plate C, with length equal to 20 mm.
The curves in Figure 4.1(p) reflected improved reinforcement of the plates through assembly. In Figure 4.1(p), the 20 mm distance resulted in combined maximum gaps between mating plates of more than 3 \( \mu m \) when subjected to previously defined maximum operation pressures. These gaps were far too large for sealing purposes. Therefore, limitations on reservoir pressures may be imposed during testing for proper function of the fuel injector.

To verify the deflection values presented above, the secondary model using the Equations 2.5.3. and 2.5.4. This model was applied to two different plate sizes. Case 1 modeled the deflection of the reservoir assuming no internal fasteners. It was a circular geometry of radius 32 mm with a uniformly distributed load and it was constrained (clamped) in the circular pattern the model describes. The second case was to assume the four fasteners that were located about each swirler behaved like a circular clamping perimeter with radius of 10 mm, which was half the fastener distance. The results of these calculations are listed in Table 4.1(f). Due to the equal maximum design pressures on both sides of Plate C, the deflection would be zero.

<table>
<thead>
<tr>
<th>Case 1</th>
<th>Case 2</th>
<th>Yield strength at 600ºC (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bending stress</td>
<td>Bending stress</td>
</tr>
<tr>
<td>Deflection</td>
<td>(µm)</td>
<td>(µm)</td>
</tr>
<tr>
<td>Plate A</td>
<td>100</td>
<td>0.95</td>
</tr>
<tr>
<td>Plate B</td>
<td>356</td>
<td>3.4</td>
</tr>
<tr>
<td>Plate C</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Plate D</td>
<td>8.6</td>
<td>0.08</td>
</tr>
<tr>
<td>Plate ABC</td>
<td>20</td>
<td>0.19</td>
</tr>
</tbody>
</table>

Table 4.1(f) indicates the vast difference in deflection values depending on the circular plate as defined by the fasteners. Case 1 significantly overestimates the
deflection since it is assumed there are no fasteners surrounding the swirlers within the reservoirs. Case 2 slightly underestimates the deflection since there are only four fasteners, not a full perimeter of them. The 3 µm deflection determined in the analysis presented above is within the range indicated by this model.

4.1.4 Thermal Expansion of Plates

There were two areas of concern when considering the effects of thermal expansion. Expansion of plate thickness may exceed expanded bolt length, resulting in plastic deformation of the bolt during heating with insufficient clamping when cooled. Additionally, deformation in the xy plane, as indicated in Figure 4.1(m), due to thermal expansion could cause buckling if the fasteners were clamped to another device, such as a combustion chamber. Further investigation of both types of expansion was necessary.

For thermal expansion in the z-direction, a preliminary calculation using the thermal conductivity of the metal plates without fluids flowing through the reservoirs revealed that the temperature difference between Plates A and D was 4°C, with a temperature of 600°C for Plate A. This temperature difference was considered negligible and 600°C was applied to all four plates. Elevated temperatures cause expansion in metals, according to Equation 4.1.1.

\[
\Delta L_z = \delta_t \cdot L \cdot \Delta T
\]

4.1.1

where \( \Delta L_z \) is the change in plate thickness, \( \delta_t \) is the linear coefficient of thermal expansion, \( L \) is the plate thickness, and \( \Delta T \) is the change in temperature, with room temperature as the reference. The coefficient of thermal expansion is material specific and can be temperature dependent. Values of coefficient of thermal expansion were
chosen with the applied temperature range. These parameters and expansion results for the design temperature of 600°C are shown in Table 4.1(g).

The bolts expand 44.5 µm more than the accumulated plate expansion. This expansion difference could create spaces for leakage if not addressed. In order to maintain tension on each bolt at elevated temperatures, a lock washer was added. Further discussion on the specifications of hardware used for clamping the fuel injector is found in Section 4.2.

There are two different effects that made thermal expansion in the xy plane unworthy of further investigation. First, thermal expansion would be uniform due to the high conductivity of each plate, which would result in subsequent uniformity of expansion upon exposure to elevated temperatures. Additionally, the fastener holes were fabricated significantly larger than the shaft diameter. Therefore, relative motion of the plates, caused by differences in thermal expansion coefficients, would eliminate buckling. Isotropic expansion effects tend to cause dimensional changes symmetric about the centroid of the piece, with increasing effects as distance from the centroid increases. Thus, relative plate motion would have negligible effects on critically aligned features since they were located close to the centroid of the fuel injector.

4.1.5 Deflection of Microchannel Walls

The original design of the air swirler included straighteners, which split the

<table>
<thead>
<tr>
<th>Component</th>
<th>Material</th>
<th>δt (µm/m·K)</th>
<th>L (mm)</th>
<th>ΔL_{600} (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plates A, B</td>
<td>Inconel 600</td>
<td>13.3</td>
<td>2</td>
<td>22.6</td>
</tr>
<tr>
<td>Plate C</td>
<td>Nickel 201</td>
<td>13.3</td>
<td>1.25</td>
<td>14.1</td>
</tr>
<tr>
<td>Plate D</td>
<td>SS 316</td>
<td>18</td>
<td>5.2</td>
<td>79.4</td>
</tr>
<tr>
<td>Total for all plates</td>
<td></td>
<td></td>
<td></td>
<td>138.7</td>
</tr>
<tr>
<td>Bolts</td>
<td>SS 316</td>
<td>18</td>
<td>12</td>
<td>183.2</td>
</tr>
</tbody>
</table>
120 µm wide channel into four microchannels. The prototype was modified to remove the microchannels, but a brief analysis was necessary to determine the strength of these walls when subjected to fluid flow. This analysis determined if the material strength of the thin wall would be adequate if a distributed force acted on it.

The dimensions of each channel wall were 16 µm wide, 75 µm high, and 3500 µm long. These fins had a length aspect ratio of over 45. To simplify the effects of the fluid inside the reservoir, two times the original reservoir pressure (2126 kPa) was used as the applied pressure for modeling. The doubling of the reservoir pressure was realistic since at any moment there may be a positive pressure of that magnitude applied to one side of the wall and a negative pressure of that magnitude applied to the opposite side of the same wall. This value was translated into the distributed load by multiplying it by the fin height. A cantilever beam was used to model the fin since the top of the walls were not designed to adhere to the cover plate (Plate B). It was assumed the cantilever beam would provide a more conservative estimate of fin deflection than a clamped-clamped beam. The dimensions of the beam in the equations correlated to fin geometry according to the drawings in Figure 4.1(q). Equations 2.6.1 through 2.6.3 were used for these calculations. The moment of inertia was determined by using the full length of the fin as the base. The results of these calculations are listed in Table 4.1(h).

Comparing the applied stress on the fin to the yield strength of the electroplated nickel, 323 MPa (Sharpe, LaVan, & Edwards, 1997), these calculations predicted fin survival in reservoirs at the maximum design pressure.
These results indicated the ability of the fins to avoid yielding in the high-pressure air channels. There was no indication whether the adhesive strength of the electroplated nickel to the substrate may be a limiting factor in fin survival. There was also the clamping force of the capping plate to consider. This force could assist in reducing deflection or it could cause buckling of the fin.

4.1.6 Conclusions

Compressible fluids calculations along with plate deflection considerations revealed competing constraints in sizing between channel dimensions and the supply pressure for the air; however, a compromise was found to reduce supply pressure. Dilution quantities needed for mixing air and fuel with a ratio of 60:1 was not maintained, since only 27:1 was possible with the maximum design air supply pressure. However, a complementary air reservoir will compensate for the reduced air inside the combustion chamber.

<table>
<thead>
<tr>
<th>Reservoir pressure (kPa)</th>
<th>$M$ (N·m)</th>
<th>$\sigma_{\text{app}}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2126</td>
<td>$4.2 \times 10^5$</td>
<td>222</td>
</tr>
</tbody>
</table>
The finalized channel dimensions were determined from these calculations and are summarized in Table 4.1(i). To preserve the micrometer scale of the nozzles, swirler length minimization was a primary priority. A 92.3 µm hydraulic diameter would yield an air channel length of 2 mm. The channel length was eventually altered from the 2 mm to 5 mm since the prototype had more space to allow for longer channels.

The next section in this chapter reveals the details of the metal fuel injector design. The design process was iterative, using calculations, design criteria, and fabrication limitations to converge on a configuration feasible for fabrication and a robust assembly.

4.2 Prototype Design

For the design to be appropriate, the fuel injector must function according to the device specifications that were detailed in Section 1.3. An effective means of moving separate fluids through their own swirl channels and swirl chamber, forcing fuel into a microhole, and then spraying the air/ fuel mixture into the combustion chamber had to be determined for a micro nozzle.

Each micro nozzle was composed of a swirler mounted on a substrate at each end of a conically-shaped microhole, which passed through the substrate as indicated in Figure 4.2(a). A swirler was composed of four swirl channels, which tangentially fed fluids into a cylindrically-shaped chamber. This chamber was designed to induce swirl in
the fluids. A three-dimensional view of an individual swirler is shown in Figure 4.2(b). Air swirlers were located at the 100 µm end of the orifice, while fuel swirlers were located at the 50 µm end.

The design of the prototype required eliminating preconceived ideas, focusing on device function, and developing creative solutions to packaging. The calculations in Section 4.1 indicate the small dimensions of the swirler channels. The swirl chambers were required to be scaled with the channels similar to the image in Figure 4.2(b), which would result in swirl chamber diameters on the order 10^2. Moving the fluids through the channels and microhole into the combustion chamber required the channels to be capped.

Figure 4.2(a) Drawing of cross-section of a single nozzle. Note the size difference between the air and fuel swirlers.

Figure 4.2(b) Three-dimensional rendering of a generalized swirler.
The caps required a hole with a maximum size equal to the swirl chamber diameter for prevention of leakage. Alignment of these small holes over the center of each swirl chamber was necessary to prevent interference of the induced vortical profile of the exiting fluids.

Separation of the fluids to prevent premature mixing was also required. Attaching the fuel and air swirlers on each side of a thin plate was beneficial for minimizing the path length between the two swirl chambers and, again, preserving the vorticity in the fuel for improved mixing with the air. In order to supply the channels with the fluids, a submersion technique was adopted. Creating reservoirs of each fluid on the corresponding side of the plate allowed control of fluid velocity through the nozzle by controlling supply pressure. The reservoirs were defined and contained by the same plates that capped the swirl channels. Additionally, the reservoirs allowed multiple nozzles to be added to the plate since many swirlers could be submerged in the same reservoir as easily as a single swirler. The number of nozzles added was limited by swirler size and fabrication methods. The elevated pressures of the supply air and fuel required plate clamping devices along with leakage prevention. The air and fuel were supplied by fittings, the number of which could be far fewer than the number of micro nozzles. The connectors could have been located at various positions around the plates, but it was considered easier to attach them on the bottom side of the prototype since this would reduce interference of the supply lines with other features of the combustion chamber. Passages within the prototype were needed to isolate and direct the fluids to the appropriate reservoirs. Another reservoir was added above the high pressure air reservoir to provide extra air needed for controlled dilution of the combustive mixture at locations
adjacent to the mixture exit site. Supply lines and passages for this added reservoir were also required. Fabrication methods were balanced with nozzle function and design. A combination of precision machining, traditional machining, and micromachining were determined necessary for prototype fabrication.

4.2.1 Swirler Dimensions

The swirler design started with the microhole size and shape and the results of the fluidic calculations. The swirl chamber diameters were estimated by taking into account the amount of space needed to allow fluid to enter the swirl chamber tangentially from four points and to establish swirl before injection. Channel dimensions determined in Section 4.1 are summarized in Table 4.2(a) along with the finalized design dimensions of both air and fuel swirler chambers. The geometry of the conically-shaped microhole is reflected in the two microhole diameters listed in the table.

Early in the project, the developing entry length of each air channel was reduced by dividing the 120 µm wide passage into four microchannels. Each microchannel width was calculated by determining the widths of the dividing walls that could survive fabrication and operating stresses. The results of Section 4.1.6 indicated a 16 µm wall thickness does not physically yield in the turbulent fluid environment. As a result, four microchannels 18 µm wide were created with three 3.5 mm long fins. Figure 4.2(c) indicates the layout of the four micro channels in the air swirler. This design is referred to as Channel Design I. Channel Design II evolved after calculations indicated that

<table>
<thead>
<tr>
<th>Design Microhole Diameter</th>
<th>Swirl Chamber Diameter</th>
<th>Channel Width</th>
<th>Channel Height</th>
<th>Channel Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fuel Side</td>
<td>50 µm</td>
<td>200 µm</td>
<td>50 µm</td>
<td>75 µm</td>
</tr>
<tr>
<td>Air Side</td>
<td>100 µm</td>
<td>600 µm</td>
<td>120 µm</td>
<td>75 µm</td>
</tr>
</tbody>
</table>
Excessive pressures were required to force the fluid through the long microchannels. The microchannels were eliminated from the design, leaving the 120 µm wide channels for supplying air to the swirl chamber. Channel Design II is the air swirler design generically referred in the remainder of this document.

4.2.2 Plates

The plates were required to house the nozzles, cap the swirler channels, and accommodate fluid delivery. Fluid delivery required high pressure air, lower pressure air, and fuel transport from the external sources to corresponding reservoirs. The delivery system also included the supporting channels that eliminated premature mixing and facilitated prescribed mixing and injection into the combustion chamber.
4.2.2.1 Reservoirs

Reservoirs were used to transport specific fluids to the prescribed locations for proper function of the nozzles. Flooding the volumes that surround the array of swirlers with pressurized fluids maintained uniform pressure to the swirlers. Additionally, reservoirs serve to reduce the number of connection sites for supply lines for each fluid. Decreasing the need for multiple connectors per fluid conserves the compact size of the array of micro-injectors.

Alternatives to reservoirs would have required interfacing micrometer-scaled features with 1.5 cm diameter fluid connections. Without reservoirs multiple fluid connections for each swirler along with logistical problems in interfacing with the combustion chamber would have added much complexity to the design of multiple nozzles.

Three reservoirs were created for the three fluids. The reservoirs were isolated and sealed by stacked metal plates, as indicated in the cross-sectional view of the metal prototype shown in Figure 4.2(d). Reservoir 1 contained and supplied supplementary air for dilution control of the injected mixture after entry into the combustion chamber. The air in Reservoir 1 was held at pressures just above the combustion chamber pressure since it was not required to flow through microchannels. A seal was needed to isolate the air in the reservoir to prevent air from leaking into the air/fuel mixture before entering the combustion chamber. A separate air supply was channeled to Reservoir 2 at pressures which depended on fuel flow rate in order to meet prescribed dilution quantities of the injected mixture. Reservoir 3 housed the pressurized fuel.
The low pressure air was injected directly into the combustion chamber at locations adjacent to and surrounding the air/fuel mixture injection orifice through Plate A, as indicated in Figure 4.2(e). The injection holes were 1mm in diameter, which was large enough to machine easily, yet small enough to provide a fine distribution of air around the mixture exit site. The cylinder needed to seal Reservoir 1, as indicated in Figures 4.2(e), was 5 mm in outer diameter with 1 mm inner diameter.

For flow rate adjustment, the reservoir pressures were controlled externally with supply line pressure. A piezoelectric actuator was originally devised to pump fuel...
through the fuel swirlers; thereby controlling the frequency of injection. This feature could be integrated into the next generation prototype.

4.2.2.2 Dimensions

The design of the reservoirs and the need to seal them led to the laminated plate assembly shown in Figure 4.2(d). The metal plates provide strong, robust containment for the corrosive, pressurized fluids. The frontal area and thickness of each plate depended on many factors: maximum reservoir pressure requirements, feature sizes, fabrication limitations, mechanical properties of plate materials, and fastener sizes.

Each metal plate face was sized to allow space for the features, but within the confines of fabrication equipment compatibility. The face area of each square plate was 10 cm x 10 cm, which was small enough to fit in most of the fabrication equipment normally used to hold 100 mm circular silicon wafers. Figure 4.2(f) indicates a plate with the corners chamfered in order to accommodate the jig for precision machining, which is discussed in section 4.3.

Since the four plates were subjected to high pressures within the reservoirs, maximizing the thickness of each plate increased the stiffness of the assembly according
to results found in Section 4.1.3. The thickness of Plates A, B, and C were minimized in order to decrease travel distance of air/fuel mixture within the limitations imposed by plate deflection. The thickness of Plate D was maximized within limits of fastener shaft length. A balance between stiffness and other parameters resulted in a compromise in final thickness for each plate. The results are summarized in Table 4.2(b).

The thickness of Plate A is 2 mm since 1 mm of material was removed to create the low-pressure air reservoir, as discussed in Section 4.2.2.3.

