OPTIMIZATION OF A LIGHT EMITTING DIODE BASED PROJECTION STEREOLITHOGRAPHY SYSTEM AND ITS APPLICATIONS

BY

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THESIS

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ABSTRACT

The rapid manufacture of complex three-dimensional components has eluded researchers for decades. Several manufacturing options have been limited by either speed or the ability to fabricate true three-dimensional structures. Over the last decade, projection stereolithography has been developed to manufacture true three-dimensional structures in a faster manner than traditional processes. This work discusses the development of a projection stereolithography system and the different process parameters that affect part quality. A functional system has been created at Lawrence Livermore National Laboratory and several unique components have been fabricated to demonstrate its capabilities. Examples of true three-dimensional structures and tunable porous materials are presented.
This thesis is dedicated to my family and the outreach programs that developed me.
ACKNOWLEDGMENTS

I would like to acknowledge my family and all the support they have given me. They have always been there to support me and push me forward to excel. My siblings have been particularly helpful through my college years and their support has been instrumental to my progress. Thank you, Karlena, Michael, and Jeremy! I would like to express my heartfelt appreciation to my wife, Renata, for encouraging my graduate work and allowing me the freedom to explore (break) many electronic devices in pursuit of knowledge.

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# TABLE OF CONTENTS

CHAPTER 1: INTRODUCTION ........................................................................................................... 1
CHAPTER 2: LITERATURE REVIEW ................................................................................................. 3

2.1 Introduction ............................................................................................................................... 3

2.2 Mask Technologies .................................................................................................................... 3

2.3 Gray Scale Support System ....................................................................................................... 5

2.4 Stop Flow and Continuous Flow Lithography ......................................................................... 5

2.5 Competitive Technologies ........................................................................................................ 6

CHAPTER 3: SYSTEM DESCRIPTION ............................................................................................. 8

3.1 Introduction ............................................................................................................................... 8

3.2 Software .................................................................................................................................. 10

3.3 Optical System .......................................................................................................................... 13

3.4 Materials .................................................................................................................................. 15

3.5 Nitrogen Environment ............................................................................................................. 15

3.6 System Resolution .................................................................................................................... 15

CHAPTER 4: OPTIMIZATION ........................................................................................................ 17

4.1 Introduction ............................................................................................................................... 17

4.2 Parameters ................................................................................................................................ 20

4.3 Method .................................................................................................................................... 21

4.4 Exposure Depth ......................................................................................................................... 23

4.5 Reproducibility .......................................................................................................................... 27

4.6 Model Description and Comparison ....................................................................................... 28

4.7 Conclusions .............................................................................................................................. 30

CHAPTER 5: APPLICATIONS ......................................................................................................... 31

5.1 Introduction ............................................................................................................................... 31
Research often involves the fabrication of components and devices with features that current technology struggles to produce. In the future, this problem will encompass components that the general public will desire to consume. Over the last two decades, researchers have utilized processes designed for the computer industry to meet design challenges. However, these processes are restricted in their capabilities to produce complex geometries and true three-dimensional structures. Several technologies were devised to bridge the fabrication gap over the last decade. This thesis will cover the design and optimization of one of these processes, projection micro stereolithography, and its applications. This technology will be compared to competing fabrication technologies in Chapter 2.

Projection stereolithography is a three-dimensional fabrication technology that is based on a photo polymerization reaction. A schematic of this fabrication technique is given in Figure 1. Typically, a series of two dimensional images are created to represent a three dimension structure. These two dimensional images are sent one by one to the machine, which projects them onto a liquid monomer surface. The portion of the image to be fabricated is represented by white pixels. The light source of traditional image processing machines, like projectors, is commonly white light; therefore, it is completely reflected or transmitted to the intended viewer. In a projection stereolithography system, light of a specific wavelength is reflected off or transmitted through a dynamically reconfigurable mask to the liquid monomer surface. A stage holding a substrate is then lowered, exposing a fresh layer of resin and another image is displayed. In order to successfully fabricate components using this technique, several parameters must be properly controlled.
This thesis covers the setup of a projection micro stereolithography (PμSL) machine in Chapter 3 and the effects of various parameters in Chapter 4. Parameters covered in this thesis include: light flux, exposure time, photoabsorber concentration, and environmental conditions. In Chapter 5, several different applications of this system will be demonstrated. It should be evident from the results presented here and in the literature that this type of fabrication method can significantly contribute to scientific advancement by providing the capability to construct complex three dimensional geometries.
CHAPTER 2: LITERATURE REVIEW

2.1 Introduction

The concept of projection stereolithography was introduced by Bertsch\(^1\) in 1997. Lithography was not a new concept, but projection stereolithography transformed it from a slow serial process to an extremely rapid and effective parallel process. This technology employs a spatial light modulator, which is also known as a dynamic or digital mask. Light passes to the digital mask, which displays an image of the layer to be fabricated, and is either transmitted or reflected depending on the mask. A series of optical devices are then introduced to focus the image on the desired build plane. The light causes a polymerization reaction in the shape of the projected image, and stacks of these images produce three dimensional objects.

The digital mask is the core technology that enables projection stereolithography. Bertsch employed the first digital mask, a Liquid Crystal Display (LCD)\(^1\) in 1997. Researchers have a choice of devices to utilize as a dynamic mask, which include Liquid Crystal on Silicon (LCoS) chips, Digital Micromirror Devices (DMD), or the standard LCD. These devices allow the manipulation of individual pixels. This provides the investigator with the ability to rapidly change the mask by sending a new digital image, rather than replacing a physical mask and painstakingly realigning the system as is standard in lithography.

In 2003, P\(\mu\)SL experienced a shift from being considered a rapid prototyping technology to being viewed as a manufacturing process. Xia\(^2\) was able to produce a three-dimension microstructure directly under a MEMS device, described by Chen\(^3\). While the fabrication tool is capable of producing complete components, it is a lower throughput technology compared to stop-flow lithography or continuous flow lithography, which will be described in section 2.4. Applications like the holhraum described in Chapter 5 will need to be fabricated at enormous rates, which will require a combination of these technologies.

