ELECTROHYDRODYNAMIC JET PRINTING: ADVANCEMENTS IN MANUFACTURING APPLICATIONS

BY

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THESIS

Submitted in partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering in the Graduate College of the University of Illinois at Urbana-Champaign, 2013

Urbana, Illinois

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ABSTRACT

Electrohydrodynamic jet printing offers advantages such as high resolution over more traditional manufacturing printing techniques, but lacks in the high throughput that is demanded by industry. At the University of Illinois at Urbana-Champaign, UIUC, a high resolution technique for printing was developed and also a desktop system designed and built. UIUC has been researching the advancements of E-jet in manufacturing applications for many years. This thesis includes work which continues to push the abilities of the current E-jet system to be more appealing for manufacturing along with developing a new printing technique utilizing the fundamental physics of E-jet printing.

A multi-nozzle array (MNA) print head is proposed which has the capability of addressing each nozzle individually or simultaneously print with the whole array. For proof of concept, a four nozzle print head is discussed and has the potential to be increased for batch pattern/image printing. By powering the nozzles with a pulse width DC voltage, the printed image quality can be controlled depending on the desired application and is also studied. The standard deviation of the achieved droplet size across the multi-nozzle print head is approximately 0.5-0.6µm. A simple cost analysis was performed to determine feasibility of a one-time use nozzle print head.

Finally, preliminary efforts of printing from an ink film by means of a generated rather than applied voltage is developed, tested, and discussed in this thesis. This work eliminates the need for a physical nozzle that is used in nearly all current E-jet printing setups. By implanting pyroelectricity to E-jet, droplet diameters as small as 300µm were printed. Although these droplets are large compared to current E-jet printing, the work discussed is a proof of concept and by further understanding the physics of pyroelectricity, it is hoped that a comparable resolution of droplet size can be achieved.
ACKNOWLEDGMENTS

I would like to thank my advisor, Professor Placid Ferreria, for allowing me to be a part of his research group and working on E-jet. His guidance and novel ideas for E-jetting applications were always exciting. His demand for quality results pushed me to work hard and efficiently which resulted in successfully completing my Master’s degree.

I would also like to thank the other members of Professor Ferreira’s research group. With the wide variety of projects our group does research in, there was rarely a question I had that would go unanswered. They always had encouraging words to help me stay focused and find joy in my research. I would specifically like to thank Ala’a Al-Okaily for setting aside large amounts of time to assist me with my experiments, he always made time to run the laser for my experiments and providing excellent discussions and ideas on the results to my experiments. I would also like to thank the other members of my group that made research and our office an enjoyable environment to work in; Numair Ahmed, Bruno Azeredo, Jorge Correra, Kyle Jacobs, and Bonjin Koo.

Keng Hsu must also be thanked for his insight on rapid prototyping and part production and Michael Philpott for providing the aPriori cost analysis on my multi-nozzle array research. I would also like to express my deepest gratitude to the late Mark Shannon. He gave me the opportunity to be involved with his research as an undergraduate which fueled my desire to continue my studies and obtain my Master’s degree.

Lastly I would like to thank my family and friends for their constant encouragement and words of motivation. Words cannot describe how grateful I am to have such a large support group to help me through the struggles of graduate school and to celebrate the accomplishments I have achieved.
# TABLE OF CONTENTS

List of Figures ................................................................................................................................ vi
List of Tables ................................................................................................................................ ix
Chapter 1: Introduction ................................................................................................................... 1
  1.1 Background of E-jet .............................................................................................................. 2
  1.2 Current Technology ............................................................................................................ 4
  1.3 Current E-jet Limitations ................................................................................................. 5
Chapter 2: Multi-nozzle array printhead ......................................................................................... 7
  2.1 Introduction ....................................................................................................................... 7
  2.2 Design of holders and mounts ........................................................................................... 8
  2.3 Nozzle Alignment ............................................................................................................. 10
  2.4 Experimental Setup ......................................................................................................... 11
  2.5 Printing Experiments ......................................................................................................... 13
  2.6 Results and Analysis of Simultaneous MNA Printing ........................................................ 16
    2.6.1 PWM Parametric Study ............................................................................................. 16
    2.6.2 Conclusion of MNA PWM printing .......................................................................... 19
  2.7 Cost Analysis of V-groove MNA ....................................................................................... 22
Chapter 3: Nozzle-less Printing .................................................................................................... 24
  3.1 Introduction ....................................................................................................................... 24
  3.2 Pyroelectricity ................................................................................................................... 25
    3.2.1 Background and History ............................................................................................ 25
    3.2.2 Physics of Pyroelectricity .......................................................................................... 26
    3.2.3 Materials and Applications ....................................................................................... 28
  3.3 Experimental Setup ........................................................................................................... 29
  3.4 Experiments with Heat source .......................................................................................... 31
    3.4.1 Soldering Iron .............................................................................................................. 31
    3.4.2 Resistive Heater Coil .................................................................................................. 33
    3.4.3 Near Infrared (NIR) Laser Diode ............................................................................... 36
  3.5 Experiments with Infrared Absorber .................................................................................. 36
    3.5.1 NIR Absorbing Dye with PDMS .............................................................................. 37
LIST OF FIGURES

Figure 1. Schematic of UIUC E-jet printer ................................................................. 3
Figure 2. Image of nozzle, nozzle reflection, and a printed droplet from 5-axis E-jet setup .............. 4
Figure 3. CAD drawings of the Teflon nozzle holder (left) and nozzle alignment setup with Teflon holders (right). ......................................................................................................................9
Figure 4. CAD drawings of the v-groove nozzle holder (left) and alignment setup (right) .............. 10
Figure 5. Final v-groove nozzle print head with four secured nozzles. ........................................ 11
Figure 6. RP E-jet mount and rotational stage attached to the 5-axis E-jet printer. ......................... 12
Figure 7. Composite image of the nozzles and nozzle reflections for alignment purposes for a four nozzle MNA. ..................................................................................................................13
Figure 8. UIUC logo used for demonstrating simultaneous printing with the MNA. ................. 14
Figure 9. Initial MNA simultaneous printing of UIUC logo with T1 parameters. ...................... 16
Figure 10. Enlarged area of printed pattern from the same area for each nozzle of the MNA print head for five experiments. ..............................................................................................................17
Figure 11. Final MNA simultaneous printing with high density of droplets with PWM T5 parameters. .............................................................................................................................................19
Figure 12. Enlarged T1 (top) and T5 (bottom) printed image of common printing defects. ....... 20
Figure 13. Printed pattern of two of the four images to display the spacing of the four MNA. 21
Figure 14. Graphical representation of relationship between thermal, mechanical, and electrical properties with the primary and secondary pyroelectric coefficients highlighted [23]. ........................................................................................................................................26
Figure 15. Ink film pyroelectric E-jet printing schematic (top) and actual image of setup (bottom)..................................................................................................................30
Figure 16. Composite of printed droplets from experiments with a soldering iron heat source and LN crystal. .......................................................................................................................32
Figure 17. Resistive heater coil printing setup .............................................................................33
Figure 18. Image of liquid bridge formed with SS resistive heater coil instead of a printed droplet ........................................................................................................................................34
Figure 19. Image of ink film deformation profile while printing and a printed droplet is seen in the ink reflection. .....................................................................................................................35
Figure 20. Droplets printed with a resistive heater coil heat source and LN crystal. .......... 36
Figure 21. Schematic of NIR dye mixed with PDMS as NIR laser absorbing material for pyroelectric E-jet printing..................................................................................................................37
Figure 22. Optical images of printed droplets from Table 5 using a pulse laser heat source and LN crystal................................................................................................................................39
Figure 23. mPDMS pulsed laser printing experiments with no glass on the mPDMS and Cu tape under entire ink resulting in multiple printed droplets from Table 6 ......................... 40
Figure 24. Printed “I” pattern using mPDMS and NIR laser heat source.............................. 41
Figure 25. Printed lines attempts with continuous NIR laser and mPDMS......................... 43
Figure 26. Composite of printed droplets from experiments with varying PDMS to NIR dye ratios A (top) and C (bottom)........................................................................................................44
Figure 27. mPDMS damage (left) and misaligned printed droplet to mPDMS damage (right) due to laser printing. ................................................................................................................45
Figure 28. NIR dye directly on crystal (far left), printed droplets (middle left & right), damaged crystal after printing due to overheating (far right)............................................ 46
Figure 29. Schematic of Si µ-square NIR laser absorbing pyroelectric E-jet printing. ........ 47
Figure 30. Laser focus on a 200x200x10µm Si µ-square (left) and three Si µ-squares with successfully printed droplets (right)..........................................................................................48
Figure 31. Schematic of altered Si µ-square location for NIR laser absorbing pyroelectric E-jet printing. ........................................................................................................................49
Figure 32. Three different Si µ-squares with printed droplets using the flipped crystal/Si µ-square setup..................................................................................................................49
Figure 33. 100x100x10µm Si µ-square with flipped crystal/Si µ-square setup. ................. 50
Figure 34. Three different Si µ-squares with printed droplets at 20Å using the flipped crystal/Si µ-squares setup. .................................................................................................50
Figure 35. Three different Si µ-squares with successfully printed droplets using a LT crystal. 53
Figure 36. Two Si µ-squares with successfully printed droplets using the flipped LT/Si µ-square setup..................................................................................................................54
Figure 37. Alternative setups for pyroelectric E-jet printing from an ink film directly to the LT crystal (left) and printing away from the LT crystal to glass (right)............................... 56
Figure 38. Printed droplet from Setup A and NIR laser with Si µ-square absorber......... 56
Figure 39. Setup B print attempt with an image captured immediately after laser is off (left), after approximately 11s (middle) and when there is no more observed changes (right). ............................................................................................................................................57

Figure 40. Alternative schematics for ground electrode placement in pyroelectric E-jet printing from an ink film................................................................................................................58

Figure 41. Two printed droplets using Setup A from Figure 40................................................. 59
LIST OF TABLES

Table 1. Initial jetting voltages and gaps for a single printing experiment (the gaps are approximated through image processing and may not be accurate to actual distance) ...............15
Table 2. Varying PWM parameters for each printed test run ......................................................15
Table 3. Average size and standard deviation of counted printed droplets from enlarged area in Figure 10 ............................................................................................................................18
Table 4. Average size droplets printed with a soldering iron as the heat source and LN crystal 32
Table 5. Parameters for pulsed laser pyroelectric E-jet printing and droplet diameters of successful prints .............................................................................................................................38
Table 6. mPDMS printing experiments with the pulsed laser ......................................................40
Table 7. Printed I with mPDMS and NIR laser droplet analysis ................................................42
Table 8. Different mPDMS ratios and thickness, labeled A - D ...............................................43
CHAPTER 1: INTRODUCTION

Printing technologies such as thermo or piezo inkjets, Xerography, and 3D printing have made advancements in manufacturing processes by increasing resolution and throughput. With 3D printing tools such as selective laser sintering (SLS), fused deposition modeling (FDM), and stereolithography (SLA), direct writing is achievable at low costs and at relatively good resolutions (typically 20-50µm). However, there is a demand for direct write technologies that can achieve even higher resolution. These demands can be met by using electrohydrodynamic jet (E-jet or EHD) printing techniques which can achieve resolutions unattainable with the more traditional printing capabilities. E-jet not only provides advantages in high resolution, but also has the capability of printing a variety of materials including nanoparticles, polymers, and bio-material suspensions onto a varied set of conducting and non-conducting substrates including bare silicon, polymers, oxides and metal films [1-6].

To enhance the appeal of E-jet for manufacturing, a multi-nozzle array holder is designed and tested for increasing the throughput of printing a single image. There are benefits to developing a multi-nozzle array (MNA) for any printing tool. One benefit is to have each nozzle in a print head print a separate but identical image and therefore increase the throughput of printing multiples of the same image. Instead of decreasing the time to print a single image, the time to print an array of images will be decreased. Another advantage to a multi-nozzle array is if the proximity of the nozzles are close, each nozzle could print different subsections of one image simultaneously therefore aiding in decreasing the time to print a large single image. However, there are many issues associated with trying to have nozzles in close proximity of each other though. The main obstacle is the electrical interference or cross talk of the nozzles while printing, which has been studied by many [7, 8]. When the nozzles are placed in too close proximity, the electric fields generated will affect the neighboring nozzles printing, and the droplets will no longer be printed directly below the nozzle orifice, but will be displaced depending on how much the electric fields interact. Simultaneous printing multiples of the same image is discussed in Chapter 2.
Further improvements to E-jet printing can be achieved by removing the need for physical nozzles to print the ink material. Increasing the number of printing nozzles by fabricating or assembling a high density array possesses many challenges and requires significant time and capital investment to develop. The ability to print from an ink film eliminates the issues associated with nozzles such as alignment, electrical crosstalk, or requirements for a high voltage power supply or amplifier, and offers the advantages of printing viscous inks, reducing material costs, and making E-jet more appealing for manufacturing applications. Successful printing from an ink film using pyroelectricity is discussed in Chapter 3 where the goal is to simply modify the printing setup to obtain a desired size droplet. Using the developed techniques of pyroelectric E-jet printing from an ink film allows for multiple droplets varying in size to be patterned on a substrate surface without having to change nozzles sizes.

