GENERATION OF MICRO-SIZE CONDUCTIVE LINES USING THE DIRECT WRITE OF NANO-PARTICLES

by **Daniel John Ketchum**

A Thesis submitted to the Graduate Faculty of the University of Michigan in partial fulfillment of the requirements for the Degree of Master of Science

UNIVERSITY OF MICHIGAN DEPARTMENT OF MECHANICAL ENGINEERING

Ann Arbor

December 12, 2005

APPROVED BY:

ABSTRACT

The idea of this project is to generate micro-size conductive lines using the direct write of nano-particles and laser sintering processes. More specifically, the goal is to generate gold lines 8-12 µm wide, which can conduct as close to the bulk material as possible using direct write and a laser for sintering. Prior to the start of the project, research had been done on this process using silver nano-particles instead of gold. It was determined that silver would not be able to conduct enough electric current. However, the use of the silver nano-particles was useful in that it familiarized the direct write process and worked the kinks out of that machine.

The first half of the research on the gold nano-particles revolved around using direct write to generate a continuous line in the 8-12 µm wide range. It was decided that these thin lines were not very reproducible for many reasons including inconsistent solvent ratios and machine error. Experimental results show the horizontally thin lines need to be thick vertically to be able to meet the conductivity requirement.

The second half of the research was dedicated to using direct write to create wide, thick lines. Reproducibility is no longer a problem because line over 40 µm wide can be easily generated. These wide lines could then be sintered with a laser to the correct size. The excess gold is then washed away.

The laser sinter process works well on glass samples. However, on a polycarbonate substrate, the laser caused major damage to the surface of the substrate. After much investigation, it was found that the wavelength of the laser was too short and substrate

was absorbing a large amount of energy causing substrate damage. Working operating parameters were found, but they were not practical for operation. The only working laser operating parameters were at very low speeds, around 0.2 mm/s. Conclusions of this research were that micro-sized conductive lines could be generated, but the current Nd: YAG, solid state laser was not optimal for this process.

Resistance measurements were taken from a few on the generated micro-sized lines.

Although the measurements were very high, they did show that the lines were conductive and the process was a capable one. Future work will be conducted to bridge the gap between the current high resistance measurements and the near bulk conductivity the project statement requires. The fact that the process is capable of producing conductive lines is important and a good starting point determined from this project.

Conclusions to be Drawn from Work Conducted at University of Michigan

- The laser at GRC is not optimal for this process.
- Show how a conductive line, 8-12 µm wide can in fact be created
- Find current working operating parameters for direct write and laser

BIOGRAPHY

Daniel Ketchum was born in Pennsylvania in October of 1981. He has lived in New York, Florida, Indiana, Ireland, and Ohio. He has spent he last five and a half years at the University of Michigan. At Michigan he completed his undergraduate degree in Mechanical Engineering while being a part of the Michigan Varsity Swim Team. During his 4.5 years of undergrad he had one internship with Logik Research and Engineering. He swimming accolades include Olympic Gold Medal, Varsity Team Captain, and National Champion. He is now wrapping up his Master of Science Degree in Mechanical Engineering under the guidance of Albert Shih. In January he will begin to work with General Electric, the company he did most of his research for this report with.

ACKNOWLEDGEMENTS

First and foremost I would like to thank Dr. Albert Shih. If he hadn't convinced to stay on for my masters and supported me along the way, I probably would have never received such an amazing job, and reached my full academic potential. I need to thank Marshall Jones for his help with all my research, as well as his help in actually finding interesting research for me to do. A special thanks to Steve Rutkowski and all his help. He did his best to show me how things really are. Thank you Carl Erikson for your patience and help with all my laser problems. Finally thank you to everybody else who supported me through this last year, especially Lori, my Mom and Dad, General Electric, and all my friends and colleges at the University of Michigan.

Table of Contents

| List of | Figures and Tables | vii |
|----------------|----------------------------------|-----|
| 1 | INTRODUCTION | 1 |
| 1.1 | The Use of Nano-Ink | 1 |
| 1.2 | The Direct Write Process | 2 |
| 1.3 | The Laser Sintering Process | 4 |
| 1.4 | The Overall Process and Goals | 6 |
| 2 | EXPERIMENTAL TEST SETUP | 8 |
| 2.1 | Direct Write Lines | |
| 2.2 | Laser Sintering | |
| 2.3 | Testing Procedures | |
| 2.3.1 | Visual Testing Procedures | |
| 2.3.2 | Solvent Testing Procedures | |
| 2.3.3 | Resistance Measurements | |
| 2.3.4 | Stress Craze Test | 18 |
| 3 | TYPES OF PROBLEMS | 20 |
| 3.1 | Gas Ratio | 20 |
| 3.2 | Solvent Evaporation | |
| 3.3 | Changing Parameters | |
| 3.4 | Laser Set-Up Problems | |
| 3.5 | Examples of Poor Line Quality | |
| 3.5.1 | Overspray | |
| 3.5.2 | Too Dry or Too Wet | 27 |
| 3.5.3 | Excessively Large Lines | |
| 3.5.4 | Droplets | 29 |
| 4 | RESULTS AND DISCUSSIONS | 31 |
| 4.1 | Line Shape Results | 31 |
| 4.1.1 | Line Thickness | 31 |
| 4.1.2 | Adjusting Table Speed | 34 |
| 4.1.3 | Multiple Passes | |
| 4.1.4 | Solvent Ratio | |
| 4.1.5 | Optimal Direct Write Parameters | 40 |
| 4.2 | Laser Sintering Results | 42 |
| 4.2.1 | Laser Sintering on Glass | 11 |
| 4.2.2 | Laser Sintering on Polycarbonate | 46 |
| 4.2.3 | Explanation for Laser Results | 52 |
| 4.3 | Resistance Measurements | 55 |
| 5 | | 57 |
| 5.1 | Conclusions | |
| 5.2 | | |
| | RENCES | 60 |
| APPE | NDIX | 61 |
| Appen | dix A | 61 |
| Appen | dix B | 67 |
| Appen Appen | dix C | |
| Abben | uix D | 68 |

| Appendix E | 68 |
|------------|----|
| Appendix F | |
| Appendix G | 69 |
| Appendix H | 70 |
| Appendix I | |
| Appendix J | |
| Appendix K | |
| Appendix L | |
| Appendix M | |
| Appendix N | |
| Appendix O | 73 |

List of Figures and Tables

| Figure 1 | Atomic Force Microscope (AFM) image of clusters of 20 nm gold nano-particle | es 1 |
|-----------|--|-------|
| Figure 2 | Close-up view of the nozzle with spray coming out. | 2 |
| Figure 3 | View of ultrasonic bath, notice the test tube filled with ink and the attached tube | e 3 |
| Figure 4 | Potomac set-up | 4 |
| Figure 5 | set-up used in this specific process | |
| Figure 6 | The ideal effects of laser sintering, a line width reduction to about 1/3 the origin | ıal 6 |
| Figure 7 | Gold being deposited on a 2 x 3inch glass substrate via direct write | 9 |
| Figure 8 | Laser focused by simply moving the laser up and down from the substrate | 12 |
| Figure 9 | Optical microscopy of a line at 240x | 14 |
| Figure 10 | Optical microscopy of lines at 50x. | |
| Figure 11 | SEM picture at 15000x | |
| Figure 12 | An example of the line profile measured using an AFM | 15 |
| Figure 13 | Results from the optical profilometer_ | 16 |
| Figure 14 | TGA test results | 17 |
| Figure 15 | The Agilent 4155 for resistance measurements | 18 |
| Figure 16 | Actual stress craze setup inside an oven | 19 |
| Figure 17 | The load set up for the stress craze test | 19 |
| Figure 18 | A line run with the same parameters can change drastically. | _21 |
| Figure 19 | Suddenly droplets start forming | 22 |
| Figure 20 | Clumping of particles due to high solids content | _23 |
| Figure 21 | The effect of excessive amount of overspray | 27 |
| Figure 22 | Dry lines | 28 |
| Figure 23 | Dry lines | 28 |
| Figure 24 | Wet and excessively large line | 38 |
| Figure 25 | Excessive large lines | . 29 |
| Figure 26 | Close up picture of a droplet | 30 |
| Figure 27 | Ideal curve of line width and thickness | |
| Figure 28 | Actual experiment data of line width and thickness | |
| Figure 29 | Adjusted data point | .32 |
| Figure 30 | Peak thickness and average thickness | 33 |
| Figure 31 | Lines written at 5mm/s speed | 35 |
| Figure 32 | Lines written at 25mm/s speed | 35 |
| Figure 33 | Lines written at 50mm/s speed | 35 |
| Figure 34 | The effect of adding an extra pass | _36 |
| Figure 35 | The logarithmic function of number of passes vs. line thickness | 37 |
| Figure 36 | The solvent ratio compared with the line peak thickness | 39 |
| Figure 37 | The solvent ratio compared with the line average thickness | |
| Figure 38 | Profile of a non-sintered line | |
| Figure 39 | Profile of a sintered line | 43 |
| Figure 40 | Sintered Results | |
| Figure 41 | Burnt | |
| Figure 42 | Burnt | |
| Figure 43 | Mild Burn_ | 48 |
| Figure 44 | Slight Raise | 48 |

| Figure 45 Figure 46 Figure 47 Figure 48 Figure 49 | Slight Raise Color Change Color Change Insulators and Steels are both very unstable Absorption depth of gold nanoparticles | 49 49 54 |
|---|--|----------------|
| Table 1 | The increase in solvent ratio over time | 23 |
| | The increase in solvent ratio over time | |
| Table 2 | The effect of table speed on line thickness. | |
| Table 3 | Optimal direct write parameters | 41 |
| Table 4 | Details of the progressive line change | |
| Table 5 | The laser parameter at low laser powers | |
| Table 6 | The laser parameter results at high laser powers | 50 |
| Table 7 | Complete laser parameter results | |
| Table 8 | The resistance measurements | |
| | | |

1 INTRODUCTION

1.1 The Use of Nano-Ink

Gold nano-particles are starting to be experimented with all over the world in various research facilities. This is due to their high potential of impact on an ever increasing nano-world. Gold nano-particles have two very attractive features. They are very small (7-20 nm, as shown in Figure 1) and can be manipulated into many small nano or micro sized shapes, and they are electrically conductive and are a perfect fit for many micro sized electrical structures [1]. Gold nano-particles have already found their way into the medical device and computer chip markets.

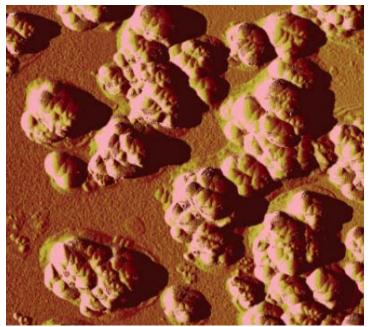


Figure 1: Atomic Force Microscope (AFM) image of clusters of 20 nm gold nano-particles

The nano-particles used for this project came in an ink form. The particles where mixed in an AF-7 solution and provided in 5 ml vials. The AF-7 chemical was an unknown chemical from a Japanese distributor. Originally the AF-7/gold nano-particle solution

was mixed with hexane or heptane to further dilute, however after some time the solution was diluted with more AF-7. The dilution levels of the nano-particles play a large role in how well they flow in the direct write process.

1.2 The Direct Write Process

Direct Write is a process that involves spraying metallic inks on to a substrate (see Figure 2) [2]. The process starts with an ink composed of metallic nano-particles. This ink is placed inside a large test tube that is connected to a tube with a diameter of about 0.25 inches as shown in Figure 3. The test tube is then placed in side a water bath attach to an ultrasonic transducer. The ultrasonic bath turns the liquid ink into a vapor which then flows through the attached tube. The metallic vapor then encounters a pressurized carrier gas. NO₂ was used as the carrier gas. The carrier gas forms a spiral as it moves through the attached tubes and into a nozzle. The nozzle size can vary but the carrier gas is capable of forming a spiral so tight that it can force the metallic vapor out at a size 1/20th that of the nozzle diameter. This process results in a spray of metallic nano-particles, similar to that of spray paint, onto a substrate a few millimeters below the nozzle creating micro-sized metallic designs. A common use of direct write is to spray thermocouples onto auto or aircraft test specimens.

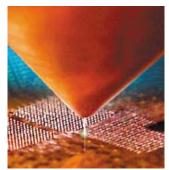


Figure 2: Close-up view of the nozzle with spray coming out.



Figure 3: View of ultrasonic bath, notice the test tube filled with ink and the attached tube

The specific direct write set-up that was used in for this specific process was Optomec's Maskless MesoscaleTM Materials Deposition (M3D) Technology [2]. M3D is revolutionary because previous direct write technology had been created for relatively large electric features in the order of 100 μm and above (such as thermocouples). There had also been technologies that use thin films to create electronic devices smaller than 1 μm. There had not, however, been any device created capable of producing metallic devices that can conduct electricity in the 1 to 100 μm range. Optomec filled that market gap with their M3D direct write technology. Their marketing website describes it as a moderately priced device capable of operating at low temperatures on a verity of inked materials and substrates. It is ideal for micro features about 25 μm.

The set-up used was slightly different from Optomec's set-up. The differences can be clearly seen in Figures 4 and 5 below. Since most of the equipment used in Optomec's version was already available, only the programmed part, the enclosed wires and tubes, and the ultrasonic bath were needed from of their M3D direct write. The programmed,

computer part of the M3D was connected to all the various wires and tubes and then mounted onto a CNC controlled table of an old milling machine. The M3D computer was than hooked up to an existing computer, in which all the parameters were able to be controlled and monitored from.

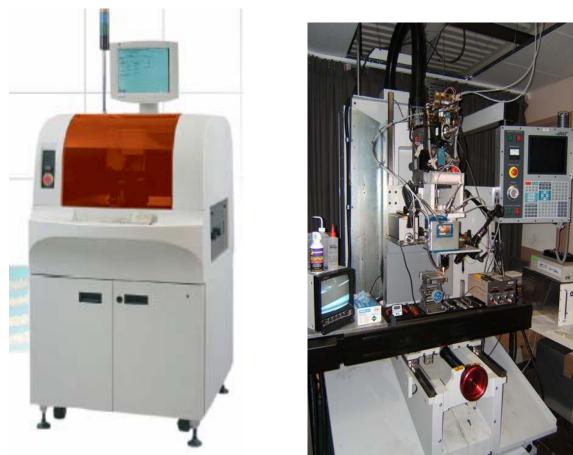
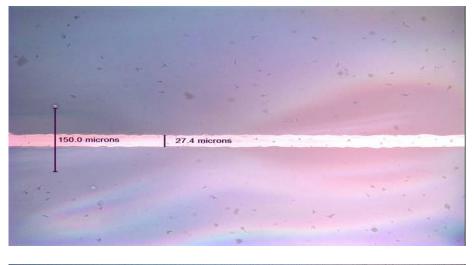


Figure 4 and Figure 5: On the left is the Optomec M3D Potomac set-up, on the right is the set-up used in this specific process [2].