2.2 Mask Technologies

Three different mask types have been utilized in projection micro stereolithography: the standard LCD, the DMD, and the LCoS chip. In an LCD system light is transmitted through the mask and specific pixels are designated electronically as either opaque or transparent. The first
projection stereolithography system employed an LCD chip as described by Bertsch in 1997. Bertsch reviewed the common fabrication techniques available at the time. LCD chips were limited by their low transmission of light and low contrast ratios. The common LCD chip is still characterized by these disadvantages.

In 1999, Beluze developed the first system to utilize DMD as a dynamic mask for three-dimensional fabrication. These devices reflect light at a fixed angle, typically ± 13°. This creates a much higher contrast ratio than the LCD chip and since the light is reflected rather than transmitted; it does not suffer the light flux problems associated with an LCD chip. The two chip technologies were compared by Sun et al. in 2005. Sun achieved impressive results, in terms of resolution, by demonstrating components with 0.6µm features. The DMD chip provided a solution capable of producing high quality components by improving the contrast and resolution.

The LCoS chip was developed in 1999 and is technologically similar to the LCD. Liquid crystals are deposited on top of a highly reflective surface; by electrically controlling the polarity of these crystals the light flux of each pixel can be defined. The LCoS and DMD display chips are both capable of gray scale imaging, have similar contrast ratios, and have similar resolution. The LCoS is more affordable and easier to implement.

The above masks have been tried and tested in various projection stereolithography machines. New mask technologies are evolving and may provide a better option for future researchers. LCD and LCoS technology has been improved using ferro electric crystals, creating FLCoS and FLCD options. FLCoS are fabricated using CMOS process and LEDs can be fabricated on the same chip as the device. Currently, these options are not readily available and have not been used in any projection stereolithography device. Table 1 compares and contrasts the different mask technologies available.

Table 1: Comparison of different mask properties important to PµSL.

<table>
<thead>
<tr>
<th>Technology</th>
<th>Light Loss</th>
<th>Contrast</th>
<th>Resolution</th>
<th>Cost</th>
<th>Speed</th>
<th>Power</th>
<th>Optics</th>
<th>UV</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCD</td>
<td>High</td>
<td>Low</td>
<td>Low</td>
<td>Lowest</td>
<td>Low</td>
<td>Low</td>
<td>Easy</td>
<td>Poor</td>
</tr>
<tr>
<td>DMD</td>
<td>Lowest</td>
<td>Highest</td>
<td>High</td>
<td>High</td>
<td>Highest</td>
<td>High</td>
<td>Difficult</td>
<td>Good</td>
</tr>
<tr>
<td>LCoS</td>
<td>Low</td>
<td>High</td>
<td>High</td>
<td>Mid</td>
<td>Mid</td>
<td>Low</td>
<td>Medium</td>
<td>Okay</td>
</tr>
<tr>
<td>FLCoS/FLCD</td>
<td>Low</td>
<td>High</td>
<td>Mid to High</td>
<td>High</td>
<td>High</td>
<td>Lowest</td>
<td>Easy</td>
<td>Okay</td>
</tr>
</tbody>
</table>

The aspects considered in Table 1 are defined as follows. The light loss is the greatest in technologies where light is transmitted through the device like the LCD and lowest in reflective
technologies like the DMD chip. The contrast describes how well the light of one pixel is confined to that pixel. The speed of the system refers to the switching speed of the device, this is important for gray scale fabrication and for reducing exposure times. The optic setup is more complicated for a DMD system since the light is reflected at a small angle. Additional setup and space in the system is required. An LCD system can be very simple since light passes through the mask. Since an LCoS system reflects light, it requires more components than an LCD system. UV light can be very harmful to the different chip technologies. The materials in DMD are able to tolerate the higher energy light better. Extreme UV can damage all of the technologies described, but the amount of time to failure is lowest for the DMD technology.

### 2.3 Gray Scale Support System

The main advantage of projection stereolithography is the ability to fabricate layer-by-layer compared to the serial method employed by other technologies. However, this advantage has its limitations. Fabrication layers with features that are spatially separated and not connected by a previous layer are unconstrained and will move freely within the monomer resin. Rapid prototyping and lithography techniques employ support scaffolds made of soluble materials to support features that are orphaned during fabrication. This solution is impractical for projection stereolithography systems which are typically constrained to one material.

LCoS chips can manipulate the polarity of light, thereby allowing it to modulate the flux of the light it reflects. This technique adds a new dimension called gray-scale fabrication. Prior to gray-scale fabrication, a binary system of white and black images was employed. White images created fully cross-linked components and black images did not polymerize the resin. Parts fabricated using a gray image have lower cross-linking density and can be chemically dissolved without affecting the fully cross-linked, white imaged, structures. Xia has demonstrated the value of gray-scale fabrication as a method to create disconnected features and to aid in the build process. This technique solved a major problem for projection stereolithography and improved its effectiveness as a fabrication technique. This technique was demonstrated using a DMD chip for the standard lithography process by Totsu.

### 2.4 Stop Flow and Continuous Flow Lithography

In 2007, Dendukuri et al. introduced stop flow lithography and continuous flow lithography. Stop flow lithography incorporates a traditional fixed pattern lithography setup, but integrates it with a microfluidic system to transport the components. The monomer flows
through a polydimethylsiloxane (PDMS) microchannel to the fabrication area. The flow is stopped using a valve and light passes through a fixed mask to polymerize the monomer. The microchannel is made of PDMS to ensure that the monomer does not adhere to the channel after polymerization. PDMS contains oxygen that inhibits the polymerization reaction, which is discussed further in section 4.1. The valve is released and the two-dimension assembly line continues.

Continuous flow lithography is similar to stop flow lithography with the exception that the material continues to flow during fabrication. Light passes through a fixed mask as the fluid moves. Since the fluid is moving, a mask of a circle would produce a pill shape and a square mask would produce a rectangle. Multiple immiscible fluids, exhibiting laminar flow, can create multi-material components. Dendukuri et al.\textsuperscript{10} demonstrated this technique with two different materials.

These systems can create a large volume of two-dimensional parts. However, a new mask must be fabricated and installed in the system in order to change the pattern. The combination of stop flow or continuous flow lithography with projection stereolithography would remove a major disadvantage of both techniques. The realization of a dynamic micro assembly line could have a very significant impact on micro device fabrication by PµSL. The drawback of using a dynamic mask with these two techniques is a more complex setup, but it has the significant advantage of being able to change the shape of the fabricated components while the machine is operating. The integration of a digital mask is particularly advantageous to the continuous flow lithography. The mask and the fluid flow can be used to fabricate a much wider variety of parts. Rather than digital images being sent to the dynamic mask, a video could be utilized to create a continuous part. With the additional of gray-scale fabrication, a very complex 2.5 dimensional part could be created.