1.1 Background of E-jet

The concept of electrohydrodynamic jet (E-jet) printing has been documented in literature as early as 1986 by Hayati et al. [9] but didn’t become a popular area of research until about 5 years ago. Since then, it has been a very active area of research with an increasing understanding of the physics behind e-jetting along with advancements in printing resolution, printable materials (ink and substrate), scaling, and so forth to advance E-jet from a research setting to a mass manufacturing technique.

The driving mechanism for E-jet printing is an electric field generated by a voltage difference between the conductive nozzle and the substrate. Figure 1 is a simplified schematic of the UIUC E-jet printing setup. Glass micro pipettes are coated with a gold palladium alloy, Au/Pd, and connected to a voltage source to act as the print head top electrode. A metallic chuck that supports the substrate acts as the grounded electrode.
Figure 1. Schematic of UIUC E-jet printer.

Inks to be printed are loaded into a syringe and are supplied to the pipette orifice under a small back pressure. When a positive (negative) voltage is applied to the pipette tip, the fluid at the tip meniscus is polarized with cations (anions) migrating toward the substrate and the anions (cations) traveling in the opposite direction. Due to the ion interactions (Coulombic repulsion), a positive (negative) ion concentrated pendant drop will form [10]. At a low voltage which is dependent on nozzle diameter and air gap, the pendant droplet has balanced electrostatic stress and surface tension forces and will not print. As the voltage is increased, the meniscus is deformed to become conical and can be modeled as a Taylor cone with both a normal and tangential component to the electric polarization stress due to the electric field [11, 12]. When the voltage is increased to a “jetting voltage”, the electrostatic shear stress the ions exert on the meniscus will overcome the surface tension and a droplet is released from the tip of the Taylor cone and propelled towards the substrate. If the initial jetting voltage is surpassed, the E-jet can be operated in a continuous spraying mode. The jetting voltage depends on not only the ink properties, but also the printing substrate material, the distance between the nozzle tip, and the printing substrate [12]. While printing with a DC voltage, the droplet size can be controlled by altering the pipette orifice/nozzle diameter, applied voltage, ink, and substrate materials [13].
1.2 Current Technology

The current UIUC E-jet system uses a function generator (Agilent 33220A) to generate the data signal for printing. A camera is set up to provide feedback on successful printing and is also used to set the air gap, or working distance, by viewing the reflection of the nozzle as shown in Figure 2.

![Image of nozzle, nozzle reflection, and a printed droplet from 5-axis E-jet setup.](image)

**Figure 2.** Image of nozzle, nozzle reflection, and a printed droplet from 5-axis E-jet setup.

The E-jet machine is a custom built, five degree of freedom (DOF), computer controlled substrate positioning system using Aerotech stages and a standard manual z stage for adjusting the nozzle syringe holder’s distance to the substrate. To reduce the effects of external vibrations, the entire E-jet system is assembled and operated on an isolation table. Using a LabView interface created specifically for the UIUC E-jet system, the 5-axis E-jet stage is operated either manually by moving the stage in the x and y directions or in a computer numerical control (CNC) mode. When using the CNC mode, a G-code (EIA RS274) program is generated to control the direction of stage movement in the X and Y direction, stage speed/feed rate, turning
on and off the amplifier and the dwell duration at a desired coordinate while the amplifier is on or off. A DC signal with high impedance is supplied to determine the initial jetting voltage of a specific nozzle. The initial jetting voltage is recorded and then a pulse width modulated (PWM) signal is used to print-on-demand a line of single droplets [12]. The frequency/period, pulse width/duty cycle, high/low voltages are specified using the function generator. The high and low voltage for PWM are set typically to ± 10-20V of the initial jetting voltage. Altering the specified printing parameters set by the function generator, the stage speed, and the working distance will change the size and spacing of the printed droplets.

1.3 Current E-jet Limitations

Currently, E-jet printing cannot compete with inkjet or Xerox printing when it comes to high throughput because typically only a single nozzle is used to print [1, 4, 12-15]. At UIUC, success in decreasing the single nozzle printing time of a pattern using a pulsed signal has been reported [12], but efforts must now be directed in developing either a high density nozzle print head or finding alternative means of precisely delivering small droplets using similar physics as E-jet, dispensing inks using electrostatics. Also at UIUC, there have been developments in multi-syringe holder type devices to increase the throughput of printing with E-jet, Sutanto et al. developed a single nozzle “carrousel” which allows for multiple materials to potentially be printed in the same area [4]. Multiple materials can be loaded into different syringes and the carousel can be rotated to call upon each syringe to print. This achievement allows for multiple materials to be printed with ease but does not significantly decrease the printing time of an image.

Fabrication of a high density nozzle array can be expensive if made with traditional silicon processing. There would be a high initial cost to develop a process for fabrication but could be batch fabricated later. Having the actual printing orifices fabricated from a single substrate can significantly reduce the issues of nozzle alignment. There is however the issue of cross talk that could potentially occur with a high density array with strong electric fields in close proximity of each other.
There is a minimum size droplet that can be produced for a given nozzle inner diameter. To further decrease the size of the droplet, the nozzle size would also have to be decreased. The issue of nozzle clogging becomes more apparent when decreasing the size of the nozzle. Clogging can be a result of many variables such as the humidity of the environment, the viscosity and/or vapor pressure of the ink material, and also the composition of the ink, whether it is a suspension of nanoparticles or polymer chains. For these reason, to print at high resolutions with current E-jet printing techniques, the ink selection is restricted to low viscous materials.
CHAPTER 2: MULTI-NOZZLE ARRAY PRINTHEAD

2.1 Introduction

Fabricating a high density array to compete with an inkjet printer head is challenging due to how E-jet works. Alignment of the nozzle tips and cross talk are key issues when trying to achieve uniform printing with multiple nozzles. The nozzles used in the following experiments are pre-pulled, thin walled, borosilicate glass pipettes commercially purchased from World Precision Instruments (WPI) and can vary in total length, therefore individual nozzle alignment in the print head is necessary. As previously discussed, there are many advantages to being able to print multiple nozzles simultaneously. The motivation for the work discussed in this chapter is to design an inexpensive print head which can secure nozzles in a linear array to demonstrate simultaneously printing of a pattern. The total printing time of an array of nozzles can be roughly estimated using the time to print an image with a single nozzle divided by the number of nozzles in the array. The capability of the work done on a MNA is demonstrated and later discussed by simultaneous printing a four nozzle print head made from off the shelf products and low cost custom mounting pieces.

With the desire to increase printing throughput, many issues have to be addressed in order to have successful multi-nozzle printing. The most significant factor for determining successful printing with a single nozzle is finding the correct printing voltage for the given air gap between the nozzle tip and the printing substrate. A one micron difference in air gap can change the printing voltage by 1-10V, which is enough to start or stop E-jetting, therefore alignment is the most dominant issue that must be addressed when scaling to multiple nozzles. Unlike an entirely fabricated micro-array print head, alignment of the commercially purchased nozzles must be done while building the print head. Once a print head is created, it is important to test the array to demonstrate that the print head operates identically to how a single nozzle would perform. Since the goal is to increase the throughput of printing, the nozzles must be able to print simultaneously and must have uniform droplet size across the array. By increasing the number of printing nozzles, the cost of print manufacturing can decrease so a low cost print head must be designed and produced to show this approach of increasing the number of printing nozzles is feasible.
There have been many advancements in developing a multiple nozzle E-jet delivery system to decrease production time [4, 7, 10, 16, 17]. As previously mentioned, Sutanto et al. developed a single nozzle “carrousel” which allows for multiple materials to potentially be printed in the same area. This tool advances the ability to repeatedly print multiple materials in the same spot without having to change out nozzles, but it is still single nozzle printing [4]. Lee et al. fabricated a three nozzle array using PDMS and an FR-4 substrate using standard printed circuit board processing. This array eliminates the use of glass micropipettes and reported a printed line width of 100µm or greater from a 120µm opening [10]. Choi et al. developed a five multi-nozzle array made from molding PDMS to allow for glass micropipettes to be inserted with a pitch of 3mm and the reported average line width with this array was approximately 30 µm. In Choi’s setup, copper electrodes are fed directly into the ink in each nozzle to supply voltage and each nozzle has an individual ink inlet [7, 16, 17]. However, there is currently no reported array that can achieve the density of nozzles in a print head comparable to inkjet printing.

### 2.2 Design of holders and mounts

Initially the nozzle holder, or print head, was a custom design machined out of Teflon. Teflon was chosen because it is chemically inert which allows for a set of nozzles to be secured in the holder and then later removed to reuse the holder. To keep the pitch of the nozzles small, every other nozzle required to be bent in a 90° angle to allow for enough tolerance for tubing to be secured to the ends of the nozzles. Nozzles were bent using a butane torch but it was difficult to ensure the bend was in the same location of each nozzle because the total length of each nozzle is not the same. To keep the nozzles as vertical as possible once mounted in the holder, a second Teflon holder was machined to assist in the horizontal alignment. Figure 3 shows a computer aided design (CAD) drawing cross section of the initial holder design in which the nozzles were inserted vertically through the designated holes. Alignment was done by inserting the nozzles, tip first, through the Teflon holders and allowing them to rest on a parallel PDMS spun glass slide. This was a physical alignment process which resulted in severe damage to the tips rendering then unusable for printing. In order to demonstrate the feasibility of producing a MNA, the alignment must be precise with minimal damage to the nozzles themselves so an alternative nozzle holder design was made.
The second and final print head design utilizes predefined v-groove slots for the nozzles to rest in. Figure 4 shows the design of the v-groove print head in a CAD software. The “V” spacing is defined by the desired pitch of the nozzles, which in turn is limited by the outer diameter of the pipettes (1mm) and the tubing (1mm), which supplies ink to each nozzle. By incorporating the tubing thickness and a clearance of 0.25mm on either side of the pipettes, a pitch spacing of 2.5mm will also ensure electrical isolation and eliminate cross talk while printing. Due to the small size and complicated geometry, machining the v-groove holder would be very difficult and expensive for this proof-of-concept device so it is built using 3D rapid prototyping (RP) techniques. By designing the print head in a CAD software, it is compatible with the RP machines and allows for changing the number of grooves required to be done with ease. The RP material chosen is ProtoGen O-XT 18420 white which is an ABS-like photopolymer. This material gives good accuracy in resolution but is not chemically resistant and therefore is a one-time use part. The used nozzles cannot be removed without permanent damage to the v-groove holder therefore a whole new print head with nozzles would be required to print more images.
To simplify the mounting of a small nozzle print head to the actual E-jet system, each print head has compression tabs located on either side of the part. An E-jet mount is designed to have slots allowing for the tabs to be press fitted into and is also made from RP techniques. Only one E-jet mount needs to be built for a given width of the print head. Should the number of nozzles increase, the E-jet mount will need to be adjusted to accommodate the increase or decrease in the v-groove nozzle holder width.

### 2.3 Nozzle Alignment

A camera with a high magnification lens is mounted perpendicular to an optical grating used to align the nozzles. Once the nozzles are laid into each groove, a differential adjuster fixed to a linear stage is used to push the ends of each nozzle individually to the desired line on the grating. Figure 4 shows the alignment setup assembled in a CAD software on the right. Norland Optical Adhesive (NOA74) is selected as the adhesive to secure the nozzles because it remains a liquid until cured with UV light and has low shrinkage. Due to the groove shape, each nozzle can be cured independently of the others. Although this alignment process yields much lower damage to nozzles, it is still a manual process which leaves room for error. Ideally with high precision alignment, all nozzles mounted will be printed both individually and simultaneously because they will all require the same jetting voltage. Figure 5 shows the final alignment of four nozzles in the v-groove holder with copper tape around them. Based on the optical alignment technique and using a differential adjuster, the nozzle tips are aligned to within ±1.6µm standard deviation.
2.4 Experimental Setup

The v-groove MNA print head is successful in eliminating any misalignment in the horizontal direction, out of plane, and the need for physical contact to ensure alignment. However there is the issue of alignment of each nozzle to be perpendicular to the printing substrate. In addition to the 5 DOF stages and manual z stage, the current UIUC E-jet is equipped with an additional rotating stage that is mounted to the manual z stage. To address the potential perpendicular misalignment issue while printing, the designed E-jet mount attaches to the rotating stage to ensure the nozzles are all perpendicular to the printing substrate and require roughly the same voltage to jet. Figure 6 shows the rotational stage with the E-jet mount.