1.3 The Laser Sintering Process

There were two parts to this project. The first was to direct write micro sized lines on to various substrates. The second part of the project was to laser sinter the lines with the hopes of reducing their size and improving their physical characteristics. For laser sintering a UV Diode pumped frequency-tripled Nd: YAG laser (considered a solid state

laser) made by Spectra Physics, that operates at 80 mHz repetition rate [3]. It had a wavelength of about 355 nm and had an operating power in the range of 8-360 mW. However, due to the optical path only a max power of about 260 mW is attainable. This laser was mounted on an Anorad 3 axis gantry, capable of moving in the range of 0.01 to 24 mm/s and beyond [4]. Sintering is a process that involves heating the sprayed nanoparticles causing them to condense together while evaporating the remaining solvent that was combined with the original nano-particles to form an ink. This caused the observed lines to condense forming lines up to 1/3 their original size. Other adding physical changes to the lines are an increased conductivity of 3-10x the original line and particles that have actually combined enough with the substrate that a tape test could be passed. The conductivity increases based on the fact that with particles condensed and less solvent remaining, there will be fewer gaps in the lines and electricity will be able to flow more freely between them. The stronger bond between the substrate and metallic particles stems from the curing that occurs in the moments after sintering [5]. Sintering can be done by simply placing a sample in an oven and letting the heat of the oven cause the line geometry to change. However, in an oven the whole substrate is affected by the heat and often unwanted damage was caused. Laser sintering focuses the heat onto a specific place with a diameter as small as 12 µm. Both processes were used, sometimes separately and sometimes together. Figure 6 below shows the affect of an oven treated line. Notice the sintered line is about 1/3 the size of the original non-sintered line.



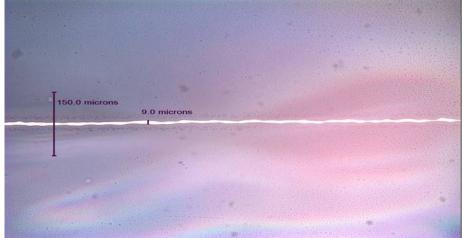


Figure 6: The ideal effects of laser sintering, a line width reduction to about 1/3 the original

1.4 The Overall Process and Goals

The idea of this project is to generate micro-size conductive lines 8-12 μm wide, which can conduct as close to the bulk material as possible using direct write and a laser for sintering. Direct write is ideal for producing lines about 25 μm wide but has the potential to produce lines as small as 6 μm . Laser sintering has the potential to reduce line width down to a third of its pre-sintered form. Combined these two technologies clearly have the potential to produce lines in the in the 8-12 μm range.

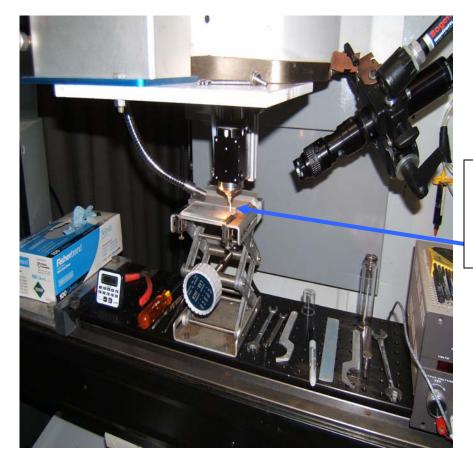
Gold and silver nano-inks were chosen for this project because of ability to conduct electricity. Silver was scratched early in the project in favor of gold nano-particles. The gold lines will allow a maximum amount of electricity to flow through them. Getting close to the bulk conductivity of gold is about as high of conductivity one could get from these lines. An Agilent 4155 was used to take resistance measurements of these lines [6]. The second goal of this project will ideally be met through the use of gold nano-particles.

2 EXPERIMENTAL TEST SETUP

Generating the thin metal lines using direct write, laser sintering methods and the test procedures of the thin line are discussed in this chapter.

2.1 Direct Write Lines

The micro-sized gold lines can be generated using Optomec's Maskless Mesoscale™ Materials Deposition (M3D) Technology, as shown in Figure 7. The lines were written onto various substrates. These substrates included 2 x 3 inch glass lab slides, 1 x 7 inch polycarbonate samples, and 100 mm diameter quartz slides. The substrates were placed 4 mm below the nozzle of the M3D direct write. The nozzle tip size was 100 µm. The substrates moved on a CNC controlled old milling table. For these experiments the table speed was adjusted between 3 to 50 mm/s, although most lines were written at a standard speed of 5 mm/s. The sheath gas pressure was varied between 30-45 cc/min, while the carrier or atomizer (ATM) gas pressure was between 6-20 cc/min. The gas pressure was sometimes changed in the middle of running a test. This was done if the lines were starting to change in shape or width. Although for the most part, the parameter settings remained the same for all the lines on a single substrate. The goal of most experiments was to create the same line over and over and the substrate translated back and forth. Line consistency was important. The amount of lines per substrate ranged from about 10 up to 100. The lines were placed 500 µm apart. The process of writing all these lines on a single slide could take up to 1 hour. This is because it was always done manually.



Glass substrate with gold ink being deposited from the above nozzle

Figure 7: Gold being deposited on a 2 x 3 inch glass substrate via direct write (note the camera and the light in the setup).

It was done manually so adjustments could be made if the line shape or width started to change. Also, the direct write operator could note any changes in atomization of the gold nano-ink, or other details that may affect flow. Some details that were found to affect flow were tube clots, nozzle clots, and sudden pressure changes in gold ink flow. If a clot formed, the test was stopped and all the tubes and nozzles were thoroughly cleaned with hexane in an ultrasonic cleaner. This could take up to several hours and was a problem if more than one full slide was attempted in a day. Clotting would normally occur by the second slide.

Most direct write tests were run at different settings. The settings that were changed were as follows:

- Nano-ink Solvent
- Substrate
- Stand off Distance
- Time Machined
- Machine Speed

- Sheath Gas Pressure
- ATM. Gas Pressure
- Ratio Sheath/ATM
- Tip Size

The observations taken and measurements made after direct writing lines were as follows:

- Line Quality
- Line Width
- Line Thickness

- Line Variation
- Run Date

Line quality is determined by the line shape and whether it is wet or dry looking, straight or wavy, or has any clumps. Any other notable features about the appearance or shape of a line were noted in this category. In observing line variation, notes were made on whether a line changes quality, width, or thickness on a single substrate. Results of these observations were put into a spreadsheet which can be viewed in Appendix A.

2.2 Laser Sintering

The laser sintering was done on an Nd: YAG solid state laser with a maximum power output of about 260 mW and a wavelength of about 355 nm. The laser was mounted on an Anorad 3 axis gantry with a Keyence distance sensor and camera attached and in line with the laser [7]. The camera was in line with the laser not only to be able to follow the laser via video monitor, but it was also used to zoom in on a single line. The cameras zoom was how the laser was focused on such a small line. This was a technical marvel how the camera was set up. It was set up in such a way that its focus was through the same lens that the laser was focused through. Therefore, when 1 was focused, so was the other. The laser had a separate, very weak red laser that was attached to a sensor that could be used to focus the laser to within 1 μ m. This meant that the camera could as well be focused to within 1 μ m. This setup was very unique to this specific laser.

The parameters monitored in this phase of the project were as follows:

- Laser Power
- Beam Width

• Laser Traverse Speed

The results observed and noted were the following:

- Substrate Damage
- Amount of Substrate Damage
- Line Width Reduction

- Observed Beam Width
- Line Quality Change
- Line Thickness Change

The laser power and traverse speed were the most often changed parameters. The laser power was controlled by placing the laser over a power measuring device made by Molectron Labmaster and changing the laser power via a computer. The laser power was controlled on a computer which uses LabView as the interface to the Acusto Optic Modulator. The traverse speed was controlled by the same computer program. The beam width was changed by adjusting the focus of the laser (See Figure 8) [8]. However, we could only estimate the beam width with the equipment in the lab and only through the use of a high power microscope could an actual micron (µm) measurement of the beam width be made. Some tests were done on just a substrate sample with no micro-sized gold lines on them. This was done to access substrate damage. The same tests were obviously run with gold lines on the substrate.

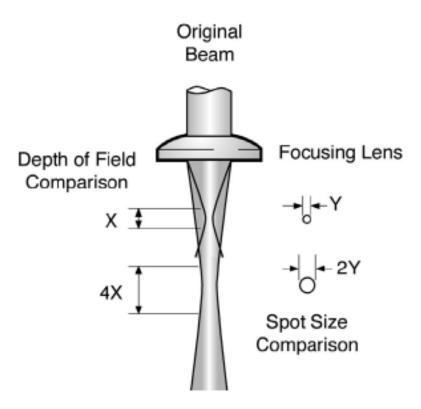


Figure 8: Laser focused by simply moving the laser up and down from the substrate. The distance between the laser and the focusing lens does not change, it only matters what the spot size is when the laser hits the substrate.

2.3 Testing Procedures

The testing procedures for the lines before and after laser sintering were fairly similar. They mostly involved using various microscopes, profilometers, and other methods of visual observation. A few tests were done to measure the nano-fluid, its solvent, and the effects of its solvent. After visual tests were performed some resistance measurements were taken of the lines as well as a stress craze test. This section will detail all those testing procedures.

2.3.1 Visual Testing Procedures

To measure line width and shape several different types of microscopes were used. For most purposes, different high powered microscopes were used at a magnification of 240x (see Figure 9). This microscope was adjusted between 100x and 500x for a few other observations. This microscope was attached to a camera so pictures were able to be taken while real time observations were being recorded. Several pictures of silver lines and early gold lines were taken on a Motic K-series 50x microscope (see Figure 10). To try and analyze the alignment of nano-particles in a given line, a Scanning Electron Microscope (SEM) was used (see Figure 11). In an attempt to determine the actual profile (within an error of 0.5 nm) of a line, an Atomic Force Microscope (AFM) was used (see Figure 12). All four of the listed microscopes were linked to a camera and able to be calibrated so measurements could be placed on a picture (see Figures 9-12).

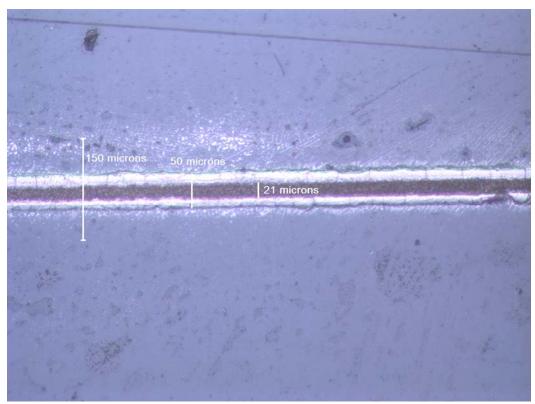


Figure 9: Optical microscopy of a line at 240x.

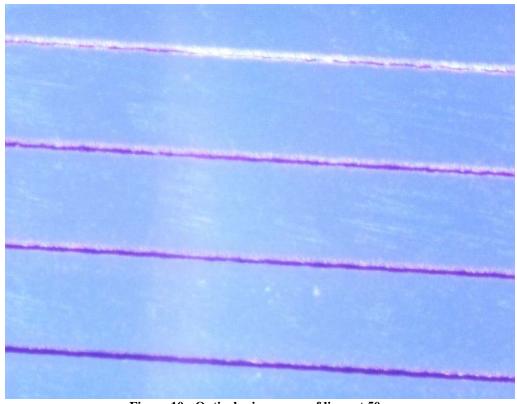


Figure 10: Optical microscopy of lines at 50x.



Figure 11: SEM picture at 15000x.

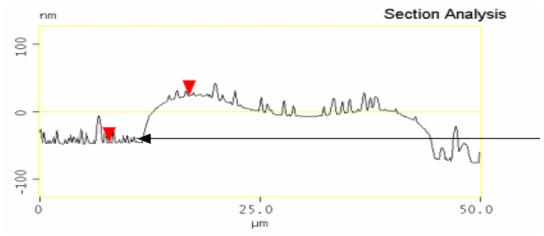


Figure 12: An example of the line profile measured using an AFM.

Another visual method of testing was the use of a profilometer. For the most part an optical, non destructive ADE Phase Shift profilometer was used [9]. However, as a method to confirm the optical profilometer's results there were a few measurements taken on a needle profilometer. The profilometer was used to take measurements on the line thickness. However, this was also a tool capable of producing width, shape, and profile measurements. An example of these results can be seen in Figure 13.

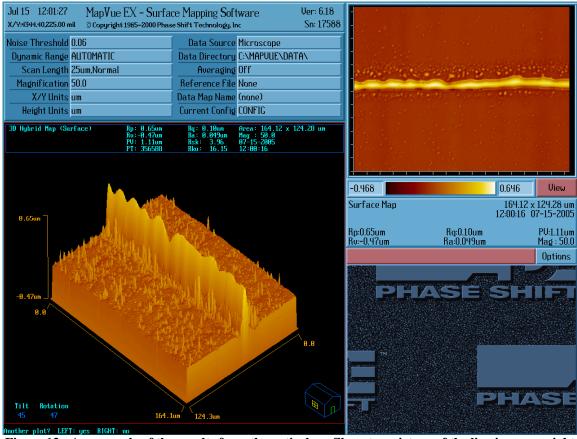


Figure 13: An example of the results from the optical profilometer, picture of the line in upper right and line profile in bottom left.

2.3.2 Solvent Testing Procedures

After several weeks on the project and slowly observing an overall change in line shape, it was thought that the solvent ratio of gold to AF-7 to Hexane was somehow changing. To test this we compared an unopened bottle of gold nano-particles with one that had been opened for several weeks. We wanted to test the solids content to prove the shelf life of an opened bottle of gold. To do this we used a thermo gravimetric analysis (TGA) test. The process involved slowly evaporating the solvent until there was only solid left in the sample. This process was monitored and plotted on a graph of % weight vs. time as shown in Figure 14.

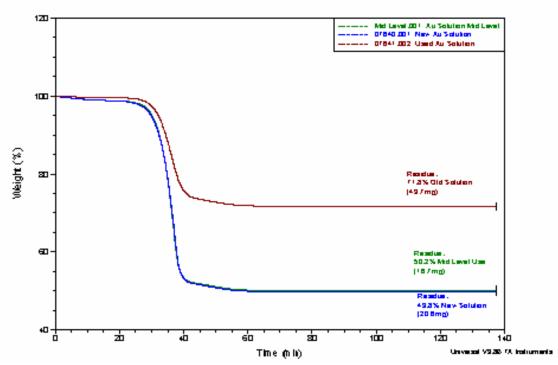


Figure 14: TGA test results.

2.3.3 Resistance Measurements

The resistance measurements were taken using an Agilent 4155 Semiconductor

Parameter Analyzer. The Agilent 4155 used two very small needles, in the order of 50 to

200 nm, to measure the current through one of the micro sized lines. Current was sent
through of the needles, while a computer program used sensors to calculate and convert
current into a resistance measurement. A picture of the Agilent 4155 can be seen below
in Figure 15. Resistance measurements were taken on lines that had been created through
any number of different parameters, but also on lines that had been pre-heated in a
furnace or by a laser, or by both.



Figure 15: The Agilent 4155 for resistance measurements [6].

2.3.4 Stress Craze Test

The stress craze test was not very pertinent to this specific research; but, if the stress craze test was failed, it would have meant a different material would be needed for the specific research and for that reason it is mentioned. The stress craze test simply tested the effect of the gold and its solvents on the particular polycarbonate used in this project. If the polycarbonate was affected in such a way that it could no longer pass this test then a new solvent would be needed. The setup for this test was very simple and is illustrated in Figures 16 and 17 below. The jig was mounted in a forced hot air oven maintained at 180 °F (82.2 °C). The polycarbonate samples used for the test were 6 x 1 x 0.25 inches and one bar had seen exposure to hexane and AF-7 solvent via direct write gold lines, while the second was a control bar. The lines were laid down in a checkered pattern that is shown in Appendix B.



Figure 16: Actual stress craze setup inside an oven. This test was run for 48 hours.

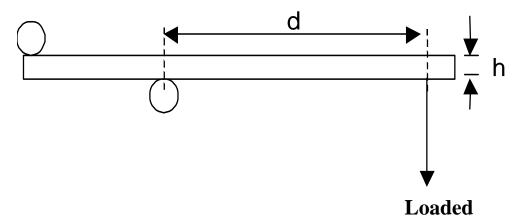


Figure 17: The load set up for the stress craze test.