Plate B was minimized in thickness within the limitations of dimensional stability. Plates generally have internal stresses which can cause deflection if they are too thin. The limit to plate thickness for the Inconel 600 and Nickel 201 plates was determined to be approximately 1.5 mm. This value varies by ±0.5 mm depending on the origin of the metal and the skill of the machinist performing the grinding.

The thickness of Plate C was dictated by the aspect ratio of the microholes, microhole drilling method, and capabilities of the machinist. The diameter of the microhole was originally 50 µm. EDM plunge drilling was the method chosen for drilling a conically-shaped microhole. Aspect ratio limitations for EDM plunge drilling were 17 for the experienced operator, Dean Jorgensen (personal communication, June 1999), with a nickel substrate, which is impressive when compared to conventional length to diameter ratios of 7 for mechanical drilling. As the diameter of the microhole decreased, the plate thickness was required to decrease by a multiple of 17. The plate

<table>
<thead>
<tr>
<th>Plate</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (mm)</td>
<td>2.00 ± 0.05</td>
<td>1.25 ± 0.05</td>
<td>1.250 ± 0.025</td>
<td>5.20 ± 0.05</td>
</tr>
</tbody>
</table>
was required be thick enough to maintain stiffness, flatness, and dimensional stability, yet thin enough to drill the microhole. A thickness of 1.25 mm with a minimum diameter of 75 µm was the most acceptable compromise that was compatible with the drilling process for Plate C. A tolerance of ±25 µm was imposed on the plate thickness, since variations would affect microhole aspect ratio and drilling results.

The face size and thickness of the plates became more important as fabrication methods were determined. The main difficulty encountered with fabricating metal plates was excessive weight. Some of the microfabrication equipment use automated wafer transport devices, which were designed for the standard 10 gram silicon substrates. The metal plates described in this section easily exceeded this limit. The plate area also posed problems for various precision machining and microfabrication jigs since it strayed from the standard 100 mm circular silicon wafer size.

4.2.3 Fluid Passages and Connectors

External connections and internal paths were required to direct fluids through the prototype while avoiding leakage and premature mixing. The fluid connections to the device were located on the bottom side of the prototype in order to minimize disorder of supply tubing and avoid interference with the combustion chamber. Internal passages were required to isolate the fluids as they traveled to their respective reservoirs.

Connectors for the external fluid supply lines were five compression fittings attached to the bottom side of Plate D, shown in Figure 4.2(g). One fitting for the fuel lines and two fittings of each air pressure line were connected. The number of air inlets were doubled since pressure distribution of the compressible fluids was a concern early in the project. Increasing the number of inlets ensured a uniform pressure distribution of air
inside Reservoirs 1 and 2. The locations of passage and connection sites are discussed in Section 4.2.8.

Specifications for the compression fittings are summarized in Appendix A. The fittings were attached by tapping Plate D at the entry sites and screwing in the compression fittings. Three and a half threads held the tapped portion of Plate D for each compression fitting. Strength was enhanced by welding the base of each connector at the plate interface. Since the fluids were designed to travel through the joint at a maximum pressure of 1417 kPa, the weld also sealed the joint to prevent fluid leakage and improve strength.

Internal passages were needed to move high pressure air past Reservoir 3 to reach Reservoir 2. Additionally, low pressure air had to pass through Reservoirs 3 and 2 to reach Reservoir 1. In order to eliminate leakage between the fluids in the passages and the reservoirs, annular seals between the passages and around the passage holes were machined in the plates, as indicated in figure 4.2(h). Plate D entered Reservoir 3 without bypassing another reservoir. The seals were required to align with the corresponding

Figure 4.2(g) The back side of Plate D. The five compression fittings are situated to connect with fluid supply lines.
fluid hole within a generous tolerance that was coarse compared to alignment requirements of other features on the plates.

4.2.4 Plate Fasteners

A fastening mechanism was needed to seal the reservoirs and maintain alignment between the plates. In choosing the fastening method, plate condition and fastening effects were important. Since sealing involved plate flatness and surface finish, these parameters had to be preserved. Fastening strength between the plates needed to be strong enough to withstand the separating tendencies of the maximum reservoir pressures without deflection. Bolts secured with nuts were fastening mechanisms used for this prototype.

Bolting the four plates together was a good choice since the fastening strength could be ensured to meet requirements. There were no modifications, such as heating or

Figure 4.2(h) Schematic indicating leakage prevention seals located between the reservoirs and internal passages. The low pressure air bypassed two reservoirs before entering Reservoir 1 and the high pressure air bypassed the fuel reservoir before entering Reservoir 2.
machining, to the plates during assembly which could compromise plate strength or ruin features. Disadvantages of using bolts included additional efforts to design appropriate sealing between the plates. Reduction in work area, such as reservoir volume and sites for more swirlers, resulted from designation of space for the bolt shafts and seals, as discussed in Sections 4.2.5 and 4.2.8.

Ultrasonic welding was a fastening alternative considered for the prototype. This method uses vibration and pressure to weld the two plates at their points of contact. A benefit to this method was its sealing capabilities. Ultrasonic welding was rejected since it introduces severe vibration to the weld site. The vibration would have deformed, damaged, or delaminated the smaller microfabricated features.

Bolt size was determined by balancing bolt strength, shaft diameter, and shaft length. Bolt strength depended on the material used and shaft diameter. Bolt shaft length was dictated by shaft diameter, since length to diameter ratio is standardized in order to reduce the possibility of buckling. Shaft diameter was minimized due to congestion of the other features within the limitations of bolt strength. Additionally, shaft length was required to be long enough to accommodate required plate, nut, and lock washer thickness.

Each plate was stacked along the fastener shaft, according to the image in Figure 4.2.b. The fasteners were 316 stainless steel M2 x 12 bolts, meaning a 2 mm shaft diameter and 12 mm shaft length. This was the longest standard length for the M2 bolt.

According to Figure 4.2(i), the bolt length was shorter than the required length to properly bolt the stacked components by 0.2 mm. This shortage was acceptable since it excluded half of a thread rotation, out of possible four, at the nut/bolt interface.
4.2.5 Sealing

Sealing was necessary to contain the fluid within each reservoir while it was forced through swirler channels. Leakage through various sites could contaminate the reservoirs, undermine the function of the swirlers, and reduce stagnation pressure in an unpredictable manner. Figure 4.2(j) shows a potential leakage path for a fastener hole. Fastener sites within the reservoirs were sealed by annular leakage seals located around the drilled holes, similar to the sealing for fluid passages discussed in Section 4.2.3. These cylinders compress against the mating plates at the fastener and fluid passage orifice sites.

The reservoirs, swirler channels, fluid passages, and fastener sites were capped and sealed through flatness and smoothness of the mating plates and parts. This procedure required two flat, smooth surfaces joined under compression to prevent leakage. Seals created by plate flatness demanded flat, smooth surfaces of mating parts.
The parameter used to measure the degree of flatness was waviness. Waviness is a recurrent deviation from a flat surface (Kalpakjian, 1990). It was measured and described by wave width and height, which are the horizontal distance between adjacent crests and the vertical distances between crest and valley, respectively. Waviness is commonly measured by an optical flat. Units are given in number of wavelengths of the incident monochromatic light. For this project, waviness was induced primarily by the curling of the thin plates and imperfect leveling by the lapping machine.

Surface roughness consists of closely spaced, irregular deviations on a smaller scale than the waviness parameter (Kalpakjian, 1990). Roughness is expressed in terms of its height, width, and the measured distance along the surface.

Analysis of the required surface parameters to avoid leakage was beyond the scope of this project. For an extensive review of theoretical sealing along with experimental data analysis, see Bhushan (2001). The use of the Bhushan parameters were sufficient for this project since surface parameters of the gas chromatograph (GC) project cannot be obtained with the large substrates used for the fuel injectors. The GC developed in the Bhushan thesis, had a footprint of approximately 25 mm x 50 mm, which allowed the use of polishing wheels for surface finishing. These wheels were too

Figure 4.2(j)  Example of leakage between mating structures surrounding a fastener hole.
small for the fuel injector substrate size. Additionally, Bhushan did not include waviness, which was important with a broader substrate. Finally, the GC internal pressure requirements were maximum 709 kPa, compared to the maximum reservoir pressures of 1418 kPa for the fuel injector. The elevated pressure would require improved surface requirements to contain the fluids. The Bhushan values were used as guidelines to fuel injector prototype parameters since even those finishes could not be obtained with the same surface finishing options available for the fuel injector plates. Specifications and results of surface finish are presented in Section 4.3.1.4.

Leakage through the fastener sites was detected with a submergence test since no fluids were expected to flow out of these holes. Of course, similar leakage could also occur over the swirlers, circumventing the channels, as shown in Figure 4.2(j). This problem would be difficult to detect since the fluid would continue to flow through the micro hole as anticipated. Particle image velocimetry (PIV) could be used to compare experimental results with expected injection profile since expected swirl would not be generated with this type of leakage.

Gaskets were considered as an alternative for sealing. It was decided the gasket thickness and the required deformation of the gasket material during assembly would interfere with reservoir volumes.

Interlocking mechanical seals were not used since design simplicity was a priority. It was determined that the flat plates would be less complicated than mechanical seals since the bond strength of electroplated structures to the plate was unknown. Adhesive strength to the substrate was a parameter vital to the success of mechanical seals since the mating materials are exposed to various lateral forces.
4.2.6 Alignment

The location of the plates relative to each other in the assembly required procedures for alignment. The air/fuel mixture exiting the nozzle passed from the air swirler and microhole through Plates B and A before injecting into the combustion chamber, as shown in Figure 4.2(k). Subsequent studies on the completed prototype would involve fluid profile characterization. Interference with the exiting mixture caused by misalignment between Plates A, B, and C could result in misrepresented experimental data. Additionally, the connection sites and fluid passages must be aligned in order to avoid leakage. The critical plate-to-plate alignment involved clearing the path for mixture injection without disrupting flow from the microhole to through Plate A.

Alignment pins were used in coordination with precision-drilled holes in each plate to achieve the prescribed alignment between the plates. Alignment of each plate relative to the others was obtained by inserting the pins in the same corresponding hole as those in the other plates, as indicated for Plate A in Figure 4.2(l). Once aligned, the plates were then fastened with the bolts and nuts. The alignment holes were needed for alignment only during assembly of the fuel injector, after which they served the purpose of fastener sites for connection of the fuel injector to the combustion chamber. Design parameters were not necessary for this secondary purpose of the alignment holes, since the fuel injector was expected to be retrofitted to the combustion chamber.

Proper plate alignment involved consideration of tolerances on size, location, and relative orientation of alignment and atomizing holes. In order to achieve uniform flow results from the exiting orifices, total error of less than ±50 µm was available for the misalignment between any two plates.
Tolerances based on machining capabilities were accumulated for the alignment holes and microhole drilling, as summarized in Table 4.2(c). Alignment holes were mechanically drilled, while the microholes were electrodischarge machined (EDM), as discussed in Section 4.3.

Figure 4.2(k) Schematic of the cross-section a single nozzle assembly with capping plates and reservoirs. The center line of the microhole, fuel swirler, air swirler, reservoir seal and each plate must be aligned relative to each other.

Figure 4.2(l) Photograph of Plate A as viewed from the side in contact with Plate B. The alignment holes are indicated.
Tolerances of the alignment holes were determined by allocation of total error available between plates and machining tolerances. The originating feature was the diameter of the alignment pin. Stainless steel dowels, which were machined to the accuracy indicated in Table 4.2(d), were used for the alignment pins. The resulting tolerances placed on the diameter of the alignment holes are indicated in Table 4.2(d), while the tolerance indicated in Table 4.2(c) was used to located the alignment hole positions. It is important to remember that the x-y positional error combined into a larger radial error.

EDM drilling locations were oriented to a datum created for the previously drilled alignment holes. The precision on EDM hole diameter was determined by the skills of the EDM machinist. The critical tolerance feature was the relative distance between the microholes. The error involved with relative location of these holes was ±8 μm. Once the first microhole was located against a datum, the other microholes were located with high precision. When comparing the air/fuel exit orifices in Plates A and B to the microhole centers, a maximum error of 160 μm was calculated. Though this error is outside of the ±50 μm total error for allocation, it is an error of 16% when viewed through the 1 mm exiting orifice. This error was acceptable to the design.

<table>
<thead>
<tr>
<th>Feature</th>
<th>Traditional drilling</th>
<th>EDM microhole drilling</th>
</tr>
</thead>
<tbody>
<tr>
<td>X/Y translation</td>
<td>±13 μm</td>
<td>±10 μm</td>
</tr>
<tr>
<td>Diameter</td>
<td>±13 μm</td>
<td>±7 μm</td>
</tr>
</tbody>
</table>

Table 4.2(c) Tolerance of drilling capabilities

<table>
<thead>
<tr>
<th>Feature</th>
<th>Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dowel size</td>
<td>3175 μm +5, -0 μm</td>
</tr>
<tr>
<td>Alignment hole size</td>
<td>3223 μm ±25.4 μm</td>
</tr>
</tbody>
</table>
To quantify alignment results, the degree of concentricity of the four air/fuel exiting orifices through Plates A and B were measured using a Nikon MM-22U Measuroscope (CAMD, Baton Rouge). This instrument provided distinct measurement with accuracy of ±5 µm through the combination of an optical microscope, camera, and measuring software. Images in Figure 4.2(m) show the swirlers photographed through Plate B. The small depth of focus common to microscopes required focusing on the swirlers for measurement, then, without moving the sample, focusing on Plate B for measurement, as shown in the bottom image of the figure.

All tolerance assumptions were based on drilling perpendicular to the surface. These were reasonable assumptions since the mechanical drilling was completed with high precision, the plates were flat to within ±5 µm waviness, and the microholes aligned with the microfabrication mask. The features on the microfabrication mask is a good check against plate machining since they were fabricated independently and were designed to align with each other.

For elevated temperatures, the resulting temperature distribution was effectively uniform through the fuel injector volume due to the high conductivity of each metal plate. Therefore, thermal deformation in the xy-plane was simultaneous and uniform for each plate. Relative plate alignment was maintained despite temperature variation.
4.2.7 Feature Layout

The nozzles, fluid passages, and fastener sites were located according to space available within the limitations imposed by fabrication. Consideration of fabrication techniques was vitally important in determining feature layout. Figure 4.2(n) 4.2(q) the features and their relative locations on Plate A. The shop drawings in Appendix B indicate the exact locations of the features.
The number of fasteners and their prescribed maximum distance apart was the original parameter for determining feature layout. The fasteners were required to be in contact with Plate B. The image indicates the drilled fastener, fluid, and alignment holes. Plate A has the low pressure air reservoir, which was milled 1mm into the 2mm thick Inconel plate. The supplementary air passages leading to the combustion chamber are shown surrounding the nozzle exits. The six alignment holes are shown on the periphery of the reservoir fastener holes.

Figure 4.2(n)  Photograph of Plate A as viewed from the side in contact with Plate B. The image indicates the drilled fastener, fluid, and alignment holes. Plate A has the low pressure air reservoir, which was milled 1mm into the 2mm thick Inconel plate. The supplementary air passages leading to the combustion chamber are shown surrounding the nozzle exits. The six alignment holes are shown on the periphery of the reservoir fastener holes.

The number of fasteners and their prescribed maximum distance apart was the original parameter for determining feature layout. The fasteners were required to be in contact with Plate B. The image indicates the drilled fastener, fluid, and alignment holes. Plate A has the low pressure air reservoir, which was milled 1mm into the 2mm thick Inconel plate. The supplementary air passages leading to the combustion chamber are shown surrounding the nozzle exits. The six alignment holes are shown on the periphery of the reservoir fastener holes.

Figure 4.2(o)  Drawing of Plate B, which has the two large low pressure fluid holes.
the same locations on all the plates and to be maximum 20 mm apart on center to minimize plate deflection due to reservoir pressure. Fasteners were needed to surround each swirler to reduce the possibility of fluid leakage between the swirler and cap plates. Four fasteners in a diamond pattern surrounded each swirler, as shown in Figure 4.2(p).

To seal the outer edges of the reservoir, fasteners and their seals were needed outside of the reservoir diameter, yet within the 80 mm working diameter imposed by microfabrication mask work areas. This parameter resulted in the reservoir diameter of 64 mm.

It was estimated that maximizing the reservoir volume within the prescribed diameter would allow easy maintenance of reservoir pressure uniformity. Four nozzles were determined sufficient for flow analysis and would leave adequate reservoir volume.

The air swirlers were the largest nozzle features, which limited how closely the fasteners could be positioned to the nozzles. The nozzles were centered in the reservoir and positioned closely in order to share fasteners, as indicated in Figure 4.2(p).