Figure 5. Final v-groove nozzle print head with four secured nozzles.
Once the nozzles have been aligned and secured, the print head is press fitted into the designated slots on the E-jet mount. The nozzles are then lowered into a hydrophobic coating solution to help pin the meniscus to the inner diameter of the nozzle to aid in forming a Taylor cone and printing. The coating is a mixture of 0.1% percent weight of 1H, 1H, 2H, 2H-perfluorodecane-1-thiol (Sigma-Aldrich) to dimethylformamide (DMF) (Sigma-Aldrich) where the thiol component binds with the gold coating on the nozzles creating a monolayer of a hydrophobic carbon chain. For the stage alignment to the nozzle array, the mount is leveled to the machine itself. To confirm the alignment of the printing substrate to the nozzles, the tip and tilt of the stage is adjusted with respect to only one nozzle. If there is poor nozzle tip alignment, the working distance (air gap) is set based on the nozzle closest to the substrate. The working distance determines the printing voltage and can be at a variety of heights but the closer the nozzle is to the substrate, the lower the required printing voltage. Figure 7 is a composite of the four nozzle MNA tip (N1-N4) images from a single print head taken with the E-jet camera. The pitch spacing of the nozzles is too large to view simultaneously with the installed optics on the camera. To power each nozzle, a small alligator clip is attached to the high voltage electrode and clamped.
onto the copper tape wrapped around each nozzle. The capability to address each nozzle is important for determining which nozzle(s) will actually print and at what jetting voltages. An array of gold spring loaded pogo pins can also be installed to replace the copper tape by making a RP holder to align each pin with an Au/Pd coated nozzle. The pogo pins allow for good contact to the nozzles and by eliminating the use of tape, the risk of damaging the nozzles is also reduced.

![Composite image of the nozzles and nozzle reflections for alignment purposes for a four nozzle MNA.](image)

**Figure 7.** Composite image of the nozzles and nozzle reflections for alignment purposes for a four nozzle MNA.

### 2.5 Printing Experiments

The ink selection for all experimental printing with the MNA is NOA74. NOA74 is selected due to its good charge mobility and dielectric constant. The printing substrate for all experiments is a single side polished silicon wafer. Both 10µm and 2µm inner diameter nozzles are tested with the MNA but the printing results presented here will reflect an experiment only using 2µm nozzles. The 2µm nozzles are selected because the droplet sizes are more suitable for the production of high density droplet images for printing fine or intricate features.
After ensuring each nozzle individually successfully prints in both DC and PWM mode, simultaneous printing is achieved by connecting a single electrode to each of the functioning nozzles. The capability of printing is determined by whether or not the nozzle is broken or clogged and can be determined either visually or simply by not being able to print. The voltage required to print all four nozzle simultaneously did not vary from the jetting voltages of an individual nozzle. To demonstrate the full capability of the linear multi-nozzle array print head and the current E-jet system, a G-code was generated and used to print with all four nozzles.

The image chosen to print is the UIUC logo of an “I” and can be seen in Figure 8. As previously stated, the G-code CNC feature on the UIUC E-jet was used to print this image automatically.

![UIUC logo](image)

**Figure 8.** UIUC logo used for demonstrating simultaneous printing with the MNA.

The air gap is set to 20µm for the nozzle closest to the substrate, which is nozzle 4 (N4) in Figure 7. The initial jetting voltages and estimated air gaps can be found in Table 1. The air gap for each nozzle is approximated because they are determined with image processing and is dependent on the camera resolution. For the N4 set to a 20µm air gap, the initial jetting voltage is ~130V. N2 and N3 have roughly a 24 µm air gap requiring voltages of 136 and 141V
respectively to print. This confirms the correlation between smaller working distances requiring less voltage.

**Table 1.** Initial jetting voltages and gaps for a single printing experiment (the gaps are approximated through image processing and may not be accurate to actual distance).

<table>
<thead>
<tr>
<th>Nozzle #</th>
<th>Vertical Spacing [µm]</th>
<th>Initial Jetting Voltage [V]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>~22</td>
<td>132</td>
</tr>
<tr>
<td>2</td>
<td>~24</td>
<td>136.4</td>
</tr>
<tr>
<td>3</td>
<td>~24</td>
<td>140.8</td>
</tr>
<tr>
<td>4</td>
<td>~20</td>
<td>130.4</td>
</tr>
</tbody>
</table>

The goal for all the experiments is to have all nozzles simultaneously print by determining the optimal printing parameters for a print head with a given nozzle size and set air gap. For the purpose of this work, the desired printed image is one with a high density of uniform size printed droplets across the print head. By changing the PWM components, the effect of an individual parameter can be studied so an optimal printing signal for a given print head can be achieved. Highlighted in yellow in Table 2 are the varied PWM parameters changed from each test print. The PWM parameters were iteratively changed after each CNC print to achieve the desired results.

**Table 2.** Varying PWM parameters for each printed test run.

<table>
<thead>
<tr>
<th></th>
<th>High/Low [V]</th>
<th>Period [ms]</th>
<th>Pulse Width [ms]</th>
<th>Speed [mm/s]</th>
<th>Line Distance [µm]</th>
<th>Dwell On/Off [µs]</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>120/148</td>
<td>10</td>
<td>5</td>
<td>1</td>
<td>10</td>
<td>100/100</td>
</tr>
<tr>
<td>T2</td>
<td>120/148</td>
<td>10</td>
<td>5</td>
<td>0.7</td>
<td>10</td>
<td>50/100</td>
</tr>
<tr>
<td>T3</td>
<td>120/148</td>
<td>10</td>
<td>5</td>
<td>0.5</td>
<td>10</td>
<td>10/100</td>
</tr>
<tr>
<td>T4</td>
<td>120/148</td>
<td>10</td>
<td>5</td>
<td>0.6</td>
<td>5</td>
<td>10/100</td>
</tr>
</tbody>
</table>
| T5    | 120/148      | 10          | 5                | 0.6          | 5                  | 10/1
2.6 Results and Analysis of Simultaneous MNA Printing

From the experimental initial jetting voltages found in Table 1, the first print (T1) has a low and high voltage for simultaneous PWM of 122/148V respectively, a pulse spacing of 10ms, pulse width of 5ms, and the on and off dwell times are both 10ms. The feed rate is 1mm/s in both the x and y direction and the image line spacing in the x direction is 10µm. These parameters can also be found in Table 2, along with the five other printing tests ran (T1-T5). Figure 9 shows the printed images of an MNA with T1 parameters. There are some defects such as missing droplets and enlarged first droplets on a line. These defects can be attributed to the signal generation rather than the MNA print head because the defects are consistent across all four printed images. The only differences in the printed images between the nozzles are the droplet sizes, which is to be expected due to the different initial jetting voltages of each nozzle.

![Image](image.png)

**Figure 9.** Initial MNA simultaneous printing of UIUC logo with T1 parameters.

2.6.1 PWM Parametric Study

All five printing tests were carried out and Figure 10 shows an enlarged portion of the printed pattern for the five different experiments with modifying different PWM parameters such as dwell on time, stage speed, and line spacing. The images are taken from the same area for each nozzle printed pattern with the same number of lines. This enlarged area is where the image
analysis of consistency across the print head is done. Post processing image analysis is
performed in ImageJ to determine the variation of printed droplets between the nozzles in the
print head.

Figure 10. Enlarged area of printed pattern from the same area for each nozzle of the MNA print
head for five experiments.

T2 had a decrease in stage speed and half the on dwell time compared to T1 and resulted in
smaller initial droplets and can be seen in Figure 10. In Table 3, the standard deviation of
droplet size decreased from 1.84µm to 1.08µm for the total print head (across all four nozzles
combined). Although this decrease in standard deviation is desired, the value is still too large
given the actual droplet size is approximately 2.6µm. The large standard deviation in droplet
diameter is due to the large initial droplets: the initial droplet for T2 is approximately 7.21µm
and T3 is 5.19µm with a standard deviation of 0.54µm for just the large initial droplets of both
printed images. The initial first droplets for T1, T2, and T3 were measured to confirm the correlation of decrease in dwell on time to smaller droplets but are not presented.

**Table 3.** Average size and standard deviation of counted printed droplets from enlarged area in Figure 10

<table>
<thead>
<tr>
<th></th>
<th>N1</th>
<th>N2</th>
<th>N3</th>
<th>N4</th>
<th>Print Head</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>T1</strong></td>
<td>Average [µm]</td>
<td>2.97</td>
<td>2.34</td>
<td>3.15</td>
<td>2.68</td>
</tr>
<tr>
<td></td>
<td>Std. Dev. [µm]</td>
<td>1.72</td>
<td>1.75</td>
<td>1.89</td>
<td>2.00</td>
</tr>
<tr>
<td></td>
<td># Droplets</td>
<td>218</td>
<td>269</td>
<td>222</td>
<td>265</td>
</tr>
<tr>
<td><strong>T2</strong></td>
<td>Average [µm]</td>
<td>2.42</td>
<td>2.16</td>
<td>2.77</td>
<td>2.68</td>
</tr>
<tr>
<td></td>
<td>Std. Dev. [µm]</td>
<td>0.97</td>
<td>1.07</td>
<td>1.19</td>
<td>1.23</td>
</tr>
<tr>
<td></td>
<td># Droplets</td>
<td>312</td>
<td>327</td>
<td>259</td>
<td>269</td>
</tr>
<tr>
<td><strong>T3</strong></td>
<td>Average [µm]</td>
<td>2.63</td>
<td>2.53</td>
<td>2.86</td>
<td>2.88</td>
</tr>
<tr>
<td></td>
<td>Std. Dev. [µm]</td>
<td>0.43</td>
<td>0.49</td>
<td>0.46</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td># Droplets</td>
<td>313</td>
<td>317</td>
<td>298</td>
<td>300</td>
</tr>
<tr>
<td><strong>T4</strong></td>
<td>Average [µm]</td>
<td>2.54</td>
<td>2.48</td>
<td>2.97</td>
<td>2.96</td>
</tr>
<tr>
<td></td>
<td>Std. Dev. [µm]</td>
<td>0.46</td>
<td>0.51</td>
<td>0.57</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td># Droplets</td>
<td>615</td>
<td>640</td>
<td>576</td>
<td>564</td>
</tr>
<tr>
<td><strong>T5</strong></td>
<td>Average [µm]</td>
<td>2.30</td>
<td>2.24</td>
<td>2.72</td>
<td>2.82</td>
</tr>
<tr>
<td></td>
<td>Std. Dev. [µm]</td>
<td>0.54</td>
<td>0.63</td>
<td>0.53</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td># Droplets</td>
<td>630</td>
<td>625</td>
<td>529</td>
<td>527</td>
</tr>
</tbody>
</table>

Table 3 also has the calculated average printed droplet size and standard deviation for the measured number of droplets for each individual nozzle, N1-N4, for all five tests. By decreasing the dwell on time and slowing down the printing speed, more uniform droplets are printed. For T3 shown in Figure 10, the average droplet size is 2.75µm with a standard deviation of 0.53µm. Further decreasing both the stage speed and line spacing results in more droplets printed per line and more lines printed in a given area as seen by the T4 images. Increasing the frequency or decreasing the pulse spacing will also increase the number of droplets printed per line, but the pulse width will then need to be adjusted to maintain the same size droplets. For simplification, the speed was decreased to achieve the same result. For T5, the dwell off time was decreased to try and eliminate the issue of missed droplets. The final printed pattern for this set of experiments, T5, can be seen in Figure 11 with an average droplet size of 2.56µm and a standard deviation of 0.64µm.
2.6.2 Conclusion of MNA PWM printing

When comparing Figure 9 of the initial print (T1) to Figure 11 of the final print, the MNA demonstrates the capability of printing with all nozzles simultaneously and an optimal PWM signal was generated. As previously discussed, the initial printing setup had large first droplets, and they were eliminated using T5 printing parameters. Figure 12 is an enlarged portion of a section of the printed pattern from the initial print, T1, and the final print T5. Not all the issues of extra and skipped droplets were addressed, which can also be seen in Figure 12, but there was an overall improvement in printing, with a high density printed image.
The average droplet size was approximately 2.66µm for the entire printing session and it is not expected to change because the pulse width and voltage, which determine the droplet size, are never altered. The defects which were not corrected for in the final printing test can be attributed to the signal generation and not the MNA itself. If defects were due to the MNA, they would be present in all the printed images regardless of the PWM parameters, but that was not the case. An example of a defect in the MNA is that nozzle 3, N3, always seemed to have larger printed droplets compared to the other three nozzles, see Table 3. A possible explanation is that this nozzle was most likely either closer to the substrate or the inner diameter of the nozzle was larger than the others. It is possible that the low resolution images resulted in analysis error as well as performing the analysis after printing. T3 shows the lowest standard deviation while the higher droplet density prints, T4 and T5, had a higher standard deviation, which could be due to the image analysis software. For example, the droplets are more pronounced in T3 than in T5, so they are more easily identified as a single droplet during analysis. Ultimately, being able to tune all the PWM parameters will determine how well the pattern is printed.