3 TYPES OF PROBLEMS

Technical problems in generating the thin lines are summarized in this chapter. The problems are discussed and solutions are briefly explained. The problems range from problems with the initial ink, to difficulty with the set-up, to struggles with the direct write and laser themselves.

3.1 Gas Ratio

The sheath to carrier, (ATM) gas ratio was the cause of many problems in this project. The pressures of these two gases were the most difficult parameters to control as well as the most important towards the quality of line produced. All the other parameters, solvent, substrate, tip size, stand off distance, machine speed, machine time, and ultrasonic power are very easily measured and defined. However, if one small clog gets in the way or the nano-particles flow a little different for some reason, the gas pressure readings can become off. To understand this better, a new pressure monitor was placed at the end of the tip and it became clear that without changing the gas pressures the pressure of the flow was changing. This meant that at the same gas ratio, a very different line could be produced from one trail to the next (see Figure 18). The process was nearly impossible to reproduce.

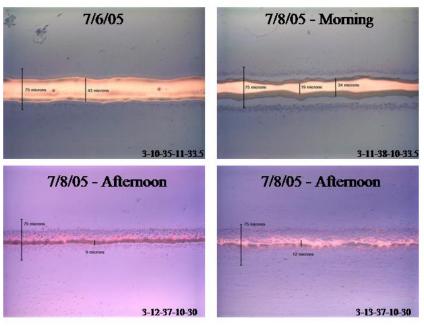


Figure 18: A line run with the same parameters can change drastically.

Some of the causes for the difficulty in reproducing tests came from dirty tubes that caused changes in pressure. The dirty tubes were the results of particle build up instead the tubes or previously clumped particles being to big to flow properly. To help minimize this, the direct write was taken apart everyday and thoroughly cleaned, that included all tubes, tips, and test tubes used. However, when dealing with such small lines, the smallest change in pressure could cause noticeable changes. This is why lines often varied through a single sample. This is also why somebody was constantly monitoring the machine while in use, to make small changes in gas pressure in an attempt to generate consistent lines. Changes in the gas pressure could happen very rapidly as shown in Figure 19. The change would only be seen by the pressure gauge on the nozzle tip, not in the monitors controlling the sheath and carrier gases. For this reason, the gas ratio was a major problem in line consistency.



Figure 19: Notice the top line is straight, and then suddenly droplets start forming due to a clog in a tube causing the build up and sudden release of powder.

3.2 Solvent Evaporation

The solvent in this project was constantly changing and very difficult to keep constant. Already mentioned was the AF-7 solvent the gold nano-particles came in. Originally, hexane was added to the gold and AF-7 to make the jump from liquid to gas easier. Heptane was used to replace hexane for a while. It was found to be no better and possible worse than hexane. Finally, some excess AF-7 was obtained and the original gold solution was mixed with more AF-7. There was also a completely different solvent added to the silver solution that was used briefly.

With all the changes in solvents, it took a while to notice a problem. It became clear over a matter of weeks that more and more hexane was being added to the gold/AF-7 solution to produce the same line (see Table 1). Eventually, it did not matter how much hexane

was added, the line appeared clumpy as in Figure 20. It was then evident that there was a problem with solvent evaporation. To prove this a thermo gravimetric analysis (TGA) test was preformed. To see the results refer back to Figure 14. The results showed that an unopened gold/AF-7 solution contained 49.8% solids, a container opened for 1.5 weeks had 50.2% solids content, and a container opened for 5 weeks had a solids content of about 71.8%. The recommended solids content for direct write use was between 49-54%. After this it was determined that this solution had a shelf life somewhere between 2-5 weeks. This is because some of the solution would evaporate with the slightest break in the container seal. As the AF-7 evaporated the solids became more prevalent in solution. An example comparing an original valve of gold/AF-7 solution with a four week old one can be scene in Appendix C.

Table 1: The increase in solvent ratio over time

| | Solvent |
|-----------|---------|-----------|---------|-----------|---------|-----------|---------|-----------|---------|
| Date | Ratio |
| 5/9/2005 | 5.0:1 | 5/20/2005 | 30.0:1 | 6/26/2005 | 25.0:1 | 6/8/2005 | 30.0:1 | 6/27/2005 | 17.5 |
| 5/10/2005 | 7.5:1 | 5/23/2005 | 25.0:1 | 6/26/2005 | 25.0:1 | 6/8/2005 | 30.0:1 | 7/5/2005 | 30.0:1 |
| 5/12/2005 | 12.5:1 | 5/23/2005 | 25.0:1 | 6/26/2005 | 25.0:1 | 6/9/2005 | 27.0:1 | 7/5/2005 | 17.5:1 |
| 5/12/2005 | 12.5:1 | 5/25/2005 | 22.0:1 | 6/8/2005 | 30.0:1 | 6/9/2005 | 27.0:1 | 7/6/2005 | 40.0:1 |
| 5/16/2005 | 17.5:1 | 6/25/2005 | 22.0:1 | 6/8/2005 | 30.0:1 | 6/11/2005 | 10.0:1 | 7/6/2005 | 40.0:1 |



Figure 20: Clumping of particles due to high solids content.

This drastic increase in solids content had an effect on the initial atomization of the nanoparticles as well as an effect of the line quality. Initial atomization became difficult because as the solids became more prevalent in solution they collided with each other eventually causing clumps of particles to form in solution. These clumps were too heavy to flow with the atomized gas and would instead stay in the test tube. After using a solution with high solids content, the test tube would look dirty after atomization due to clumped particles staying behind in the test tube. In an attempt to minimize this problem, more AF-7 was obtained to hopefully extend shelf life and original containers were never used longer than 3 weeks. Solvent evaporation proved to be a solvable problem by controlling its use in this manor.

3.3 Changing Parameters

There were a few parameters that have not been previously listed that were able to be changed. Some of these parameters were controllable, while others were not. A controlled parameter was the cleanliness of the substrate. Some substrates came cleaner than others. The quartz substrates for example had already been thoroughly cleaned, while the glass slides often had visible smudges on them. The polycarbonate substrates were somewhere in between. The questions that came from this were should we reclean all the substrates in the same way to create a controlled environment, and is it worth it to do that? A controlled clean substrate was tried, but eventually scraped do to what appeared to be minimal effect. The lines were so inconsistent as it was; it was very difficult to tell what effect, if any, a clean substrate had on line quality.

There were two uncontrollable parameters that caused problems for this project. The first being the use of silver and gold and the second being the protective coating on the polycarbonate. Silver was originally thought best for this projects overall purpose. It was then found otherwise and the silver nano-particles were replaced in favor of gold. The silver research proved helpful in working out some of the kinks in the direct write. However, a lot of time was spent working with silver that could have been spent on gold, and gold turned out to be much more difficult to work with. The protective coating on the polycarbonate was also uncontrollable. It changed four times in a matter of three months. Each time the coating would react slightly different with the written gold lines. It also led to problems when trying to do the craze test, and understanding how the hexane and AF-7 would react with the polycarbonate and its coating. This problem was so far out of control that it became more of an annoyance than a real problem. Besides, it had minimal effect of the quality of the lines.

3.4 Laser Set-Up Problems

Most of the overall laser problems will be discussed as results and findings; however, there were some problems the occurred with the set-up. The biggest problem that occurred during set-up was a water leak above the laser. This leak did minimal damage to the laser but did cause problems with the camera that was crucial to laser use. The camera had to be replaced but there were some other problems that came along with the camera and laser alignment.

As already stated the laser and camera were aligned in such a way that the camera would focus the same as the laser. That way one could use the camera's picture to determine

laser focus. This became a problem when trying to focus on such a small line. The first problem was actually finding a line. The camera feed was black and white and a line looked like a scratch on the table below. A line was very difficult to find without a strong light aimed to reflect off the gold micro lines in such a way that they were distinguishable from any other scratch or nearby line. The other problem came from our clean substrates. All three glass, quartz, and polycarbonate were clear. This made it hard to determine if the focus was on the front or the back of the clear substrate. When the camera appeared to be in focus, it was often in focus with the surface below the substrate. If not careful, the laser would focus on the surface below the substrate which was anywhere from 1 to 65 mm below the top of the substrate and where the lines were located. This caused the beam width to be off, as shown in Figure 8. Further problems with the laser wavelength and power will be discussed in results and conclusions.

3.5 Examples of Poor Line Quality

A good quality line has already been described as a line that is $8-12~\mu m$ wide, and as close to bulk resistance as possible. To have a line be the least resistive it was determined that it should be as thick as possible. Also, in the best case scenario the line would be very straight. This section will show examples of poor quality lines and explain why they are so.

3.5.1 Overspray

In Figure 21 the lines clearly have too much overspray. Overspray is when a dense intended line is formed and due to high pressure out of the nozzle the spray almost bounces off the substrate and forms overspray. It is an unintended extension of the line

width. The overspray is not very dense in gold particles and is therefore not very conductive. Overspray is almost always present when using direct write, however when it exceeds 50% of the actual dense line width than the line is considered to have too much overspray.

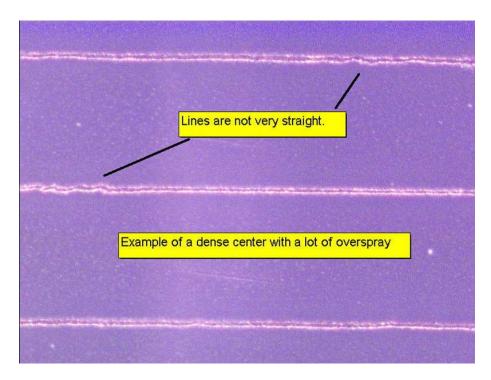


Figure 21: The effect of excessive amount of overspray (note the dense center and all the spray around it).

3.5.2 Too Dry or Too Wet

The lines in Figures 22 and 23 are far too dry to be adequate. If a line is dry looking like this, it means there was not enough solvent used, and the lines in turn look like spots of gold instead of a nice flowing line. A dry line will not conduct very well, if at all, because it is not continuous. The current will not be able to go this line. The other side of this is a line that is too wet. A wet line is caused by doing the opposite and having a very high solvent ratio. When there is an excess of solvent, the gold comes out of the

nozzle and then will spread out as a running liquid, instead of forming a straight equally consistent line. An example of a wet line is shown in Figure 24 and another of a wet and dry line together can be scene in Appendix D. The same effect happens when using spray paint. If you move the spray paint can too fast, or there is little left it won't make a solid painted line. The opposite is true if you hold it in one spot for a while or if it is not shaken up, the spray paint will run.



Figures 22 and 23: Dry lines: overview (left) and close-up view (right).

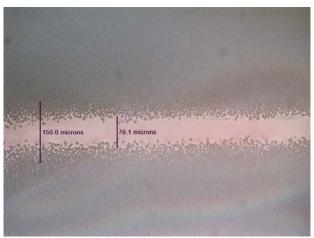


Figure 24: Close-up view of a wet and excessively large line (notice the little fingers that run off the main line).

3.5.3 Excessively Large Lines

Excessively large lines are a problem for obvious reasons since one of the goals in the project statement was to have lines that are 8-12 µm. It was found along the way that with the use of the laser creating lines that were 25-40 µm wide was not necessary a bad thing, however lines any bigger than that were of little use. Figure 24 above shows a good example of an excessively large line that is 76.1 mm wide. Figure 25 below shows another example. Large lines often came with pretty good quality, but because of there size, they were unacceptable for this project.

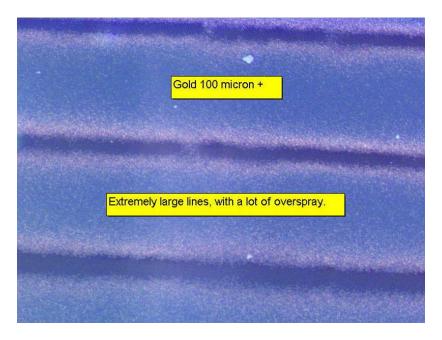


Figure 25: Excessive large lines (over 100 µm wide).

3.5.4 Droplets

Droplets have already been described earlier, and a picture of them can be seen in Figure 19. They are caused by a build up of gold particles and then a sudden release of them. Figure 26 below shows another example of a droplet. It is clear why these lines are considered poor. They are too large, too inconsistent, and clearly are of poor quality.

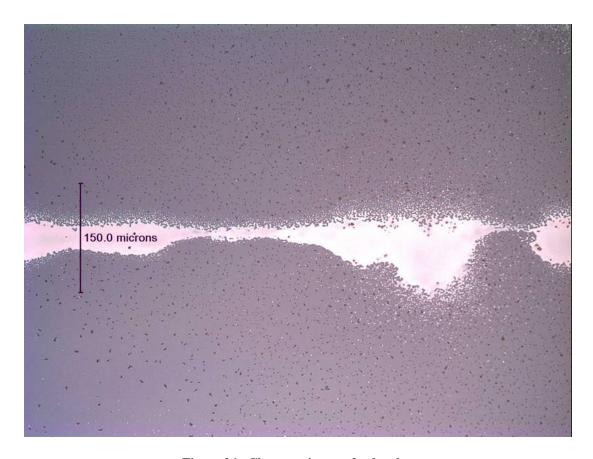


Figure 26: Close up picture of a droplet.

Smears were another type of line defect. They were caused by an existing smudge on the substrate or something contacting the line after it has been written. Often a fingerprint could cause a smear. One is shown in Appendix E.

4 RESULTS AND DISCUSSIONS

4.1 Line Shape Results

The line shape results come from the work done on the direct write. This section discusses what was done to get an optimal shape, and which was eventually considered the best way to get an optimal line. As already stated an optimal line was one that was 8-12 µm wide and conductive. It was also preferable if the line was reproducible. In order to achieve this several experiments were run. Some tests that were executed were changing table traverse speeds, doing multiple passes of the same line, changing solvent ratios, as well as a few others. The most important thing that came out of studying the line shape was the importance of measuring the line thickness.

4.1.1 Line Thickness

Line thickness, which is different from line width, is important because it is very closely correlated to both line width and how conductive a line is. If a line is thick, then it would make sense that it would not be as wide. This is assuming that the volume of gold nanoparticles flowing from the direct write nozzle was constant. From Figures 27 to 29, it is clear that it is a reasonable approximation. There are three graphs, the first describes what the graph would look like if our assumption of equal flow was true, the second and third show what is really happening. Comparing the Figure 27 and Figure 29, it is clear that assuming the flow is of a constant volume is reasonable. Even with the relatively small amount of data points and the lack of error bars, the point is that if the width decreases the thickness increases. This is good, and makes decreasing line width even more important.

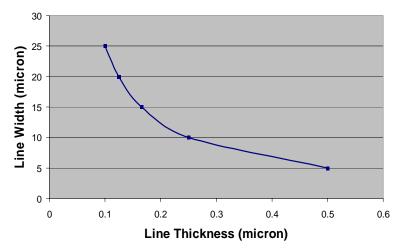


Figure 27: Ideal curve of line width and thickness assuming the constant flow volume.

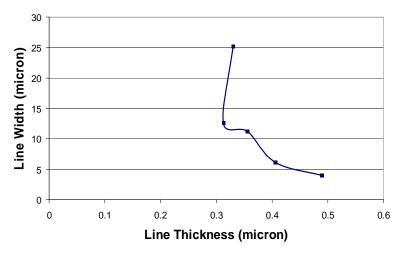


Figure 28: Actual experiment data of line width and thickness.

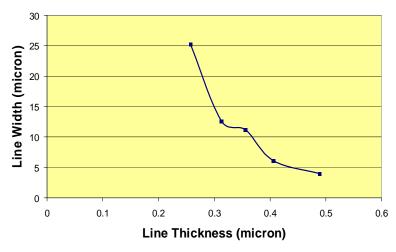


Figure 29: Actual experiment data of line width and thickness with adjusted data point.