Figure 4.2(p) The images show features for both sides of Plate C. The air and fuel swirlers are Plate C Air side he corresponded by Plate C Fuel side t diameter circle in each drawing.
The alignment holes were located in the same position on all the plates. They were the six holes located outside of the prescribed working area. It was important to maximize the distance between alignment holes to improve positional accuracy. It was determined acceptable to locate the alignment holes outside of the microfabrication mask area since these holes did not have to be sealed, which means they were not included in the microfabrication features, as discussed in Section 4.3.

The fluid passages were drilled through the plates as indicated by the large holes shown in Figure 4.2(n) through 4.2(q). The passage seals were sized 6 mm outer diameter and 2.5 mm inner diameter, so that the seals would be robust. The seals were strategically placed, since they were not needed for fluid passages leading to the

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Figure 4.2(q) Drawing of Plate D with fluid supply lines and connectors indicated. The alignment holes are not indicated in this drawing. The supply lines are labeled on Plate D and the large holes on the other plates correspond to the labeled supply lines indicated on Plate D.
corresponding fluid reservoirs. The number of fluid holes drilled in a specific plate depended on how many fluids passages were by-passing and entering a reservoir.

Plate D had all five passages since the external connection sites were included on this plate. The center hole was the fuel supply, which supplied Reservoir 3, as shown in Figure 4.2(r).

The remaining four passages drilled into Plate C provided the path to bypass the fuel reservoir by including seals on the fluid supply passages, as shown on the fuel side of Plate C.

The high pressure air was supplied to the Reservoir 2. The absence of seals on the high pressure supply passage on the air side of Plate C facilitated flow into Reservoir 2, as shown in Figure 4.2(p). The low pressure air bypassed this reservoir through the two seals shown on the air side of Plate C.

Plate B capped the features on the air side of Plate C, so the low pressure air passages were drilled through. Plate A contained the complementary air supply holes that lead to the combustion chamber adjacent to the sealed air/fuel mixture exit orifice. An array of these 1 mm diameter holes was located in Plate A at each nozzle exit.

Figure 4.2(r)  Schematic of the cross-section of the prototype assembly with a single nozzle, capping plates, and reservoirs.
4.3 Prototype Fabrication

Precision machining methods along with updated microfabrication techniques were used to create the prototype metal fuel injector. Traditional methods included mechanical milling and drilling, tapping, welding, and polishing. The fastener and alignment holes were drilled simultaneously since they were common to all four plates. The supplementary air reservoir was milled out of Plate A. Fittings were assembled on the backside of Plate D. Surface finishing was accomplished for sealing. Less common methods were needed for the atomizing holes and the swirlers. Plunge EDM was used to drill the conical microholes. Microfabrication using the UV-LIGA technique was used to form swirlers and supporting structures.

Microfabrication was used to create the swirlers, the fuel and high pressure air reservoirs, and the seals associated with those reservoirs, because conventional machining could not yield the precision and accuracy required in this project to form the swirler channels and chambers. The materials compatible with microfabrication techniques make microfabrication attractive for high temperature applications. Nickel can be electroplated on metal substrates and ceramics can be injection molded into micro molds.

Mounting the microfabricated structures on both sides of Plate C was preferred over single-sided mounts on two different substrates due to the material properties of the plates. Double-sided structures on Plate C required only one plate that was compatible with microfabrication techniques. The alternative of single-sided mounts would have required the use of two substrates that were compatible with microfabrication. The best metal for the high temperature fuel injector was Inconel 600. Nickel 201 was the most
acceptable metal for meeting the design standards for the prototype and acting as a microfabrication substrate. However, the physical properties of Nickel 201 were inferior compared to the Inconel. Thin Inconel 600 plates had more strength at high temperatures with reduced deformation. However, Inconel 600 made a poor substrate for microfabrication due to the tendency of low-content nickel alloys to form passive oxide layers that inhibit adhesion to the microstructures. Maximizing the number of plates composed of materials with strength at high-temperatures was a step towards a more robust assembly. Thus, Plate C was composed of Nickel 201 and was the only plate with microfabricated structures formed on both sides.

There was the additional option of placing the microfabricated structures on Plate D. Although Plate D was stainless steel, which is not an uncommon microfabrication substrate, adding microfabrication to the many additional steps involved in completion of Plate D would have reduced the chances of successfully completing that component. The substantial thickness and weight of Plate D would have been difficult to accommodate in microfabrication equipment. The addition of protruding fluid fittings would have presented further difficulties at each step in the microfabrication process to make this option prohibitive.

Double-sided microfabrication added to the complexities involved with device fabrication. There were no previous examples to date in which thick (>50 µm) mechanical structures were microfabricated and machined on both sides of an opaque substrate. There is good reason for this: the problems involved in preserving the first side during processing the second side were so complex that other assemblies are designed for single-sided microstructure formation. These problems include process
chemical contamination, abrasive equipment, isolating the structures during electroplating, and mechanically destroying features on the first side during surface finishing. However, it was decided the benefits of double-sided microstructures outweighed the obstacles and the air and fuel swirlers and reservoirs were designed to mount on either face of Plate C.

The material removal methods of necessary precision machining are presented in Section 4.3.1. UV-LIGA was the preferred microfabrication method, as discussed in Section 4.3.2

4.3.1 Precision Machining

Material removal was most effectively accomplished by traditional machining methods. Drilling holes, milling cavities, and grinding and polishing surfaces were the most cost effective and common techniques available for creation of most features on the fuel injector plates. However, even the traditional machining methods were time-consuming and costly due to the stringent tolerances, discussed in Section 4.2, and the prototypical nature of the project. Microholes were drilled with a non-traditional EDM plunge technique.

4.3.1.1 Cutting

A water jet was used to cut the four plates 100 mm square, nominally. The corners of each plate were removed as shown in Figure 4.3(a) in order to fit the plates for the lapping jig.
4.3.1.2 Grinding

Grinding was necessary in order to obtain the proper thickness and flatness before drilling the microholes. Plates B and C were designed to be the thinnest plates at 1.25 mm. Considerable skill was required to prevent these plates from curling during grinding. Grinding induces surface stresses on the plates. These stresses encourage bending, or curling, tendencies at the stress site. If the plates are thin enough and the yield strength low enough, these surface stresses may deform the plate. Double-sided grinding reduced curling in the thin plates since both faces were simultaneously stressed, which tended to offset the stresses. This method was used for creation of Plate B and Plate C (Don’s Grinding, Houston, TX). Obtaining the required thickness and flatness of Plates A and D by grinding was not as demanding since these plates were significantly thicker. Appendix B indicates the thickness, flatness, surface roughness requirements for initial grinding for each plate. The results of surface finishing are discussed in Section 4.3.1.4.
4.3.1.3 Traditional Drilling

A CNC (Computer Numerical Control) drilling/milling machine (Precision Industries, Baton Rouge, LA) was used to mechanically drill the alignment holes. The four plates were clamped together for simultaneous drilling of the holes common to all plates, which were the fastener and alignment holes. This method reduced the error in relative plate alignment. It also reduced burr formation, thereby conserving plate grinding finish. The remaining holes, which were the large fluid passages, were drilled through each individual plate as specified. Tolerance analysis on critical features was necessary to ensure adequate alignment for the final assembly. In Section 4.2.6., the tolerances for drilling the alignment holes were determined by accumulating tolerances on the alignment system, microholes, and exit orifices.

Reservoir 1 was milled from Plate A with the CNC milling machine to a depth of 1 mm (Precision Industries, Baton Rouge, LA). The 6 mm diameter cylindrical fastener seals were milled with nominal dimensions. Nominal refers to the ideal dimension with undefined accuracy. Since error in the dimensions of the seal did not affect the function of the prototype, unspecified accuracy was acceptable.

Fastener holes for each plate were drilled according to the shop drawings found in Appendix B. These holes were drilled with the CNC milling machine and the tolerances of location and size were nominal. The holes were drilled with a diameter of 2.5 mm for bolts that have a 2 mm shaft diameter in order to easily clamp the plates with the bolts.

Plate B was drilled with 600 µm diameter air/fuel exits, as shown in Figure 4.3(b).
Plate C was drilled similarly to Plate B, but with two more fluid passage holes and the four microholes substituted for the larger mixture exits found in Plate B.

The holes in Plate D were drilled and the connectors attached as shown in Figure 4.3(c). The fluid connection sites were tapped to screw the compression fittings and then welded along the perimeter to strengthen and seal the joint.

4.3.1.4 Leveling and Polishing

Leveling and polishing were surface finishing steps required to obtain the flatness and smoothness of sealing surfaces. Leveling refers to reduction in waviness over the plate face. Polishing refers to decreasing surface roughness.

A single-sided lapping machine was used to level and polish Plates A and D, since only one sealing side needed polishing for each plate. Both sides of Plate B were also single-side polished.

Figure 4.3(b) Plate B-2 has the large fastener and low pressure air inlets along with 600 µm diameter air/fuel exit holes.
Single-sided polishing was used for Plate C because the microfabrication procedures required it. Since both sides of the plate needed polishing after the microstructures were formed on the surfaces, it would seem advantageous to lap both sides simultaneously in order to ensure parallel faces. The lithographic photoresist, which is structurally strong, was retained during lapping in order to protect the metal structures from damage. When the second side of the plate was processed, the resist from the first side was removed to avoid contamination of the process chemicals. Without the resist on the first side, double sided polishing could not be accomplished. Thus, single-sided lapping was necessary for final surface finishing of Plate C.

Additional difficulties with double-sided lapping involved supplying “dummy” plates to balance the machine. These extra plates were required to be the same thickness and material as the substrate. Calculation of plate and machining monetary costs along with the time to obtain the extra plates made single-sided lapping a relatively simpler and cheaper solution.
The single-sided lapping machine used for surface finishing was an Engis 15LM/V (Engis Corporation, Wheeling, IL), which is shown with accessories in Figure 4.3(d). The pictures indicate the lapping wheel, slurry and sprayer, control panel, and roller yoke arms. The substrate was mounted onto the bottom, flat surface of a 4.5 kg cylindrical weight, as indicated in Figure 4.3(e). The weight was placed, with the substrate facing down, inside a ceramic-backed stainless steel ring. The ring was cradled by the roller yoke arm. Rotation of the lapping plate induced rotation of the ring and substrate within the roller yoke arm. The rotation of the sample on the rotating wheel provided polishing over the whole sample face and polishing wheel surface. The sample was polished under the influence of two parallel axes of rotation with different origin locations and angular speed.
Figure 4.3(e) Ceramic-backed stainless steel lapping jigs are shown with the mounting weights in place on the lapping wheel.

The rotation rate of the ceramic-backed rings set the substrate rotation speed, due to friction between them. The roller yoke arm guided the rotation rate of the ceramic-backed rings. Two roller yoke arms were available: manual and automatic. The manual version allowed the substrate to rotate with speed induced by the rotating lapping wheel, though at times the substrate could slow due to friction between the ring and the polishing wheel. The automated version had a variable speed motor which set the ring rotational speed. The automated version was used to polish all of the plates in order to obtain a consistent substrate rotation rate.

Components for the polishing machine included the controller for the lapping plate, which controlled the angular rate of the lapping wheel and synchronized the spraying timer with the lap plate motion. The oil-based slurry was continuously mixed by the magnetic stirrer. The oil-based slurry was a slight improvement over the water-based slurry for the nickel and nickel alloys that were being polished. Compressed nitrogen was needed to spray the slurry. The 15” outer diameter (OD) and 4” inner
diameter (ID) annular lapping wheel was composed of a copper composite with a waffle surface texture. Diamond covered cylindrical rings of 5.75” diameter were used to condition and clean the wheel.

Operator experience with the lapping machine dictated the degree of success of the resulting surface conditions. Consultation with experienced machinists originally revealed a few key parameters that must be controlled in order to obtain the required flatness. Maintenance of wheel flatness was critical. During lapping, the sample eroded the polishing wheel. Over time, this wear can be significant and can alter the shape of the wheel, resulting in unsatisfactory sample topography. Incremental level checking was required in order to correct deviations from level before the sample was ruined. After experimenting, the polishing wheel was determined to wear uniformly during lapping due to the large substrates used for this prototype. Initial leveling of the lapping wheel was required, and then it was self-maintained to within ±12 µm.

Wheel level monitoring was achieved with a two-point level. This level was composed of two individual gauges mounted in tandem along with supports through a granite leveling bar. The bar spanned the outer diameter of the 15” polishing wheel. The supports contacted two opposite points on the wheel OD. The gauges contacted a single point on both the ID and the surface center of the wheel, which was half the distance between the ID and OD, as shown in Figure 4.3(f). It was assumed the wheel level was uniform in the tangential direction. Due to symmetry about the wheel center, the height of two radially varying points relative to the OD height was sufficient to determine the degree of waviness.
Conditioning referred to the process of leveling the wheel. Conditioning was achieved with a diamond-grit conditioning ring and lubricant. If the ID was higher than the OD, the wheel was considered concave. The amount of waviness was the height difference between the polishing wheel OD and ID. To level a concave wheel, the ring was shifted off of the polishing center towards the wheel ID. The amount of shift was determined by trial-and-error. It was found that extreme shifting towards the wheel OD such that the ring OD was over the wheel center of rotation was necessary to level an exceptionally concave wheel. If the OD was higher than the ID, the wheel was considered convex. To level a convex wheel, the ring was shifted towards the OD of the polishing wheel. The best leveling obtained was 2 µm as measured between the ID and polishing center of the wheel.

The wheel became abrasive during lapping through charging of the wheel surface. Charging was achieved by embedding abrasive particles into the wheel surface by spraying the diamond slurry onto the polishing wheel intermittently during lapping. The slurry consisted of specific-sized diamond particles mixed in oil. The spray time consistently used for lapping the solid plates was 3 seconds every 33 seconds, with a wheel speed of 5.75-6.25 rad/sec. To avoid delaminating the electroplated structures,
increasing the frequency of slurry spray to every 28 seconds and reducing wheel speed to 4.70 rad/sec provided more gentle material removal.

To avoid burrs or sharp edges that could scratch the lapping wheel, 540 grit sandpaper was used to hand sand the electroplated surface before leveling on the lapping machine. It was important to maintain flatness when hand-sanding. A piece of ½” thick and 7” square Plexiglas, was used as a sanding block, with the sandpaper taped to it. The Plexiglas was used since it is quite flat, wears slowly, and the large thickness prevented deflection while in use. If there was expansion of the Plexiglas block due to heat or moisture that made the sanding uneven, it was inconsequential since the piece was subsequently leveled on the lapping machine.

Leveling and polishing was achieved on the lapping machine by beginning with larger grit sizes and incrementally decreasing to the finest grit. The finest grit used for these samples was 0.1 µm, which was reached by stepping down through 9 µm, 6µm, and 3 µm particle sizes. These slurries removed material at an accelerated rate compared to finer grits. Then 1 µm, 0.5 µm, and 0.1 µm sizes were used, which further reduced surface roughness.

A surface profiler (KLA-Tencor Alpha Step 500, San Jose, CA) located at CAMD was used to view resulting waviness parameters. The profiler was a metrology tool that generated a two-dimensional profile of the surface of a sample. This was accomplished by a stylus, which touched the surface of a sample and was scanned across a specified length. Waviness values extracted by the surface profiler were suspect due to the overall plate curling. With the drift associated with the profiler, it was impossible to determine
the difference between mechanical/electrical drift and surface slope. Therefore, waviness values were too unreliable to be included in this document.

Surface roughness was expressed through the root mean square average, $R_q$. Equation 4.3.1 indicates the mathematical relationship.

$$R_q = \sqrt{\frac{a^2 + b^2 + c^2 + \ldots}{n}} \quad 4.3.1$$

The roughness data obtained from the profilometer was a series of waves traced by the stylus. The root mean square (RMS) was related to the average wave height as indicated in Figure 4.3(g). The values of the RMS roughness are equal to or slightly greater than the average of the surface deviation from zero. Table 4.3(a) summarizes mean values of $R_q$, averaged from ten separate scans per plate at locations which varied radially from the center. The elevated standard deviation values indicated the difficulty in reliably determining surface roughness with the profiler. Additionally, the stylus tip had a radius of curvature of 12.5 µm, which resulted in a trace that was smoother than the actual surface roughness.

As indicated in Table 4.3(a), the surface roughness for the electroplated structures was much higher than the other plates. These electroplated portions were subjected to less lapping in order to prevent loss of adhesion between the nickel structures and substrate. Previous adhesion problems demanded delicate handling of these structures.