2.5 Competitive Technologies

Competitive technologies to PµSL include two-photon polymerization, conventional stereolithography (STL), and digital three-dimensional printing. For a history of STL and early PµSL please reference Bertsch\textsuperscript{11}. PµSL’s ability to fabricate the entire layer in one step makes it suitable to replace serial STL concepts except two-photon polymerization (TPP). Two-photon polymerization was developed by Maruo et al. in 1996\textsuperscript{12}. TPP utilizes focused lasers to precisely polymerize small volumes resin. The volume is only polymerized if it is excited by two different
photons within a very short time period. TPP is much slower than PµSL, but has successfully created components with 100 nm features\textsuperscript{13}. TPP is limited to polymers because it requires a clear resin to function; suspended particles would scatter the laser beams. PµSL can fabricate components with polymers and metal or ceramic polymer suspensions.

Cox\textsuperscript{14} carried out a comparison of technologies competitive with PµSL in 2006. Technologies compared included: microstereolithography, 3D printing, Selective Laser Sintering, Fused Deposition Modeling, Soft Lithography, Micromachining, and Laser Machining. None of the three-dimensional technologies listed above are competitive with PµSL for polymers. PµSL has also been demonstrated with ceramic\textsuperscript{15} and metal suspensions. Several parallel fabrication technologies have been developed that focus more on micron and submicron fabrication. High-resolution electrohydrodynamic jet printing, first introduced in 2007 by Park \textit{et al.}\textsuperscript{16}, has been able to achieve a resolution of 200 nm. It is limited right now to two-dimension fabrication, but it has a much larger working area than PµSL. Electrophoretic assembly has adapted projection mask and could benefit from the mask usage of projection stereolithography\textsuperscript{17}. 
CHAPTER 3: SYSTEM DESCRIPTION

3.1 Introduction

The basic system schematic for the Lawrence Livermore National Laboratory’s (LLNL) PµSL system is given in Figure 2. The major components are identified and a schematic for how they are connected is provided. The setup was constructed on a Newport isolation table. The components are controlled using a National Instruments’ Labview program. The graphical user interface (GUI) is shown in Figure 3. This GUI shows the current image being fabricated along with a continuous stream of still images of the fabrication surface for exposures over 800ms in length. The build sequence is controlled by a user generated text file discussed in section 3.2. The LCoS, camera, and LED are fixed on stages to adjust their positions for alignment. The camera can only capture a fraction of the build plane since the charge-coupled device (CCD) chip is smaller than the LCoS chip in this system. The camera is able to capture approximately 30% of the LCoS’s display area. For comparison, the bottom star in Figure 3 is the CCD image and the top star is the image being displayed by the LCoS.
Figure 2: Schematic of LLNL’s PµSL system. System designed by Steven Gemberling, George Farquar, and Chris Spadaccini from LLNL and Nick Fang from the University of Illinois at Urbana-Champaign. Design contributions were also made by the author.
Figure 3: Graphic User Interface for the PµSL system, GUI created by Steven Gemberling, LLNL

3.2 Software

The projection of two dimensional images by the system greatly reduces the complexity of software necessary. Microsoft Power Point was used to generate a series of images to be fabricated. The bitmap file standard was used and images were saved at a resolution of 1920 x 1200 to match the LCoS. As an example, the bitmap files for the bridge structure, discussed in Chapter 4, are shown in Figure 4 below. The files are loaded by the system and these images are sent to the LCoS chip. A profile is created in a txt file. This file determines the order in which the images are displayed, the movement of the z axis, the amount of photons delivered to the build plane, and it tells the system how long to wait until the liquid bath is stabilized after a movement.
Figure 4: Bitmap images displayed on LCoS to fabricate bridge structures shown in Figures 12, 13, and 15. The predicted width of the bridge is 96 µm.

An example profile is given in Figure 5. Lines 1 and 2 of the profile direct the machine to coat or recoat the substrate. The example profile checks the focus of the fabrication plane on the liquid monomer resin. Since the resin and build substrate is replaced or moved after each build, it is necessary to re-tune the focus prior to fabrication. The profile in Figure 5 lowers the build substrate into the polymer to fully coat it in resin. This step is important because it breaks any menisci created by surface tension and then raises the stage back to zero. The operator checks the camera image and looks for a slightly out of focus image, like the image in Figure 3. This profile utilizes a very low light flux to avoid polymerizing the material. The camera is set to automatically adjust its light levels to accommodate for this low intensity. In the fabrication of parts this movement is repeated after each layer is fabricated. The stage travels down 1.5mm and then back up the appropriate layer thickness. The reason for this movement is discussed in section 4.1. The full procedure is included in the appendix.
**GENERAL FAB INFO BEGIN**

Fabrication Files Folder Pathway: C:\Documents and Settings\fabuser1\Desktop\Bitmap Files Folder\NanoFab\Profile_parts\Matt_January_2010\Part2_Jan5_2010_RectangStar

Fabrication Starting Position (mm): 0.000
Sample Removal Position (mm): -0.000

**GENERAL FAB INFO END**

**COLUMN NAMES BEGIN**

Column 1: Layer Number  
Column 2: Bitmap Filename  
Column 3: Layer Thickness (mm)  
Column 4: LED Intensity (watts)  
Column 5: LCoS ON Time (mS)  
Column 6: Polymerization Time (mS)  
Column 7: Settling Time (mS)

**COLUMN NAMES END**

**PROFILE BEGIN**

<table>
<thead>
<tr>
<th></th>
<th>Bitmap Filename</th>
<th>Layer Thickness (mm)</th>
<th>LED Intensity (watts)</th>
<th>LCoS ON Time (mS)</th>
<th>Polymerization Time (mS)</th>
<th>Settling Time (mS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>blank.bmp</td>
<td>1.5</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>blank.bmp</td>
<td>-1.5</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>10000</td>
</tr>
<tr>
<td>3</td>
<td>slide2.BMP</td>
<td>0.5</td>
<td>1000</td>
<td>100</td>
<td>100</td>
<td></td>
</tr>
</tbody>
</table>

**PROFILE END**

Figure 5: Example focusing profile – template created by Steven Gemberling, LLNL
For high volume items, features like refocusing during a build cycle and automated x and y stages might be necessary. Objects in this study are very small compared to the polymer bath; consequently, this functionality has not been added.