From the v-groove print head spacing, the images should be 2.5mm apart, and the actual printed images are ~2.43mm apart. The small error can be attributed to either horizontal misalignment
(nozzles not seated in the v-grooves properly) or the v-groove part was not designed with sufficient tolerances to accommodate for the RP build inaccuracy. Figure 13 shows two of the UIUC logos printed using the 2µm nozzle MNA. Overall the error in spacing can be accommodated for in the future by improving the alignment setup.

![Figure 13. Printed pattern of two of the four images to display the spacing of the four MNA.](image)

Even though larger nozzles like 10µm are easier to align because they are larger and more robust, it is challenging to get simultaneous printing to occur with all four nozzles in the print head. Most failed printing occurred due to poor alignment or broken nozzles. By using a higher magnification lens, the alignment is improved and a set of 10µm nozzles printed simultaneously with initial jetting voltages averaging 360.5V and a standard deviation of 22.66V. There was still some difficulty with controlling the printing parameters, so to simplify the print and to lower the required voltages, 2µm nozzles were used. However, the design of the v-groove does not
restrict the size of the inner diameter nozzle as long as the outer diameter (OD) remains the same. This specific v-groove design is for an OD of 1mm. With the 2µm nozzles, the printing experiments which were successful for all four nozzles had much lower standard deviations like the discussed experiment. The average printing voltage was 134.9V with a standard deviation of 4.68V. Since the determination of accurate working distance is difficult, a more accurate representation of the quality of alignment is reflected by the initial jetting voltages. Nozzles being damaged or having poor alignment was and is still an issue when making the proposed multi-nozzle print head.

2.7 Cost Analysis of V-groove MNA

A simple cost analysis of the MNA print head can done based on the work completed thus far. The RP machined used to build the v-groove holder is a Viper SLA Project 6000 Professional 3D Printer with a printing envelope of 240x240x240mm. The ProtoGen O-XT 18420 material costs $0.35/gram and the final v-groove piece weighs 0.7 grams resulting in a unit cost of $0.25. The v-groove holder is printed vertically with dimensions of 14x25x3.25mm so with a 4mm clearance around each part, up to 441 four nozzle v-groove units could be built in one print/build session. Using RP technologies allows for the ease of altering the v-groove holder for a custom print head. If the width of the print head is to change, a new E-jet mount will also need to be constructed. For the experiment discussed above, the E-jet mount is 70x120x7mm, so if this part is built on the same RP platform as the v-groove units, potentially 371 v-grooves could be built. Each glass micro-pipette nozzle from WPI costs $12.50 and has to be coated in Au/Pd to be conductive. The metal deposition is done using a sputtering tool and costs $10.54/min including overhead for the material. Up to 25 glass nozzles can fit on the sputtering platform and are typically coated for 120secs, resulting in approximately 12nm of deposited Au/Pd. An amount of $5 includes all other materials such as the adhesive, tubing, luer locks and the copper tape used for a print head. The unit cost of one four nozzle print head based on this bill of materials is approximately $60.

Using a photopolymer material and the NOA adhesive, the current v-groove holder is a one-time use holder. The lifetime of the MNA is difficult to determine due to the numerous factors that can affect printing. The greatest factor in ceasing the ability to print is nozzle clogging. Ink
material properties that are suspensions of nano-particles, highly viscous, high vapor pressure, or high surface tension can cause clogging after a short time of printing, less than two hours. The printing environment can also affect nozzle clogging. For example, it has been observed that nozzles have a tendency to clog faster if the humidity is low. The hydrophobic coating that is applied to the nozzles is not a permanent coating and the effects can be reduced over time which could lead to failure of the print head. For the presented experiments, using NOA as an ink, the total printing time was approximately five hours with no signs of nozzle clogging or variation in printing.

With the current alignment setup, it takes roughly 45-60 minutes to align and secure four nozzles. The RP build time based on university resources will be approximately three hours which includes the pre-processing, printing and post processing steps. If the manufacturer of the print head were to pay an employee the federal minimum wage reported by the US Dept. of Labor of $7.25, then the fabrication of a four MNA print head would cost minimum of $90. This total does not include the cost of the equipment such as the lens, camera, and computer required for the alignment process or the cost of the RP machine itself.

RP manufacturing parts is useful when making custom print heads or a few thousand a year, but if the number of print heads demanded were to increase, plastic injection molding would have to be used. For the purpose of mass production, aPrioir software is used to perform a cost analysis based on injection molding 500,000 units/year. There is a large capital investment for the hard tooling, but if ABS is to be used as the material, the unit price for an injection molding the v-groove holder is $0.16. This price per unit takes into account variable costs such as labor, material, and overhead, unlike the simplified cost analysis performed for using RP machines to produce the v-groove holders.
CHAPTER 3: NOZZLE-LESS PRINTING

3.1 Introduction

Electrohydrodynamic jet printing has the capability of enhancing printing capabilities in manufacturing processes, but many issues still remain. In the previous chapter, the issue of throughput was addressed by developing a print head that increases the number of nozzles available to print. The alignment issues during the fabrication of the print head with commercially purchased nozzles proved to be a significant challenge. If the alignment issue of the physical nozzles could be completely eliminated, the appeal of E-jet for manufacturing would increase dramatically. In this chapter, research has been done to print small droplets from a thin liquid film using similar physics as E-jet. With E-jet printing, an electric field is used as the mechanism for printing which essentially causes small quantities of ions within a liquid to accumulate and print resulting in much smaller droplets compared to inkjet. The goal is to still use of an electric field and the formation of a Taylor cone to print small droplets consistently on a substrate but find an alternative way of delivering ink to the system.

There are many advantages to eliminating the need for physical nozzles for E-jet printing. As previously mentioned, eliminating the need for nozzles will eliminate the alignment issues of multiple printing orifices. With the proposed setup, the only alignment required is between two stationary parallel surfaces. Further, the electric field is locally generated, eliminating the need for a high voltage power supply or amplifier. This technique may have a higher capital cost but may have a lower consumables’ cost as one does not need to prepare printing nozzles. Further, the issue of nozzle clogging is completely eliminated. If the ink has changed in material properties over a printing session, the ink film only needs to be replaced or refreshed to continue printing. With typical nozzle E-jetting, either new nozzles or a quick way of cleaning the nozzles without damaging them is required to carry on printing. With the potential advantages of printing from an ink film, E-jet can become more attractive for common manufacturing processes such as roll-to-roll printing. The desired final setup used will be similar to Xerox printing with rollers, continuous ink supply, and the ability to print at any location on a substrate.
The initial goal for printing from an ink film is to prove that it can actually be achieved. This study focuses on using pyroelectricity as a high voltage source to pull droplets from an ink film. There are many advantages to using pyroelectrics such as the fast response time, operation in a wide range of temperatures, and relatively low cost [18]. After demonstrating feasibility, the problem of controlling the size of the droplet is addressed. The desired droplet size was determined after initial experiments were conducted to know what an achievable range is when using pyroelectrics. The current desired diameter of droplets for printing from an ink film using pyroelectricity is 500µm or smaller.

### 3.2 Pyroelectricity

#### 3.2.1 Background and History

Pyroelectricity is the conversion of thermal flux to an electrical quantity, such as current or voltage, and is often described in a vector form [19]. The first documented observation of the pyroelectric phenomenon can be traced back all the way to the late 4th to early 3rd century BC [20]. The observation of a lyngourion stone being charged to electrostatically attract materials like straw and metals when it was heated is documented in a treatise titled “On Stones” [18, 20]. The author of the treatise is unknown but the material content has been credited to the Greek philosopher Theophrastus [20]. It wasn’t until 1717 when the chemist and physician Louis Lemery first wrote a scientific paper on the electrostatic phenomena of pyroelectricity. Throughout the 18th and 19th centuries, there was much advancement made in both pyroelectricity and the field of electrostatics using a stone called tourmaline. The term “pyroelectricity” wasn’t first used until 1824 when David Brewster defined the phenomena as such. After the electrometer was developed in 1859, John Mothée Gaugain made the first precise measurement of the charges generated from a pyroelectric material. However, when ferroelectricity was discovered in 1920, all interests and efforts in the research of pyroelectricity nearly disappeared until 1938 when Yeou Ta reignited the field by publishing a paper with the application of pyroelectric materials in IR sensors [18]. In 1974, Rosenblum et al. were the first to study the pyroelectric effect in materials by observing the “thermally stimulated electron emissions” or “thermally stimulated field emission” (TSFE), from lithium niobate [21, 22]
3.2.2 Physics of Pyroelectricity

Pyroelectricity is the reversible coupling of mechanical, electrical and thermal responses. In the discussed work, the relationships between thermal and electrical properties are studied while any mechanical responses are neglected. Figure 14 is adapted from Yuan et al. published work to graphically explain the relationship of pyroelectrics and is found in many other references [18, 23]. The green highlighted path represents the primary pyroelectric coefficient and the red dashed path is representative of the secondary pyroelectric coefficient. These two coefficients are discussed in the later in this section.

![Graphical representation of relationship between thermal, mechanical, and electrical properties with the primary and secondary pyroelectric coefficients highlighted](image)

**Figure 14.** Graphical representation of relationship between thermal, mechanical, and electrical properties with the primary and secondary pyroelectric coefficients highlighted [23].

The resultant charge that is generated due to a thermal change is proportional to the cross sectional area of the crystal [18]. The generation of charge is due to the asymmetric interaction of the atoms within the pyroelectric material [18, 19, 24]. For example, in the material lithium niobate, the cations (lithium and niobium) shift relative to anions (oxygen) to the “centre of gravity” of the unit cell [18, 24]. This leads to a horizontal alignment of the electron dipole moments or spontaneous polarization, $P_s$, when the material is heated (or cooled) which causes a
surface charge density to build up on one of the material surfaces. If the material is immediately cooled (heated), then the charge will essentially go to zero because equal amounts of charge is generated when cooled but with the reverse sign than when heated [18, 21].

The pyroelectric effect is only present if the temperature change applied on the material is below the Curie temperature. The Curie temperature, $T_c$, is the transition temperature of a material at which the spontaneous polarization will approach zero [24]. For most single crystals and ceramics, increasing the temperature decreases the spontaneous polarization due to the decrease in the charge bound at the surface. The redistribution of the free charges then tries to compensate for this depletion of charge at the surface causing current to flow [18]. Rosenblum et al. compared what occurs when a pyroelectric material is continuously heated to a gas discharge by field ionization of a residual gas or the environment. Before the steady-state equilibrium of electron field emission is reached, there is field ionization. The discharge will neutralize most of the surface charge and reduce the electric field but if heat is continuously supplied to the system, a periodic process will repeat [21].