Figure 4.3(g) Definition of roughness parameters (KLA-Tencor, San Jose, CA).
The optical profiler at CAMD (model NT3300, Veeco, Woodbury, NY) was used to determine surface roughness and to compare the data with the stylus version, as shown in Table 4.3(a). This machine used interference optical light waves, which reflected from the surface of the sample, to determine vertical changes in surface topography. The optical profiler had a resolution of 1.0 nm for the average $R_q$. Since the polished metal plates had good reflectivity, the precision of the data was within 2 nm. Reproducibility of the data was also determined experimentally on the optical profiler. A mean and standard deviation of a rescanned site ten times was 96.742 nm and 0.605 nm, respectively. An image from data taken with the optical profiler of a rough sample is indicated Figure 4.3(h).

<table>
<thead>
<tr>
<th></th>
<th>Stylus Profilometer</th>
<th>Optical Profilometer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$R_q$ (nm)</td>
<td>Standard deviation (nm)</td>
</tr>
<tr>
<td>Plate A</td>
<td>18.7</td>
<td>5.6</td>
</tr>
<tr>
<td>Plate B: A interface</td>
<td>15.2</td>
<td>4.8</td>
</tr>
<tr>
<td>Plate B: C interface</td>
<td>13.7</td>
<td>3.2</td>
</tr>
<tr>
<td>Plate C: sandblasted substrate</td>
<td>610.8</td>
<td>317.3</td>
</tr>
<tr>
<td>Plate C: air side</td>
<td>38.0</td>
<td>7.4</td>
</tr>
<tr>
<td>Plate C: fuel side</td>
<td>57.7</td>
<td>22.3</td>
</tr>
<tr>
<td>Plate D</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
The values determined from each profilometer were quite different, though they are both consistent in determining smoother and rougher surfaces. The stylus profiler values indicate smoother surfaces on the rougher samples compared to the optical profiler. The values were almost the same for the smoother samples. The radius of curvature of the stylus tended to reduce surface roughness significantly. Analysis of surface roughness with the surface profiler was determined unacceptable for valid reporting of surface roughness. The data from the optical profiler were the valid surface roughness data.

During the lapping procedure, there were difficulties in using a solid mounting weight to hold the sample. Removing the substrate from the mounting weight could cause the plate to warp. If a plate warped, then it was ruined, since it could not be sufficiently flattened again. Double-sided sticky tape held the substrate to the weight. Acetone was used to dissolve portions of the tape in order to gently pry off the plate.
tape was approximately 1.5 mm thick and it was soft. The variation in tape thickness depended on the pressure applied. Efforts were made to evenly tape the backside of the substrate and maintain uniform load. There were no adverse effects to the plate polishing surface while using the tape, since the tape was carefully adhered between the sample and weight over the entire area of the plate. With full contact of the plate to a single layer of tape, there were no bumps or trapped air pockets to produce flaws on the polished surface. A vacuum chuck would have eliminated these problems.

The height of the electroplated structures was gauged relative to the resist, since it was impossible to use a profilometer on a sample with a 4.5 kg weight attached to the back side of it. It was not practical to periodically remove the substrate from the weight. Therefore, when the electroplated structures were level with the resist, determined by running a finger along the interface, the structures were at an acceptable height and finer polishing could be started.

Deep, random scratches were found on the lapped areas. These scratches were caused by contamination of the polishing wheel with coarse particles.

The lapping machine sometimes removed material at an angle relative to the reverse side surface, which would be compounded when lapping the second side. Plate C was assumed flat with parallel faces before micromachining. The thickness varied by $\pm 5\mu m$, as measured with a digital micrometer with 1 $\mu m$ accuracy. The electroplated surface was sometimes sloped relative to the plate face. It could be thinner close to the waterline and thicker near the bottom of the tank. When lapping the first electroplated side, the surface was flush with the polishing wheel with the mounting weight on the back side of the plate. Thus, the electroplated surface was initially leveled at an angle,
and removal continued at that same angle. The difference in swirler heights on one measured sample was 20 µm. When lapping the reverse side, the angle between the two faces could increase if the same edge was the waterline edge. However, this effect could also be offset if the edges were reverse during electroplating and similarly sloped electroplated structures were formed. Modifications with the electroplating process were also used to reduce the sloped features, as discussed in Section 4.3.2.8.

The flatness of the back of the plate dictated the pressure distribution of the lapping wheel on the substrate. When PMMA filler was ‘puddled’ around the holes on the reverse side of the plate, a bumpy, hard surface was formed on the plate surface, as indicated in the photograph in Figure 4.3(i). When the weight was applied to the bumpy surface during lapping, the applied pressure was concentrated at the bumps on and the increased pressure in this localized region was indicated on the polished side, as shown in Figure 4.3(j).
4.3.1.5 Microhole Drilling

The fabrication method for creating the microholes was determined by considering drilling aspect ratio, the quality, size, and geometry of the holes, positional tolerances, and localized heating and deformation. Electrodischarge machining (EDM) wire plunge drilling was the best method for the conically-shaped holes. The specified 50 µm holes were actually drilled to approximately 75 µm in diameter at the fuel swirl chamber to 115 µm in diameter at the air chamber.

Traditional drilling could not form the microholes due to their large aspect ratio and conical geometry. Drill bits commonly have shank length to diameter ratios of 7, while the atomizing holes required an aspect ratio of 20. Electrodepositing the plate with the holes was also considered. However, this method would create holes with vertical sidewalls. A number of other methods were reviewed, as summarized in Table 4.3(c).

Localized heating and deformation were complications to avoid since plate flatness and material strength were critical. Hole quality, involving surface characteristics at both ends of the hole was also considered. ECM etches nickel
isotropically, which forms a hemi-spherical geometry. LBM and EBM result in localized heating and blow-out of the exit end.

EDM plunge drilling involved the use of thin wire used as the electrode sized slightly smaller in diameter than the prescribed hole. The substrate was submerged in a dielectric fluid and a dc power supply connected the electrode and substrate. When the potential difference between the charged electrode and nickel plate was appropriate, a transient spark arced from the fluid causing localized erosion of the substrate. The critical gap between the wire electrode and substrate, which dictated potential difference, was controlled by a servomechanism as it continued to drill vertically through the substrate. Throughout the procedure, the diameter and length of the electrode was also continuously eroded. Upon penetrating the backside of the plate, the reduced diameter of

<table>
<thead>
<tr>
<th>Process</th>
<th>Characteristics</th>
<th>Process Parameters and Material Removal Rate (MRR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrochemical machining (ECM)</td>
<td>Complex shapes with deep cavities; highest rate of material removal among nontraditional processes; expensive tooling and equipment; high power consumption; medium to high production quantity.</td>
<td>Potential: 5-25 dc Current: 1.5-8 A/mm² MRR: 2.5 – 12 mm/min, depending on current density</td>
</tr>
<tr>
<td>Electrical-discharge machining (EDM)</td>
<td>Shaping and cutting complex parts made of hard materials; some surface damage may result; also used as a grinding and cutting process with traveling wire; expensive tooling and equipment.</td>
<td>Potential: 50-300 V Current: 0.1-500 A MRR: Typically 0.15 cm³/min; 0.1-0.25 cm³/min for grinding.</td>
</tr>
<tr>
<td>Laser-beam machining (LBM)</td>
<td>Cutting and hole making on thin materials; heat-affected zone; does not require a vacuum; expensive equipment; consumes much energy.</td>
<td>MRR: Typically 0.006 cm³/min</td>
</tr>
<tr>
<td>Electron-beam machining (EBM)</td>
<td>Cutting and hole making on thin materials; very small holes and slots; heat-affected zone; requires a vacuum; expensive equipment.</td>
<td>MRR: 0.0008-0.002 cm³/min</td>
</tr>
</tbody>
</table>
the wire determined the exit size. The entrance of the hole eroded during drilling such that the resulting entrance size was approximately 20% larger than the original wire diameter. The resulting cross-sectional shape of the orifice was conical.

Two different substrates were EDM drilled for the prescribed microhole geometry and size. The first one, known as Plate C-1, was drilled with the four micro holes after the large holes were drilled. The second one, known as Plate C-2, had only the four micro holes drilled through the substrate. The fastener, fluid, and alignment holes for Plate C-2 would be drilled after micromachining.

The microholes were drilled by the plunge EDM method by the machinists at Optimation, Inc (Midvale, UT). For a starting wire diameter of 100 µm, the holes were drilled to an average diameter of 117 µm at the entrance side of the plate and 78 µm at the exit side. Photographs of the microholes are shown in Figure 4.3(k). Table 4.3(d) indicates the average entrance and exit diameters of each hole drilled by this method. The standard deviation indicates that the accuracy of the exit diameter was less than the accuracy of the entrance hole diameter. This was due to the variation in erosion of each electrode for the individual holes. The surface quality of the microhole wall is assumed rough. Since it would destroy the plate to characterize the sidewall roughness of the hole, this parameter was not investigated. The hole sizes for the fuel side of Plate C-2 could not be obtained since the plate was processed and the microholes were overplated before measurements were made.

The microholes were drilled within 25 µm relative position to each other for Plate C-1, which was within the tolerance specified. This was verified by use of the UV mask during alignment.
In order to determine the most efficient method of microfabrication, process steps were evaluated for compatibility with the fuel injector substrate and feature characteristics. Efforts to use X-ray lithography were made since the solid resist, polymethyl methacrylate (PMMA), was simple to use with the unconventional Nickel 201 substrate. During the design phase of this project, the X-ray exposure station for the LIGA fabrication process had limitations on wafer size and geometry. X-ray exposure was accomplished by scanning the substrate with a horizontal beam of a given span and stroke length, which were predetermined by the exposure station used at the LSU CAMD synchrotron facility. Early in the project, the X-ray beamline was 5 cm wide with a

![Optical photographs of micro holes created by EDM plunging. Shown are the exiting diameters of the cone-shaped profile of orifice on the fuel side (left) and the air side (right) of Plate C.](image)

**Figure 4.3(k)** Optical photographs of micro holes created by EDM plunging. Shown are the exiting diameters of the cone-shaped profile of orifice on the fuel side (left) and the air side (right) of Plate C.

### 4.3.2 Microfabrication

Table 4.3(d) Average diameters of drilled microholes of both Plate C samples as measured through a microscope. The small diameters of the fuel side measurements indicate wire exit and the air side corresponds to wire inlet location.

<table>
<thead>
<tr>
<th>Plate</th>
<th>Diameters (micrometers)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hole 1</td>
</tr>
<tr>
<td>C-1</td>
<td></td>
</tr>
<tr>
<td>Fuel Side</td>
<td>65</td>
</tr>
<tr>
<td>Air Side</td>
<td>119</td>
</tr>
<tr>
<td>C-2</td>
<td></td>
</tr>
<tr>
<td>Fuel Side</td>
<td>N/A</td>
</tr>
<tr>
<td>Air Side</td>
<td>110</td>
</tr>
</tbody>
</table>
10 cm stroke length. In order to expose the 10 x 10 cm$^2$ square plate, at least two separate scans with overlap would have been necessary. Additionally, at that time fabrication of X-ray masks was time-consuming, expensive, and unreliable. Delicate titanium masks were the standard for X-ray mask production. Fabrication of these masks involved thousands of dollars with production times of up to 9 months. Development of the graphite mask using gold absorber was progressing (Desta et al., 2000), but was not a reliable alternative at that time. Additionally, due to the opaque nature of the graphite, an alignment system including a modified graphite mask and precision-machined markings on the substrate would have been necessary in order to align the X-ray mask to the substrate. Decreased reliability accumulates with additional steps in microfabrication processing.

Compatibility problems associated with the mask and exposure station for X-ray lithography were less severe for optical lithography. The UV exposure jig accepted large-area substrates with thicknesses of less than 3 mm. The flood-exposure system covered the entire area of the mask, which eliminated the overlapping exposure seams in the resist. The UV mask, which was composed of soda lime glass with chrome absorber, was readily patterned with a pattern generator. Although UV lithography generally does not generate the sidewall quality that X-ray offers, the requirements on structure quality for this project were within UV-LIGA capabilities. Mask alignment to the substrate was simplified by visually aligning the microholes, which were viewed through the clear SU-8 resist, directly with the swirl chamber etched on the clear UV mask. A microscope and an alignment stage were used in this method, with expected alignment accuracy within 20 µm.
UV lithography introduced difficulties that did not exist in X-ray lithography. UV lithography required use of liquid resists which were spun onto the substrate, whereas a solid sheet of PMMA is glued to the substrate as the resist for X-ray exposure. The procedure for processing PMMA was simpler and more reliable than the liquid process.

UV-LIGA, excluding molding, was selected as the better fabrication technique for this microfabrication project after consideration of the information available during the design phase. Facilities located at CAMD and the μSET laboratory on the LSU campus (Baton Rouge, LA) provided the fabrication equipment.

The microfabrication portion of this project was the most time-consuming, even compared to the extensive amount of time taken with traditional machining. Considerable effort was directed towards obtaining acceptable results for the structures formed on both sides of Plate C. Through experimental trials, two slightly different machining sequences were developed which significantly impacted the reliability of the microfabrication process. The two sequences are compared in Table 4.3(e). Sequence 1

<table>
<thead>
<tr>
<th>Step</th>
<th>Sequence 1</th>
<th>Sequence 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cut plate to size, grind/polish flat and thin</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Traditional drilling of fastener holes for all the plates</td>
<td>No traditional drilling</td>
</tr>
<tr>
<td>3</td>
<td>EDM drill microholes</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Microfabrication of structures on air side of Plate C</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Level and polish structures</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Microfabrication of structures on fuel side of Plate C</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Level and polish structures</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>No drilling needed</td>
<td>Traditional drilling of fastener holes for all the plates</td>
</tr>
<tr>
<td>9</td>
<td>Assemble the four plates</td>
<td></td>
</tr>
</tbody>
</table>
used a pre-drilled substrate, which included all of the required drilling for Plate C, including the microholes. Sequence 2 was developed after extensive processing on the pre-drilled substrate. This sequence involved a solid microfabrication substrate with only the microholes drilled through. After metal structure formation and polishing on both sides were completed, the fluid, fastener, and alignment holes were to be drilled. Sequence 2 is discussed in detailed in Section 4.3.2.12.

4.3.2.1 Microfabrication Processing Sequence 1

Table 4.3(f) summarizes the steps for microfabrication and surface finishing for Sequence 1. Temporarily filling each hole, as indicated in Step 3, was required in order to create a continuous surface for spinning liquid resist and to prevent electroplating.

<table>
<thead>
<tr>
<th>Step</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Sandblasting substrate on both sides</td>
</tr>
<tr>
<td>2</td>
<td>Cleaning substrate</td>
</tr>
</tbody>
</table>
| 3    | Filling holes  
|      | a. fastener / fluid holes  
|      | b. micro holes |
| 4    | Final substrate preparation |
| 5    | Evaporation of metallic layers |
| 6    | Resist processing |
| 7    | Preparation of substrate for electroplating |
| 8    | Electroplating Nickel |
| 9    | Leveling |
| 10   | Polishing |
| 11   | Resist / filler removal |
| 12   | Repeat steps 2-11 for the reverse side with slight modifications for protection of existing structures. |
inside the holes. Strong adhesion between the developed resist and substrate was required in order to survive the electroplating process at elevated temperatures. Metallic layers evaporated onto the clean substrate surface provided an intermediate layer for improved adhesion between resist and the substrate. Resist processing involved spinning the liquid resist, pre-baking, UV exposure, post-baking, and chemical development. Etching the metallic layers in the cavities that were provided by developed resist improved adhesion between the electroplated nickel and substrate. Electroplating nickel structures and leveling/polishing the deposited nickel to the final structure height were the final steps in processing. In order to form microstructures on the reverse side of the plate, the resist and fillers were removed for subsequent creation of a smooth substrate on the reverse side.

4.3.2.2 Sandblasting

Early experiments revealed that the SU-8 UV resist adhered better to a rough substrate than a smooth one. After many trials attempting to bond the resist to a smooth metal plate, Plate C was sandblasted. Low surface roughness was originally desirable for predictable flow through the swirler channels. However, the elevated surface roughness of the swirl channel floor actually assisted in maintaining turbulent conditions, which encouraged fully-developed flow.

4.3.2.3 Initial Substrate Cleaning

Substrate cleanliness was critically important for adhesion of the evaporated metallic layers. The only way to determine if the substrate was cleaned properly was to continue resist processing. If the resist did not peel the metallic layers from the nickel substrate, then the substrate was considered properly cleaned. Rarely would a tape-
peeling test of the evaporated metallic layers onto the substrate indicate inadequate cleaning. A reliable sequence of cleaning mechanisms was developed. The cleaning procedure is summarized in Table 4.3(g). With this procedure, the surface was clean enough for adhesion of the metal layers 80% of the time on the pre-drilled substrate. This method applied to both the smooth and sandblasted substrates.