The reason for the adjustment on Figure 29 is because of the way the optical profilometer takes measurements. The profilometer gives an average line thickness for a segment of the line as well as a peak thickness. In most cases they are the same. In Figure 28, all data points came from a peak thickness. However, if a line is fairly dry, or at least has the gold particles really spread out, then an average thickness is more appropriate. This is because these spread out clumped particles make peaks on the profilometer. The peaks are what is shown for a for a peak thickness. However the majority of the line is not that thick. When the line is not as wide these clumps are closer together and the peak thickness and average thickness are very close. This phenomenon is best explained in Figure 30 below.

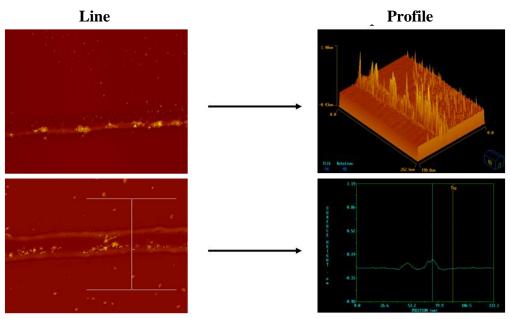


Figure 30: The difference of peak thickness and average thickness on many wide lines.

Back to the point, line thickness is a very important line characteristic because of its impact on line width and conductivity. If a line is thicker, the gold particles will be closer together and current will be able to flow through the line easier. The fewer gaps

between gold nano-particles the less resistance a line will have. It is possible to take this information and the relationship between line width and thickness and focus on making lines that are 8-12 µm wide knowing that will increase the thickness and conductivity. However, it was decided to focus on the line thickness. This was because there was evidence showing that a wide line could be hit with a laser in such a way that the line width would reduce to the size of the laser's beam. This process will be discussed more at a later time. But, if a line had no restrictions on width then making it a thick as possible was the challenge, and in turn the focus of the line shape tests.

4.1.2 Adjusting Table Speed

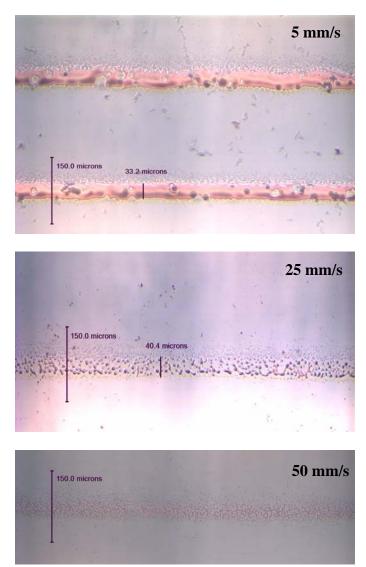
One would expect as the table traverse speed is increased, the line thickness would decrease and the opposite. The results of the table speed vs. line thickness are shown in Table 2.

Table 2: The effect of table speed on line thickness increases for two different solvents

| Solvent | Speed | Thickness |
|---------|--------|-----------|
| Au-Hep | 50mm/s | <.02µm |
| Au-Hep | 25mm/s | <.02µm |
| Au-Hep | 5mm/s | 0.031µm |
| Au-Hex | 5mm/s | 0.028µm |
| Au-Hex | 4mm/s | 0.051µm |
| Au-Hex | 3mm/s | 0.114µm |

From the above table, it is clear that changing the table speed has the expected effect on line thickness. The only problem with this is that, in order to increase line thickness this way, the manufacturing time is increased. The ideal situation would involve moving the

table as fast as possible while achieving a line of necessary thickness. Necessary thickness is arbitrary and therefore not a calculated number. The effect of table speed is illustrated further in the Figures 31 to 33 that show actual lines direct written at various speeds.



Figures 31 - 33: Lines written at 5, 25, and 50mm/s speed

4.1.3 Multiple Passes

Adding multiple passes over the same line, like changing the traverse speed, has an obvious effect on the line. By running back and forth over the same line the thickness of

the line will increase as well as the width of the line. This was originally thought not to be an option because of the goal to minimize line width. However, once the focus changed to maximizing the thickness, taking multiple passes became more plausible. The same problem with using slower speeds occurs when several passes are made over the same line. It takes longer to complete the process if many passes are needed. For this reason, multiple passes was still not considered the best option, although it was a very clear, easy way to increase line thickness. Figure 34 shows the effect of taking an extra pass. Notice how even after four passes of the same line, a limit is starting to form. This is almost intuitive, as objects are stacked gravity will force more material to move to the side of the line and cause an increase in width instead of thickness (see Figure 35). An example of this occurs when trying to build a sand castle, the first scope of sand stacks up nicely, and slowly each consecutive stack will flow off to the side of the castle.

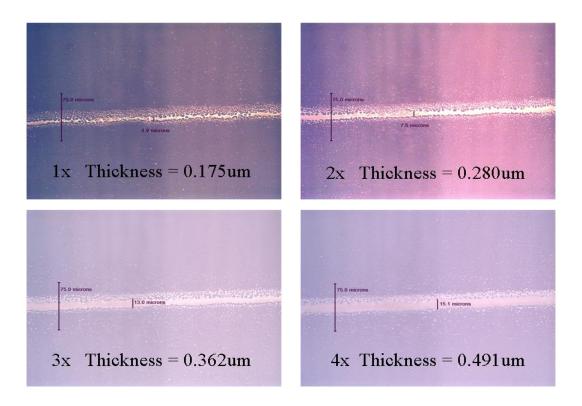


Figure 34: The effect of adding an extra pass over the same line

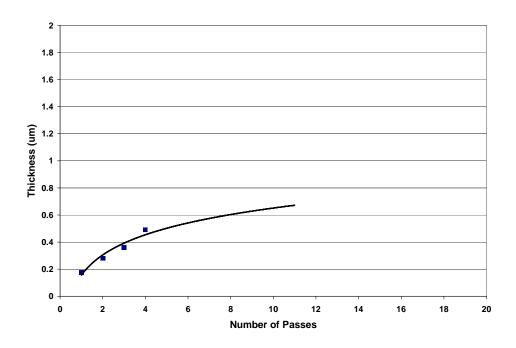


Figure 35: The logarithmic function of number of passes vs. line thickness.

Although it is of minimal importance, it is worthwhile to point out that the thickness and width of the line appears to increase at the same rate as the number of passes increases. From one pass to four passes, line thickness increases 280% while the width increases 308%. The percent increase remains very close at each pass added. The same table is included in Appendix F explains this in more detail. A much more significant detail worth noting is whether an extra line pass will increase the thickness more than reducing the speed in half. Both should take the same amount of time. From analyzing a very minimal amount of data, it appears that reducing the traverse speed will increase thickness the most. This guess comes from Table 2 showing that if the speed reduced from 5 to 3mm/s, the increase in line thickness is over 400%. This type of increase in line thickness takes more than four passes. Four passes would take more than twice as long as just moving 3 mm/s instead of 5. However, by intuition, reducing the traverse

speed could also be approximated logarithmic curve, and therefore will reach a limit on how thick a speed reduction could make a line.

There is very little actual data to prove this theory that a traverse speed reduction is a more effective way to increase line thickness than making multiple passes. However, using the little data that is available and a bit of intuition, it is a very reasonable assumption that this is true. It would not take much future work to prove this theory either. Neither method by itself is the best option to increase line thickness, because both methods add time to the process. However, one of those processes combined with the best direct write operating parameters might be.

4.1.4 Solvent Ratio

The solvent ratio is the ratio of how much solvent, hexane, heptane, or AF-7, is added to the gold nano-fluid. The gold nano-fluid (or nano-ink), again is the original mixture of gold nano-particles and AF-7. For the research to understand the correlation between solvent ratio and thickness hexane was used as the added solvent. During this research two different original nano-fluids were used. They are labeled as 1st Ink and 2nd Ink in Figures 36 and 37. The 2nd ink was new and still had the correct solids content. The 1st Ink was near the end of its life and its solids content was above the recommended 49-54% (see section 3.2 on page 24 for more information on solids content). Figures 36 and 37 show that as the solvent ratio increases line thickness decreases. This is especially evident with the 2nd Ink. However, there are several other factors that go into choosing a solvent ratio.

As already mentioned to little solvent causes clumps of material and the gold will not spread out evenly. Another problem with a small amount of solvent is the tubes for the direct write get dirty quicker and therefore line consistency is compromised. This is yet another example of why a machine operator is needed for the direct write, to monitor which solvent ratio works, and for how long. There is a minimum amount of solvent that can be used and still allow the gold to be atomized into a gas. So there are several factors that need to be considered other than simply choosing the lowest possible solvent ratio because it will make the line the thickest. The overall recommendation based on all this information is to use a solvent ratio in the range of 15:1 - 25:1. This range factors in all the aforementioned. Due to the constantly changing solids content of the nano-ink, the ratio may not be consistent within the ratio from one day to the next.

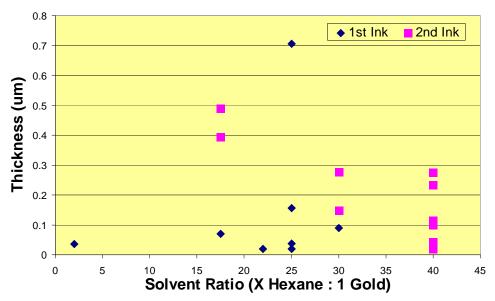


Figure 36: The solvent ratio compared with the line peak thickness.

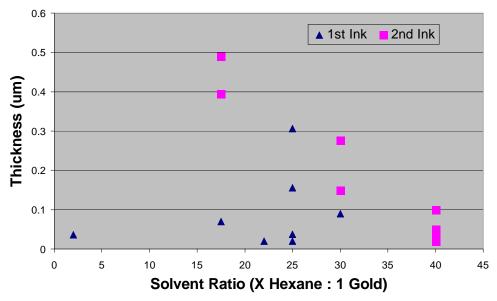


Figure 37: The solvent ratio compared with the line peak thickness.

It is ideal to have the peak thickness and the average thickness about the same. In Figures 36 and 37, this is true for the most case. However, especially on the 1st Ink it is clear that they are not at a 25:1 ratio. This is due to high solids content. On the 2nd Ink, it is the same at the 40:1 ratio. This is again because the clumps of particles get very spread out and there are parts of the line with lots of gold and parts with hardly any.

4.1.5 Optimal Direct Write Parameters

The optimal direct write parameters have been determined to produce the best line. The best line is first and foremost the thickest line. However, the line should also have a minimal width in order to be less dependent on the laser. It should be of good quality, shape, and consistent. As described in Chapter 3, consistency is very difficult and is another perk to focusing on thickness instead of width. A small width is difficult to make consistent, but a large thickness will always occur if the number of passes is increased.

Still, it was considered when trying to find the optimal parameters. The optimal parameters are shown in Table 3.

Table 3: Optimal direct write parameters

| Ultrasonic Power | 30 – 33 V |
|----------------------|----------------|
| Machine Speed | < 5 mm/s |
| Stand Off Distance | 4 mm |
| Sheath Gas Pressure | 30 – 40 cc/min |
| Carrier Gas Pressure | 8 – 13 cc/min |
| Solvent Ratio | 15:1 - 25:1 |
| Number of Passes | 1 – 4 |
| Tip Size | 100 µm |

Most of the parameters have been determined as a range of values. This is because the operator may need to change a value during the direct write process. Also with changing factors such as nano-ink solids content and the substrate there needs to be a range of operating parameters. These parameters are not even finite, but more a guide. It is possible that the optimal settings for a given day are outside these ranges. However, these ranges have been created from many data points and there is a 75% chance that any given parameter will fall within the optimal range listed. Most parameters have a greater than 90% chance the optimal is within the given range. Overall there is a 54% chance that the optimal for all parameters are in the ranges listed in Table 3.

The values listed for ultrasonic power came from thousands of lines being written and monitoring the power so that it creates a nice ultrasonic bath that converts the nano-ink into gaseous form. Machine speed was discussed in Table 2 shows that above 5 mm/s the lines get extraordinarily thin. Below 5 mm/s the lines actually get much thicker and the optimal may be considerably less than 5 mm/s but there is not currently data to prove

what that value is. Most tests were run at 5 mm/s traverse speed. The stand off distance was determined early in the project and held constant throughout. The optimal may be slightly different than what is listed, but due to the inconsistency of the direct write, it would be very difficult to prove such a slight change. The sheath gas pressure is one of the parameters that can change mid test. It should be adjusted to help create the best line viewable to the necked eye. For this reason it has a relatively wide range. However, many tests were done far outside that range to prove the accuracy of that range. All tests can be seen in Appendix A. The same could be said about the ATM gas pressure, although its range is a little smaller. There is minimal data to show if there is a consistent relationship between the sheath to ATM gas ratio. The solvent ratio was explained in Section 4.1.4 and the number of passes in Section 4.1.3. The tip sizes come in increments of $50 \,\mu m$. So a $50 \,\mu m$, $100 \,\mu m$, and a $150 \,\mu m$ tip were tried. The $100 \,\mu m$ tip proved to be the best, leaving little doubt about a bigger or smaller tip size.

4.2 Laser Sintering Results

Direct Write was used to create the lines. The laser sintering was used to improve the quality of them. The goal of the direct write deviated from the project goal in that it focused on thickness instead of line width. This was because of the laser's potential to reduce the line thickness. Laser sintering works by sending a low powered laser beam over the gold line and having the nano-particles in the line be forced to expand towards the heat of the laser. In other words, the nano-particles want to get as close to the heat source, which is the laser beam, as possible. To do this particles outside the beam width contract to inside the beam. Also, the particles inside the beam rearrange in order to form a single peak in the middle of the heat source. The nano-particles are written to the

substrate in such a way that they hit the substrate and almost bounce away from the center. This phenomenon was described earlier and results the bulk of the gold particles to be on the edges of a line. When the line is hit by the laser, the particles on the edges move to the middle thus reducing the line width and increasing its thickness. This laser sintering rearrangement can be best described in Figures 38 and 39 [10].

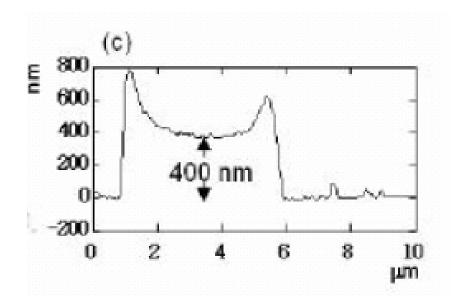


Figure 38: Profile of a non-sintered line (disregard the scale in this diagram).

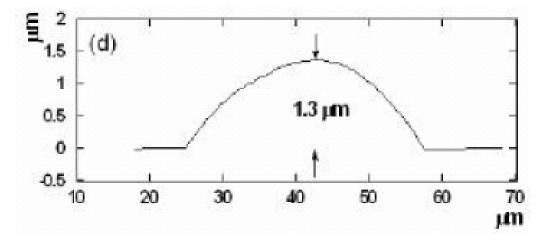


Figure 39: Profile of a sintered line (disregard the scale in this diagram).

Figures 38 and 39 show the ideal effect of laser sintering. This effect is what makes line width less important in the direct write phase of this project, even though it is one of the essential points in the project statement. Another outcome of laser sintering is that the sintered part of the gold line should bond with the substrate [11]. This means that the non-sintered part of a line should be able to be washed away while the bonded, sintered part remains. This will leave a line with a width equal to that of the laser beam width, and a thickness larger than what was achieved from just using direct write. This ideal effect from sintering was not always easy to achieve and in some cases it was not possible without new equipment.

4.2.1 Laser Sintering on Glass

Most direct write samples were done on glass or quartz because of availability and consistency. The types of coating on the polycarbonate changed several times throughout the project and that was uncontrollable. In order to remain consistent, lines were written onto glass. For this reason, gold lines on a glass substrate were the first to be sintered. The results were about as good as could be expected. The line width decreased to the beam width and the thickness increased slightly, as the shape of the line changed. Figure 39 gives a very clear example of how this happened.