### 3.3 Optical System

The optical system consists of a light source, a dynamically reconfigurable mask, and a series of optical components. An overview of this system is given in Figure 6. The system resolution and working area is limited by the selection of the reconfigurable mask, the wavelength of light, and reduction optics.

The LumiBright Light Engine, a 395 nm LED array, was selected for its low cost, extended life, compact size, and wavelength specificity. An LED source is also modular and can easily be replaced or transferred to a new system. The light produced by an LED source is more divergent than light produced by a laser. An LED system requires the investigator to consider the light intensity and contrast of the system, as they are adversely affected by the light divergence.

The system has a Holoeye HED-6001 LCoS chip with 1920 x 1080 pixels over a 15.36 x 8.64 mm area. The LCoS has a contrast ratio of 2500:1 and reflects 95% of the light when active. The LCoS is part of an OEM HDTV kit that includes the control board for the system. This board was developed by Aurora Systems Inc. This allows the LCoS to function the same as an external monitor.

An LCoS chip has a reflective surface with an array of individually controlled pixels on top. The pixel polarity can be adjusted and the amount of outgoing light controlled by applying a voltage. This is regulated in the same manner as any normal projection system. The closer the displayed image is to white, the higher the light flux received. Only black and white images were used. A minimum contrast ratio of 4:1 was achieved at the fabrication surface after passing through all of the optical components. This was measured by comparing the light flux at the build substrate of a white image versus a black image. This is much lower than the maximum capable contrast ratio of the LCoS chip. Additional improvements are required to increase the polarization of the light, which will increase the contrast ratio. Adding another polarizing optic after the cubic will increase the contrast ratio; however, it will also decrease the light flux at the fabrication plane. A collimator can be employed to collimate the light prior to the LCoS, but this
is difficult to implement correctly in a confined space. Thus, there is a tradeoff between improving the quality of light at the fabrication plane and light flux.

UV projection optics from Coastal Optical Systems (part numbers 0745200, 0508100, and 0452200) reduce the image by a factor of 6:1 and therefore our approximate resolution is 1.3 \( \mu \text{m} \). In this case, our resolution does not approach the diffraction limited resolution of 200 nm.

The beam splitting cubic is from Tower Optical Corporation and was designed for 405 nm light, part number 13397. It contains a polarization film on the hypotenuse and an anti-reflective coating with an R <0.25% per face. The cube is made from BK7 Grade A material. The Pellicle Beam Splitter is a 92% transmitted 8% reflected beam splitter. This is selected to direct most of the light to the build plane for fabrication.

![Figure 6: Schematic representation of the optical system.](image)

We used a CCD camera to ensure the projected image was focused on the monomer and build substrate. By focusing the camera on a scored substrate and adjusting the optics until the image was in focus, we ensured that the two image planes were properly aligned. Prior to fabrication, the camera is used to view the focused image on the liquid resin. Since the substrate and image planes are aligned, the investigator only needs to move the resin bath to this plane. This step enables the system to create parts that maintain high fidelity with the image to be
fabricated. The camera was also employed to monitor fabrication. Variations in the picture feed can be interpreted by the investigator and will help identify a failed build.

3.4 Materials

All objects were fabricated on a gold plated silicon wafer. This wafer was lightly sanded and cleaned before being bonded to the substrate holder. Qualitative experiments indicated that objects had a stronger bond to the substrate when it was scored.

All models were fabricated using 1, 6-hexanediol diacrylate (HDDA; CAS 13048-33-4) and mixed with 1-phenylazo-2-naphthol (Sudan 1; CAS 842-07-9), a photoabsorber that varied in concentration from 0 – 1.5% by weight in HDDA, and phenylbis (2, 4, 6-trimethylbenzoyl) phosphine oxide (Irgacure 819; CAS 162881-26-7), a photoinitiator fixed at a concentration of 2% by weight. Irgacure 819 has an absorption peak at 370 nm and is soluble up to 9% by weight with HDDA. Sudan 1 is a photoabsorber that limits the exposure depth and spread of the polymerization. This increases the effective contrast ratio and reduces the minimum step size, which brings the system closer to its maximum resolution.

3.5 Nitrogen Environment

Two separate conditions were tested in this setup. In one case, the machine was open to the atmosphere and in the second case parts were fabricated in a nitrogen environment. The overall system setup was the same except the material container was covered and nitrogen was pumped into the chamber. Slits were cut for the projected image to enter the container and for the substrate holder to move freely. The nitrogen was pumped in at a rate sufficient to maintain a positive pressure in the container. This is needed to prevent oxygen from entering the system. Oxygen will react with the free radicals generated by the photoinitator; reducing the amount available for polymerization. In short, oxygen inhibits the polymerization and reduces the cure depth for a given set of parameters. This will be discussed in Chapter 4.

3.6 System Resolution

In this set of experiments, only the Z stage moves during fabrication. A linear stage was utilized for the Z-axis. This stage has a resolution of .1μm and a minimum step size of .3μm. The maximum build height is on the order of several centimeters, although no parts were manufactured over 1.5mm. Manual stages exist for the x, y, and focusing axis. The y axis was
used to reposition the stage to fabricate multiple parts on the same substrate. The monomer bath sits on a focusing stage and is manually adjusted prior to fabrication.

The system has an approximate resolution of 1.3 microns as estimated from the LCoS size, resolution, and reduction lenses. To test the resolution, several base layers were created and the phrase “LLNL & ILLINOIS” was projected at various font sizes. The result of this test is shown in Figure 7. The line width of the letters is 4.2 µm. The fine detail of each letter suggests that we were close to the predicted resolution. The exposure conditions will alter the image if it is overexposed. The x and y dimensions and their dependence on exposure conditions were not determined; therefore, an exact determination of the system resolution cannot be made from this image.