The spontaneous polarization is not only dependent on the temperature change of the material, but also the pyroelectric coefficient, $\gamma$, which is an experimentally determined material constant. The relationship of the change of spontaneous polarization with respect to heating time and pyroelectric coefficient at constant electric field and elastic stress is [19, 24].

$$\Delta P_{s,i} = \gamma_i \cdot \Delta T$$ in a 3D space (i=1,2,3) \hspace{1cm} (3.1)

The pyroelectric coefficient is an experimentally determined number so depending on the setup, material properties, and assumptions made, this value can vary but not by a significant amount. Typically the value of the pyroelectric coefficient reported is the total effect, $\gamma_i^{\rho}$, which is the sum of the primary and secondary coefficients at constant pressure or stress as seen in Equation 3.2 [19, 24].

$$\gamma_i^{\rho} = \gamma_i^{e} + d_{ijk}^{T} e_{jklm}^{T} \alpha_{lm}^{E}$$ \hspace{1cm} (3.2)
The primary pyroelectric coefficient, \( \gamma_f \), is measured at constant strain and the secondary pyroelectric coefficient is calculated by the piezoelectric, \( d \), elastic stiffness, \( c \), and thermal expansion, \( \alpha \), coefficients which result from thermal deformation at a given temperature. Typically, if the pyroelectric material is free and not fixed, the secondary pyroelectric effect can be neglected. However, if just a small area of the entire crystal is heated, then the bulk of the material will essentially act as a clamp and the secondary effect must be considered [24]. There is also a tertiary coefficient in which a change in temperature generates shear stress in the material, but this coefficient is commonly neglected when discussing the total pyroelectric coefficient due to the assumption of constant stress. The tertiary coefficient is also highly depended on the experimental setup so the value is not commonly reported [19, 24].

### 3.2.3 Materials and Applications

Due to the coupling of mechanical, thermal, and electrical properties, all pyroelectric materials are piezoelectric, but the reverse is not true [24]. Lang *et al.* explains that there are three criteria that must be met for a material to be considered pyroelectric. “The molecular structure must have a nonzero dipole moment, the material must have no center of symmetry, and lastly, the material must have either no axis of rotational symmetry or a single axis of rotational symmetry that is not included in an inversion axis” [18]. As previously mentioned, only materials with a polar point group symmetry, a dipole moment, can exhibit the pyroelectric effect [18, 19, 24]. Of the 32 point symmetry crystalline groups, only 10 have vectorial polarization. These groups are 1, 2, m, 2mm, 4, 4mm, 3, 3m, 6, and 6mm [24]. There are also ionic ceramics and crystalline or covalently bonded polymers that are considered pyroelectric materials, but they have less possibilities of polar axis which will lower the spontaneous polarization and in turn the pyroelectric coefficient because of the relationship derived in Equation 3.1 [24].

Pyroelectric materials can be categorized as non-ferroelectrics or ferroelectric. Non-ferroelectric pyroelectric materials are those that the polarization cannot be switched with an applied electric field while ferroelectrics polarizes with an external electric field [19]. Ferroelectrics are more temperature dependent and polarization occurs at temperatures below the Curie temperature. If the temperature exceeds the material Curie temperature, then the element will behave like a non-ferroelectric material and lose the ability to spontaneous polarize. Ferroelectrics also are more
dominated by the primary pyroelectric coefficient which is typically negative because of the spontaneous polarization decreasing with increasing temperature [19].

Some common and favorable pyroelectric materials for applications are triglycine sulfate (TGS) due to its high pyroelectric coefficient, \(550 \frac{\mu C}{m^2 K}\) [19], and low permittivity [18]. Lithium tantalate (LT) and lithium niobate (LN) also have high pyroelectric coefficients, -176 to -230 \(\frac{\mu C}{m^2 K}\) and -83 \(\frac{\mu C}{m^2 K}\) respectively, and can be used in high temperature applications due to their high Curie temperatures [19]. There are also polymers that exhibit the pyroelectric effect, like polyvinylidene fluoride (PVDF). Even though PVDF has a very low Curie temperature, the low thermal conductivity and dielectric constant, along with a pyroelectric coefficient of 27 \(\frac{\mu C}{m^2 K}\) [19], can be desirable if used in the appropriate application. For ceramic materials, lead zirconate titanate (PZT) is the most commonly used due to its robustness in both mechanical and chemical applications. PZT is a unique material in that changing the component ratio, specifically the zirconium and titanium, not only alters the mechanical properties but also the pyroelectric coefficient to give a possible range of -50 to -268.0 \(\frac{\mu C}{m^2 K}\) [18].

Currently, the use of pyroelectric materials can be found in infrared detectors, vidicons, point detectors, and fast pulse detectors [19, 24]. They can also be used as sensors for electromagnetic radiation and thermal imaging systems [24]. There have been many applications of pyroelectrics in sensors but none yet in a printing manufacturing process which is what is motivating the presented research.

3.3 Experimental Setup

Drawing analogies to E-jetting, the high voltage source is the pyroelectric material, the printing substrate will be glass, and the ink used, NOA74, will be the same as the MNA experiments. The setup is simple with ink spun onto a piece of cover glass in order to control the thickness. Glass spacers are used to set the air gap between the ink and the glass printing substrate. On top of the printing substrate, the pyroelectric material is placed to be in direct contact with the heat source. Unless specifically mentioned, all pyroelectric material thickness is 500µm and all ink
thickness is approximately 20µm. Figure 15 is a simplified schematic of the pyroelectric electrohydrodynamic jet printing and an actual image of the setup to show the printing stack. Grounded copper tape is placed along the perimeters of the bottom substrate surface where the ink is spun. Varying the different parameters of the setup, the heat source material, and the pyroelectric material are studied and discussed in this chapter.

![Figure 15. Ink film pyroelectric E-jet printing schematic (top) and actual image of setup (bottom).](image)

For the initial experiments, the above described printing stack was mounted on the same 5-axis E-jet machine in Chapter 2, and a horizontal camera was used to view printing. The goal was to not only view the droplets being printed, but also see the profile of the deformed ink to see if a Taylor cone profile is generated. However, due to the small desired size of droplets and the quick rate of printing using E-jet, this camera placement and capture rate were not ideal to view actual printing of small droplets. Instead a top-down camera is implemented in later experiments to view through the pyroelectric material and printing substrate to give feedback of successful
printing and also to measure the size of the printed droplet in real time. The printing setup in Figure 15 is altered during experiments and the modified schematics will be further discussed in the following sections. All the reported diameters are estimated based on optical microscope measurements or from post image processing.

3.4 Experiments with Heat source

As previously explained, pyroelectric materials generate a temporary charge when a temperature difference is applied to the material surface. For this work, an effective heat source had to be found to produce consistent printing results. The desire for this work was to not only achieve printing of small droplets less than 500µm in diameter, but also the heat source had to be able to print droplets with high throughput to be implanted in a manufacturing printing process similar to Xerox printing.

3.4.1 Soldering Iron

To begin the work of using pyroelectricity to print from an ink film, lithium niobate (LN) was chosen as the pyroelectric material due to its commercial availability and moderate pyroelectric coefficient. There has also been some work done with a LN crystal by Ferraro et al. who demonstrated using lithium niobate to print ink from a small liquid reservoir and studied the wavelength of a ink film with respect to film thickness and working distance [25]. The initial heat source chosen was a variable temperature soldering iron to determine if the LN crystal could in fact generate enough surface charge to print. The printing stack can be seen in Figure 15 with the spacer thickness of 500µm and an ink thickness of approximately 20µm resulting in an air gap of 450µm. The soldering iron was set to 450°F and was allowed to rest directly on the LN crystal. Table 4 has the average printed droplet diameters corresponding to Figure 16 which shows a composite of many single droplets that were printed. Droplets A1-A3 and B1-B3 are printed with the soldering iron set to 450°F and droplet C1 is printed at 500°F. As can be seen in Figure 16 and noted in Table 4, the droplet diameters varied and were not consistent. There also were many satellite droplets which are not desired and are an indication of too much voltage being applied and not quick enough dissipation of the charges carried in the droplet.
Table 4. Average size droplets printed with a soldering iron as the heat source and LN crystal

<table>
<thead>
<tr>
<th>Droplet</th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>C1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avg. Diameter [µm]</td>
<td>524.8</td>
<td>443.9</td>
<td>441.3</td>
<td>576.8</td>
<td>437.8</td>
<td>367.6</td>
<td>354.7</td>
</tr>
</tbody>
</table>

Figure 16. Composite of printed droplets from experiments with a soldering iron heat source and LN crystal.

The initial experiments with the soldering iron confirmed successful pyroelectric E-jet printing from a liquid film using the LN crystal. The variation in size is most likely due to the inability to hold the soldering iron at a single point without deflecting the printing substrate toward the ink. With the weight of the soldering iron resting on the glass, the air gap was decreased and
therefore the results could not be reliably replicated. A more controlled method of resting a heat source onto the LN crystal had to be developed in order to print consistently size droplets and to decrease the heat spot size by decreasing the heat tip diameter.

**3.4.2 Resistive Heater Coil**

Since the application of the soldering iron to the crystal is very difficult, a resistive heater coil was made using nichrome wire and wrapping it around a small diameter steal metal tube. A 22 gage stainless steel (SS) hypodermic needle is then inserted into the metal sleeve. As current passes through the wire, there will be joule heating, and in turn, the needle becomes heated which will now act as the heat source. By mounting the heater sleeve on the current E-jet 5-axis system, the heat source can be lowered onto the crystal to allow for better control over location. Additionally, there is less force exerted on the printing substrate because the SS needle is small and is not fixed in the sleeve. Figure 17 shows the schematic of pyroelectric printing with lithium niobate and the resistive heater coil. The SS needle was filed to have a larger contact area to the LN crystal and is approximately 74µm wide.

![Resistive heater coil printing setup.](image)

Even with better control of the heat source placement, printing was not consistent. Controlling the heat to the LN crystal was challenging because the heater sleeve is the source of heat, so the heat applied to the crystal is more dominated by the distance of the heater sleeve to the crystal,
δy, rather than the heat in the SS needle tip. Many tests resulted in a liquid bridge, seen in Figure 18, instead of a printed droplet from the liquid steaming or Taylor cone regime. When a liquid bridge formed, there was no droplet printed and the ink did not relax to its original profile.

Figure 18. Image of liquid bridge formed with SS resistive heater coil instead of a printed droplet.

Figure 19 shows a still frame from a video of printing with the heater coil and LN as the pyroelectric material. As seen in the image, the ink deformation profile is large and a printed droplet is seen in the reflection in the ink film. The printing occurs too quickly to see if a Taylor cone is formed. A high speed camera is required to view the droplet being ejected from the deformed ink apex because it occurs in less than 1ms. These experiments showed that if the ink behaves in the liquid streaming regime, the ink film will relax nearly immediately after a droplet is printed. If a liquid bridge has formed and made contact to the printing substrate, then the ink will not relax to its original thickness. The ink deformation is expected to relax due to the physics of pyroelectricity. When temperature is applied, there is a buildup of charge generated on the surface and the crystal behaves similar to a capacitor. The capacitor, or in this case the LN crystal, will build up charge and once the circuit is closed, the capacitor or surface charge, will be drained. For the specific example captured in Figure 19, this occurs roughly 300ms after the droplet reflection is observed. During the 300ms after the droplet reflection appears, is
difficult to tell if there were multiple droplets being printed or just a single one. Only one droplet is expected to print, but the temperature is still changing due to the applied heat source so there is a possibility that multiple droplets have printed which could result in larger diameters and in some instances, a liquid bridge.

![Image of ink film deformation profile while printing and a printed droplet is seen in the ink reflection.](image)

**Figure 19.** Image of ink film deformation profile while printing and a printed droplet is seen in the ink reflection.

In Figure 20, increasing the temperature of the SS needle would sometimes result in the droplet diameter increasing, but this trend was not consistent across experiments. The feedback of successful printing was done visually, and once a droplet was printed, the heat source was removed. However, removing the heat source was not an efficient or consistent way of printing, and the application of heat needs to be more controlled with respect to time. Although the use of a soldering iron and resistive heater coil produced the desired outcome of printing a droplet, a more controlled method of supplying heat in a localized manner had to be developed.
3.4.3 Near Infrared (NIR) Laser Diode

To better control the duration of heating the pyroelectric crystal, a 805nm near infrared LED laser was used as the heating source. Using a laser eliminates the need for physical or conductive heat transfer to the crystal, allows for precise control of the power of the heating element, and also reduces the applied heat time down to milliseconds. Both pulsed and continuous laser settings were tested to determine which would result in smaller droplets. Two laser power settings were used, 20A and 10A which are approximately 8W and 3.16W respectively. Now that the heat source is the conversion of power/intensity to heat, the wavelength of the light has to be absorbed by either the pyroelectric material directly, or by an absorber. Two of the pyroelectric materials used in this work, lithium niobate and lithium tantalate, are fully transparent in the NIR range so a NIR absorbers was required in order to convert the laser energy to heat to generate the temperature gradient on the crystal surface. All the following experiments were done using the NIR laser and discussed in depth in the following sections.