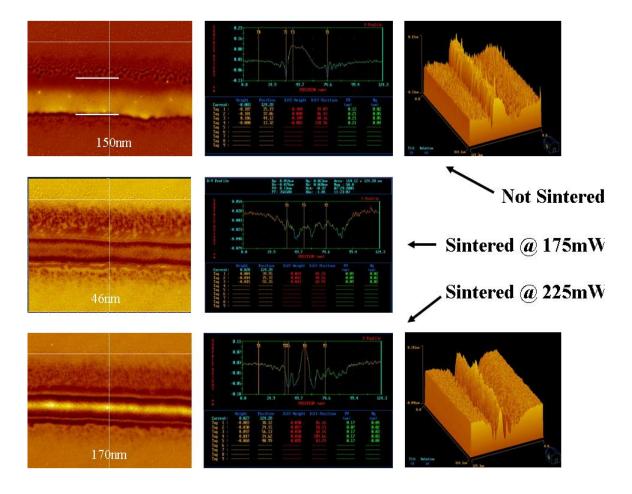


Figure 40: (Top) a non-sintered line, (middle) sintered at 175 mW, and (bottom) a line sintered at 225 mW.

In Figure 40 the top row shows how the line had most of its material pushed to the bottom by direct write. The top line is 40 μ m wide. In the middle row of pictures, the gold nano-particles have started to rearrange and a dense center is beginning to form at 175 mW. When the laser power was increase to 225 mW, a very bright center is visible and the line width is now 16 μ m. Notice in the picture in the bottom right that particles have arranged themselves into a peak in the center of the beam. The area around the gold line was also affected by the beam which is why it appears lower than the rest of the substrate. This occurrence will be described in detail in Sections 4.2.2 and 4.2.3.

Despite this small amount of substrate damage, sintering on glass or quartz was a complete success. The details of the line change are shown in Table 4 below. It was now possible to create thick lines that are conductive, and after changing the beam width, lines that sintered to 8-12 μ m wide. After this success with glass, polycarbonate samples were next.

Table 4: Details of the progressive line change that occurred during sintering

| Laser Power | Line Width | Line Thickness |
|-------------|------------|----------------|
| 0mW | 40µm | 150nm |
| 175mW | 20μm | 46nm |
| 225mW | 16µm | 170nm |

4.2.2 Laser Sintering on Polycarbonate

Unlike on the glass where substrate damage was viewed as a minor problem, it was a big deal on the polycarbonate. On the glass the substrate the surface level dropped at most 100 nm. On the polycarbonate, the surface level would sometimes rise, sometimes drop and it was in the order of up to $10~\mu m$. The effect of the laser on the polycarbonate surface was up to 100 times greater than on glass. When the substrate surface is changing several μm , it makes the gold line with a thickness of a few hundred μm seem insignificant on the surface of the substrate. It also severely damages the quality of the line.

Once it became clear that it was not going to be possible to sinter the lines at the same power used for the glass and quartz substrates, new powers and laser traverse speeds were tested. On the glass, the best sintering occurred at 225 mW at a speed of 0.2 mm/s. However nothing near that power and speed was possible on polycarbonate. Before that

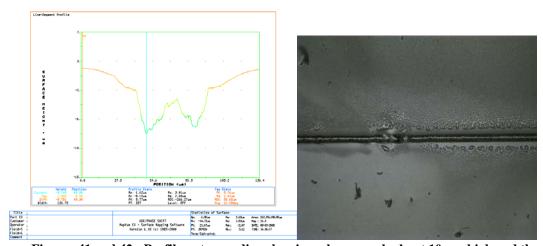
can be explained though, first the damage done to the polycarbonate surface must be explained.

The most recent results show that the substrate is burning, or at least slightly raising the polycarbonate surface every time the laser is used on it. The power and speed only play a role in how bad it is burned. The results of this are shown in Tables 5 to 7, and the pictures and descriptions in Figures 41 to 47 will be used to explain them.

All of these results are determined from using the laser on Polycarbonate coupons with no gold lines on them. Through a microscope there have been 4 different types of burns able to be classified:

1) Burnt

The line that has been hit by the laser is black, and the surrounding area has been affected causing the polycarbonate (or its coating) to burn or bubble up. It has been raised or lowered at least 2 µm.



Figures 41 and 42: Profilometer reading showing a burn mark about 10 μ m high and the microscope picture showing the black line with bubbling around it.

2) Mild Burn

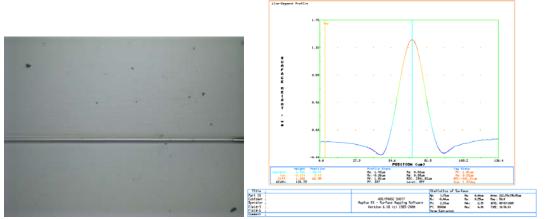
A mild burn shows just a black line. It does not change shape a much as when burnt (only raises 1-2 µm) and no bubbling to the surrounding area is caused.



Figure 43: The mildly burnt is black in color, but not raised as high and no bubbling.

3) Slight Raise

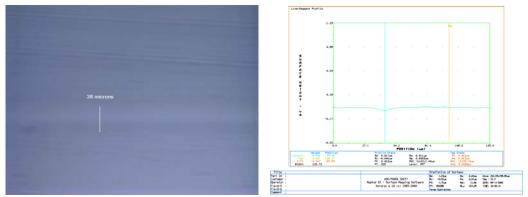
When the polycarbonate is raised slightly, there is very little black. The mark appears much smaller, as if only the area that has been hit by the laser is affected. It appears as a very thin line with a little yellow around it. The optical profilometer shows that the area hit by the laser has been raised $0.1-1~\mu m$.



Figures 44 and 45: The etched line with no dark black and the thin line in the middle and the yellowish coloring, about 1 µm raise.

4) Color Change

The only sign the laser has hit the polycarbonate is a slight color change visible at 200x or higher. The OP registers a small increase or decrease in material height, but in most cases this is less than 50 nm, and is hard to determine if you are measuring laser sintered material or a scratch.



Figures 46 and 47: (left) A hardly visible color change is shown beneath the white calibration marker and (right) the very slight change in material height.

5) Discrepancies

There are few problems with this data. The problems occur with really burnt lines or in the color change category. For really burnt lines it appears that the polycarbonate will thermally expand to a certain point causing the material to rise. Then suddenly it will drop and a burn mark or indention will happen, showing up as a negative thickness. This trend can be seen clearly in data shown in Table 7, and there is one point that appears to fall in between this phenomenon, and has a thickness of $1.862~\mu m$ that makes it appear just a mild burn.

The other problem is whether color change marks are raised or lowered. They are almost invisible on the optical profilometer and therefore some measurements are nothing more

than scratches. So the thicknesses of color change marks are undetermined, but are with confidence less than 0.02 $\mu m.\,$

Table 5: The laser parameter at low laser powers

| | | Speed (mm/s) | | | | | | | | | | | | |
|----------|----|--------------|------|-----|-----------|-----|-----|-----|-----|---|-----|-----|-----|-----|
| | | 0.03 | 0.05 | 0.1 | 0.15 | 0.2 | 0.4 | 0.6 | 0.8 | 1 | 1.3 | 1.6 | 1.9 | 2.2 |
| <u></u> | 8 | E | Е | Е | Е | С | C | С | С | С | С | С | С | С |
| (mW) | 11 | С | С | С | Е | Е | Е | E | E | Е | Е | Е | Е | С |
| <u>-</u> | 13 | | | | | Е | | | | | | | | |
| l ei | 16 | | | | | Е | | | | | | | | |
| Power | 19 | | | | | М | | | | | | | | |
| <u>а</u> | 23 | | | | | В | В | В | М | В | М | М | М | M |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | B= | Burnt | | | | | | | | | |
| | | | | M= | Mild Burn | | | | | | | | | |
| | | | | E= | S. Raise | | | | | | | | | |
| | | | | C= | C. Change | | | | | | | | | |

Table 6: The laser parameter results at high laser powers

| | | Speed (mm/s) | | | | | | |
|------------|----|--------------|-----------|----|----|----|----|----|
| | | 4 | 8 | 12 | 16 | 20 | 24 | 28 |
| | 50 | В | В | В | М | М | М | М |
| | 60 | В | В | В | В | В | М | М |
| ≥ | 70 | В | В | В | В | В | В | М |
| Power (mW) | | | | | | | | |
| We | | | | | | | | |
| 9 | | | | | | | | |
| _ | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | B= | Burnt | | | | | |
| | | M= | Mild Burn | | | | | |
| | | E= | S. Raise | | | | | |
| | | C= | C. Change | | | | | |

Table 7: Complete laser parameter results.

| Power (mW) | Speed (mm/s) | Effect | Height (µm) | | | |
|------------|--------------|--------|--------------------|----|--------------|-----------|
| 8 | 0.03 | E | 0.352 | | | |
| 8 | 0.05 | E | 0.267 | | | |
| 8 | 0.1 | E | 0.28 | | | |
| 8 | 0.15 | E | 0.212 | | | |
| 8 | 0.2 | C | <.05 | | | |
| 8 | 0.4 | C | <.05 | | | |
| 8 | 0.6 | C | <.05 | | | |
| 8 | 0.8 | С | <.05 | | | |
| 8 | 1 | С | <.05 | | | |
| 8 | 1.3 | С | <.05 | | | |
| 8 | 1.6 | С | <.05 | | | |
| 8 | 1.9 | С | <.05 | | | |
| 8 | 2.2 | С | <.05 | | | |
| 11 | 0.03 | С | <.05 | | | |
| 11 | 0.05 | С | <.05 | | | |
| 11 | 0.1 | С | <.05 | | | |
| 11 | 0.15 | Е | 0.21 | | | |
| 11 | 0.2 | Е | 0.534 | | | |
| 11 | 0.4 | Е | 0.434 | | | |
| 11 | 0.6 | E | 0.365 | | | |
| 11 | 0.8 | Е | 0.332 | | | |
| 11 | 1 | E | 0.293 | | | |
| 11 | 1.3 | E | 0.25 | | | |
| 11 | 1.6 | E | 0.202 | | | |
| 11 | 1.9 | E | 0.163 | | | |
| 11 | 2.2 | С | <.05 | | | |
| 13 | 0.2 | M | 1.128 | _ | D1 | 0 |
| 16 | 0.2 | M | 1.264 | B= | Burnt | > 2um |
| 19 | 0.2 | В | -7.386 | M= | Mild Burn | 1 - 2um |
| 23 | 0.2 | B B | -10.746 | E= | Slight Raise | 0.1 - 2um |
| 23 | 0.4 | В | -10.197 -11.335 | C= | Color Change | <0.1um |
| 23 | 0.8 | M | 1.862 | | | |
| 23 | 1 | В | 14.406 | | | |
| 23 | 1.3 | M | 2.555 | | | |
| 23 | 1.6 | M | 2.411 | | | |
| 23 | 1.9 | M | 2.549 | | | |
| 23 | 2.2 | M | 2.462 | | | |
| 50 | 4 | В | 5.126 | | | |
| 50 | 8 | В | 2.503 | | | |
| 50 | 12 | В | 8.877 | | | |
| 50 | 16 | M | 1.874 | | | |
| 50 | 20 | M | 1.603 | | | |
| 50 | 24 | М | 1.244 | | | |
| 50 | 28 | М | 1.256 | | | |
| 60 | 4 | В | 3.863 | | | |
| 60 | 8 | В | 7.252 | | | |
| 60 | 12 | В | 8.589 | | | |
| 60 | 16 | В | 6.938 | | | |
| 60 | 20 | В | 2.149 | | | |
| 60 | 24 | М | 1.67 | | | |
| 60 | 28 | М | 1.211 | | | |
| 70 | 4 | В | 2.536 | | | |
| 70 | 8 | В | 7.252 | | | |
| 70 | 12 | В | 8.589 | | | |
| 70 | 16 | В | 6.938 | | | |
| 70 | 20 | В | 6.741 | | | |
| 70 | 24 | В | 3.5 1.542 | | | |
| 70 | 28 | M | | | | |

From the data in Tables 5 to 7, it can be seen that to try and minimize the damage to the substrate the power was greatly reduced to a value less than or equal to 23 mW. The very low laser powers were tested at very low speeds in hopes that a low powered laser that is in contact with the gold line for longer will sinter better. The opposite was true at higher powers, 50-70 mW. At high powers that laser ran over the line quickly in hopes that a shorter exposure at a relatively high power would be effective.

From the results in Table 5, it is clear that the only way to not damage the substrate was at very low powers and at reasonably fast speeds. Unfortunately at those speeds and powers the amount of sintering done to the gold line was negligible. After several tests with similar results, and trying matters such as having the laser go over the only the gold line on the polycarbonate, and hence never touching the actual polycarbonate, it was determined the laser was inappropriate for the process. It was inappropriate due to its unusually short wavelength. Most other lasers used for a similar process had wavelengths between 488 and 530 nm, the average being 510 nm, which is in the green light spectrum [1, 5, 10, and 11]. The laser being used for this process only had a wavelength in the UV spectrum at 355 nm.

4.2.3 Explanation for Laser Results

The effect a laser has on a given substrate is related to the amount of light the substrate transmits, absorbs, and reflects. The transmittance, absorption, and reflectivity also depend on the wavelength of light hitting the surface. A good example of this is the earth's ozone layer. The ozone layer will reflect or absorb most of the suns UV (ultra violent) rays, but will transmit all its rays with wavelengths in the yellow or orange

spectrum. This means that very few of the dangerous UV rays reach the earth's surface and the wavelengths that do transmit cause the sun to look yellow and orange. The same is true for any material or gas; certain wavelengths of light will transmit, absorb, or reflect.

In the case of a laser that emits a beam of light at a constant wavelength. This wavelength will react the same with polycarbonate every time. It will act differently with every other material. This is good for laser sintering in some senses. For the glass, it allowed the gold to absorb the lot of the laser's energy while the glass absorbed a minimal amount. When a material absorbs energy it will change. The energy has to go somewhere and do something. This is the intended purpose of laser sintering, because it will cause the shape of the gold lines to change. However, it is not intended that the substrate absorb any significant amount of energy. Metals and insulators have very different reactions to the same wavelength. In fact they are almost opposites as shown in Figure 48. At the 355 nm wavelength used for this process polycarbonate aparentantly absorbed a lot of the lasers energy. The difference of about 150 nm between the laser used, and lasers currently being used in other sintering applications, is enough to cause a significant difference in the amount of damage the polycarbonate incurs. This is conceivable, and also likely due to the unstable absorption coefficients of metals and insulators that occur at short wavelengths (<1000 nm). While all materials are unstable in their reactions to short wavelengths, all materials react very differently. Figure 48 is a generalization and not true for every metal, nor every insulator. The transmission, reflection, and absorption of the polycarbonate at 355 nm are very different from that at 510 nm. For that reason its reaction to an otherwise similar laser beam is very different.

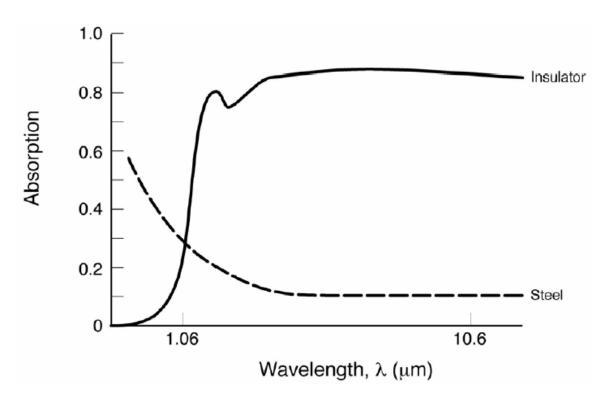


Figure 48: Insulators and Steels are both very unstable at wavelengths less than 1 μ m. Insulators and steel are also opposites throughout the light spectrum [8].