Figure 7: System capability test. This image contains features smaller than 4.2 µm.
CHAPTER 4: OPTIMIZATION

4.1 Introduction

In order to create successful components using a projection stereolithography machine a few issues need to be addressed. Since the system relies on polymerizing material on a substrate within a liquid bath, these two planes must be level or a system must be in place to correct for it. Since the monomer is a liquid, it can be used as a reference level surface. It would require additional stages to physically level the build substrate. A base is built on top of the substrate to create a level structure for component fabrication. Figure 8 shows two fabrication attempts. Attempt A was built directly on a scored substrate, while Attempt B was built on a base. The desired structure in Attempt A is distorted because the substrate was not level. In Attempt B, the first layer of the base is distorted and the structure is unaffected. The stage is manually positioned for the first layer and the actual thickness of the first layer is unknown. It is typically less than 50 µm, but an excess amount of exposure dose is used to produce this layer since the thickness is unknown. Exposure dose is defined as light flux multiplied by exposure time. For this reason, the first layer in Attempt B is very thick compared to the layers above it. Several layers are built on top of this layer to ensure a flat surface is obtained.
Figure 8: Justification for using a self-leveling base.

The next challenge stems from the viscosity of the fluid, surface tension, and the small layer thicknesses attempted in projection microstereolithography. The basic operation in projection stereolithography is to lower the substrate into a monomer bath, polymerize the layer, and repeat this process. However, the stage cannot simply be lowered to the next desired layer thickness. The star pattern on the left in Figure 9 shows one potential problem of only lowering the stage to the desired layer thickness. If the wait time is not long enough for a given viscosity, the material will not coat the base layer and a hole will result. In some cases, a meniscus will form, as depicted, and the material will not level at all. By lowering the substrate far enough to break the meniscus, the resin will flow and the stage can be raised to the desired height. The right star in Figure 9 was lowered 1.500 mm into the material bath and then raised 1.495 mm. This allows the monomer to coat the surface and break any menisci formed by the monomer and the component being fabricated. A wait time is still necessary for the free surface disturbance to settle.
Figure 9: Meniscus and viscosity effects are avoided by lowering the stage deep into the resin.

The above process steps are the basis for creating successful parts in projection micro stereolithography. The next step is to understand the underlying chemical reaction in photo polymerization. Figure 10 outlines the steps that occur during polymerization and provides a mathematical description for how it occurs. When the photoinitiator, Irgacure 819, is irradiated with light, free radicals are released. In an ambient environment these radicals have two choices; either bind with a monomer or react with oxygen. When it binds with a monomer, the reaction propagates as it recruits more monomers and forms a polymer chain. When two of these chains meet, the radicals recombine and the chain propagation is terminated, which forms a higher molecular weight polymer. This process is described by equations 1-5 in Figure 10. Equation 1 describes how the absorption of light is dependent on the amount of photoinitiator and the amount of photoabsorber. Equation 2 describes the consumption of the monomer; this reaction is used to calculate the total time for the other processes. The photoinitiator depletion is
described by Equation 3 and is dependent on photoinitiator diffusion and the generation of radicals. The generation of free radicals in Equation 3 is proportional to intensity. The consumption of radicals is dependent on the diffusion of radicals, the generation of radicals, and the rate of chain termination as shown in Equation 4.

\[
\frac{dI}{dz} = -(\varepsilon[S] + \varepsilon_a[S_a])I
\]

\[
\frac{d[R]}{dt} = -k_p[R][M]
\]

\[
\frac{\partial[S]}{\partial t} = \nabla(D_e \nabla[S]) - \frac{1}{2} \sigma \varepsilon[S] I
\]

\[
\frac{\partial[R]}{\partial t} = \nabla(D_p \nabla[R]) + \sigma \varepsilon[S] I - k_t[R]^2
\]

\[
\frac{\rho C_p}{\partial T} = k \nabla^2 T - \Delta H \frac{d[M]}{dt}
\]

\( I = \text{Irradiance} \)
\( D_e = \text{Diffusion coefficient} \)
\( D_p = \text{Light penetration depth} \)
\( \sigma = \text{of radicals activated per photon} \)
\( \varepsilon = \text{Molar extinction coefficient of photo-initiator} \)
\( H = \text{Enthalpy} \)
\( T = \text{Temperature} \)

Figure 10: Polymerization process and mathematical description. Process and equations from Odian\(^\text{18}\).

4.2 Parameters

Since projection stereolithography is based on photo polymerization, there are a large number of parameters that need to be controlled. This study reviews: exposure time, exposure dose, exposure flux, Sudan I concentration and qualitative environment. An iterative process was used to gradually improve the machines performance and reduce the layer thickness. Exposure dose was varied by controlling LED light flux and exposure time. The Sudan I
concentration was varied between .08% and 1.5%. The material was either open to air or under a constant nitrogen atmosphere.

Great care needs to be exercised to ensure the optical system produces uniform light while maintaining sufficient light flux for polymerization. To maximize the amount of polymerization, the wavelength of light used should match the photoinitiator. Optimal results are obtained at a lower light flux and a longer exposure time. A quick and intense burst of energy tends to create underexposed and, therefore, not fully cross-linked gel-like results. This is due to the diffusion limitation of the polymerization reaction; a short burst of energy does provide enough time for the radicals to be generated, diffuse, and terminate below the surface.

Sudan I is a photoinhibitor and prevents the exposure dose from penetrating deep into the polymer, which helps control the layer thickness. The second action of Sudan I is to increase the threshold light flux needed for polymerization. This decreases the sensitivity and makes it more difficult for stray light to polymerize the solution, effectively increasing the contrast of the system.

Irgacure 819 is a photoinitiator that releases free radicals when it absorbs energy. This causes the monomers in solution to bind and polymerize. The presence of oxygen limits the number of free radicals available for polymerization. For this reason, in order to minimize the amount of exposure dose required, the system should be designed to minimize oxygen at the reaction surface. Experimental data have been collected for experiments in an oxygen environment and in a nitrogen environment.

4.3 Method

The method to improve the system resolution and document the fabrication parameters is described in Figure 11. For the comparison studies, if the depth was incorrect the build conditions would still be recorded for model comparison. In this study, for a given photoabsorber concentration, the light flux and exposure time would be varied. A photoabsorber concentration was chosen and the conditions optimal for that concentration were determined. Then the concentration was changed and the process repeated. Each concentration was optimized according to the method outlined in Figure 11.