3.5 Experiments with Infrared Absorber

As stated in the previous Section 3.4.3, the laser used in these experiments has a wavelength of 805nm so a NIR absorber had to be used when using the LN and LT pyroelectric crystals.
Initially used to absorb the laser power, a NIR organic dye (5768 Epolight Company) was mixed in PDMS to control the concentration. Micron size silicon dies were also used to absorb the laser and both methods are discussed in this section. Lithium niobate was the pyroelectric crystal material chosen for all the experiments in this section unless if otherwise mentioned. In theory, if enough power and heat is applied to the pyroelectric crystal, then the crystal will generate enough charge to print. However, if the required parameters were at or beyond the limits of the laser and optics used, then the setup was deemed to be unsuccessful for printing.

### 3.5.1 NIR Absorbing Dye with PDMS

PDMS (Dow Corning Corp. Sylgard 184) was chosen as a material to mix with the NIR dye so that different parameters that affect the amount of absorption of the NIR laser could be controlled. Once the NIR dye was diluted in toluene, it was mixed with PDMS (1:5 ratio of curing agent to polymer base), spun onto cover glass and cured. The cured modified PDMS, mPDMS, is placed directly onto the lithium niobate crystal surface to have good thermal contact between the two surfaces. Figure 21 shows a schematic of the printing stack with the mPDMS in green directly on top of the pyroelectric material. For each experiment, the printing stack does not change when modifying experimental parameters so that altering a variable, such as heat source, can be evaluated for its effect on the printing process.

![Schematic of NIR dye mixed with PDMS as NIR laser absorbing material for pyroelectric E-jet printing.](image)

**Figure 21.** Schematic of NIR dye mixed with PDMS as NIR laser absorbing material for pyroelectric E-jet printing.
Initially, the laser parameters are 20A, 2Hz, 100ms pulse width, and an air gap of approximately 576µm because four cover glasses were used as spacers. The PDMS was mixed with a 3:1 ratio with the NIR dye in solution. Once spun, the resultant thickness for the mPDMS was 65µm and for testing, the mPDMS is still attached to the glass slide it was spun and cured on. Copper tape was placed directly under the ink to ground the ink itself but undesired secondary droplets were printed. These secondary droplets were believed to be attributed to the reflection of the laser off the Cu surface and heating another location on the crystal. This is the reason for placing the copper tape around the perimeter of the stack to ground the base rather than under the ink. Table 5. has the printing results of how the pulse width, number of pulses, and the laser power were changed using this setup and if a droplet was printed successfully. If there were successful droplets printed, the approximate diameter of the droplets was also recorded. It can be seen that printing could only be achieved at higher powers with the number of pulses to print varying. The number of pulses and the size of the droplets may be related as noted in Table 5 and Figure 22, but that relationship would require further testing in order to be validated.

Table 5. Parameters for pulsed laser pyroelectric E-jet printing and droplet diameters of successful prints.

<table>
<thead>
<tr>
<th>Test #</th>
<th>Current [A]</th>
<th>Pulse Width [ms]</th>
<th># of Pulses</th>
<th>Average Droplet Diameter [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>10</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>50</td>
<td>1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>100</td>
<td>1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>100</td>
<td>1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>20</td>
<td>100</td>
<td>6-7</td>
<td>826.82</td>
</tr>
<tr>
<td>8</td>
<td>50</td>
<td>10</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>100</td>
<td>10</td>
<td>882.81</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>100</td>
<td>11-12</td>
<td>911.52</td>
<td></td>
</tr>
</tbody>
</table>
In an effort to try and reduce the size of the droplets while maintaining the desired circular shape, the spacer thickness was changed to three cover glass slips or approximately 435µm thick, which results in an air gap of approximately 227µm. This air gap was kept constant for all experiments discussed in this thesis unless specified. The laser parameters were kept constant at 20A, 2Hz and 100ms pulse widths. Table 6 shows selected mPDMS and NIR laser printing experimental results for pyroelectric printing with modified PMDS. Test A, in Figure 23, shows multiple droplets printed, which occurred once the glass slide with the mPDMS was removed. Multiple droplets being printed with the laser center at a set position was an undesired effect, but the droplet size did decrease to about 400-450µm as noted in Table 6. Another possible cause of multiple droplets is the uniformity of NIR dye in the mPDMS within the laser spot size (approximately 600µm). With Test B, it was noticed that even after the laser had been pulsed, there was always a secondary droplet being printed in the same general location. As mentioned earlier, once the copper tape was removed from under the ink and moved to the perimeter, the secondary droplet was eliminated. The unpredictable printing can be seen in Figure 23 because there was no change between the actual mPDMS between Test A and Test B, yet the results are very different.
Table 6. mPDMS printing experiments with the pulsed laser.

<table>
<thead>
<tr>
<th>Droplet</th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>A4</th>
<th>B1</th>
<th>B2</th>
</tr>
</thead>
<tbody>
<tr>
<td># Pulses</td>
<td>6</td>
<td>6-7</td>
<td>6-7</td>
<td>6-8</td>
<td>9</td>
<td>-</td>
</tr>
<tr>
<td>Avg. Diameter [µm]</td>
<td>401.04±10</td>
<td>450.52±26</td>
<td>392.15±45</td>
<td>402.34±40</td>
<td>432.29±10</td>
<td>402.13±6</td>
</tr>
</tbody>
</table>

Figure 23. mPDMS pulsed laser printing experiments with no glass on the mPDMS and Cu tape under entire ink resulting in multiple printed droplets from Table 6.

Additional experiment achieved better control over printing by using a continuous laser rather than a pulsed laser source. To determine the time required to achieve printing with the mPDMS, the laser power was lowered to 10A and was left on until printing occurred. Using the 65µm mPDMS with a ratio of 4:1 to NIR dye, a letter “I”, as seen in Figure 24, was printed to demonstrate control over droplet placement, printing time, and size. The droplets were printed with 1mm pitch spacing.
A more detailed analysis was performed with the printed “I” image compared to the previously discussed results of a single droplet. Since the ink used is NOA74, the droplets were cured with a UV light source so the size and volume of each droplet could be measured using a profilometer (KLA-Tencor Alpha-step IQ). Table 7 has the height and width of all eight printed droplets that form the “I”. The average height of the pattern was 38.52µm with a standard deviation of 3.36µm, and the average diameter was 446.22µm with a standard deviation of 20.86µm. The volume of the droplets was also calculated using a spherical cap model, and the average volume was 12.12nL with a standard deviation of 1.78nL.
Table 7. Printed I with mPDMS and NIR laser droplet analysis.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Height</td>
<td>Width</td>
<td>Height</td>
<td>Width</td>
<td>Height</td>
</tr>
<tr>
<td>1</td>
<td>40.30</td>
<td>444.02</td>
<td>39.49</td>
<td>425.68</td>
<td>39.90</td>
</tr>
<tr>
<td>2</td>
<td>40.26</td>
<td>472.97</td>
<td>40.04</td>
<td>444.50</td>
<td>40.15</td>
</tr>
<tr>
<td>3</td>
<td>34.90</td>
<td>444.02</td>
<td>33.49</td>
<td>412.64</td>
<td>34.20</td>
</tr>
<tr>
<td>4</td>
<td>41.43</td>
<td>492.28</td>
<td>39.87</td>
<td>489.39</td>
<td>40.65</td>
</tr>
<tr>
<td>5</td>
<td>32.61</td>
<td>426.64</td>
<td>32.04</td>
<td>458.98</td>
<td>32.33</td>
</tr>
<tr>
<td>6</td>
<td>43.25</td>
<td>432.92</td>
<td>40.17</td>
<td>427.12</td>
<td>41.71</td>
</tr>
<tr>
<td>7</td>
<td>39.92</td>
<td>453.67</td>
<td>38.66</td>
<td>447.39</td>
<td>39.29</td>
</tr>
<tr>
<td>8</td>
<td>39.50</td>
<td>434.36</td>
<td>40.34</td>
<td>432.92</td>
<td>39.92</td>
</tr>
</tbody>
</table>

Figure 23 and also from the Table 7 show that the droplets are fairly uniform but some have satellite droplets. Since the mPDMS is a mixture with the NIR dye, the dye may not have been uniformly distributed throughout the entire mPDMS mixture, and there may be locations in the mPDMS that require lower power or pulse width to achieve enough heating to print a droplet. This “extra” heat will generate more charge which can result in satellite droplets.

After successfully printing droplets in a controlled manner with a continuous laser source, a printed line was attempted. Figure 25 shows images of two attempts to print a continuous line by moving the laser at 100µm/s which resulted in droplets. Unfortunately, a continuous line was not achieved because when the ink makes contact with the pyroelectric material, it essentially removes all the surface charge that was generated which causes the ink to relax resulting in a single droplet, see Figure 19. Attempts at printing lines showed that even if the first droplet was printed, the laser continued to stay on while it was moving. By having the laser on and moving locations on the mPDMS, there was enough charge built up along the crystal which caused another droplet to print quickly but also very close to the previously printed droplet. Printing was faster most likely due to the ink film profile not relaxing completely, therefore the air gap is smaller requiring less voltage to print. Although a continuous line was not achieved, it can be seen that the spacing between printed droplets can be less than 1mm, and in these experiments, a 500µm pitch was achieved.
To further attempt to decrease the size of the printed droplets, the mPDMS thickness and ratio of the dye was altered. Having thicker mPDMS would result in more dye in a given volume, so it was expected that it would absorb more of the laser power and therefore generate more heat. The ratio of curing agent to base ratio was not altered for all experiments done with mPDMS. Two different ratios of NIR dye and PDMS (1:4 and 1:2) were mixed and spun at two different speeds (300 and 500 RPM). The four different combinations of NIR dye and spin speeds are listed as A-D in Table 8 with the measured thickness of the resultant mPDMS. Increasing the amount of NIR dye in the PDMS resulted in thinner, less viscous mPDMS because of the toluene.

Table 8. Different mPDMS ratios and thickness, labeled A – D.

<table>
<thead>
<tr>
<th>Ratio NIR dye:PDMS</th>
<th>Spin Speed [rpm]</th>
<th>mPDMS thickness [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1:4</td>
<td>300</td>
</tr>
<tr>
<td>B</td>
<td>1:4</td>
<td>500</td>
</tr>
<tr>
<td>C</td>
<td>1:2</td>
<td>300</td>
</tr>
<tr>
<td>D</td>
<td>1:2</td>
<td>500</td>
</tr>
</tbody>
</table>

Out of the four mixtures, only A and C resulted in successful printing. It was concluded that for a given amount of NIR dye mixed into the PDMS, there is a minimum thickness required for
enough heat to be generated through absorbance of the laser. For mPDMS A, droplets were consistently printed with 10A and a 3.5s laser pulse. Images of the droplets can be found in Figure 26. The droplets in Figure 26 are not perfectly circular, but the diameters are similar at approximately 567.87µm and 590.71µm for droplets A1 and A2, respectively. For mPDMS C, droplets were printed with 10A and a 2.5s laser pulse. These droplets were slightly smaller, C1 and C2 have diameters of approximately 468µm and 508.47µm. Even though mPDMS C is significantly thinner than mPDMS A, the printing results are similar with respect to the printing time and the size of droplet. Further experimentation and study will need to be done to determine the optimal mPDMS ratio of dye and PDMS and the thickness to achieve the smallest size droplets.

![Figure 26. Composite of printed droplets from experiments with varying PDMS to NIR dye ratios A (top) and C (bottom).]

Using the mPDMS allows for a droplet to be printed essentially anywhere on the crystal surface because the entirety of the mPDMS should absorb exactly the same amount. This allows for any pattern to be printed because there is no restriction to where the laser can strike the surface and generate heat to induce pyroelectricity. However, some damage on the mPDMS where the laser strikes prevents that exact location to be used again for printing. Figure 27 shows the damage in
the mPDMS while printing with the NIR laser diode, which corresponds to the laser spot size of approximately 600µm. It can be seen in the right image in Figure 27 that the center of the laser spot and the droplet center were not always aligned with up to a potential 200µm center offset.