The 9µm-grit sandpaper was used to remove localized chemical films or oils from the surface. The 800-grit was used to eliminate films from deeper crevices of the rough samples.

For cleansing the plate after completion of the full cycle of fabrication requirements for the first side, another step was included in the above procedure. Since the main component of the polishing slurry was oil, a de-greaser was necessary. TrichloroEthylene (TCE) was used to wipe the reverse side after Step 3 in Table 4.3(g). A thorough DI rinse, IPA rinse, and then DI rinse followed this additional step.

<table>
<thead>
<tr>
<th>Step</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Acetone bath / Trichloroethylene (TCE)</td>
</tr>
<tr>
<td>2</td>
<td>IPA (Isopropyl Alcohol) rinse*</td>
</tr>
<tr>
<td>3</td>
<td>Water rinse*</td>
</tr>
<tr>
<td>4</td>
<td>Sand with 9µm-grit sandpaper, then 800-grit sandpaper for 10 minutes each</td>
</tr>
<tr>
<td>5</td>
<td>Rinse with water</td>
</tr>
<tr>
<td>6</td>
<td>Wipe with IPA thoroughly to remove any particles</td>
</tr>
<tr>
<td>7</td>
<td>MicroSoap wash, DI rinse (2 times)</td>
</tr>
<tr>
<td>8</td>
<td>IPA rinse*</td>
</tr>
<tr>
<td>9</td>
<td>Thorough de-ionized water (DI) rinse*</td>
</tr>
</tbody>
</table>

* Rinse refers to pouring copious amounts of liquid on the plate surface at many orientations.
4.3.2.4  Hole Filling

A solid, uniform surface was required in order to create a smooth, level film of liquid resist on the substrate surface. Since the pre-drilled substrate included the larger traditionally machined fluid and fastener holes and the microholes, creating a consistent surface for spinning resist was not trivial. The filler materials for both types of holes were required to have relatively low viscosity for hole filling, remove easily after fabrication of each side, maintain adhesion to evaporated metals at the surface, have adequate stiffness to support the resist during spinning and handling, and survive resist processing. The limiting steps for filler survival during resist processing included thermal cycling and exposure to corrosive chemicals.

Initially, an experiment was run to determine if the microholes needed plugging. Electroplating inside the holes could ruin the expensive microholes. There was also a possibility of clogging the 75 µm diameter conical holes permanently with resist, due to the difficulty in removing some resists from small crevices. However, it was thought that the viscous SU-8-25 resist might span the microhole and spin smoothly over the substrate.

Due to the lack of microholes available for experimentation, larger 400 µm diameters test holes were mechanically drilled on a smooth Nickel 201 substrate. The hypothesis to be tested by experimentation was that if the resist did not penetrate the larger holes, then it would not penetrate the micro holes. It was found that the SU-8-25 photoresist quickly filled the test holes. Subsequent observation revealed that the test holes did not electroplate due to the presence of resist inside the holes. It was also determined that the microholes could be cleaned using an ultrasonic acetone bath.
Therefore, deliberate plugging was not necessary since the resist would passively protect the microholes. The material and procedure for plugging the microholes are presented in the following two sections since the procedure could apply to future applications.

4.3.2.4.1 Choosing Filler Material

Two types of resists were chosen to fill the two different types of holes. First, liquid PMMA was used to fill the larger holes and SJR 5740 optical resist was chosen as the filler for the microholes. The composition and recipe of the liquid PMMA used is indicated in Table 4.3(h).

Liquid PMMA was selected for the large holes due to its relatively low viscosity in the liquid phase and its high stiffness when hardened. In the solid phase, this material had strong adhesion to the nickel sidewalls and to the evaporated metallic layer at the surface. PMMA was easily removed from the large holes by softening in an acetone soak for 10 minutes then pushing it out with a tool. This filler material was also unaffected by the baking cycles and UV exposure encountered during processing. However, the PMMA was vulnerable to acetone and SU-8 Developer, which were both used during processing. Contact with these chemicals caused the filler to dissolve and produce a film over the substrate surface and resist structures. The film contaminated the SU-8

<table>
<thead>
<tr>
<th>Substance</th>
<th>Composition (by weight percent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methyl Methacrylate</td>
<td>85%</td>
</tr>
<tr>
<td>Polymethyl Methacrylate</td>
<td>13.4%</td>
</tr>
<tr>
<td>Benzoyl Peroxide</td>
<td>1.6%</td>
</tr>
<tr>
<td>Dimethyl Analine</td>
<td>Additional 1%</td>
</tr>
</tbody>
</table>

The first three ingredients were added together and stirred for 6 hours. The resulting mixture was then stored at just above 0°C. At time of use, the Dimethyl Analine must be thoroughly stirred into the mixture. The composition began to harden within 15 minutes.
developer, and interfered with subsequent processing. To avoid these problems, careful planning for cleaning the substrate in order to avoid the use of acetone after the PMMA was inserted was necessary. In order to prevent the difficulties encountered upon contact between PMMA and SU-8 developer, a barrier of exposed SU-8 was created to protect the filler on the working side of the substrate. For improved protection of PMMA, both of the UV masks were modified to provide resist coverage of an area slightly larger than the area of the filled hole. Visualization of this coverage is shown in the sketch in Figure 4.3(l).

As Figure 4.3(l) indicates, the developed resist on the right covered exactly the area over the PMMA-filled holes in the nickel substrate. This configuration was vulnerable to the caustic chemicals since they leached past the resist and through the metallic layers to attack the PMMA. Thus, the thin metal layers covering the PMMA were insufficient prevention of PMMA dissolution and subsequent removal of the metallic layers. The configuration on the left of Figure 4.3(l) adequately covered the PMMA with the broader developed resist geometry. For the reverse side of the plate,
electroplating tape (Harmon Corporation, Pittsburgh, PA) was an effective barrier between resist and chemicals during developing.

A UV resist, SJR 5740 (Shipley, Somerville, NJ), was chosen as the filler for the microholes since it stayed in the holes during processing and dissolved instantly when exposed to acetone. The viscosity of this resist was low enough to move into the microholes, yet high enough to stay. When baked at 90°C for 1 hour after filling, the cross-linked resist swelled slightly and hardened resulting in a press-fit plug that did not interfere with subsequent processing. The red color of SJR 5740 allowed confirmation of complete filling without the use of a microscope.

4.3.2.4.2 Procedure for Filling Holes

For filling the large fastener and fluid inlet holes, a smooth surface was created without the need for subsequent sanding by using electroplating tape. The tape was placed over the holes on the plate face to be processed. Liquid PMMA was prepared by adding three drops of Dimethyl Analine (DMA) into 10 ml of the premixed solution. After stirring with a small magnetic stirrer for 2 minutes, the mixture was then gently injected into the larger exposed cavities through a syringe. The syringe had an inner diameter of 500 micrometers. A vacuum environment with pressure of up to 70 kPa was used to remove trapped air bubbles. The PMMA was allowed to harden for a minimum of 8 hours under an exhaust hood. The electroplating tape was then carefully removed to reduce the amount of adhesive residue remaining on the surface.

The microholes were filled with SJR 5740 photoresist by forcing the resist through the slender orifice. On the reverse side of the substrate, four small reservoirs were created by electroplating tape. These reservoirs were attached, adhesive sides
together, at strategic locations to a single large piece of electroplating tape, which was used to seal the reservoirs over the micro hole sites. A drop of SJR 5740 was placed on each small reservoir. Carefully, the reservoirs were matched to the microhole sites and the large tape was used to seal around each reservoir. The red resist was forced through the microholes by pressing the reservoirs lightly. The assembly was then baked to harden the photo resist.

4.3.2.5 Final Substrate Preparation

It was necessary to subsequently clean the front side of the substrate since the electroplating tape used for assistance in PMMA filling left a film after removal. Gently scraping the SJR 5740 resist bubble off the top surface decreased smearing, which can leave a film that destroys adhesion of metal layers to the substrate. The surface was sanded with 9 µm-grit sandpaper for 5 minutes. After rinsing with water, IPA, and DI water, the sample was dehydrated at 90°C for 30 minutes. At this point, Plate C was clean with the larger orifices and microholes filled.

4.3.2.6 Evaporation of Metallic Layers

The next step in microfabrication processing was thin film deposition. Since SU-8 resist did not adhere to the Nickel 201 substrate, an intermediate layer was required. The resist adhered well to gold, and a chromium layer served to bond the gold to the substrate. The typical thicknesses of these intermediate layers were 50 Å of chromium and 300 Å of gold. Evaporation of these metals produced controlled layer thicknesses at relatively high speed with low contamination.

Evaporation is a form of physical vapor deposition in which deposition of the material onto the substrate is an impingement-type deposition. A surface not exposed to
the line-of-sight will not be covered. (Madou, 1997). The evaporation process can be divided into three steps: 1) the solid metal must be changed into a gaseous vapor, 2) the gaseous metal must be transported to the substrate, and 3) the gaseous metal must condense onto the substrate. E-beam evaporation used a high-intensity electron beam gun to heat the target. As the beam was magnetically directed into the source area, the metal was heated to its melting point, and eventually, evaporation temperature. A Temescal BJD-1800 E-Beam Evaporator, owned and operated by CAMD, was used to evaporate the chromium and gold layers onto Plate C.

Adhesion between the evaporated metallic layers and a nickel substrate was typically strong enough to withstand multiple attempts at resist processing for most nickel substrates. However, failed resist procedures required resist removal with the chemicals that attack the fillers through the metallic layers. Thus, each attempt at resist processing required refilling the holes and deposition of new gold and chromium layers.

If the cleaning procedures were ineffective, a film on the drilled substrate surface sometimes interfered with adhesion to the metallic layers. Residue from the SJR 5740 filler and from the electroplating tape used during PMMA filling caused flaking of the metallic layers upon SU-8 development.

4.3.2.7 Resist Processing

The UV resist chosen for UV-LIGA was a type of SU-8-25 (MicroChem, Inc, Newton, MA). This resist was tailored to yield resist structure heights of approximately 40 µm to 150 µm. Table 4.3(i) summarizes the steps developed for creating SU-8-25 photo resist structures 100 µm high. This resist height was needed to yield final metal structures 75 µm high.
A Headway Research PWM 103 heavy-duty photo resist spinner located at CAMD was used to spincoat the resist. This spinner had a circular platform with a 125 mm square counter-sink. A spincoating jig was needed in order to snugly fit the nickel plates into the milled area. A jig of ¼” Plexiglas was cut to the dimensions indicated in Appendix B. The spinner held wafers of the same size as the outer dimensions of the jig. A slight curvature was required for the inner corners to avoid stress concentrations and subsequent fracture of the Plexiglas jig.

<table>
<thead>
<tr>
<th>Process</th>
<th>Parameter</th>
<th>Length of Time</th>
<th>Equipment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spin resist 100 µm thick</td>
<td>850 rpm</td>
<td>20 sec</td>
<td>Spinner</td>
</tr>
<tr>
<td>Sit</td>
<td></td>
<td>10 min</td>
<td></td>
</tr>
<tr>
<td>Pre-exposure Bake</td>
<td>60°C</td>
<td>10 min</td>
<td>326 Convection oven</td>
</tr>
<tr>
<td></td>
<td>ramp to 96°C</td>
<td>hold 1.5 hrs</td>
<td></td>
</tr>
<tr>
<td></td>
<td>ramp to 65°C</td>
<td>25 min.</td>
<td></td>
</tr>
<tr>
<td>Post-exposure Bake</td>
<td>Energy (E) = 380 mJ/cm²</td>
<td>Time (t) = E / P</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Wavelength = 220-400 nm</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>326 Convection oven</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>10 min</td>
<td></td>
</tr>
<tr>
<td>Exposure</td>
<td>60°C</td>
<td>10 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>ramp to 96°C</td>
<td>hold 25 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>ramp to 65°C</td>
<td>25 min.</td>
<td></td>
</tr>
<tr>
<td>Cool</td>
<td></td>
<td>5 min.</td>
<td></td>
</tr>
<tr>
<td>Development</td>
<td>Large vessel of developer</td>
<td>7 – 10 minutes</td>
<td>Nano-Developer by MicroChem</td>
</tr>
<tr>
<td></td>
<td>IPA bath / rinse</td>
<td>1 minute</td>
<td></td>
</tr>
<tr>
<td></td>
<td>DI bath / rinse</td>
<td>30 sec</td>
<td></td>
</tr>
<tr>
<td>Hard Bake</td>
<td>60°C</td>
<td>3 min</td>
<td>326 Convection oven</td>
</tr>
<tr>
<td></td>
<td>ramp to 96°C</td>
<td>12 min</td>
<td></td>
</tr>
<tr>
<td></td>
<td>ramp to 70°C</td>
<td>20 min.</td>
<td></td>
</tr>
</tbody>
</table>
The most significant problem involved with UV resist processing on the pre-drilled nickel substrate was the variation of resist height over the surface. This significant variation was caused by surface tension. For a silicon or solid metal substrate, SU-8 resist normally pulled back from the edges due to high surface tension. The resulting film was level over the working area and thinner near the substrate edges. Though the fillers in the large holes created a smooth spincoating surface, there was a distinct interface between the PMMA-filled holes and the nickel surface. The resist reacted to this interface as if it were an edge, resulting in resist thickness variations of 100 µm over the substrate area. The resist thickness was a maximum of 140 µm at locations between the holes. In the area proximal to the holes, the film thickness was reduced to less than 40 µm.

Figure 4.3(m) CAD drawing of UV mask for (left) fuel side and (right) air side indicating locations of features on Plate C. The shaded areas will eventually be electroplated nickel. The features indicated are typical of the remaining structures. [1] are fastener holes, [2] are fastener holes with seals. [3] are holes for alignment pins. [4] are seals for high-pressure air passages, and [5] are seals for low-pressure air routes. [6] are air swirlers and [7] are fuel swirlers.
4.3.2.7.1 UV Mask Fabrication

UV mask fabrication was drawn with a computer aided drawing software, AutoCAD, (Autodesk, Inc.) and fabrication. The design consisted of determining which features would be electroplated and the type of resist used. The mask was composed of soda-lime glass with a thin layer of chrome used to absorb the UV light during radiation. Since SU-8 was a negative resist, the exposed portions generate the inverse of the final metal structures. The absorber was patterned according to the layout of features discussed in Section 4.2.7 where the electroplated structures were needed. The holes in the mask aligned with the drilled holes. Thus, the holes for the air side are the mirror image of the configuration of holes for the fuel side. The mask was patterned similar to the drawings in Figure 4.3(m).

The areas around the holes for the masks indicated in Figure 4.3(m) are the same size as the drilled holes. After resist processing and electroplating, it was learned that the electroplated nickel was collecting around the edges of the holes and adhering, due to the lower resist heights at those sites. Two more masks were designed that simply expanded the areas adjacent to the circumference of the holes by 0.5 mm radially. This allowed more resist at the edges during electroplating. Figure 4.3(n) shows the electroplated results of the offset resist from the hole perimeter.

The UV mask patterns were exposed with a Pattern Generator (PG). This station translated the lines on the drawing into step points and micrometer-sized flash points. For features larger than 5 μm and radii of curvature larger than 20 μm, the resulting UV mask was as accurate as the CAD drawing. For this project, all UV masks were
fabricated by LSI Photomask (Chandler, AZ) with CAD files that were completed by the author.

4.3.2.7.2 UV Exposure

Exposure was accomplished on an Oriel UV flood exposure station (model 82421, Stratford, CT) located at CAMD with radiation in the 280-450 nm wavelength range, with the most intense peak at 365 nm. The system allowed vacuum contact printing, which was preferred in order to minimize diffraction. However, vacuum exposure was not achievable since the seal was weak and there was insufficient space for the thicker metal plate. After alignment between the microholes and the swirl chambers, the stage was raised high enough to provide full support of the glass UV mask on the resist surface. Without vacuum contact, diffraction softened the sharp corners of developed resist. Since the structures for the fuel injector were large by microfabrication standards, the resulting diffraction was acceptable.

An exposure level of 450 mJ/cm² and other resist processing parameters were
used from guidelines published by Linke, et al (2000) for 100 µm resist thickness. This energy level resulted in well-defined structures on single-crystal silicon and solid nickel substrates without holes. The lithographic resolution, exposure dose sensitivity, and development time were sensitive to resist height variations. For the pre-drilled substrates and varying resist heights, the prescribed dose of 450 mW/cm² was overexposing the thinner areas and underexposing the thicker areas resist on the plate. The results were loss of adhesion between the resist and the gold covered substrate. The resist was required to survive many processing steps including oxygen plasma etch, wet etch of gold and chrome, the electroplating baths, and the violent surface finishing techniques.