Overexposure was determined via measurements from a Scanning Electron Micrograph (SEM) and a comparison was made to the desired layer thickness. The layer thickness is specified in the build profile and is the thickness of the liquid monomer above the previous layer.
or build substrate. Overexposure can occur when there is an excess amount of photoinitiator, insufficient amounts of photoabsorber, or if the exposure energy and time is too high. Since the amount of photoinitiator was fixed in this study; the photoabsorber concentration, exposure time, or light flux can be adjusted to reduce the exposure depth. The light flux should be reduced and the exposure time increased if the fabricated component has a hard shell, but is soft and gel-like inside. Two examples of overexposure are presented in Figure 11. The top example is a case of background overexposure. The intensity of the background image became greater than the threshold intensity for polymerization, causing the desired image and the background to be polymerized. The light flux can be reduced, more photoabsorber added, or the contrast ratio of the system could be improved to alleviate this problem. The second overexposure case is more useful than the first. It is still a successful build because the polymerization depth can be recorded and incorporated to create parts with the measured layer thickness of this bridge. One problem that should be noted is the slight curving of the structure. Rapid polymerization or non-uniform light can cause distorted structures. The rapid polymerization of the structure causes the top layer to highly crosslink, but does not allow sufficient time for the light and radicals to diffuse and crosslink the material below it. This can cause the top of the layer to shrink more than the bottom portion and curve the beam. Similarly, non-uniform light causes the same variation in crosslinking density in the x or y direction and can tilt the beam.

Underexposure was rarely observed except in the case of gel-like components described above. Generally underexposure causes the entire structure to fail or in the case of bridge studies, no bridge would be present. An underexposed part can rarely support the weight of the structure and will collapse. The underexposed part in Figure 11 collapsed under the weight of the bridge span. Correcting an underexposed part can be achieved by increasing the exposure time or the light flux. However, there is a tradeoff that must be considered. Increasing the exposure time can significantly increase the build time for very tall structures with a significant number of layers. Increasing light flux too much while decreasing the exposure time could lead to gel-like structures. Since thinner layers are generally desired, reducing the photoabsorber is not recommended.
4.4 Exposure Depth

A series of bridge structures were created to determine the polymerization depth at different Sudan 1 concentrations and exposure energy. The bridge structure was chosen because it allows multiple comparisons and checks. The center beams can be measured to check exposure depth and confirm light uniformity. In addition, the side overhangs present a profile for the polymerization. Figure 12 shows the effect of exposure time when light flux, photoinitiator concentration, and photoabsorber concentration are held constant. The results indicate that exposure time controls cure depth in a near linear fashion. A series of depth studies conducted in an ambient environment can be found in Figure 13. This study suggests that light flux can be used to make large modification in curing depth. Exposure time can then be used to tune the depth to the appropriate thickness. Exposure dose should not be used to estimate depth.
The data in Figure 13 clearly indicates that exposure dose is not an accurate predictor of cure depth.

The results presented in Figure 12 and 13 cannot be compared directly because they were taken at different times and under different light conditions. A different LED light source was used for the nitrogen study in Figure 12 and therefore the light uniformity and other characteristics between the two could be different.

![Figure 12: Polymerization depth for HDDA with 1% Sudan I in a nitrogen environment.](image)

<table>
<thead>
<tr>
<th>Time (ms)</th>
<th>Depth (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>12.88</td>
</tr>
<tr>
<td>200</td>
<td>14.52</td>
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<td>400</td>
<td>19.22</td>
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<tr>
<td>600</td>
<td>20.25</td>
</tr>
<tr>
<td>800</td>
<td>21.88</td>
</tr>
<tr>
<td>1000</td>
<td>23.11</td>
</tr>
</tbody>
</table>

Flux 173.15 mW/cm²

HDDA under Nitrogen
Figure 13: Depth study under ambient environment at constant photo absorber for three flux conditions. Column A, B, and C represent fluxes 15.89, 23.74, and 29.20 MW/cm² respectively.
The amount of photoabsorber limits the penetration depth of the light and therefore reduces the cure depth for a given photoinitiator concentration and light flux. Figure 14 demonstrates this trend. The bridge structure was fabricated for this experiment. Structures that were confirmed to have produced an incomplete bridge were given a depth of 0. A Sudan I concentration of .25% did not produce a successful bridge at any of the light fluxes given in Figure 14. This study was conducted under ambient conditions.

Figure 14: The effect of Sudan I on cure depth vs. exposure time and light flux under ambient conditions.

The shape of the depth profile is dependent on the concentration of photoabsorber. The structures in Figure 13 and Figure 8 have different profile shapes due to the amount of photoabsorber. The bridges in Figure 13 contained .08% Sudan I while the bridges in Figure 15 contained .5%.
4.5 Reproducibility

The ability to produce consistent results is critical in manufacturing. Hence, several bridge structures were produced to test the Reproducibility. Bridge depth and width measurements were taken on three bridges. The results are shown in Figure 15. The three bridge structures were uniform and consistent in their dimensions. Figure 15 demonstrates the process can produce components with consistent dimensional precision. The predicted bridge width, based on the number of pixels in the bitmap and the reduction optics, is 96 microns.

As previously mentioned, these parts were made before the recoat procedure was finalized and the bases are damaged in the center. This problem does not affect the measurements because the bridge structure is of lower surface area than the base. Therefore, it can be coated by the polymer.

<table>
<thead>
<tr>
<th>Structure</th>
<th>Depth (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Left</td>
<td>68.89</td>
</tr>
<tr>
<td>Center</td>
<td>68.89</td>
</tr>
<tr>
<td>Right</td>
<td>69.85</td>
</tr>
<tr>
<td>Flux 78.72 (mW/cm²)</td>
<td></td>
</tr>
<tr>
<td>HDDA under Ambient</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Structure</th>
<th>Width (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Left</td>
<td>82.29</td>
</tr>
<tr>
<td>Center</td>
<td>82.29</td>
</tr>
<tr>
<td>Right</td>
<td>83.25</td>
</tr>
<tr>
<td>Flux 78.72 (mW/cm²)</td>
<td></td>
</tr>
<tr>
<td>HDDA under Ambient</td>
<td></td>
</tr>
</tbody>
</table>

Figure 15: Reproducibility study of three bridge structures made under the same conditions. The material is HDDA with .5% Sudan I and 2% Irgacure 819.
4.6 Model Description and Comparison

In order to produce quality components using projection micro stereolithography (PμSL), it is useful to model the effects of the different process parameters in section 4.2. The researchers at Lawrence Livermore National Laboratory have created a process model that incorporates the chemical kinetics of photo polymerization, fluid dynamics, and the different optical effects present in the system described in Chapter 3. The model includes the effects of light attenuation, polymerization kinetics coupled with heat and mass transport phenomena, and diffusion. This model can help predict the importance of the different process parameters. Figure 16 is a sensitivity plot used to demonstrate the relative importance and effect of light intensity, exposure dose, photo initiator concentration, UV absorber concentration and quantum yield on cure depth. The quantum yield was not studied in the experimental results presented in this chapter since it is a material property that is difficult to measure and vary.