Another reason the polycarbonate may be so susceptive to absorbing the laser energy is because the gold lines are not thick enough to absorb enough of the laser themselves. Depending on the wavelength of the laser, gold will have a related absorption depth. This is best described in Figure 49. At 355 nm wavelength the laser operates at the laser energy will be absorbed to a depth of a little over 1 μ m. The thickest lines that were created were only about 0.7 μ m thick. This means the polycarbonate was seeing a good portion of the lasers energy through even the thickest of the gold lines. However, when a laser operates at wavelength of 510 nm, that is right were the graph spikes down towards an absorption depth of zero. While the absorption depth never reaches zero, it is quite obvious that at a wavelength 510 nm the absorption depth is much less. This means lines that are only 0.3 – 0.4 μ m thick will be able to absorb all the laser's energy, and a very

small amount would be seen by substrate. This is probably the leading case to explain why the laser used in this process was not optimal, and why laser sintering of gold lines on polycarbonate has been successful else where.

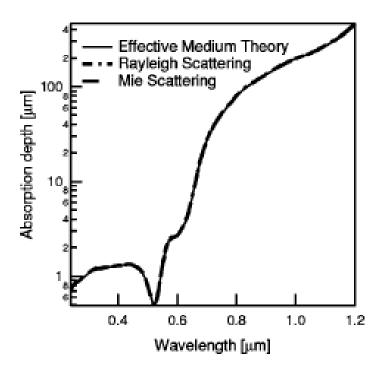


Figure 49: Absorption depth of gold nanoparticles compared to laser wavelength [12]

4.3 Resistance Measurements

Conductivity was one of the two main parts of the project statement. It was very important that these gold lines could conduct and have as little resistance as possible. Very few resistance measurements were actually taken due to the limited availability of the Agilent 4155 machine. Also quartz was preferred for taken resistance measurements and the quartz samples took a while to arrive. However, a small amount of resistance measurements were taken, enough to prove the lines were in fact conductive. Measurements were also taken from a solid gold nugget formed from evaporating all the

AF-7 in the original nano-ink solution. The measurement taken from the gold nugget

would serve as the bulk resistance of gold. The problem statement said to get as close to bulk resistance as possible. Other researched bulk gold measurements were found for comparison. Finally measurements were taken from some thick, wide gold lines that had been created by the direct write supplier Optomec. These lines proved to have a much smaller resistance than our initial measurements and served as a benchmark for future lines. All of the resistance measurements are displayed in Table 8.

Table 8: The resistance measurements of several different lines, the bulk gold nugget, and the researched bulk gold values using Agilent 4155

| Sample | Resistance (Ohms) |
|-----------------------------|----------------------|
| Non-Heated Line | 1.0×10^{11} |
| Furnace Heated Line @ 250°C | 1.0×10^8 |
| Bulk Gold Nugget @ 600°C | 6.0×10^{-3} |
| Bulk Gold on Glass | 1.0×10^{-2} |
| Bulk Gold on Alumina | 2.0×10^{-2} |
| Optomec Line 1 | 32.9 |
| Optomec Line 2 | 30.7 |
| Optomec Line 3 | 139 |
| Optomec Line 4 | 280 |

The small amount of data also supported thicker gold lines producing lines with a higher conductivity. The two lines shown in Table 8 listed as Non-Heated and Furnace Heated (see Appendix G) both had thicknesses of about 50 nm. All of the Optomec lines had thicknesses in the order of 0.5-1.5 µm. Optomec's lines were 10-30 times thicker and had resistances around a million times better. This is because when the lines were only 50-100 nm thick they had many gaps in them. As the gaps between the gold nanoparticles fill in with greater thickness the resistance improves in an exponential fashion. While there is limited data to actually prove this theory, more exists and can be found in Appendix H.

5 CONCLUSIONS AND FUTURE WORK

5.1 Conclusions

The biggest setback in producing micro size conductive lines was that they are so inconsistent (see Appendix I-M). Direct write was designed to produce lines about 25 µm wide. It can produce lines of this width fairly consistently. However, when the line gets smaller in width and approaches the 8-12 µm range the consistency fades. This is because lines that small are affected by the slightest change of nano-ink flow. The flow was affected by so many different things, including ink solids content, dirty tubes, and slight changes in sheath and ATM gas pressures, that became very difficult to control enough to have any consistency. In order to avert this problem, it was decided to focus on line thickness and let the direct write machine produce lines in its normal range of 25-40 µm. The lines would then be reduced to an acceptable width through laser sintering.

With the focus off of the line width, line thickness became the most important characteristic. Line thickness was directly related to the width of the line and most importantly the conductivity of the line. A good majority of time was spent trying to improve and figuring out how to improve line thickness.

Line width and shape was useful knowledge, but no longer considered a priority after the initial laser sintering results. It was useful knowledge because it gave insight into how the direct write worked and why it was so inconsistent. The laser sintering on glass proved that it was possible to drastically improve a line's width and shape. Although similar results were not shared with the polycarbonate, it was still believed that with the

use of the appropriate laser sintering could be used to aid line shape and width on any substrate. For this reason laser sintering made line quality less important.

The laser sintering process can be greatly improved with the use of a different laser. At the 355 nm wavelength the current laser was operating where too much energy was being absorbed by the substrate. At times, this was causing major damage to the substrate, especially if it was polycarbonate. Other research has shown that the use of a laser with a wavelength near 510 nm will be much more effective on a polycarbonate substrate. The Nd: YAG laser that was used for this project was not optimal and in turn, did not produce optimal results.

The stress craze test was briefly mentioned in Chapter 2. The stress craze test was passed, as no extra crazing occurred due to the addition of the chemicals in the solvent solution. This had no barring on the considerations listed in the problem statement. However, if the craze test was failed than it meant the nano-ink was unacceptable and therefore the test was very important to this research.

Despite the small amount of testing, the lines have been shown to be conductive and there resistances can be measured. In the few tests conducted, the lines showed very high resistances. However, it is the fact that the lines are able to conduct that is important. There is a good understanding of how to reduce resistance, and confidence is high that it can be done. Optomec has shown it is possible to get reasonable line resistances. It has also been made clear that by simply increasing line thickness that resistance in a given line will fall drastically. These lines are able to conduct electricity and will meet the criteria in the problem statement.

5.2 Future Work

It was briefly mentioned that other research has shown that once a line has been sintered, that it will bond with the substrate allowing the other material to be washed away. This is a consideration for the future. If the appropriate laser is used for sintering and more work tests can be run, than it would be interesting to start testing how well the gold nanoparticles bond with the substrate. Different solutions could be used to wash away the excess, non-sintered, material. Tape tests could be performed to test the strength of the bond. Quite a bit of research could be done to perfect this process and fully complete the sintering aspect of this project.

Another laser is needed for sintering on polycarbonate. A 510 nm laser has been previously used and works; however, it may not be the optimal source. More information could be found and studied to better understand just what type of laser is optimal for sintering on polycarbonate. Is it a laser that currently exists? Is it a laser that is optimal for other substrates or just polycarbonate? Would one want a laser that is only optimal for polycarbonate? These are all questions that could be answered with extensive future work on lasers and their absorption factors.

Improving the consistency of direct write would be a good idea for the future. If the direct write was consistent it is conceivable that sintering would never have to be done. That would allow the direct write to operate on its own without an observer standing by. If it is able to be consistent at 25-40 µm, there should be a way to make it the same at 8-12 µm. This would not be an easy task. Nor would any of this future work, however, these are all problem areas that were either diverted in the project, or left unsolved.

REFERENCES

- [1] Juntao Xu, Changgong Zhou, Sheila Grant, Edward Nadgorny, and Jaroslaw Drelich, "Laser Guidance Deposition Technique for Patterning Microstructures Made of Nanoparticles with Varying Surface Functionality", Materials Research Society Symp. Proc. Vol. 728 (2002).
- [2] Optomec Website, http://www.optomec.com/, "M3D Applications, Microelectronics Manufacturing", updated 2005.
- [3] Spectra-Physics Website, http://www.spectra-physics.com/, "High Energy Pulsed Lasers, MOPO Series Diode Lasers", updated 2005
- [4] Rockwell Automation Website, http://www.rockwellautomation.com/anorad/, "Anorad 3 Axis Gantry", updated 2005.
- [5] J. Chung, N.R. Bieri, S. Ko, C.P. Grigoropoulos, and D. Poulikakos, "In-tandem deposition and sintering of printed gold nanoparticle inks induced by continuous Gaussian laser irradiation", Appl. Phys. A 79, 1259-1261 (2004)
- [6] Materials Development Corporation Website, http://www.mdc4cv.com/CVplotters.htm, "Product Information, Agilent 4155", updated 2005.
- [7] Keyence Website, http://www.keyence.com/, "Laser Displacement Sensor", update 2005.
- [8] Elijah Kannatey-Asibu Jr., "ME586 Coursepack, Laser Materials Processing", University of Michigan, Chapter 14, p. 10-31, (2005).
- [9] ADE website, http://www.phase-shift.com/#menu, "Product Information, Optics/Precision Eng Surfaces, Profilometry", updated 2005.
- [10] Tae Y. Choi, Dimos Poulikakos, and Costas P. Grigoropoulos, "Fountain-Pen-Based Laser Microstructuring with Gold Nanoinks", Proceedings of NANO2004, Integrated Nanosystems: Design, Synthesis and Applications, (2004).
- [11] Edward M. Nadgorny, Changgong Zhou, Jaroslaw Drelich, and Randy Zahn, "MTU Laser-Based Direct Write Techniques: Recent Development and Nanoparticles Patterning Results", Materials Research Society Symp. Proc. Vol. 758 (2003).
- [12] N.R. Bieri, J. Chung, S.E. Haferl, D. Poulikakos, and C. P. Grigoropoulos, "Microstructuring by printing and laser curing of nanoparticle solutions", Applied Physics Letters, Vol. 82, N. 20 (2003).

APPENDIX

Appendix A: Spreadsheet of all direct write line data (Use dates to follow data)

| | I | | T | | I | |
|-----------|----------|-----------|----------|-------------|--------|----------|
| | | Stand off | Time | Machine | Sheath | 4714 0 |
| Run/Date | Material | Distance | Machined | Speed | Gas | ATM. Gas |
| 4/8/2005 | Ag | | | | 21 | 19 |
| 4/8/2005 | Ag | | | 500 | 21 | 19 |
| 4/11/2005 | Ag | | | 560 | 22 | 14 |
| 4/11/2005 | Ag | | | | 21 | 19 |
| 4/11/2005 | Ag | N | | 4.701.41755 | 22 | 14 |
| | | NEW | | ATOMIZER | NEW | ATOMIZER |
| 4/14/2005 | Ag | | | | 19 | 21 |
| 4/14/2005 | Ag | | | 750 | 21 | 19 |
| 4/14/2005 | Ag | | | | 28 | 18 |
| 4/15/2005 | Ag | | | | 22 | 22 |
| 4/20/2005 | Ag | | | 850 | 25 | 23 |
| 4/21/2005 | Ag | | | 850 | 25 | 21 |
| 4/21/2005 | Ag | | | 850 | 28 | 21 |
| 4/21/2005 | Ag | | | 850 | 30 | 20 |
| 4/21/2005 | Ag | | | 850 | 32 | 19 |
| | | | | | | |
| | | Stand off | Time | Machine | Sheath | |
| Run/Date | Material | Distance | Machined | Speed | Gas | ATM. Gas |
| | | Box 2 | | Box 2 | | Box 2 |
| 4/21/2005 | Ag | | | 999 | 27 | 25 |
| 4/27/2005 | Ag | 0015 | | | 30 | 26 |
| 4/00/000 | | GOLD | | Gold | | GOLD |
| 4/28/2005 | Au-Hex | | | | 30 | 24 |
| 5/4/2005 | Au-Hex | | | 750 | 43 | 6 |
| 5/4/2005 | Au-Hex | | | 750 | 40 | 10 |
| 5/9/2005 | Au-Hex | | 30 min | 800 | 33 | 11 |
| 5/10/2005 | Au-Hex | | 30 min | 800 | 33 | 11 |
| 5/12/2005 | Au-Hex | | 15 min | 800 | 35 | 14 |
| 5/12/2005 | Au-Hex | | 15 min | 800 | 45 | 8 |
| 5/13/2005 | Au-Hex | | | | 34 | 8 |
| 5/16/2005 | Au-Hex | | | 800 | 44 | 9 |
| 5/20/2005 | Au-Hex | | | | 40 | 6 |
| 5/23/2005 | Au-Hex | | | 999 | 36 | 9 |
| 5/23/2005 | Au-Hex | | 30 min | 999 | 36 | 9 |
| 5/25/2005 | Au-Hex | | 40 min | 999 | 40 | 6 |
| | | 01 1 11 | - | | | |
| Dum/Data | Metarial | Stand off | Time | Machine | Sheath | ATM O |
| Run/Date | Material | Distance | Machined | Speed | Gas | ATM. Gas |
| 0/05/0005 | A 1.1 - | Box 3 | 001- | Box 3 | 40 | Box 3 |
| 6/25/2005 | Au-Hex | slide 1 | 20 min | 999 | 40 | 6 |
| 6/26/2005 | Au-Hex | slide 2 | 15 min | 999 | 32 | 10 |
| 6/26/2005 | Au-Hex | slide 3 | 15 min | 999 | 32 | 10 |
| 6/26/2005 | Au-Hex | slide 4 | 15 min | 999 | 32 | 10 |
| 6/8/2005 | Au-Hex | slide 5 | 5 min | 800 | 28 | 19 |