Two modifications were adopted to promote adhesion: exposure dose adjustment and improved mechanical bonding. Further experimentation led to successful resist adhesion at the reduced exposure level of 380 mJ/cm². The exposure energy was decreased to induce surface microcracking. It was hypothesized that delamination of resist during processing was due to incompatibility of the coefficient of thermal expansion (CTE) between the nickel substrate and the UV resist. Micro cracking at the surface alleviated some of the internal stresses within the resist resulting in improved adhesion.

While reducing stress by microcracking helped improve adhesion, the actual mechanism was probably a softer, more pliable resist. The amount of polymer cross-linking can affect adhesion through a number of mechanisms. The level of exposure energy was directly correlated to the extent of cross-linking in the SU-8. This chemical reaction resulted in the hard, insoluble portions of developed resist. Hardness and internal stresses in the resist increased with further cross-linking. Though small amounts
of microcracking resulted at the reduced exposure levels, the reduction in cross-linking led to a softer, more flexible resist, which resulted in improved adhesion through resist processing. Figure 4.3(o) shows a photograph of a view of the resist through a microscope after development to verify definition and adhesion. Adhesion was verified by the view of the full image in focus, since delamination caused the lifted portions to look blurred.

Though the previous techniques did improve adhesion of resist to the substrate, the yield for successful resist processing was on the order of 5%. In order to further improve adhesion between SU-8 and the substrate, mechanical bonding was generated by increasing uniform surface roughness, which was created by sandblasting, as discussed in Section 4.3.2.2. With the improvements made in exposure dose and the sandblasted surface, the rate of successful resist adhesion was 100% of each processing attempt on a clean pre-drilled substrate. Often small white particles were seen through the optical microscope. These pieces of silicon (sand) were embedded in the nickel substrate during

Figure 4.3(o) Optical photograph focused on the top of the resist surface for preliminary indication of resist structure definition and adhesion.
sandblasting. The particles did not compromise adhesion of the resist or structures.

Alignment of the substrate with the mask was accomplished by two methods. For smooth nickel plates, alignment was achieved by aligning the microholes to the swirl chambers. The microholes could be easily viewed through a split field microscope, which allowed optical manual alignment. This microscope was used with the mask/substrate holder and three micrometers for stage adjustment. Accuracy for the Oriel substrate/mask holder with the smooth substrate was ±5µm.

When aligning to the sandblasted substrate, the microholes could not be viewed with the alignment microscope since the surface roughness camouflaged them. However, the air side microholes could be seen without assistance of the microscope. The large fastener holes were used for coarse alignment, which was accurate to within ±40µm. Verification of alignment was accomplished by looking for the microhole inside the swirl chamber prior to flood exposure without assistance of the microscope. When processing the fuel side on a sandblasted substrate, the microholes could not be seen at all since they were so small. Alignment was accomplished in the same way as with the air side; however, the only verification of alignment was after resist development. Photographs of this verification are shown in Figure 4.3(p). These photographs demonstrate improved imaging over the alignment microscope images. All four microholes were aligned with the fuel swirl chamber to within 50 µm. Table 4.3(j) indicates the resulting alignment offsets of each swirler and corresponding microhole.
4.3.2.7.3 Remaining Resist Processing

The ten-minute “sit” indicated in Table 4.3(i) allowed the resist to complete chemical and physical reactions before subjecting it to other operations.

Bake times were experimented with, but eventually the parameters returned to those suggested by Ling, et al (Ling, Lian, & Linke, 2000). Pre-baking reduced the residual solvent level in the resist, which decreased the risk of exposed resist loss, swelling, and delamination. Increasing the pre-bake time also tended to increase the development time, which was suitable for obtaining well-defined resist structures. Post-exposure baking assisted in the cross-linking reaction, which was initiated by the UV exposure. The additional post-exposure bake time served to reduce internal stresses in the SU-8, which improved adhesion.

Table 4.3(j) Summary of distance between the centers of the micro hole and swirl chamber for each swirler for the final sample on Plate C-1.

<table>
<thead>
<tr>
<th></th>
<th>Distances (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hole I</td>
</tr>
<tr>
<td>Air side</td>
<td>30.7</td>
</tr>
<tr>
<td>Fuel side</td>
<td>19.3</td>
</tr>
</tbody>
</table>
The hard-bake step was critical for improving adhesion between resist and substrate after development. On a few occasions following development, portions of peeling resist re-adhered to the substrate during the hard-bake. Adhesion was improved during the hard-bake due to relaxation of internal stresses, but it was insufficient to complete the fabrication cycle required. These samples all survived electroplating, but slight delamination occurred again at the end of surface finishing.

Removal of the gold and chromium layers was necessary in order to expose the nickel substrate on the floor of the resist cavities that were to be electroplated. After development of SU-8, a thin film of developer and resist covered these areas. Oxygen plasma etch was used to remove the film in order to allow uniform wet etching of the metal thin films. The Branson RF Plasma Asher (Plasma Etch, San Jose, CA) owned by CAMD was used. During ashing, the temperature in the plasma chamber slowly increased since there was no cooling mechanism. A temperature limit of 65°C placed on the ashing yielded complete cleaning while preserving resist adhesion.

After cleansing in the asher, the gold layer was quickly removed with a potassium iodide gold etch solution. After thoroughly rinsing the substrate with DI water, chrome etch (Arch Chemicals, Norwalk, CT) readily removed the chromium layer. A final rinse in DI water completed resist processing, and exposed the nickel plating base.

Further sample preparation was required before electroplating. There were many areas that the resist did not cover and should not be electroplated. Electroplating tape was used to seal these areas against contact with the electroplating solutions. Use of the tape required care to ensure the electroplating fluids did not leak and the tape maintained adhesion.
4.3.2.8 Electroplating Nickel

Nickel deposition within the cavities that were formed by developed resist comprised the Galvanoformung step in the UV-LIGA process. To maximize bond strength between the electroplated structures and the substrate a nickel/nickel interface was created.

Electrodeposition rates for nickel electroplating were calculated with Faraday’s law. Equation 4.3.2 indicates this relation modified to solve for electroplated height.

\[ m_{\text{max}} = \frac{itM}{\delta Fz} \]

Where, \( m_{\text{max}} \) is the plated metal height in micrometers, \( i \) is the current density, \( t \) is time in minutes, \( M \) is molecular weight of the deposited metal, \( F \) is Faraday’s constant (96,500 Coulombs), \( \delta \) is density of deposited metal, and \( z \) is gram equivalent of each ion. The electroplating time required to achieve the design height can be solved using Equation 4.3.3.

\[ t = 48.7 \frac{m_{\text{max}}}{i} \]

This relation assumed 100% electrodeposition efficiency. Some of the electrical current went into hydrogen evolution at the cathode site. The amount of hydrogen evolving and competing with Ni deposition depended on the pH, the temperature, and the current density. It was found that very close to 100% efficiency was common with the electroplating conditions presented in the following sections.

The electroplating area was calculated in order to accurately determine the current density. The complex shapes of the resist structures required the use of a CAD system to accurately determine the areas. AutoCAD (Autodesk Inc., San Rafael, CA) was
previously used to layout the UV mask and it was also used to add areas. This technique worked well for the unique shapes in the working area of the substrate. Calculation errors arose from the use of electroplating tape for coverage and the location of the water line in the plating bath. Since the total plating area was so large, the sensitivity of the electroplating time was lower than with most other MEMS devices.

The time required for electrodeposition of 100 µm height was calculated using Equation 4.3.3. Electrochemical dynamics created situations that create uneven structures and make it difficult to predict the final height. Electroplating formed the nickel structures in a concave geometry inside the crevices, instead of in even layers as the structural height increased. The solution to this problem was overplating. The time for 100 µm with a current density of 10 mA/cm² was 7.9 hours. The samples were usually allowed to electroplate for 8.5 hours to ensure the minimum structure height of 100 µm. The resulting structures were approximately 100 µm higher than resist at the edges, reducing to 60 µm towards the center of the larger electroplated features.

4.3.2.8.1 Electroplating Procedures

Early adhesion problems required electroplating modifications for bond improvement between the nickel structures and the Nickel 201 substrate. Activation using C12 (Alpha Aesar, Ward Hill, MA) and Wood’s strike significantly improved adhesion between electroplated structures and the substrate (Lowenheim, 1963; Schlesinger, Morderchay, & Paunovic, 2000). The parameters for each bath used in electrodeposition of nickel onto a Nickel 201 substrate are summarized in Table 4.3(k) and listed in order of use.
Wood’s strike is a nickel chloride bath. Relative to nickel sulfamate baths, the chloride solution generated an improved adhesive layer between the nickel structures and various substrates. Nickel sulfamate was used for forming the electroplated structures since lower stresses in the features were obtainable compared to nickel sulfate or nickel chloride solutions.

The recommended Wood’s Strike plating procedure was 2 minutes of reverse plating at 30 mA/cm², then 6 minutes of forward electroplating at the same current density (Lowenheim, 1963). The reverse plating was an etching step which was recommended in order to remove remaining oxides or resist contaminating the surface. The etch step created small pits in the surface, which may improve mechanical adhesion to the substrate. However, the reverse plating caused delamination of the resist during this step, as shown in a test sample in Figure 4.3(q). To remove the oxides on the substrate surface activation of the surface by C12 (PCT Technologies Inc., Wantagh, NY), a 60% chlorine activator, was substituted in place of the reverse plating with Wood’s strike. The nickel adhesion layer was then plated with the Wood’s strike as specified in Table 4.3(k), followed by immediate immersion and electroplating in the sulfamate bath.

<table>
<thead>
<tr>
<th>Bath Type</th>
<th>Current Density</th>
<th>Time for process</th>
<th>Temperature / pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>C12 Activator</td>
<td>20 mA/cm</td>
<td>2 minutes</td>
<td>25°C / 1.5</td>
</tr>
<tr>
<td>Wood’s Bath</td>
<td>30 mA/cm</td>
<td>6 minutes</td>
<td>25°C / NA</td>
</tr>
<tr>
<td>Nickel Sulfamate</td>
<td>10 mA/cm²</td>
<td>8 hours (for 100 µm tall structures)</td>
<td>55°C / 4.0</td>
</tr>
</tbody>
</table>

Table 4.3(k) Procedure for electroplating nickel structures onto a nickel substrate using galvanostatic mode.
The C12 activator was mixed by following the manufacturer’s instructions. A 40:1 ratio of H₂O:C12 composition was required for 6 liters. Sulfuric acid was used to lower the pH to 1.5.

Composition of the Wood Strike is listed in Table 4.3(l). The three components were stirred together at room temperature for approximately 2 hours for complete dissolution of the salts. When electroplating, nickel rounds were used for the anode and plating temperature was 25°C.

Six liters of nickel sulfamate (1.8M) was mixed according to the compositions listed in Table 4.3(m). Boric acid was used as a buffering agent to minimize pH elevation during electroplating. Lauryl sulfate served as a wetting agent to reduce the

Table 4.3(l) Composition of Wood Strike electroplating solution.

<table>
<thead>
<tr>
<th>Component</th>
<th>Contribution to total volume of 6 liters</th>
<th>Manufacturer</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel (II) Chloride Hexahydrate (NiCl₂•6H₂O)</td>
<td>1440 g</td>
<td>Avocado Research Chemicals</td>
<td>98% coarse powder</td>
</tr>
<tr>
<td>HydroChloric Acid (HCl)</td>
<td>402 ml</td>
<td>generic</td>
<td>50 %</td>
</tr>
</tbody>
</table>
The surface tension of the solution, which contributed to low-stress plating. Stirring at elevated temperatures without boiling for more than 10 hours thoroughly dissolved the powders.

When preparing to electroplate, the nickel sulfamate bath temperature must be stabilized at 55°C and the pH adjusted to 4.0. During electroplating, the pH tended to rise. However, this was not a concern since the 8.5-hour plating time span resulted in a 0.2 rise in pH, which was insignificant. Nickel rounds gathered in a titanium basket were used as the anode.

All of the plating parameters for activation and electroplating were controlled in galvanistatic mode. In order to set the correct current on the potentiostat, a test plate was used. The plate was placed in the electroplating bath and the electrodes were connected. The current was then set to the prescribed value.

### 4.3.2.8.2 Electroplating Setup

The Amel 2055 Potentiostat (Milan, Italy) was required for some of the required pulsing since the plating currents were greater than 1A. Table 4.3(n) indicates the calculated electroplating areas for four masks used on this project. Due to the need to prevent electroplating of the electrical connection, which leads from the substrate to the

<table>
<thead>
<tr>
<th>Component</th>
<th>Contribution to total volume per liter</th>
<th>Manufacturer</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel (II) Sulfamate (Ni(SCNH₂)₂)</td>
<td>450 mL</td>
<td>Alfa Aesar</td>
<td>50% aqueous solution, Reagent Grade</td>
</tr>
<tr>
<td>Boric Acid (H₃BO₃)</td>
<td>42 g</td>
<td>Fisher Scientific</td>
<td>powder</td>
</tr>
<tr>
<td>Lauryl Sulfate</td>
<td>3 g</td>
<td>Sigma</td>
<td>Powder</td>
</tr>
<tr>
<td>Deionized water</td>
<td>Remaining volume</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Component</th>
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<th>Manufacturer</th>
<th>Specifications</th>
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<tr>
<td>Lauryl Sulfate</td>
<td>3 g</td>
<td>Sigma</td>
<td>Powder</td>
</tr>
<tr>
<td>Deionized water</td>
<td>Remaining volume</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
potentiostat, the top of the substrate must be held above the water line. The actual plating area was the value in Table 4.39(n) reduced by the taped electroplated areas and area held above the water line. The latter two measurements were approximated after taping was completed and were accurate within 1 cm². Figures 4.3(r) through Figure 4.3(u) indicate features of the plating setup and the jigs used.

The electrode was positioned near the top edge of the substrate, as shown in Figure 4.3(t). The working electrode lead was attached to the top of the electrical connection on the substrate holder.

Table 4.3(n) Electroplating areas exposed by each mask.

<table>
<thead>
<tr>
<th>Mask Design</th>
<th>CAD Plating Area (cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air (without straighteners)</td>
<td>53.9</td>
</tr>
<tr>
<td>Air (with straighteners)</td>
<td>64.5</td>
</tr>
<tr>
<td>Fuel (modified)</td>
<td>51.7</td>
</tr>
<tr>
<td>Fuel (original)</td>
<td>62.8</td>
</tr>
</tbody>
</table>

Figure 4.3(r) Photographs of electroplating station in μSET laboratory.
Figure 4.3(s) Photograph of the electroplating tank of nickel sulfamate bath with leads connected to the anode and cathode.

Figure 4.3(t) Photographs the substrate holder indicated the site for the cathodic connection.
A slide mechanism was included in the substrate holder so that variable substrate sizes may be electroplated while maintaining the top part of the plate above the water line. The anode consisted of the adapting jig, titanium basket, cloth, and nickel anodes. The anode jig may be positioned at four different submerging distances above the height of the tank by using the steel bar with the holes drilled in the sides of the Plexiglas jig. The cloth bag was used as a filter to trap particulates settling out of the anode.

4.3.2.8.3 Electroplating Results

The electroplated features were well formed without significant flaws. The SEM images in Figure 4.3(v) indicate slight curvature of the sidewalls. Due to the relatively large size of the microfabricated structures, the curvature was determined to not affect fluid flow. The resist adhered to the substrate throughout the electroplating process. Adhesion of the nickel structures to the nickel substrate was also sufficient when the procedures described previously were followed. Feature adhesion was tested and found acceptable when the structures survived the surface finishing part of the fabrication procedure. There was no pitting or unfilled features observed in the electroplated structures.
Figure 4.3(v) SEM images of nickel electroplated samples with resist removed. The sidewall of a channel appears smooth and straight (top). Upon closer inspection, some sidewall curvature is indicated (bottom) on the sharp swirler corner. The white particles are pieces of abrasive material remaining after surface finishing.
Optical photographs of electroplating results with resist removed are presented in Figure 4.3(w). There was a large difference between the optical photographs and the SEM images. The SEM yielded more information on sidewall formation, diffraction, and remaining contaminants and resist on the surface. The optical photographs indicated the remaining resist, and the electroplated structures looked deceptively good through the microscope.

The effects of thinned resist near the filled holes were apparent after the electroplating step. Figure 4.3(x) shows the bubbled portions of plated nickel at sites which were meant to be covered by 100 µm thick resist. These bubbles nickel usually...
fell off during surface finishing or filler removal. However, sometimes these pieces adhered to the surrounding cylinder. When attempts were made to remove the extra pieces, the cylinder was sometimes removed with it. The pieces were filed around the hole with a needle file to reveal the full diameter of the hole. This was insufficient for some of the alignment holes since the tolerances on these holes leave no room for burrs. For subsequent electroplating, the alignment holes were sealed against contact with electroplating solution. This was a good solution since the alignment holes are not required for sealing.