An analysis of the sensitivity plot provides a wealth of information regarding which parameters are important to investigate. The plot indicates that light intensity and UV absorber concentration have the greatest effect on exposure depth. An increase in light intensity will produce a deeper cure depth for a given exposure time. For a constant light flux and exposure time, the cure depth will decrease with an increase in UV absorber. With a constant exposure dose and UV absorber concentration, the cure depth marginally increases with an increase in photoinitiator. This suggests that limited gains will be achieved by using more than the 2% photoinitiator concentration in this study.

A depiction of the model and a fabricated component is presented in Figure 10. It describes a cantilever structure with the same characteristics as the fabricated structure on the left. This model was used to predict the thickness of a single layer. Experimental data were collected for comparison to the model. Figure 11 displays three bridge structures created using different exposure times. The depth of these structures were measured with the SEM and compared to the results predicted by the model. The model matches the SEM data within 25 percent. The experiments compared to the model were conducted in an ambient environment. The model does not account for the presence of oxygen. It would consequently predict a greater depth, since oxygen inhibits the polymerization.
Figure 16: Sensitivity plot of polymerization depth to various input parameters. Figure created by Todd Weisgraber, Lawrence Livermore National Laboratory.

Figure 17: Comparison of a PµSL fabricated part and process model results for model validation: a) side view of a fabricated bridge geometry which includes cantilevered structures and b) a cross-section of the process model showing the curing profile during formation of a cantilevered structure from the bridge. Figure created by Todd Weisgraber, Lawrence Livermore National Laboratory.
4.7 Conclusions

The results presented in this chapter indicate that the ΠμSL system presented in Chapter 3 can produce reproducible components in a controlled fashion. Data were presented that demonstrates, for the flux and time periods tested, that light flux and exposure time need to be considered independently as opposed to being thought of as one quantity, exposure dose. The model sensitivity predictions for Sudan I concentration and light flux were confirmed.

It is the author’s belief that long exposure times at a lower intensity will produce more consistent results as shown in Figure 8. Controlling the layer thickness by vary the Sudan I concentration may be the best option, rather than trying to control the light flux and exposure time. The images in Figure 8 were the most uniform parts produced and this may be due to the exposure conditions rather than the uniformity of light. A future study should be done to verify these conclusions.
CHAPTER 5: APPLICATIONS

5.1 Introduction

The applications of projection stereolithography cover a very broad range. A few examples are given in Figure 19. Structure A demonstrates a high aspect ratio component, demonstrating the three dimensional capabilities of this technique. Structure B shows a porous material. This technique provides a method to fabricate structures with a prescribed density profile. The remaining figures demonstrate a complex pattern as a proof of concept for a light scattering surface.

Figure 19: A) High aspect ratio components, B) variable density foams, C-E) complex patterns.

5.2 High Aspect Ratio and Arbitrary Three Dimensional Structures

Projection stereolithography allows the user to create arbitrary three-dimensional and potentially high aspect ratio structures. The highest aspect ratio attempted with this system was 6:1. The process is not limited in the z-direction. The only limitation that affects build height is machine components that can be moved or replaced. An example arbitrary structure is shown in Figure 20. This structure was fabricated using the parameters from the repeatability study. Without an accurate prediction of the penetration depth of the light, it is very difficult to create these types of structures. If the exposure conditions are too low, the rectangular structures will not be polymerized and will miss the pillar supports. If the exposure time and/or light flux are too high, it will create a solid structure. For these reasons, it was beneficial to use the depth study data to create a successful arbitrary structure.

Figure 20 was built before the recoating steps described in section 4.1 were fully understood. A hole can be observed in the large base that this structure was built on. This is due
to the polymer’s inability to flow over the previous layer in the time allotted. The recoating process ensures even large bases are coated correctly as the entire structure is lowered 1.5 mm into the polymer bath. Surfactants may reduce the surface tension and the necessary depth, but this concept was not explored.

Figure 20: 3D structure with features as small as 3 \( \mu \)m

The projection stereolithography technology is under review as a potential fabrication technique to create multimaterial components at the meso scale with nanometer features. A potential application for this technology is the fabrication of complex fuel capsule assemblies for Lawrence Livermore National Laboratory’s laser fusion campaign and efforts in laser fusion energy. Figure 21 is a demonstration hohlraum for these two systems. The actual hohlraum can be viewed in Figure 22. A hohlraum, German for “hollow space”, is a cylinder used to contain and concentrate laser energy. The requirement for this proof of concept was a simple 2:1 aspect ratio cylinder. This goal was achieved under both a nitrogen environment and ambient conditions.
Figure 21: Hohlraum proof of concept. The top and bottom figure is under ambient and nitrogen respectively.
5.3 Tailored Materials

Projection micro stereolithography allows the user to build a custom microstructure from scratch. Using the dynamic mask, a seemingly continuously variable structure can be created. Several porous structures have been created for demonstration. A sample of these structures is displayed in Figure 23. The top row shows two examples of a lattice pattern and the corresponding optical photographs. From left to right, the bottom row displays two different depth lattice structures, a triple layer lattice structure, and a lattice structure with cuboid cells. This technique could produce porous structures with tunable densities by varying the thickness of the lattice components, their packing density, or the depth. A structure could also be created where the density changes as a function of the x, y, or z direction.

The light uniformity during the fabrication of foam A in Figure 23 created a variable density structure by accident. It is clear in the photograph that the horizontal beam has a gradient
in the horizontal direction. This structure could have been intentionally fabricated by creating variations in the bitmap image. Light uniformity was a major problem during the creation of foams in general. The light in the center area of the system has a higher degree of uniformity than the edges. The foams were attempted over a larger area and were affected more by the non-uniformity.