![Image of mPDMS damage and misaligned printed droplet](image)

**Figure 27.** mPDMS damage (left) and misaligned printed droplet to mPDMS damage (right) due to laser printing.

### 3.5.2 NIR Dye

From the previously discussed experiments of mixing a near infrared absorbing dye with PDMS, the idea of placing the NIR dye directly on the crystal was explored. Figure 28 shows an image of the NIR dye on the lithium niobate crystal on the left. The required pulse of the laser to achieve printing was found similarly to the previous experiments. Small pulse times were tested ranging from 1ms to 300ms in intervals of 100ms. Droplets were printed at 200ms and 300ms but as can be seen in Figure 28, there was no control over the location of the droplet, there were significant amounts of satellite droplets. Also seen in Figure 28, the LN crystal sustained irreversible damage and the dye is completely used. This application of the NIR material for absorbing the laser was not further explored.
3.5.3 Silicon Micro Dies

Although using mPDMS offers the advantage of printing essentially anywhere within the area of the crystal, the irreversible damage due to printing and possible non-uniformity of the NIR dye in the mPDMS was less than ideal. Within the NanoCEMMS center, research is currently being done with transfer printers printing a variety of prefabricated structures and micro silicon squares, referred to as silicon inks [23, 26, 27]. Silicon is an excellent absorber of infrared light so a variety of sizes of Si µ-squares were printed directly onto one of the pyroelectric material surfaces to act as the heat source. Figure 29 shows a schematic of pyroelectric E-jet printing using the NIR laser and Si µ-squares. The hope for using Si µ-squares is to use an IR absorbing material that will not be damaged and can be used for multiple prints. Additionally, the heat affected zone is expected to be much smaller because when using the mPDMS, the entire 600 µm laser spot diameter was absorbed by the mPDMS and therefore creating a large heating spot on the crystal. By using these small Si µ-squares, the spot size for heating the pyroelectric material will be limited to the surface area of the Si µ-squares themselves when using pyroelectric crystal materials that do not absorb in the IR range.
Four different size Si µ-squares were fabricated and tested, 200x200x10µm, 100x100x10µm, 100x100x3µm, 50x50x3µm. Thicker inks were also fabricated but could not be printed successfully on the pyroelectric crystal for testing. Through experimentation, it was discovered that only the 200x200 squares could successful absorb enough NIR laser to generate the required amount of heat to induce the pyroelectric response to print using the setup in Figure 29. As previously discussed, the reason the other inks were deemed unsuccessful for printing is because the lasers’ and optics’ limit was reached. The air gap is kept constant at 227µm and a 10A, 2.5s pulsed laser was required to achieve successful pyroelectric E-jet printing using a lithium niobate crystal. As shown in Figure 30, the droplets printed are larger than the Si µ-squares and had many satellite droplets. Three µ-squares were printed approximately 1mm apart in a corner configuration. As labeled in Figure 30, Si I droplet was approximately 582.81µm, Si II 557.81µm and Si III 576.56µm in diameter, which is not smaller than when using the mPDMS. With these results, and the fact that the smaller inks could not print, it was concluded that the droplet size is controlled by the profile of change in temperature on the crystal face closest to the ink, not the one closest to the laser. Even though the heat spot size on one surface is roughly 200µm, the heat will dissipate and spread within the crystal so by the time it is seen on the opposing face, the one closest to the ink, it is much larger than where the heat was initially generated. From here, two setup alterations were explored. The first moved the location of the
Si $\mu$-square closer to the ink surface and the second decreased the crystal thickness, which is discussed later in Section 3.6.

Figure 30. Laser focus on a 200x200x10\(\mu\)m Si $\mu$-square (left) and three Si $\mu$-squares with successfully printed droplets (right).

Figure 31 shows a schematic of reorienting the crystal so the Si $\mu$-square is now also in contact with the printing substrate and is closer to the ink surface. All the other printing parameters were kept the same from when the Si $\mu$-square was on top of the crystal, exposed to air. By changing the location of the Si $\mu$-square, the hypothesis is the droplet size and time for printing will both decrease because the surface closes to the ink will generate charge in a smaller area. Even if the heat is dissipated into the pyroelectric material, the printing will not be as affected as the previous configuration like before where the heat had to travel through the material to achieve printing.
This new configuration did yield a smaller printed droplet size and a shorter time that the laser had to be on to print. The laser was set to 10A and a 1s pulse. Figure 32 shows three Si µ-squares used for printing and the droplets formed. Si I had a droplet diameter of approximately 264.20µm, Si II 282.37µm, and Si III was 292.61µm. The Si µ-squares appear darker because the squares are printed onto the crystal, and the crystal is flipped showing the underside of the squares. The backside of the Si µ-squares revealed that the Si adhesion is another possible parameter that can affect printing. The adhesion of the Si µ-squares to the crystal surface is not the same and can contribute to variations of the droplet size while printing. As seen in Figure 32, Si II has lighter corners while Si III is completely dark. Studying the correlation of printed droplets to Si µ-square adhesion will be addressed in future work.
Since printing was more successful with a shorter pulse of the laser when the Si µ-square was placed closer to the ink film, the smaller 100x100x10µm Si µ-squares were used again as the NIR absorber. Using the laser at 10A was not sufficient enough power to achieve printing so it was increased to 20A with a pulse of 1s. As seen in Figure 33, the droplet size did not decrease from the 200 Si µ-squares. The droplet diameter was approximately 286.93µm.

![Figure 33. 100x100x10µm Si µ-square with flipped crystal/Si µ-square setup.](image)

With the laser power set to 20A, the 200x200x10µm µ-squares were used again with the hopes of even further decreasing the droplet diameter. Smaller droplets were printed with a 0.2s pulse but it was difficult to measure the diameter because the shapes of the droplets were not very circular and were mostly covered by the Si µ-squares. Figure 34 shows the three Si µ-squares and the droplets printed. Although the goal is to print the smallest achievable droplets, the shape is also very important.

![Figure 34. Three different Si µ-squares with printed droplets at 20A using the flipped crystal/Si µ-squares setup.](image)
3.6 Experiments with Pyroelectric Material

Initially, lithium niobate was chosen as the pyroelectric material because of the work done in current literature with respect to pyroelectricity and availability of the material [25]. This section includes the discussion of exploring other pyroelectric materials to get a shorter printing time and a smaller droplet. Lead zirconium titanate and lithium tantalate were two options explored as alternative materials.

3.6.1 Lithium Niobate (LiNbO₃ or LN) Crystal

LN is a common material chosen when studying pyroelectricity and is also commercially available. All the reported results in the previous sections were done using a LN crystal. The total pyroelectric coefficient of LN is $-83 \frac{\mu C}{m^2 K}$ which is measured experimentally and is reported elsewhere [18, 19]. Since this is an experimentally determined value so there are varying values of the pyroelectric coefficient for all the mentioned materials. The LN crystal was purchased from MTI Corp. and is poled in the z direction, 500µm thick, optical grade, and double sided polished. It is part of the 3m point symmetry group and has a Curie temperature of 1160°C. LN does not absorb in the IR range and therefore an IR absorber was required. The smallest droplet achieved using LN as the pyroelectric material was found to be approximately 264µm and is reported in the previous section using a Si µ-square closest to the ink film.

Since reorienting the Si µ-square and crystal resulted in smaller drops, decreasing the thickness of the crystal will hopefully decrease the amount of heat that can dissipate in the crystal. Since printing occurs due to the surface charge generated from a change in temperature, the thickness of the crystal was not taken into account initially as a variable to affect printing. For this reason, a 100µm LN crystals was investigated, but printing was not demonstrated using either the mPDMS or the Si µ-squares. However, an interesting “side effect” was observed when printing with the Si µ-squares. At both higher powers and longer laser pulses, the Si µ-squares would actually move and sometimes enough to move out of the field of view of the camera. In the future, a deeper understanding of the physics behind pyroelectricity is required to determine how the thickness of the material affects the charge generation and therefore the jetting.
3.6.2 Lead Zirconium Titanate (PZT) Ceramic

Lead zirconium titanate (PZT) was chosen as an alternate pyroelectric material because it not only has a higher reported pyroelectric coefficient but also absorbs in the infrared range. The pyroelectric coefficient for PZT can range from -50 to -268.0 $\text{C/m}^2\text{K}$ depending on the composition of each component in PZT [19]. Unfortunately the exact ratio of composition of the ceramic PZT that was purchased from STEMiNC (Steiner & Martins, Inc.) for our experiments was unknown, so the exact pyroelectric coefficient is unknown. A wide range of laser power and pulse durations were used to achieve printing with the PZT but no combination resulted in successful printing. During the heating of the ceramic, it was observed that there was an unknown material precipitating out of the PZT so the decision to not pursue this as a pyroelectric material was made. PZT also has a very large secondary pyroelectric coefficient which can lower the overall total pyroelectric coefficient because they are opposite signs. This is why the ratio of the PZT causes such a large change in coefficient because of thermal and piezoelectric coefficients that vary based on the composition.

3.6.3 Lithium Tantalate (LiTaO$_3$ or LT) Crystal

A material that is similar to LN but has a higher pyroelectric coefficient was desired to study the effects of increasing the coefficient with respect to printing. It is expected that if the material properties are exactly the same, then a higher pyroelectric coefficient should just decrease the printing time. To test this theory, a lithium tantalate, LT, 500µm thick, z cut, double sided polished crystal was purchased. LT is also part of the 3m point symmetry group and has a Curie temperature of 610°C which is much lower than LN. The pyroelectric coefficient is almost twice that of the LN crystal at -176 to -230 $\text{C/m}^2\text{K}$ [19]. LT absorbs only in the UV range, similar to LN, so IR absorbing materials had to be used again.

The first experiment with the LT crystal is the same setup as seen in Figure 29. The 200x200x10µm Si µ-squares are printed directly on top of the LT crystal as the laser absorbing material. With this setup, the laser was set to 10A and took a pulse of 1s to print, compared to the 3s required for LN. The droplets printed are shown in Figure 35. Si I droplet diameter was approximately 554.05µm, for Si II 516.22µm, and for Si III 518.92µm. These values are slightly
smaller than reported using LN as the pyroelectric material but further tests would have to be performed to confirm that LT will consistently result in smaller droplets to be printed. Comparing these droplets in Figure 35 to those printed using the LN crystal in Figure 30, the shape of these droplets are more circular which is a desired feature when printing.

![Figure 35](image)

**Figure 35.** Three different Si μ-squares with successfully printed droplets using a LT crystal.

Similarly to the experiments with the LN crystal and Si μ-squares, the crystal was flipped so the Si μ-squares are now closer to the ink film. The observed advantage to changing Si μ-square location that appeared with the LN crystal was similar to what was observed for the LT crystal. The print time for the LT crystal was however roughly the same for all the tests, whether the Si μ-square was on the top or bottom of the crystal, but the droplet size did decrease. Figure 36 has two images of two droplets from two different Si μ-squares. Si I printed in 1s and has a diameter of approximately 297.3µm while Si II droplet printed in 0.75s with a resultant droplet diameter of approximately 305µm. These droplets also had undesired satellite droplets.
3.7 Studying Effects of Printing Setup

Throughout the presented research, the printing stack was kept as consistent as possible so the effects of changing the NIR absorbing and pyroelectric material could be isolated and studied. This section discusses the few experiments performed which study the effects of changing the printing stack with respect to the air gap, stack orientation and where the ground electrode was placed. Optimizing the setup with respect to the above mentioned parameters of the stack formation will most likely result in better printing performance. More experiments would need to be performed to conclude which parameters of the stack is the most dominant factor in successful small droplet diameter pyroelectric E-jet printing from an ink film.

3.7.1 Air Gap

To study the effects of air gap and pyroelectric E-jet printing from an ink film, the setup was modified to allow for the glass printing substrate to be actuated in the vertical direction. The glass spacers were eliminated, and the glass printing substrate was mounted to a z-stage. The pyroelectric material, is lithium niobate crystal with a 200x200x10µm Si µ-square, was placed on top of the glass printing substrate. The NOA74 ink was spun to approximately 16-20µm
thick. The laser was set to 10A of power, and the air gap was changed to determine the optimal working distance to produce the smallest droplet for a 3s pulse. A 3s pulse was chosen because from the previous experiments with a 270µm gap, a 3s pulse was required to achieve printing.