| 6/23/2005 | Au-Hex | slide 6 | | | 30 | 15 |
|--|---|--|---|---|---|--|
| | | | | | | |
| Run/Date | Material | Stand off Distance | Time Machined | Machine Speed | Sheath Gas | ATM. Gas |
| | | Polycarbonate | | Polycarbonate | | |
| 6/8/2005 | Au-Hex | | 10 min | 800 | 28 | 19 |
| 6/8/2005 | Au-Hex | | | 800 | 30 | 10 |
| 6/8/2005 | Au-Hex | | 40 min | 800 | 30 | 10 |
| 6/9/2005 | Au-Hex | | 35 min | 999 | 33 | 15 |
| 6/9/2005 | Au-Hex | | 35 min | 999 | 33 | 15 |
| 6/11/2005 | Au-Hex | | 20 min | 800 | 35 | 10 |
| | | | | | | |
| | | Stand off | Time | Machine | Sheath | |
| Run/Date | Material | Distance | Machined | Speed | Gas | ATM. Gas |
| | | New Ink | | New Ink | | - |
| 6/27/2005 | Au-Hep | slide 7 | 40 min | 800 | 44 | 9 |
| 7/5/2005 | Au-Hex | slide 8 | 60 min | 999 | 40 | 6 |
| 7/5/2005 | Au-Hex | slide 9 | 45 min | 999 | 44 | 9 |
| 7/6/2005 | Au-Hex | slide 10 | | 999 | 33 | 13 |
| 7/6/2005 | Au-Hex | slide 10 | | 999 | 35 | 11 |
| 7/6/2005 | Au-Hex | slide 10 | | 999 | 38 | 9 |
| 7/8/2005 | Au-Hex | slide 11 | | | 38 | 10 |
| 7/8/2005 | Au- Hex | slide 12 | 45 min | | 37 | 10 |
| 7/8/2005 | Au- Hex | slide 13 | 45 min | | 37 | 10 |
| | | | | | | |
| | | 0, 1, % | | | 01 41 | |
| Pun/Date | Material | Stand off | Time Machined | Machine | Sheath | ATM Gas |
| Run/Date | Material | Distance | Time Machined | Speed | Sheath Gas | ATM. Gas |
| | | Distance Box 4 | Machined | Speed Box 4 | Gas | |
| 7/13/2005 | Au-Hex | Distance Box 4 Slide 1 | Machined 20 min | Speed Box 4 999 | Gas 35 | 10 |
| 7/13/2005 7/18/2005 | Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 | 20 min 15 min | Speed Box 4 999 999 | 35 36 | 10 |
| 7/13/2005 7/18/2005 7/18/2005 | Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 | 20 min 15 min 15 min | Speed Box 4 999 999 750 | 35 36 36 | 10 8 8 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 | Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 | 20 min 15 min | Speed Box 4 999 999 750 500 | 35 36 36 36 | 10 8 8 8 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 | 20 min 15 min 15 min | Speed Box 4 999 999 750 500 750 | 35 36 36 36 36 32 | 10 8 8 8 8 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 | 20 min 15 min 15 min | Speed Box 4 999 999 750 500 750 999 | 35 36 36 36 36 32 32 | 10 8 8 8 8 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 | 20 min 15 min 15 min | Speed Box 4 999 999 750 500 750 | 35 36 36 36 36 32 | 10 8 8 8 8 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 | 20 min 15 min 15 min | Speed Box 4 999 999 750 500 750 999 | 35 36 36 36 36 32 32 | 10 8 8 8 8 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min | Speed Box 4 999 999 750 500 750 999 | 35 36 36 36 32 32 40 | 10 8 8 8 8 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 | 20 min 15 min 15 min | Speed Box 4 999 999 750 500 750 999 | 35 36 36 36 36 32 32 | 10 8 8 8 8 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min Time | Speed Box 4 999 999 750 500 750 999 999 | 35 36 36 36 32 32 40 Sheath | 10 8 8 8 8 10 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min Time | Speed Box 4 999 999 750 500 750 999 999 | 35 36 36 36 32 32 40 Sheath | 10 8 8 8 8 10 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Mu-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min Time Machined | Speed Box 4 999 999 750 500 750 999 999 999 Machine Speed Polycarbonate | 35 36 36 36 32 32 40 Sheath Gas | 10 8 8 8 10 10 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min Time Machined | Speed Box 4 999 999 750 500 750 999 999 999 Machine Speed Polycarbonate | 35 36 36 36 32 32 40 Sheath Gas | 10 8 8 8 10 10 10 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min 15 min 70 min 2 min | Speed Box 4 999 999 750 500 750 999 999 999 | 35 36 36 36 32 32 40 Sheath Gas | 10 8 8 8 10 10 10 10 4TM. Gas |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 7/25/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 Slide 3 Stand off Distance Polycarbonate | 20 min 15 min 15 min 15 min 15 min 70 min 2 min 35 min | Speed Box 4 999 999 750 500 750 999 999 999 | 35 36 36 36 32 32 40 Sheath Gas | 10 8 8 8 10 10 10 10 ATM. Gas |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 Slide 3 | 20 min 15 min 15 min 15 min Time Machined 70 min 2 min 35 min | Speed Box 4 999 999 750 500 750 999 999 999 999 Machine Speed Polycarbonate 800 500 Machine Speed Speed | 35 36 36 36 32 32 40 Sheath Gas 44 44 30 | 10 8 8 8 10 10 10 10 4TM. Gas |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 7/25/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Material Au-Hep Au-Hep Au-Hep Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 Slide 3 Stand off Distance Polycarbonate | 20 min 15 min 15 min 15 min 15 min 15 min 15 min Time Machined 70 min 2 min 35 min Time Machined | Speed Box 4 999 999 750 500 750 999 999 999 999 999 Machine Speed Polycarbonate 800 500 Machine Speed Quartz | 35 36 36 36 32 32 40 Sheath Gas Sheath Gas | 10 8 8 8 10 10 10 10 ATM. Gas |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 7/25/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Material Au-Hep Au-Hep Au-Hex Material Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 Slide 3 Stand off Distance Polycarbonate | 20 min 15 min 15 min 15 min 15 min Time Machined 70 min 2 min 35 min Time Machined | Speed Box 4 999 999 750 500 750 999 999 999 999 999 Machine Speed Polycarbonate 800 500 Machine Speed Quartz 500 | 35 36 36 36 32 32 40 Sheath Gas 44 44 30 Sheath Gas | 10 8 8 8 10 10 10 10 ATM. Gas 9 9 |
| 7/13/2005 7/18/2005 7/18/2005 7/18/2005 7/22/2005 7/22/2005 7/22/2005 Run/Date 6/27/2005 6/28/2005 7/25/2005 | Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Au-Hex Material Au-Hep Au-Hep Au-Hep Au-Hex | Box 4 Slide 1 Slide 2 Slide 2 Slide 2 Slide 3 Slide 3 Slide 3 Slide 3 Slide 3 Stand off Distance Polycarbonate | 20 min 15 min 15 min 15 min 15 min 15 min 15 min Time Machined 70 min 2 min 35 min Time Machined | Speed Box 4 999 999 750 500 750 999 999 999 999 999 Machine Speed Polycarbonate 800 500 Machine Speed Quartz | 35 36 36 36 32 32 40 Sheath Gas Sheath Gas | 10 8 8 8 10 10 10 10 ATM. Gas |

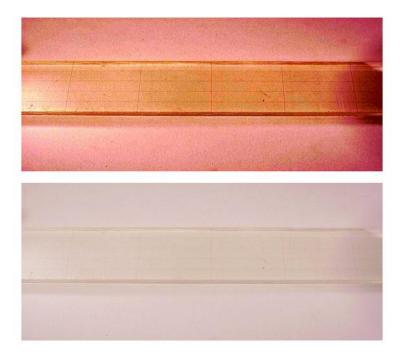
| | Ultrasonic | Ratio | Solvent | Tip | |
|------------|------------|-------------|----------------------|----------------|------------------|
| Run/Date | Power (V) | Sheath/ATM | Ratio | Size | |
| 4/8/2005 | 36 | 1.105263158 | | | |
| 4/8/2005 | 36 | 1.105263158 | | | |
| 4/11/2005 | 29 | 1.571428571 | | | |
| 4/11/2005 | 29 | 1.105263158 | | | Mid-slide change |
| 4/11/2005 | 29 | 1.571428571 | | | Mid-slide change |
| | NEW | | | | |
| | ATOMIZER | | | | |
| 4/14/2005 | 37 | 0.904761905 | | | |
| 4/14/2005 | 29 | 1.105263158 | | | |
| 4/14/2005 | 32 | 1.55555556 | | | |
| 4/15/2005 | 33 | 1 | | | |
| 4/20/2005 | 29 | 1.086956522 | | | |
| 4/21/2005 | 28 | 1.19047619 | | | |
| 4/21/2005 | 28 | 1.333333333 | | | |
| 4/21/2005 | 28 | 1.5 | | | |
| 4/21/2005 | 28 | 1.684210526 | | | |
| | | | | | |
| | Ultrasonic | Ratio | Solvent | Tip | |
| Run/Date | Power (V) | Sheath/ATM | Ratio | Size | |
| | Box 2 | | | | |
| 4/21/2005 | 29 | 1.08 | | | |
| 4/27/2005 | 34 | 1.153846154 | | | |
| | | GOLD | | | |
| | | | 10 | | |
| 4/00/0005 | 00 | 4.05 | drops/.5 | 400 | |
| 4/28/2005 | 36 | 1.25 | ml | 100um | |
| 5/4/2005 | 37 | 7.166666667 | 2:1? | 100um | |
| 5/4/2005 | 37 | 4 | 5.0.4 | 100um | |
| 5/9/2005 | 31 | 3 | 5.0:1 | 100um | |
| 5/10/2005 | 31 | 3 | 7.5:1 | 100um | |
| 5/12/2005 | 31 | 2.5 | 12.5:1 | 100um | Mid-slide change |
| 5/12/2005 | 31 | 5.625 | 12.5:1 | 100um | Mid-slide change |
| 5/13/2005 | 31 | 4.25 | 4== : | 100um | |
| 5/16/2005 | 31 | 4.88888889 | 17.5:1 | 100um | |
| 5/20/2005 | 31 | 6.66666667 | 30.0:1 | 100um | Heater to 050 |
| 5/23/2005 | 31 | 4 | 25.0.4 | 100 | Heated to 250 |
| 5/23/2005 | 31 | 4 | 25.0:1 | 100um | deg C |
| 5/25/2005 | 31 31 | 6.666666667 | 25.0:1 22.0:1 | 100um 100um | |
| 3/23/2003 | 31 | 0.00000007 | ZZ.U. I | rooum | |
| | Ultrasonic | Ratio | Solvent | Tip | |
| Run/Date | Power (V) | Sheath/ATM | Ratio | Size | |
| rtan, Date | Box 3 | JIIGUII/ATW | Ratio | 3120 | |
| 6/25/2005 | 31 | 6.666666667 | 22.0:1 | 100um | |
| 6/26/2005 | 31 | 3.2 | 25.0:1 | 100um | |
| 6/26/2005 | 31 | 3.2 | 25.0.1 | 100um | |
| 6/26/2005 | 31 | 3.2 | 25.0:1 | 100um | |
| 0/20/2003 | JI | J.Z | 20.0.1 | TOUGHT | Heated to 150 |
| 6/8/2005 | 31 | 1.473684211 | 30.0:1 | 100um | deg C |
| 0/0/2003 | J1 | 1.770007411 | 50.0.1 | TOOUTT | acy o |

| 6/23/2005 | 30 | 2 | | 100um | |
|---|---|--|--------------------------|--|-------------------------------|
| | | | | | |
| Run/Date | Ultrasonic Power (V) | Ratio Sheath/ATM | Solvent Ratio | Tip Size | |
| | Polycarbonate | | | | |
| 6/8/2005 | 31 | 1.473684211 | 30.0:1 | 100um | |
| 6/8/2005 | 31 | 3 | 30.0:1 | 100um | |
| 6/8/2005 | 31 | 3 | 30.0:1 | 100um | |
| 6/9/2005 | 33 | 2.2 | 27.0:1 | 100um | |
| 6/9/2005 | 33 | 2.2 | 27.0:1 | 100um | Heated to 150 deg C |
| 6/11/2005 | 31 | 3.5 | 10.0:1 | 100um | uog c |
| 0, 13,2000 | | 0.0 | | | |
| | Ultrasonic | Ratio | Solvent | Tip | |
| Run/Date | Power (V) | Sheath/ATM | Ratio | Size | |
| | New Ink | | | | |
| 6/27/2005 | 31 | 4.888888889 | 17.5 | 100um | |
| 7/5/2005 | 31 | 6.666666667 | 30.0:1 | 100um | |
| 7/5/2005 | 31 | 4.888888889 | 17.5:1 | 100um | |
| | | | | | mid-slide |
| 7/6/2005 | 33.5 | 2.538461538 | 40.0:1 | 100um | change |
| 7/6/2005 | 33.5 | 3.181818182 | 40.0:1 | 100um | mid-slide change |
| 7/6/2005 | 33.5 | 4.22222222 | 40.0:1 | 100um | mid-slide change |
| 7/8/2005 | 33.5 | 3.8 | 40.0:1 | 100um | |
| 7/8/2005 | 30 | 3.7 | 40.0:1 | 100um | |
| 7/8/2005 | 30 | 3.7 | 40.0:1 | 100um | |
| | | | | | |
| D /Data | Ultrasonic | Ratio | Solvent | Tip | |
| Run/Date | Power (V) | Sheath/ATM | Ratio | Size | |
| | Box 4 | | | | Dobuilt Cloop |
| 7/13/2005 | 30 | 3.5 | 30.0:1 | 100um | Rebuilt, Clean Machine |
| 7/18/2005 | 30.5 | 4.5 | 37.5:1 | 100um | mid slide change |
| 7/18/2005 | 30.5 | 4.5 | 37.5:1 | 100um | mid slide change |
| 7/18/2005 | 30.5 | 4.5 | 37.5:1 | 100um | mid slide change |
| 7/10/2005 | 30.5 | 3.2 | 37.3.1 | 100um | one line at 3x |
| 7/22/2005 | | J.Z | 1 | TOUGHT | |
| 7/22/2005 | 30 5 | 3 2 | | 100um | one line at 3v |
| 11/2/11/11/11 | 30.5 30.5 | 3.2 4 | | 100um | one line at 3x |
| 112212005 | 30.5 30.5 | 3.2 | | 100um 100um | one line at 3x one line at 3x |
| 1/22/2005 | | | | + | |
| 112212005 | 30.5 | 4 | Solvent | 100um | |
| | 30.5 Ultrasonic | 4 Ratio | Solvent Ratio | 100um | |
| Run/Date | 30.5 Ultrasonic Power (V) | 4 | Solvent Ratio | 100um | |
| Run/Date | 30.5 Ultrasonic Power (V) Polycarbonate | Ratio Sheath/ATM | Ratio | Tip Size | |
| Run/Date 6/27/2005 | 30.5 Ultrasonic Power (V) Polycarbonate 31 | Ratio Sheath/ATM 4.888888889 | Ratio 17.5 | Tip Size | |
| Run/Date 6/27/2005 6/28/2005 | Ultrasonic Power (V) Polycarbonate 31 31 | Ratio Sheath/ATM 4.888888889 4.888888889 | 17.5 42.5:1 | Tip Size 100um 100um | |
| Run/Date 6/27/2005 | 30.5 Ultrasonic Power (V) Polycarbonate 31 | Ratio Sheath/ATM 4.888888889 | Ratio 17.5 | Tip Size | |
| Run/Date 6/27/2005 6/28/2005 | Ultrasonic Power (V) Polycarbonate 31 31 30.5 | 4 Ratio Sheath/ATM 4.888888889 4.88888889 3 | 17.5 42.5:1 25.0:1 | Tip Size 100um 100um 100um | |
| Run/Date 6/27/2005 6/28/2005 | Ultrasonic Power (V) Polycarbonate 31 31 | Ratio Sheath/ATM 4.888888889 4.888888889 | 17.5 42.5:1 | Tip Size 100um 100um | |
| Run/Date 6/27/2005 6/28/2005 7/25/2005 | Ultrasonic Power (V) Polycarbonate 31 31 30.5 Ultrasonic | Ratio Sheath/ATM 4.888888889 4.888888889 3 Ratio | 17.5 42.5:1 25.0:1 | Tip Size 100um 100um 100um | |
| Run/Date 6/27/2005 6/28/2005 7/25/2005 | Ultrasonic Power (V) Polycarbonate 31 31 30.5 Ultrasonic Power (V) | Ratio Sheath/ATM 4.888888889 4.888888889 3 Ratio | 17.5 42.5:1 25.0:1 | Tip Size 100um 100um 100um | |