4.3.2.9 Final Steps for Completion of Air Side

After the electroplating phase, leveling completed fabrication. Removal of the SU-8 and the PMMA filler was necessary in order to process the reverse side. It was preferred to keep the resist intact on the first side in order to uniformly level the reverse side. However, it was discovered that the SU-8 swelled with increased temperature while
processing the second side. This swelling caused delamination of the air swirlers at the sharp corners. Thus, the SU-8 was removed from the first side before processing the second side.

Uniform pressure could be maintained on the fuel swirlers during leveling even though the resist was not available to provide uniformity over the entire face area since the fuel swirlers were so much smaller than the air swirlers and were directly behind them. The PMMA filler was removed to prevent non-uniform leveling of the working side, as shown in Figures 4.3(i) and 4.3(j).

Before leveling, the PMMA filler puddles on the opposite side of the working face were carefully leveled using acetone and sharp, flat tools. The acetone was confined to the back side of the substrate where the puddles were located in order to avoid contact with the SU-8, which was on the working side and vulnerable to delamination with acetone contact. A flat-head screw driver was used to scrape the surface flat, and an arbitrary sharply-pointed tool was used to assist in removing portions of PMMA from the holes. Subsequent sanding with 800 grit sandpaper removed any particles present on the working surface and ensured that the PMMA was at least level with the plate surface. This procedure worked well when processing the first side. The procedure was precarious when processing the second working (fuel) side since scraping the air side was forbidden to prevent scratching the polished structures. Sanding was avoided since maintenance of surface quality of the air side was imperative. Therefore, dissolution of the PMMA filler by acetone was the only way to remove these puddles.

Removal of SU-8 was always a difficult part of projects that use this resist. There have been many creative solutions devised for SU-8 removal. Incineration at
temperatures above 300°C is a thorough method, but it is forbidden in this project since the nickel plate cannot exceed 120°C. This temperature limit was placed on the substrate to reduce annealing, which would reduce strength. A method that worked slowly was to use a solution of Nano Remover (Micro Chem, Newton, MA) at 65°C with a stirring rod rotating at medium speed. The bulk of resist was removed within 1 hour. However, the channels retained small particles of resist in them. Another 10 hours were needed to remove the majority of the SU-8. Traces of resist continued to adhere to the sidewalls of the swirler channels. SEM photographs of air swirlers with the microhole in view are shown in Figure 4.3(y). The bright patches indicate remaining SU-8 that was not removed completely.

After resist removal, the gold and chrome thin films were also removed in the event these layers could not survive the working conditions of the fuel injector. The wet etchants were again used at this stage with a similar pre-cleaning method as that followed before electroplating.
4.3.2.10 Conclusion of Air side Processing

Typical swirler heights after leveling are summarized in Table 4.3(o). The standard deviation reflects the differences in height values after surface finishing of average values of the four swirlers on the same plate. The resist on the C-2 sample was spun thin, which yielded thin swirlers. This was a concern for prototype testing since the design required higher channels for the fuel flow.

Loss of adhesion between the metal structures and the substrate usually occurred during the surface finishing portion of the process. Delamination with intact resist was the most common method by which to judge insufficient bonding. Determination of a quantitative adhesion level was not explored in this project. A qualitative method was used by applying a peel test to the structures with electroplating tape before surface finishing. When SU-8 was removed with the Nano-Remover (MicroChem, Newton, MA), it swelled and pushed against the electroplated nickel. These were sources of delamination for smaller features.

Working towards satisfactory results required process iterations through many failed samples. The results of electroplating without the activator and strike were weakly bonded structures. These did not pass the peel test. Upon improvement of the electroplating sequence that included the activator and strike, bonding between the structures and substrate improved significantly. Additionally, the structures which completely peeled had no internal stresses, since they did not curl. Due to these

<table>
<thead>
<tr>
<th></th>
<th>Height (µm)</th>
<th>Standard deviation (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-2 Air swirlers</td>
<td>43.5</td>
<td>7.0</td>
</tr>
<tr>
<td>C-1 Air swirlers</td>
<td>81.5</td>
<td>10.6</td>
</tr>
</tbody>
</table>
considerations, problems from inadequate electroplating were dismissed as adhesion problems, and modified surface finishing techniques were the focus to improve structure survival.

Efforts were made to improve surface finishing techniques to avoid delamination of the structures. The wheel speed of the lapping machine was slowed since faster speed would increase shear on the structures. Increasing the frequency of slurry spray reduced friction that would tend to shear the structures. Delamination was often observed during the later stages of surface finishing, when the grit of the slurries was below 2 µm. At finer grits, it was found the sample conformed well to the wheel shape, which caused some adhesion between the wheel and sample. Suction formed between the sample and wheel when an attempt was made to remove the sample from the wheel. Pulling directly against this suction pulled the structures from the substrate. This condition was aggravated since polishing requires checking the sample often. This problem was easy to avoid once recognized by sliding the sample off the wheel. By reducing the wheel speed to 4.7 rad/sec, increasing the frequency of slurry, and carefully sliding the sample off the wheel, the structures did not delaminate.

4.3.2.11 Fabrication of Fuel Side

Processing the second (fuel) side was almost exactly like processing of the air side except that protection of the features and surface conditions on the air side was required at all times. Exposure to chemicals and materials that could damage the surface was avoided by covering the features with electroplating tape.
Final results of the fuel side processing were unsuccessful for this project. Plate C-1 was warped when the fuel side was polished, which ruined the plate since it could not be flattened. Figure 4.3(z) indicates the fuel side of Plate C-1 of when the warping was discovered. The condition of the plate shown in the figure was caused by insufficient amounts of slurry sprayed on the polishing wheel. The slurry provided particles for material removal, oil to reduce friction, and it served as a cooling agent. This plate was previously processed a number of times and the resulting thickness when it warped was 1.10 mm, which is 150 $\mu$m thinner than its original size. The plate was more dimensionally unstable at this thickness and, during one of the final steps in the process it could not withstand the rigors of surface finishing.

4.3.2.12 Microfabrication Processing Sequence 2

The alternative fabrication sequence is summarized in Table 4.3(n). This sequence was created to decrease microfabrication difficulty found with the pre-drilled

![Figure 4.3(z)](image)

Figure 4.3(z) Photograph of a lapped fuel side surface in which the plate was not level. The gray circle in the center is the location of the force concentration since it is lapped down to the substrate, while the periphery structures have not been leveled.
large holes. Hole-filling was eliminated since the microholes did not require it. Cleaning difficulties resist uniformity, and sidewall height variations were significantly reduced. However, fresh complications were introduced from postponing drilling. Substrate/mask alignment and maintenance of surface conditions during drilling were more complicated.

Cleaning was simplified to using liberal amounts of chemicals and processes in the following order: Acetone rinse, IPA rinse, DI water rinse, light sanding with 9 µm sandpaper, IPA rinse, DI water rinse, and then plasma ash to 100ºC. The difficulties associated with matching solvent compatibility with the PMMA filler were eliminated, and the cleanliness of the substrate was certain.

Spinning and baking the SU-8 on a solid, flat substrate yielded a uniform film thickness. Since this substrate was also sandblasted adhesion problems disappeared.

With the absence of the larger holes, crude alignment of the mask and substrate was eliminated. However, use of the Quintel UV exposure station (model UL-7000, San Jose, CA) at CAMD with motorized alignment made the alignment process for both the air and fuel sides easier with an error of less than 5 µm possible, but detecting the

<p>| Table 4.3(p) Microfabrication steps with surface finishing for a post-drilled substrate. |
|-----------------|-----------------------------------|</p>
<table>
<thead>
<tr>
<th>Step</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Sandblasting both sides of substrate</td>
</tr>
<tr>
<td>2</td>
<td>Cleaning substrate</td>
</tr>
<tr>
<td>3</td>
<td>Evaporation of metallic layers</td>
</tr>
<tr>
<td>4</td>
<td>Resist processing</td>
</tr>
<tr>
<td>5</td>
<td>Electroplating Nickel</td>
</tr>
<tr>
<td>6</td>
<td>Leveling</td>
</tr>
<tr>
<td>7</td>
<td>Polishing</td>
</tr>
<tr>
<td>8</td>
<td>Resist removal</td>
</tr>
<tr>
<td>9</td>
<td>Repeat steps 2-9 for the reverse side.</td>
</tr>
</tbody>
</table>
alignment holes on the rough substrate was still a problem.

The primary consequence of post-drilling the substrate was maintaining the surface finish during the final drilling step. A scratch on either side of the plate would destroy the smooth plate surfaces, without the means to re-polish. In order to protect the surface while obtaining the accuracy required for the holes, layers of electroplating tape could be used to cover the substrate at all locations except the alignment holes.

Plate C-2 was the dedicated plate for this fabrication sequence. During surface finishing of the fuel side, this plate curled, which ruined the plate since it could not be flattened.

4.3.3 Testing

The reservoir seals of metal prototype were tested with the warped Plate C-1. The plate was bolted to Plates B and D. Reservoirs 2 and 3 were then pumped at various pressures with argon gas while the prototype was submerged in water. Sealing was evaluated since the bubble pattern indicated the leakage sites. The fuel injector leaked at the edges and internally within the reservoirs at the initial pressure of 35 kPa (5 psi). As the pressure was increased up to 140 kPa, the amount of leakage increased. However, the amount of leakage as a percentage of the amount of bubbles exiting the swirler orifice was approximately 5%, which was steady at the low pressures tested. The leakage occurred due to the gap between the plates caused by the warped plate. Thus, confirmation of surface conditions to determine if they were adequate for sealing subsequent fuel injector designs could not be completed. Figure 4.3(aa) shows the assembled fuel injector, without Plate A, and gas supply lines.
Figure 4.3(aa) Partially assembled prototype prepared for leakage testing (left) and during testing in water (right).

4.3.4 Conclusions

The parameters that determined fabrication methods for the metal fuel injector were micrometer-scaled features and narrow tolerances on precision. The small features include the restricted tolerances on positions and diameters of alignment holes, the location and geometry of the port connecting the fuel and air swirlers, the alignment of the swirl chambers to the microholes, and the micrometer-scaled channels. Multiple fabrication methods were determined necessary for creation of these features. Precision drilling, milling, and surface machining were needed for the orifices, complementary-air reservoir, connectors, and sealing surfaces. The UV-LIGA method was determined necessary for creation of the swirlers and supporting features on both sides of Plate C.

During the time microfabrication decisions were made, the available resources and fabrication limitations were carefully considered. Through the efforts of CAMD staff, LSU students, and scientists around the world, improvements were realized in both SU-8 processing and X-ray lithography. Recent equipment upgrades also improved capabilities which provided more flexibility in processing. Reconsiderations in microfabrication processes due to technological advances are addressed in Chapter 6.
5 Ceramic Solutions

Ceramics provided a solution to the fastening and sealing requirements of the reservoirs. The manner in which injection molded ceramics are formed led to the idea of sealing the reservoirs with strength through a cofired assembly of the laminated plate configuration. During the sintering stage, fusion of two green pieces held in firm contact is possible. The laminated plates could be joined in this manner when firmly clamped during sintering. Cofiring originated in the packaging industry as a thermal barrier for high performance chip (Seraphim, Lasky, & Li, 1989).

In order to strengthen the uncharacterized joint, a shrink-fit joint was investigated. This mechanical adaptation would use two different ceramic mixtures with differing shrink rates to form a joint resembling a hole-and-shaft. The ceramic with the higher shrinkage would compose the annular component and the material with reduced shrinkage would comprise the shaft component, according to the images in Figures 5.1(a) and 5.1(b). The strength of both of these bonds would be vitally important to the size and geometry of the laminated plates of the ceramic prototype.

Disadvantages of the cofired bond originate from the uncertainty of working with ceramics. The bonding would be process dependent, specifically on the amount of...
pressure applied during the various heating and cooling cycles. The bond strengths would vary with the bond completeness between the two sealing surfaces and the composition of the materials fused. In the case of aligned parts for the shrink-fit joint, the precision of alignment would dictate the pressure held between the fused surfaces.

Investigation of the molded features subjected to stresses and strains would be required in order to apply the material capabilities to design parameters. Due to the elevated pressures of the reservoirs, characterization of the fastening mechanisms was required. Free body diagrams in Figures 5.1(c) and 5.1(d) were needed to isolate the stresses when a pressure load was applied to the reservoirs for each design. Tensile forces were applied to the cofired surfaces for the flat plate joint in Figure 5.1(c). Shear was the dominant stress in the shrink-fit joint. Tension was also a factor as indicated in
Figure 5.1(d), but only on the areas that top the swirlers. Since the swirler areas are small, it was valid to consider tension in this sample negligible.

Examination of joint completeness would be the first parameter investigated after

Figure 5.1(c) Free-body diagram for the contact areas of the fused components indicated in Figure 5.1(a). Tensile forces are applied to the cofired volumes when pressure is applied to the reservoirs. Figure 5.1(d), but only on the areas that top the swirlers. Since the swirler areas are small, it was valid to consider tension in this sample negligible.

Figure 5.1(d) Free body diagrams for the contact areas of the fused components under pressure load applied to the reservoir indicated in Figure 5.1(b). Tension and shear are indicated with the T and S marked forces, respectively.
accomplishing a cofired joint. The methods needed for the study would depend on the sample geometry cofired. The remainder of this chapter involves discussion of bond strength characterization with the assumption that the joint was fully formed between the two intended contact surfaces.

There were two ways to approach testing of cofire strength. Isolation of the stresses and testing the individual contributions of each is standardized. Another method would be to replicate the joint, such as the shrink-fit pressure vessel, and apply pressure until failure.

To isolate stresses, shear strength testing would be used to characterize the shear stress in the shrink-fit joint. Similarly, flexural testing would characterize the fusion of the flat plates in tension. Standardized methods for both of these tests were found in American Society of Testing Materials (ASTM) procedures. The benefits of standardized testing include specific guidelines for sample preparation and testing procedure. The authors of these procedures are experts in ceramic testing and they created the methodology for standardization than excludes potential problems of which amateurs may not be aware. Standardization also allowed for comparison of testing results with other data for improved understanding of the effects of parameters. The disadvantages of standardized testing include using standardized bar samples instead of the more geometrically accurate joint for which the test is completed. Variation of the test specimen from component size, shape, and processing reduces the accuracy of the data (Quinn, 1991). Surface preparation was also specified in the standards, which would probably differ from the surface features of the component. Surface and subsurface flaws are often strength limiting and will usually affect the mode of component failure. The
flexural strength standard available specified monolithic specimens not two fused specimens. More modifications of the standards were needed to reduce the scale of the test samples to more closely approximate component size.

To replicate the joint for more accurate strength determination, the concentric-cylinder joint would be needed. It was necessary to understand the contributions of shear and tension in this model in order to extrapolate joint strength from the vector sum of the two. The bond strength may be more complex than a simple vector sum due to joint formation and processing parameters (Quinn, 1991). The bond strength could be verified by comparing this data to the more direct experimental data from replicated component joints.

Isolation of tensile strength would require a modified version of the standard flexural test, ASTM C 1161-94 Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature (1994). The flexural test was preferred, but with caution since injection molding produces preferred orientations of grains and pores resulting in anisotropic thermal and elastic properties, which could introduce substantial errors into the extrapolation of flexure data for design purposes. Other tensile testing methods, including direct tensile testing, were less reliable for design purposes than the flexural test. A fine tolerance of eccentricity in the tensile load was acceptable depending on the strain, though the loading commonly yielded data that could not be used in strength determination (ASTM E 1012-99 Standard Practice for Verification of Specimen Alignment Under Tensile Loading, 1999).

The four-point-1/4 point configuration shown in Figure 5.1(e) was determined appropriate for testing a midline joint. The specimen size, fixture spans, and nominal
bearing distances could be scaled to 10% of recommended sizes for the first testing attempts, which are specified in ASTM C 1161-94 (1994). Loading apparatus would be possible with an ordinary tensile testing machine using the configuration specified in the standard.

For isolated shear testing, ASTM C 1469-00 Shear Strength of Joints of Advanced Ceramics at Ambient Temperature (2000) was only slightly modified for sample size to be appropriate for testing ceramic joints in shear. The test specimen size was reduced to 10% of the recommended sizes in the standard. An estimate of flexure strength and joint strength was necessary in order to determine the joint geometry. The choices for joint shape with corresponding strengths are listed in Table 5.1(a). If the joint is too strong relative to the matrix strength, there is a high probability of invalid results and this test method should not be used. The specimen and loading configuration is presented in Figure 5.1(f). Loading apparatus would require an ordinary tensile testing machine to apply the vertical load to the test fixture.