The system cannot build certain types of porous structures. Part H in Figure 23 is one example of a failed low density material. The structure is physically fine, but because the fabrication completely encloses the cells, liquid resin is likely trapped inside. Parts created with this technique need to be designed with an open structure to ensure proper resin drainage after fabrication. This limitation is found in TPP, STL, PµSL, and similar techniques. For this reason, certain microstructures are not feasible with this technique. A component of continuously variable porosity may still be achievable, but care must be taken in designing it. The gray-scale fabrication technique may be required to fabricate this type of structure.

![Figure 23: Miscellaneous examples of porous structures.](image)
5.4 Limitations

The system currently uses a fixed optical system. A fixed setup limits the maximum build area and sets the resolution limit. To make larger components at the same resolution, a larger LCoS can be installed or X and Y stages can translate the substrate in a step and repeat fashion. Parts can be fused by slightly overlapping exposures at the same Z height as described by Xia² Furthermore, a variable magnification system could be used in conjunction with X and Y stages to create large components with very fine features in targeted areas. This type of system has not been tested.

The system is constrained to materials that can be polymerized by light. Metals, ceramics, and biological materials can be incorporated, but not in their native states. The addition of suspended particles to the polymer bath does not affect the ability of projection stereolithography to fabricate components. The build conditions will change since light below the surface may still be scattered, but fabrication in general is not impeded. However, this technique cannot be used to build fully metallic or ceramic parts. The system as described is limited to one material at a time.

The components created were fabricated without a support system. This imposes some geometric limitations. While the gray-scale technique can be used, it does have potential drawbacks. The etchants used to remove the lightly cross-linked polymer could be potentially harmful to biologically sensitive environments. It also adds a time sensitive post processing step.
CHAPTER 6: CONCLUSIONS

A PµSL system using a LED light source has been described, tested, and utilized to create useful components. The major hurdles faced when optimizing a system have been described and a method has been presented to tune a new system. The major parameters important in PµSL have been described and their effect on part creation examined. Data has been presented that suggests a common trend in the literature, reporting cure depth as a function of exposure dose, is incorrect. A brief discussion of each trend and recommendations follow.

The exposure time is fairly linearly associated with cure depth when all other parameters are held constant. The equations presented and experimental observations led to the recommendation that future investigators use longer exposure times. Higher concentrations of Sudan I and lower light flux should be used to reduce layer thickness. Investigators should hold the exposure time constant and first adjust light flux and Sudan I concentrations to reduce layer thickness. Time can be used to narrow in on the desired depth after a light flux and Sudan I concentration is finalized.

Exposure dose should not be used to predict cure depth. The data presented in Chapter 4 demonstrates that exposure dose is not directly related to cure depth for the ranges tested. Light flux and exposure time should be considered separately. This is supported by the sensitivity study where a conflict is observed between the light flux and the exposure dose.

Light flux is bound by threshold intensity necessary to cause polymerization for a given amount of photoabsorber and photoinitiator. When a black image is displayed, the maximum flux must be less than the threshold intensity. When a white image is displayed, the minimum flux must polymerize the resin. The first step an investigator should follow is to find these bounds and start at the low to mid-range.

Sudan I concentration is a very important factor in determining layer thickness and should be considered first. No issues were found by increasing the amount of Sudan I in solution. The amount of Sudan I is bound both by the amount soluble in the monomer and by the light flux of the system.

Creating an environment free of oxygen increases the amount of polymerization that can occur for a given set of conditions. A nitrogen environment is easy to create, but is wasteful of resources. A nitrogen environment is not suitable for living systems. For this reason, the experiments need to be considered before the environment is chosen.
In order to create three-dimension components and complex geometries, a set of cure depth studies should be undertaken for a new system. These studies contain valuable knowledge necessary to produce complex structures.
REFERENCES


APPENDIX: Projection Micro StereoLithography Operating Procedure

Materials:
- Gold plated silicon wafer diced into ~4mm x 15mm rectangles (substrate)
- Super glue
- Sand Paper – 500 grit
- HDDA
- Irgacure 819
- Sudan I
- Isopropanol and or Acetone

Procedure:

Begin by creating your desired monomer solution. Current solution as of 1/14/2010 is 1% Sudan 1, 2% Irgacure by weight in HDDA. Mix well. Place this solution under nitrogen for 8+ hrs before use. I keep the flow meter set at 5 LPM (Air reference).

Prep the substrate by first removing all polymer and super glue from previous build with a razor blade. Then gently dab the super glue on opposing sides of the substrate holder. Too much super glue will cause the substrate to break during removal and take a long time to dry. Then gently sand the substrate and place on the tool holder. This process should take less then 30 seconds so the glue does not dry. By letting the super glue air dry for ~30 seconds you will get a quick bond. Make sure to align the substrate so it is square with the holder before contacting the glue.

Once the substrate is firmly bonded to the holder, insert the holder into stage setup and gently tighten the screw. Swing the arm down and align the substrate with the objective. Tighten the set screw.

Load the Nano Fabricator program and open the System Initiation Subs folder. Run the “stage finder.txt.” file to ensure you are building on the stage. For long builds, move the x-stage to an appropriate start location so you can move along the substrate. Plan to move 3.5mm for each part. Swing the arm to past horizontal. Uncover the monomer container and place on the focusing stage. Swing the substrate down to level position. Use Z-axis manual stage to check
level by moving the substrate above and below the liquid. Then raise the substrate to just below the surface.

The “focus.txt” file will lower the substrate 2mm below the surface and shine a very faint image unto the liquid. Using the “Manually Move Any Stage” option in the Nano Fabricator, move the Focusing stage until the image is in focus. This normally doesn’t change more than 20 microns between builds. For new materials it is best to focus on the substrate first, place the liquid and move the focusing stage to match this, and then run the focus.txt program. When you are satisfied with the focus, run the breakfocus.txt profile from the same folder. This profile brings the substrate back up to build level, runs the dip profile, and then waits for the material to settle before displaying an image. Move the manual z stage up until the edge blur is able 3mm. The system is now setup to run a profile.

Profile Locations:
C:\Documents and Settings\fabuser1\Desktop\Profile Files Folder\Nano Fab

System Initiation Subs
Focus.txt
Breakfocus.txt
Substrate Finder.txt