| - " | Line Width | | | - 15 |
|--|-------------------|----------------------------------|-------------|----------------------------|
| Quality | (micron) | Line Variation | Thickness | Run/Date |
| very dry | 30 | few lines | | 4/8/2005 |
| dense center, w/ many fingers, wet | 12.8 | few damaged lines | | 4/8/2005 |
| dense center, w/ many fingers, wet | 11.6 | few lines | | 4/11/2005 |
| good, some gaps, 10% overspray | 14.7 | inconsistent | | 4/11/2005 |
| some wet, some dry, many gaps | 18 | inconsistent | | 4/11/2005 |
| | NEW | ATOMIZER | | |
| wet but dense, 20% overspray | 21.7 | little variation | 0.068 | 4/14/2005 |
| very dry, very bad | 25 | few lines | | 4/14/2005 |
| dense but lots of gaps | 16.1 | little variation | | 4/14/2005 |
| dense but lots of gaps, fingers | 13.5 | consistently inconsistent | 0.211 | 4/15/2005 |
| good lines | 25.2 | lines got smaller | 0.537 | 4/20/2005 |
| good lines | 12.6 | consistently nice | 0.313 | 4/21/2005 |
| good lines | 11.2 | consistently nice | 0.356 | 4/21/2005 |
| a little wet, small, good | 6.1 | consistently nice | 0.406 | 4/21/2005 |
| good lines, but some gaps | 4 | to small to tell | 0.489 | 4/21/2005 |
| | | | | |
| Overlite. | Line Width | Line Venietien | | D /Data |
| Quality | (micron) | Line Variation | | Run/Date |
| | Box 2 | | | 1/01/000= |
| dense with gaps, inconsistent | 8.4 | very inconsistent | | 4/21/2005 |
| few gaps, few fingers, overall good | 8.4 | very small | | 4/27/2005 |
| | GOLD | | | |
| one big, one smaller, dense, gaps | 12.15 | only 2 lines | | 4/28/2005 |
| | | lines get better, then | | _,,,, |
| dry, huge lines 50%+ overspray | 65.5 | worse | 0.036 | 5/4/2005 |
| | | Lines start good get | | |
| dry, dense center, 50% overspray | 27.3 | worse | | 5/4/2005 |
| dry, 30-50 % overspray, dense cen. | 26.8 | Majority the same | | 5/9/2005 |
| | 0. | inconsistent, few good | | = / 4 0 / 0 0 0 = |
| very dry, all overspray | 35 | lines | | 5/10/2005 |
| Dense center, 40% overspray | 46.1 | few lines, mostly junk | | 5/12/2005 |
| Dense center, 30-40% overspray | 22.6 | fairly consistent | | 5/12/2005 |
| good but big, 20-30% overspray | 35 | droplet problem | | 5/13/2005 |
| Very Good | 25.9 | fairly consistent | 0.07 | 5/16/2005 |
| - Path and for Common and | 4.4 | lines get smaller, | | E (00 (000E |
| a little wet, fat fingers, good | 14 | droplets | | 5/20/2005 |
| wall contared lines | 10.2 | dry to dense | 0.037 | E/22/2005 |
| well centered lines very nice centered lines | 18.2 8.2 | progression some gaps, few lines | 0.037 | 5/23/2005 5/23/2005 |
| | | | | |
| gaps in lines, later dry, overspray | 16 | hard to say b/c small | | 5/25/2005 |
| | l in a \A/: alth | | | |
| Quelity | Line Width | Line Verietien | | Bun/Data |
| Quality | (micron) Box 3 | Line Variation | | Run/Date |
| too day cyaraaray | | your consistent | nogli sible | C/OF/OOF |
| too dry, overspray | 30 | very consistent | negligible | 6/25/2005 |
| dry, overspray, visible gaps | 25 6.5 | consistent on the slide | negligible | 6/26/2005 |
| very dense center, obvious gaps | 6.5 | consistent on the slide | 0.706 | 6/26/2005 |
| dense center, 45% overspray | 15.1 | line density inconsistent | .05262 | 6/26/2005 |

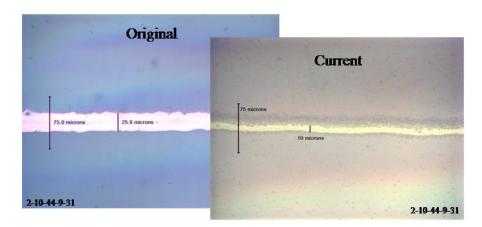
| dense, holey, large | 34.2 | consistent on the 8 lines | <.1 | 6/8/2005 |
|---|---------------------|--|---------|----------------------|
| dones, noisy, lange | U | | no | 0,0,2000 |
| dense blobs, no line, overspray | 75 (20)?? | top 5 ok, blobs on others | measure | 6/23/2005 |
| | | | | |
| | Line Width | | | |
| Quality | (micron) | Line Variation | | Run/Date |
| | Polycarbonate | | | 2/2/222 |
| dense center, 25% overspray, wet | 25-30 (20) | consistent | | 6/8/2005 |
| very wet, very dense, | 30-130 | lines got smaller | | 6/8/2005 |
| dry grainy to bordly visible | 20-30 | lines faded to barely visible | | 6/9/200E |
| dry, grainy to hardly visible small dense center, 50% overspray | 40 (10) | consistent, end is better | | 6/8/2005 6/9/2005 |
| | 40 (10) | | | 6/9/2005 |
| dense center, big, lots of overspray some dry, some kind of dense | 30-40 | consistent, small center?? very inconsistent | | 6/11/2005 |
| Some dry, some kind of defise | 30-40 | very inconsistent | | 0/11/2003 |
| | Line Width | | | |
| Quality | (micron) | Line Variation | | Run/Date |
| ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,, | New Ink | | | |
| dense center, lots of overspray | 35 (10.1) | very consistent | 0.394 | 6/27/2005 |
| Dense, but lopsided with overspray | 25 (6) | some all overspray | 0.276 | 7/5/2005 |
| Dense, but lopsided with overspray | 26 (10) | fairly consistent | 0.489 | 7/5/2005 |
| Very Nice, but big | 43 | fairly consistent | 0.114 | 7/6/2005 |
| Dense, lopsided, not as bad as 8,9 | 20 | fairly consistent | 0.042 | 7/6/2005 |
| Dense, lopsided, not as bad as 8,9 | 11 | fairly consistent | 0.019 | 7/6/2005 |
| | | Edges become less | | |
| Dense edges, non-dense center | 34 (19) | defined | 0.275 | 7/8/2005 |
| Very Dense Center, lopsided | 40 (8-9) | Gets a little less dense | 0.232 | 7/8/2005 |
| Similar to end of 12, drys up | 40 (10-12) | Drys up towards end | 0.099 | 7/8/2005 |
| | | | | |
| Quality | Line Width (micron) | Line Variation | | Run/Date |
| Quality | Box 4 | Line variation | | Kuil/Date |
| Dense, good, lopsided above line | 14 | Fairly Consistent | 0.148 | 7/13/2005 |
| Not continuous, some dense | 12 | Fairly Consistent | 0.028 | 7/18/2005 |
| More continuous/dense | 13 | Fairly Consistent | 0.020 | 7/18/2005 |
| Continuous, bottom dense, little os | 15 | dried up on the 2nd half | 0.114 | 7/18/2005 |
| Very Splotchy, not continuous | 40 | Left side slightly denser | 0.155 | 7/22/2005 |
| Less Splotchy, not continuous | 35 | Left side slightly denser | 0.0556 | 7/22/2005 |
| Continuous, nice, not thick | 25 | consistent | 0.032 | 7/22/2005 |
| , , | | | | |
| | | | | |
| | Line Width | | | |
| Quality | (micron) | Line Variation | | Run/Date |
| | Polycarbonate | | | |
| looks very nice after 4 passes | 25 | still very consistent | | 6/27/2005 |
| dense center, lopsided overspray | 35 (6.5) | one line only | | 6/28/2005 |
| Bottom very dense, top has gaps | 17 | top and bottom very diff. | | 7/25/2005 |
| | Lima NAC 141 | | | |
| Quality | Line Width | Line Variation | | Pun/Data |
| Quality | (micron) Quartz | Line Variation | | Run/Date |
| hottom very dense, ton is dry gans | Qualiz | | | 7/25/05 |
| bottom very dense, top is dry, gaps | | | | 7/25/05 |
| | 1 | | | 1/23/03 |

Appendix B: Checkered pattern of direct write lines used for the craze test

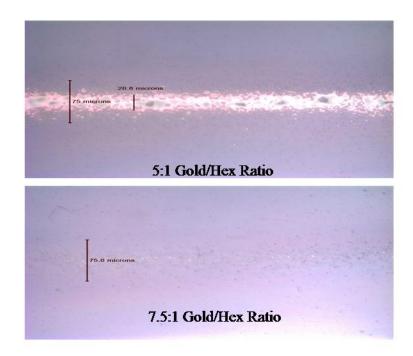


Appendix C: The original picture is of a new valve of Gold/AF-7 solution, and the current picture is one that has been open for 4 weeks.

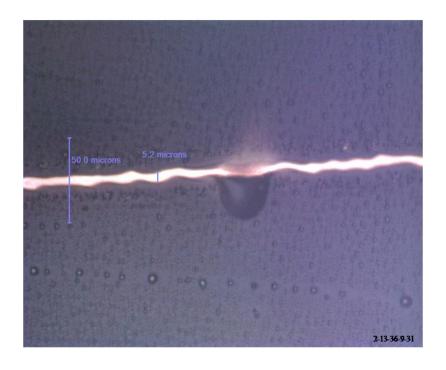
- Line size is much smaller with the current hexane
- More overspray with the current hexane
- Overspray + smaller line is approximately the same size as original hexane line



Appendix D: The top line is very wet looking, while the bottom line is extremely dry. This is a good example of the two extremes side by side.



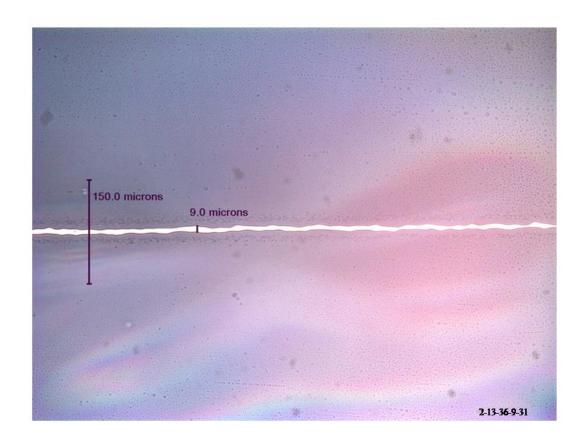
Appendix E: This is a line smear. These can be avoided by careful handling of the slides. These are caused by smudges or fingerprints.



Appendix F: Thickness and width increase almost equal amounts with each table pass

| Pass # | Thickness (µm) | Width (µm) | Thickness Increase | Width Increase |
|-----------|-------------------|---------------|-----------------------|-------------------|
| 1 | 0.175 | 4.9 | omit | omit |
| 2 | 0.280 | 7.5 | 160% | 153% |
| 3 | 0.362 | 13.0 | 207% | 265% |
| 4 | 0.491 | 15.1 | 280% | 308% |

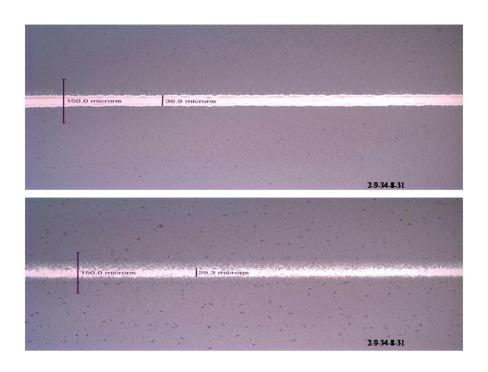
Appendix G: A very nice thin line, with good quality. This line unfortunately was not very thick, and in turn did not conduct very well. It was the furnace treated line mentioned in Table 8.



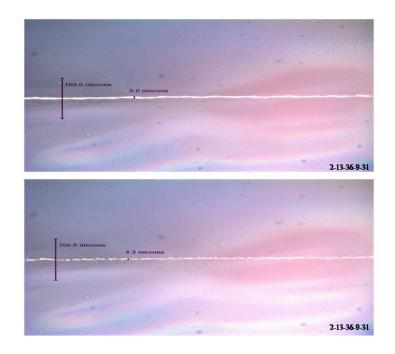
Appendix H: Optomec Resistance Measurements

| | Optomec Sample | | | | |
|---------|----------------|--------------------|------------------------|----------------------|--|
| | Width (um) | Beam Width (um) | Avg. Thickness (um) | Resistance (Ohms) | |
| 11. 4 | | 00 | 0.047 | | |
| Line 1 | 50 | 30 | 0.217 | tape test | |
| Line 2 | 45 | 17 | 0.335 | open | |
| Line 3 | 46 | 30 | 0.327 | 66.6 ohms | |
| Line 4 | 45 | 31 | 0.244 | open | |
| Line 5 | 45 | 33 | 0.282 | 32.9 ohms | |
| Line 6 | 55 | 45 | 0.530 | 30.7 ohms | |
| Line 7 | 65 | 48 | 1.271 | 139 ohms | |
| Line 8 | 55 | 48 | 1.299 | 280 ohms | |
| Line 9 | 55 | 33 | 1.198 | 111 ohms | |
| Line 10 | 50 | 21 | 1.182 | 161 ohms | |

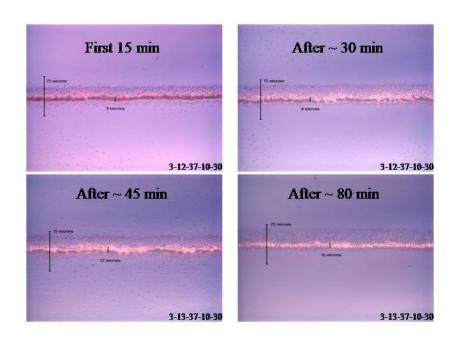
Appendix I: Line Inconsistency: The difference in line shape from the beginning of a slide to the end of the same slide.



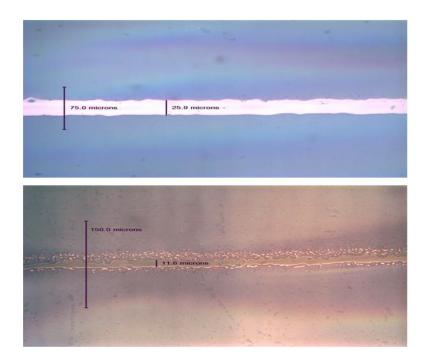
Appendix J: Line Inconsistency: The differences in line shape from the left most part of a line, the right most part of the same line on the same 3 inch slide.



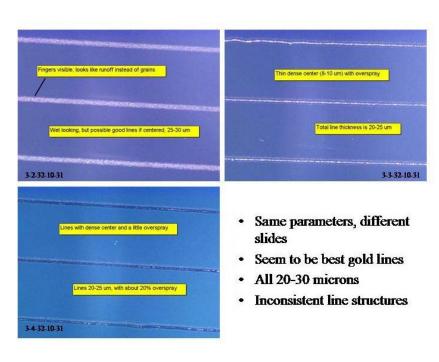
Appendix K: Line Inconsistency: In a 90 min run, the lines from the direct write change dramatically in the same settings.



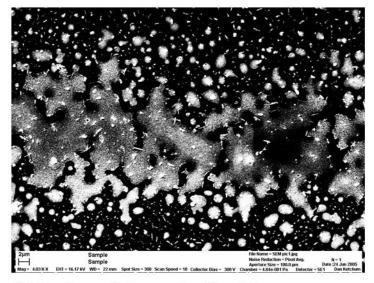
Appendix L: Line Inconsistency: The difference between a hexane line (on top), and a heptane line (on bottom). Both solvents were inconsistent and their differences in these pictures were considered coincidence and irrelevant.



Appendix M: Line Inconsistency: These are some overview pictures of inconsistent lines. They are all written at the same parameters, but were on different slides.

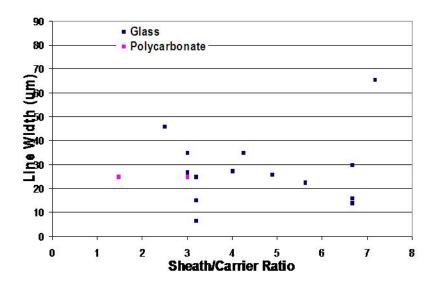


Appendix N: A 4000x SEM picture of a line.



- · Notice the small clumps of gold particles.
- The clumps range from about 100nm to 1 micron

Appendix O: The sheath/carrier ratio compared with line width appears to have no pattern. It can therefore be concluded that it has little to no effect on line width



- No pattern can be seen from initial Ratio data
- · Other parameters changing, makes this inaccurate