![Figure 5.1(e)](image)

**Figure 5.1(e)** Drawing of flexure test specimen cross-section with applied loads of the four-point-1/4 point configuration from ASTM C 1161-94. The vertical fused joint is shown in the center of the specimen. The round pins are the locations for the applied load, P.
The standards require minimum 10 valid tests for each load and configuration to be statistically valid. Fracture analysis was also required in order to understand the mode and type of fracture and location of fracture initiation for each specimen. Testing parameters such as displacement rate, specimen condition, test fixture, and testing procedure were specified in the standards.

Advantages with testing a replica of the component joint include improved accuracy and reliability in test results. The most reliable test for determination of failure thresholds for a ceramic part is test of the component itself (Quinn, 1991). This is usually an unacceptable practice since testing each component of a large through-put manufacturing process such as injection molding would be time and cost prohibitive. A

<table>
<thead>
<tr>
<th>Joint strength as percentage of flexural strength of ceramic matrix.</th>
<th>Recommended joint geometry</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 25%</td>
<td>uniform</td>
</tr>
<tr>
<td>25% - 50%</td>
<td>straight or v-notch</td>
</tr>
<tr>
<td>&gt; 50%</td>
<td>Invalid test</td>
</tr>
</tbody>
</table>

Table 5.1(a) Recommended joint geometry for various joint strengths.

Figure 5.1(f) Drawing of shear test specimen cross-section with applied loads of the asymmetric flexure configuration from ASTM C 1469-00. The vertical fused joint is shown in the center of the specimen. The round pins are the locations for the applied load, P.
representative sample of data testing in this manner could provide reliable information on joint strength. Additionally, unlike standardized testing, little extrapolation or statistical manipulation of strengths would be needed to interpret data for component design.

Test samples that were replicas of the component joint required determination of the geometry of mating components for use in the ceramic prototype. Size and scale would also significantly affect processing since thermal or pressure gradients can vary significantly with these parameters, which would affect joint strength. Testing and design would require iteration since the test results would affect the component design.

Alignment between fused parts of samples of both the standardized and the in-situ simulated testing would be important. The bar test specimen required in the standardized test would need to be perfectly aligned to ensure proper loading. The standards require no surface flaws, which would affect strength limits. A misaligned joint would be a large flaw in the specimen surface. Surface finishing could improve misaligned specimen, though consistency in finished size and surfaces would be critical. Alignment would also be important in the shrink-fit joint in order to maintain uniform pressure along the fused surfaces.

Clamping during firing would be required in order to maintain alignment and force on the dimensionally changing components. As the components shrink, the clamp would have to shrink in the direction of the applied force at approximately the same rate as the shrinkage of the components. Using the same batch for formation of the component and clamp would not ensure the same shrink since shrinkage depends on volume. The clamps would also have to survive the elevated and cycling temperatures of the kiln during firing.
In an effort to demonstrate the dimensional change between debinded and fired ceramics, two-dimensional shrinkage simulations were performed with ANSYS software (ANSYS, Inc., Lincoln, RI) using coefficient of thermal expansion (CTE) and temperature changes. Material properties were created in the simulation and the CTE was modified along with temperature until known shrinkage values were obtained. The known values were measured from disc-shaped samples obtained from Coors Ceramics (Golden, CO). Two samples were fired and four samples were green. The thickness and diameters of these samples were measured at ten different sites for each sample. The average sizes with standard deviation were recorded and are listed in Appendix A. The average values of the unfired, or ‘green’, samples were included in the simulation as the ‘hot’ metal. The average values of the fired samples were included in the simulation as the ‘cold’ metal. The results of the disc simulation which achieved known shrinkage are indicated in Figure 5.1(g).

Once the predicted dimensional changes were simulated, other two-dimensional geometries could be investigated to understand how the ceramics would shrink in assembly. Figure 5.1(d) shows an ANSYS model of a monolithic pincer after firing. The pincer was designed to shrink in a manner that would reduce the gap at the ‘fingers’ in order to hold a cofired test specimen with force during firing. It is indicated in the figure that the pincer dimensions shrink, but the gap is insignificantly reduced.
The simulation was used to understand the force concentrations and weak spots of cofired joints of two dissimilar materials with different CTE. Figure 5.1(e) indicates the stress distribution of a shrink-fit joint after firing. The sites of elevated stress are shown near the interior corners of contact. The tension and compression in the two components are also indicted. The flexural strength of the individual components should also be understood for design. The values of stress and strain in the simulation were not valid since the material properties applied loads are not accurate. The simulations demonstrated that ANSYS could be used as a tool to determine shrinkage and applied
loads. Iteration on design was not attempted for this project, but it is recommended for future work for advancement of cofiring techniques.

Ceramic testing for cofired joints was determined feasible in this chapter. Testing options were presented and test procedures were indicated through standardized test and replication of in-situ joint. Modeling was accomplished to indicate the utility of simulation for determination of dimensional changes through ceramic processing.

Figure 5.1(d) ANSYS 2-D model of pincer shrinkage. The mesh indicates the original size. The colors indicate the range of dimensional changes in meters.

Figure 5.1(e) ANSYS 2-D model indicating plane stress of a shrink-fit joint. The mesh indicates the original size. The colors indicate the range of stress in Pascals.
6 Recommendations

This project was originally focused on creating an assembly for subsequent testing of fluid flow from the swirling air/fuel mixture. It evolved into lessons in liquid resist processing on a porous substrate with double-sided structure formation and surface finishing. The difficulties in this project involved development of microfabrication for UV lithography on a pre-drilled substrate when processing both sides individually. Due to the advancement of X-ray lithographic techniques, it is recommended to use this process for future microfabrication. More work is required to solve the sealing requirements. The enormity of determining surface conditions for sealing along with achieving these specifications for both sides of Plate C was appreciated in this project.

6.1 Lapping

A solution to the problems associated with polishing the second side on the single-sided lapping machine is to use double-sided lapping along with X-ray lithography. Double-sided lapping would require altering the fabrication methods in order to keep the resist intact for processing the second side. A pre-drilled 100 mm round nickel substrate using X-ray lithography would be compatible with double-sided lapping.

6.2 Microfabrication Techniques

X-ray lithography is an attractive microfabrication method due to its use of solid PMMA resist for compatibility with processing a drilled substrate. Use of the solid PMMA resist would simplify much of the problems encountered with liquid resist. Filling the holes of the pre-drilled substrate would not be necessary since a smooth, solid surface would no longer be needed for a liquid resist. Adhesion of the resist would no longer be difficult since this is an established procedure using a methyl methacrylate
(MMA) adhesive layer. Sandblasting may not be required since adhesion of PMMA is better than SU-8, which would eliminate the alignment problems associated with the rough substrate. If the roughened substrate were needed, the procedure discussed in Section 6.2 would be useful. The developed resist areas covering the large holes would have to be larger than the holes to allow adhesion of PMMA to the periphery of the holes. The added step of fabricating an X-ray mask would be required along with configuring a clear field for alignment to the metal plate, which are all established methods when using a graphite mask mounted to a glass ring. The glass ring would provide optical transparency for alignment. The critically dimensioned features required for the masks are much larger than the current practical limits for minimum feature size of approximately 2 µm for an X-ray mask.

Reduction of plate size to the size of standard 100 mm diameter wafers would alleviate jig incompatibilities on the exposure station and the substrate holders in thin film deposition equipment. It would not be necessary to have the extra area of a 100 mm square plate if the alignment holes could be reconfigured to accommodate the working area of an X-ray mask.

Development and electroplating of both sides simultaneously would work best, but would not be necessary. Development of both sides of the plate would be easy since the resist could be exposed on one side then flipped for exposure of the second side. This exposure sequence would be possible since mounting the solid resist on both sides of the plate simultaneously is simple. The electroplating could be completed one side at a time using electroplating tape to mask the portions not being electroplated. Subsequent double-sided lapping would accomplish the finished surface on each side simultaneously.
The pressure on the substrate would be controlled with most double-sided lapping machines, which would allow gentle polishing. Flood exposure and then submersion of the plate in a beaker of acetone for a few hours would thoroughly remove the PMMA resist from the channels and large parts.

The procedure using X-ray lithography would also eliminate a number of other difficulties associated with UV lithography. Cycle time would be significantly reduced since each side would be processed simultaneously. Additionally, the resist would have consistent thickness over the entire substrate, and resist removal would not cause swelling of the PMMA.

A complication of double-sided lapping is that a number of other plates are required to balance the lapping wheel. These extra plates must be similar in geometry, thickness, and lapping surface to Plate C. Preparation of these extra plates would take time and money for cutting to size, grinding to thickness, and polishing to pre-defined surface conditions. Thus, multiple runs of processing samples would eventually lead to lapping substrates and would require a number of plates prepared. The double-sided lapping would have to be sent to a machinist, which presents uncertainties associated with lack of control of lapping conditions. However, the machinist would be more experienced and would lap the surfaces better than a student operator of the Engis machine.

6.3 Contrast of Alignment Marks

Creation of alignment features on the substrate with enough contrast to be viewed through the microscope and CCD cameras is a common problem. A reliable solution to alignment of the optical mask with a roughened substrate could not be implemented
during this project, but a procedure is presented here. The microholes are the most reliable feature of the plate against which to align. During EDM drilling of the four microholes, two more microholes with the same tolerances as the other microholes could be drilled on the plate periphery within the field of view of the alignment microscopes. The small size of the holes provides reduced alignment error and the positional tolerances are the most stringent of the precision machining. Marks that are complementary to the microholes must be developed and included on the mask with a clear field to optically align with the three peripheral microholes on the corners. Polishing the plate to improve the contrast of the microholes under the microscope would then be needed. The substrate should be sandblasted except in the locations of the two alignment holes. Alignment would then be accomplished using the peripheral holes in the substrate with the mask.

6.4 Plate Thickness

Plates C-1 and C-2 were too thin to process without eventual warping. These plates were 1.10 mm to 1.25 mm in thickness. Plate A had excellent dimensional stability and it was 1 mm thick in the reservoir and 2 mm thick in the volume surrounding the reservoir. A configuration similar to this one with a reservoir as the thinner area created from a thicker substrate could be devised. At this point, a fabrication technique is not apparent. Another solution would be to increase the thickness of the plates to 1.75 mm. While this would improve plate stiffness, the microholes would have to be larger in diameter than those drilled in the thinner holes, due to aspect ratio of the drilling process. Redesign of the swirl chambers and, possibly, swirler dimensions would be necessary with increased plate thickness.
References


Appendix A: Data Sheets

Table A.1. Composition of metals that were considered for use in metal fuel injector.

<table>
<thead>
<tr>
<th>Material</th>
<th>%Ni</th>
<th>%Cu</th>
<th>%Fe</th>
<th>%Mn</th>
<th>%Si</th>
<th>%C</th>
<th>%S</th>
<th>%Co</th>
<th>%Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel 200</td>
<td>99</td>
<td>0.25</td>
<td>0.4</td>
<td>0.35</td>
<td>0.35</td>
<td>0.15</td>
<td>0.01</td>
<td>trace</td>
<td></td>
</tr>
<tr>
<td>Nickel 201*</td>
<td>99</td>
<td>0.25</td>
<td>0.4</td>
<td>0.35</td>
<td>0.35</td>
<td>0.02</td>
<td>0.01</td>
<td>trace</td>
<td></td>
</tr>
<tr>
<td>Inconel 600</td>
<td>72</td>
<td>0.5</td>
<td>6-10</td>
<td>1.0</td>
<td>0.5</td>
<td>0.15</td>
<td>0.015</td>
<td>14-17</td>
<td></td>
</tr>
<tr>
<td>Inconel 718◊</td>
<td>50-55</td>
<td>0.3</td>
<td>17</td>
<td>0.35</td>
<td>0.35</td>
<td>0.08</td>
<td>0.015</td>
<td>1.0%</td>
<td>17-21</td>
</tr>
</tbody>
</table>

* Nickel 201 composition and data provided by Inco Alloys®. Difference between Ni 200 and Ni 201 is not made by ASME.
◊ Additional elements: 0.2-0.8% Al, 0.006% B, 2.8-3.3% Mo, 4.75-5.5 Nb, 0.015% max P, and 0.65-1.15% Ti. Composition and data supplied by Inco Alloys®.

Table A.2. Summary of hardware used for fuel injector assembly

<table>
<thead>
<tr>
<th>Function</th>
<th>Part</th>
<th>Supplier</th>
<th>Part number</th>
<th>Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluid connectors</td>
<td>Compression fitting</td>
<td>Omega Engineering, Stamford, CT</td>
<td>SSLK-316-18</td>
<td>SS 316; 1/8” NPT coupled with 3/16” compression fit.</td>
</tr>
<tr>
<td>Fasteners</td>
<td>Bolts</td>
<td>Small Parts, Inc. Miami Lakes, FL</td>
<td>MSHCX-2-12</td>
<td>SS; M2 x 12</td>
</tr>
<tr>
<td></td>
<td>Nuts</td>
<td>Small Parts, Inc. Miami Lakes, FL</td>
<td>MHNX-2</td>
<td>SS; M2 x 0.4</td>
</tr>
<tr>
<td>Lock washers</td>
<td>Washers</td>
<td>Small Parts, Inc. Miami Lakes, FL</td>
<td>MLWX-2</td>
<td>SS; M2</td>
</tr>
<tr>
<td>Alignment pins</td>
<td>Dowel pins</td>
<td>Small Parts, Inc. Miami Lakes, FL</td>
<td>DWX-2-24</td>
<td>SS; 1/8” OD x 1-1/2” length</td>
</tr>
</tbody>
</table>

SS = Stainless steel
Table A.3  Measured sizes of the ceramic samples from Coors Tek™ before and after sintering.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Unfired</th>
<th>Fired</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>Thickness (mm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.7232</td>
<td>2.7349</td>
<td>2.0079</td>
</tr>
<tr>
<td>Standard Deviation (mm)</td>
<td>0.0387</td>
<td>0.0252</td>
</tr>
<tr>
<td>Diameter (mm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Standard Deviation (mm)</td>
<td>0.034</td>
<td>0.022</td>
</tr>
<tr>
<td>D/t ratio</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Appendix B: Shop Drawings

Figure B.1 Shop drawings for grinding the Inconel 600 and Nickel 201 plates. The stainless steel plate did not require tolerances. Note the prescribed thickness of Plate B is 1.5 mm.
Figure B.2

Shop drawing for Plate D. The dimensions indicated are the same locations and sizes of the fastener and alignment holes common to all of the plates.

Plate D
(5.2 mm thick SS)

Undisclosed tolerances:
+/- 0.020"
Figure B.3   Specifications for tapping and welding compression fittings into the back side of Plate D.

Plate D
(5.2 mm thick SS)

Flycut/grind/polish/lap top surface to achieve 0.201" +/- 0.004" thickness and flat to within +/- 0.0001" after welding and all drilling is complete.

Compression fittings (just below top surface level)

Undeclared tolerances: +/- 0.010"

Weld about perimeter of each compression fitting.

Lousiana State University
Department of Mechanical Engineering
Metal Model Fuel Injection - Plate II - 1st pass
Drawing by Tracy Harris and Rachel Lachnak
Work: 388-4412 Home: 675-9764
SPONSORED BY Dr. Michael Murphy
Scale: 1/7
Plate B
\[ \times 1 \]
0.049” (1.25 mm) thick Inconel 600

Figure B.4 Shop drawing for Plate B.
Figure B.5  Shop drawing for Plate C.
Plate A
(2mm thick w/ 1mm milled as indicated)
Inconel 600

Figure B.6 Shop drawing indicating the differences between Plate A from Plate D.
Figure B.7  Shop drawing for the spinning jig used to adapt the spinner holder with the nickel plates.
Figure B.8 Proposed drawing for x-ray mask for air-side for compatibility to x-ray exposure machine. The circular ring indicates the location of a standard NIST stainless steel ring, to which the mask membrane is mounted.
Vita

Tracy Morris was born on November 1, 1969 in St. Augustine, Florida to Jane Swilley Ettel and Ray D. Ettel II. She married Chad Aaron Morris in 1989.

Upon moving to Baton Rouge in May 2001, she began attending Louisiana State University. She graduated from Louisiana State University with a Bachelor of Science degree in Mechanical Engineering in 1996. She postponed graduate school to care for her three children.

In August 1998, she began graduate studies at Louisiana State University. In September 2001, she began full-time employment with the Center for Advanced Microfabrication and Devices (CAMD) at Louisiana State University. In December 2001, she successfully defended her thesis. She expected to graduate May 2002 with a Master of Science in Mechanical Engineering, but was delayed until May 2004.