Initially the air gap was set to approximately 800µm but there were no droplets successfully printed. A droplet was not able to be printed using a 3s pulse, but required 5s so it could be inferred that going above 700µm would not result in a small droplet. The 5s pulse resulted in a droplet with the diameter of approximately 630.3µm. The gap was then lowered by 100µm to a 600µm working distance and a droplet diameter of 716.67µm was printed with a 3s pulse. The gap was further decreased to 500µm resulting in a droplet of 562.69µm in diameter. At an air gap of 450µm, the droplet formed with a 3s pulse increased to 611.94µm but with a 2s pulse printed a 508.96µm diameter droplet. At an air gap of both 400µm and 350µm, the droplets printed with a 3s were very large with many satellite droplets. It is expected that for a given ink thickness and voltage generated, there is an ideal air gap to get the smallest droplet printed. These experiments showed that the optimal thickness for this setup of ink thickness, LN crystal and with a 3s pulse is somewhere between 450-500µm gap. These results are consistent with the previously reported results of printing with LN and Si µ-squares with an air gap of 270µm in Section 3.5.3. Most of the printed droplets were circular in shape with minimal satellite droplets.

3.7.2 Stack Orientation

The stack orientation was also altered to determine if there was a better printing setup that will result in small droplets. Figure 37 shows two alternative setups that were tested. For setup A, the glass printing substrate was removed to allow the ink to be printed directly to the pyroelectric material, which in this case was the LT crystal. The air gap was set to 270µm to be consistent with most of the previously reported setups and results. Setup B has ink spun directly onto the LT crystal surface and is roughly the same thickness as if the NOA was spun onto glass, ~16-20µm. The air gap again is set to 270µm and the printing substrate is glass.
Setup A was able to print a droplet but required a longer pulse than previously reported. The power was set to 10A and a pulse of 1.85s resulted in a 444.44µm droplet being printed but with many satellite droplets seen in Figure 38. No droplets were printed when using setup B. Figure 39 shows three images taken after a pulse of the laser on a single 200x200x10µm Si µ-square. As can be seen in the first image (left), there is an observed changed that grows radially out from the Si µ-square. After the laser has been turned off and cooling occurs, the change begins to shrink towards the Si ink and after approximately 20.5s, the effect is gone. In this case, the NOA ink itself maybe deformed, but not in the normal profile required for printing. Instead, it seems the ink is being pushed away from the Si µ-square forming a void of NOA ink directly underneath the Si µ-square. It is difficult to provide a concluding remark with respect to if it is indeed ink moving or just some heat affected stress gradients in the LT.


**3.7.3 Ground Electrode Location**

The location of the ground electrode was also altered in the stack orientation to see if the printing results would vary. Initially, the ink was spun onto a silicon die and grounded, but this did not produce any printed droplets. Indium tin oxide (ITO) was then sputtered directly onto the pyroelectric material, LT, so the crystal itself could be grounded. ITO was chosen as an electrode material because it is transparent in the IR range. ITO was also sputtered onto cover glass which was used to spin NOA74. There were four setups tested and can be found in Figure 40. Once again the air gap is set to 270µm and the NOA74 ink was 16-20µm thick.
Figure 40. Alternative schematics for ground electrode placement in pyroelectric E-jet printing from an ink film.

Setup A has ITO sputtered on the top side of the LT crystal and is connected to ground while the other side has a 200x200x10µm Si µ-square printed onto it. This setup produced printed droplets with a 10A, 5s pulse which is significantly higher than the <1s required to print without ITO. The droplets printed from the µ-squares seen in Figure 41 are very large compared to what had been achieved in the previous sections. Si I produced a droplet approximately 789.10µm in diameter and Si II resulted in 741.81µm, and also there is a lot of streaking outward from the droplets. This is a common effect when there is too much like charges in the droplet and it cannot dissipate fast enough so there is repulsion if the droplet is too large.
Setup B has ink spun directly onto the ITO coated slide and both the LT crystal and ink are grounded. Setup C and D have just the ink grounded and the location of the Si ink changes from under the crystal to above respectively. Setups B, C, and D did not produce any droplets but instead had a similar effect as reported in section 3.7.2 when the NOA ink was spun directly onto the crystal.
CHAPTER 4: CONCLUSION

The current work presented is to advance E-jet printing to a large scale manufacturing process. Chapter 2 demonstrated a low cost print head multi-nozzle array for commercially purchased nozzles. The goal to successful print with four nozzles with minimal variation of droplet size in the print head was achieved. Chapter 3 explores the option of eliminating the need for physical nozzles for E-jet printing. Preliminary experiments were performed and discussed to demonstrate using pyroelectric physics as a promising alternative to generate the required voltage for printing from an ink film. Since both presented E-jet advancements are proof-of-concept, there are many directions the ongoing work can go to further improve what was presented for manufacturing applications.

4.1. Multi-Nozzle Array E-jet Printing

4.1.1 Conclusion

For applications in manufacturing, simultaneous printing can decrease the printing time, reduce cost, and increase output. The idea to have a linear nozzle array allows for multiple identical images to be printed simultaneously. A potential application for E-jet MNA is to print traces on multiple circuit boards. If the boards are identical, one could print traces on the same number of circuit boards as there are nozzles. The spacing between nozzles can be adjusted for a custom print head or the print head can have a number of v-grooves greater than the required number of nozzles and then secure the nozzles in the desired groove while leaving others blank. The results presented in this work show the consistency of a MNA print head with droplet sizes of ~2.66µm and a standard deviation of less than 0.6µm. To decrease the standard deviation, alignment will need to be improved, along with the optimizing the PWM printing parameters.

Currently, the largest cost of materials is the nozzles themselves. If one could pull nozzles “in house” this could reduce the cost of a fully assembled MNA. With a $37.62/hr fiber/pipette puller and using borosilicate thin wall glass tubes, the cost of pulling a single nozzle can be $1.60 compared to purchasing an already pulled pipette for $12.50. This simple cost analysis is based on the assumption that all the pulling parameters are set to produce a nozzle that matches
the standards from WPI (without any post processing) and it takes 5 minutes to load and unload
the glass tubes. A thorough cost analysis will need to be performed for mass production of
MNAs.

4.1.2 Future Work

Future work includes addressing the issues with nozzle alignment and minimizing the
misalignment due to human error. These proof-of-concept MNA printing experiments uses only
four nozzles, but with the ease of designing a wider print head with more grooves, up to ten
nozzles could be achieved. Before attempting to increase the number of nozzles, the alignment
issue has to be addressed to increase the probability of all the nozzles being able to print at a
given voltage. An automated alignment setup where the nozzle tips will be tracked will give the
best precision and accuracy of nozzle alignment. Currently, only one differential adjuster is used
and can only push one nozzle at a time due to size restriction. If the nozzles are pushed too far,
they have to be carefully pulled back by hand or removed from the groove and reset further back.
If the nozzles could be actuated both forward and backwards, then better alignment can be
achieved.

Since the MNA never went above four nozzles, more research will need to be conducted to study
the effects of increasing the number of nozzles simultaneously printing with a single power
supply. There were no differences in initial jetting voltage of an individual nozzle to when the
whole array was printing with four nozzles. If higher density print heads are to be fabricated,
this will most likely become a more dominant issue. If currently delivery from a single power
supply is not an issue, work can be done to increase the linear array to up to ten nozzles or
develop a 2D array such as a four by four nozzle array.

An additional future effort is to have a single power supply and function generator power all four
nozzles, but be able to print different images with each nozzle, given the printed image area is
the same. This will require more electrical work with extensive research into controls to be able
to delay a signal to only certain nozzles to get the desired printed images. This would result in
different images to be printed in a given area. Since the focus of this work is to be able to
increase throughput by printing multiple images of the same pattern, this was not studied.
4.2 Nozzle-less Printing Using Pyroelectricity

4.2.1 Conclusion

The best and most successful printing results were produced using a laser as a heat source for nozzle-less printing. By eliminating the need for a physical heat source such as the soldering iron or the resistive heater coil, any stress that is induced by the contact or weight of the heaters is eliminated. A laser also offers the advantage of precisely controlling the power and the pulse duration to find either the minimum power or time required to print a desired size droplet.

It is experimentally determined that from the three NIR absorbing materials mentioned in Section 3.5, the implantation of Si µ-squares as an absorber when printing with non NIR absorbing pyroelectric materials is preferred. Not only did the Si µ-squares produce the smallest droplets but there was less variation in the actual Si µ-squares themselves. These Si µ-squares are batch fabricated and can be printed essentially anywhere directly on the pyroelectric material surface. The only challenge to using the Si µ-squares is that nozzle-less printing from the ink film is limited to where the µ-squares are placed on the pyroelectric material.

It can be concluded for the purpose of applying pyroelectricity to E-jet printing that using a material with a high pyroelectric coefficient is desired. Using a material with a higher coefficient decreases the printing time which is a major goal for current E-jet printing. From the presented work in Section 3.6, lithium tantalate would be the material of choice out of the three tested to use for further experiments. The material properties of the pyroelectric material such as heat capacity, thermal expansion, conductivity, and other thermal quantities were not studied in the presented work but should be taken into consideration when selecting a pyroelectric material.

With respect to the actual printing stack setup discussed in Section 3.7, there is an optimal air gap for a given ink thickness and pyroelectric material. Once the optimal printing air gap is determined, if the ink thickness can be kept constant while printing, then the stack can be built without alterations. The stack orientation and location of the grounded electrode does make a significant difference when printing from an ink film. By removing the glass printing substrate and printing directly onto the pyroelectric material, it is concluded that there is not a significant
loss of charge within the glass. The location of a grounded material is important in that it should not be placed on the surface closest to the ink film or else the surface charge will be depleted instantly. No droplets were printed when the ink itself was grounded but the pyroelectric material introduces undesired effects of larger droplets with satellite droplets when it was grounded.

4.2.2 Future Work

The geometry of the Si µ-square was not explored because the readily available dies were square. Circular Si µ-dies can be used to eliminate the possible side effects of printing with a square. The expectations are with a circle, there is better uniform heating and will result in a droplet printing centered to the Si µ-circle die. The proximity limit for the Si µ-square placement to produce small circular droplets was not explored in this work. For applications in manufacturing, these experiments would have to be done to create a high density array of Si µ-squares on a pyroelectric surface. As mentioned in Section 3.5.3, it appeared that the Si µ-squares did not all have the same adhesion to the pyroelectric material so a topic for future work can also include developing a process to ensure good thermal contact between the two surfaces.

Besides the pyroelectric response, the actual material properties of the pyroelectric element should be studied. Even though the material is not clamped and the mechanical response is not of interest, the dielectric coefficient, thermal expansion, heat capacity and other values are most likely responsible for the large difference in printing with a Si ink on the top or bottom of the LN crystal as opposed to the LT crystal where the print time was almost the same. Both of the crystals were 500µm thick, but the trend of decreasing time was not the same or proportional for both materials. If the work is to be continued with the current setup, a transparent pyroelectric material will be needed to give feedback of successful printing. There are many other crystals that are pyroelectric but have different point symmetries which is another parameter that can be studied.

Due to the ink film being so thin, there is an optimal thickness desired which will overcome the boundary effects of thin films. If the ink is too thin, then the surface tension is too high and the ink will have difficultly deforming into a small peak. If the ink film is too thick, then the
deformed profile will be large and a large droplet will be printed. Preliminary experiments were performed but not presented for changing the ink thickness and also the ink property. More studies will need to be performed to provide concluding remarks on the ink properties and how it affects printing. A fluid dynamics calculation can be performed to determine if an ink film is too thin and the boundary layer effect is dominate preventing the movement of ink to deform into a Taylor cone. Studying the fluid dynamics could also provide insight to the exact reason why a liquid bridge is formed instead of printed droplets for a given setup. For a given temperature, it is expected that printing is uniform but there may be some variation in ink film thickness which most likely contributes to a liquid bridge forming.

Printing from an ink film using pyroelectrics discussed in this Thesis is essentially applying a point source of voltage and deforming a thin liquid. How a thin film of ink deforms when a point charge is introduced into the system should also be studied for future work to further the understanding of what has been presented. Some preliminary experiments were performed with applying a high voltage to an Au/Pd glass pulled rod and capturing how an ink film deforms. The goal is to simplify the setup to isolate different variables when trying to print. By applying a known voltage to an electrode, the ink film and air gap can be studied. These results can then be compared to the pyroelectric printing experiments to optimistically provide insight on how the pyroelectric material affects printing. Currently using the laser as the heat source and Si μ-squares as the NIR absorbers, the amount of heat generated and therefore the amount of voltage produced is unknown. Ideally a model would be generated to study this generation of charge, but with the simplified printing setup, if the air gap and thickness are set identically to a given pryoelectric experiment, the voltage required to pull the ink and print can be determined and then the corresponding temperature can be calculated with equations and relationships provided in the literature for pyroelectric materials.
REFERENCES


