Optimization and Characterization of a Capillary Contact Micro-Plotter for Printed Electronic Devices

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Akanksha Rohit
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This thesis titled
Optimization and Characterization of a Capillary Contact Micro-Plotter for Printed Electronic Devices

by
AKANKSHA ROHIT

has been approved for
the School of Electrical Engineering and Computer Science
and the Russ College of Engineering and Technology by

Savas Kaya
Associate Professor of Electrical Engineering and Computer Science

Dennis Irwin
Dean, Russ College of Engineering and Technology
ABSTRACT

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Optimization and Characterization of a Capillary Contact Micro-Plotter for Printed Electronic Devices

Director of Thesis: Savas Kaya

Printed electronics is emerging as an integral part of the electronic industry due to its low cost fabrication and flexibility of devices against the rigid and expensive technology using silicon. Various methods for printing have existed for a long time with inkjet printing being the most common method used for electronic devices. This thesis explores a new and innovative printed technology using a capillary based microplotting approach implemented via Sonoplot Microplotter II. Unlike the inkjet printing technique which prints in overlapping spots with resolution between 30µm-100µm, the Microplotting approach helps to prints continuous features with a higher resolution as low as 5 µm. Capillary action is used to fill picoliter amount of ink into a micropipette which is used for printing. Thus, the focus of this thesis is the optimization of this new printing technology under various conductions using different conductive inks and on a broad range of substrates and different tip diameters. In addition, passive resistive, capacitive and inductive components were printed to characterize the printing process and operation of electrical devices under different conditions. The applications of this Microplotter was further demonstrated by printing a flexible resistive strain sensor. The procedures involved for the fabrication of micropipettes using a glass puller for different diameter tips attached to the dispenser head is also explained in this thesis.
I would like this dedicate this to my family, without their love and encouragement I wouldn’t have been the person I am today!
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CHAPTER 1: INTRODUCTION

A broad introduction to the field of printed electronics, and the capillary-contact based micro-plotting approach explored in this thesis is provided. A new and versatile technology using the Sonoplot® Microplotter is used for printing devices with a higher resolution than conventional printing techniques. Forces that drive the development of this rapidly expanding field, alternative approaches and potential markets are pointed out. The motivation of the research work and the organization of the thesis are also included.

1.1 Motivation

The development of printed electronics has led to the production of innovative technologies which are lightweight, flexible, compact and cost effective. Inkjet printing has been most commonly used for printed electronic devices. However, for printing with a higher resolution, this non-contact method of printing is not suitable as printing occurs with overlapping spots and it is not possible to draw continuous features using this printing technique. The capillary contact based micro plotting approach investigated in this research is a relatively new tool for printing smaller features with a higher resolution and has not been extensively explored for printed electronics devices. Unlike inkjet printing, only picolitre amount of ink is required for the Microplotter to print continuous features at ultrasonic frequencies without any droplet complications. In addition to this, the Microplotter is capable of printing with different types of inks in a single session by changing the dispenser head which consist of a micropipette in which the ink is filled. This helps in printing multiple layers with different inks for various applications. Different type of conductive and insulating inks with a higher range of viscosities can be printed using the Microplotter. The work in this thesis focusses on the optimization of process conditions for efficient printing with different inks and tip diameters. In addition to this, characterization of passive (Resistance, Capacitance, Inductance) electronic devices and simple sensors was made with this tool on a broad range of substrates. The glass puller was used for the fabrication for different diameter tips attached to the dispenser head which was used for printing. Extremely small diameter (<5 µm) tips was pulled using the glass puller to show the resolution of printing using this device.
1.2 Nanotechnology Revolution

Nanotechnology today is an interdisciplinary field of research that is rapidly progressing in areas of medicine, healthcare, electronic devices and material science. This is a vast field with a flexibility to make novel prodigious devices. Nanotechnology also helped us to remove fabrication barriers in sustaining the so-called Moore’s law of device scaling. Nanotechnology has helped to shrink the gate length of transistors from 130 nm to 22 nm and with the help of recent advancement in fabrication techniques, researchers are aiming to make them even smaller (~ 7nm) [1]. With such rapid evolution of technology, alternative and low-cost approaches to make miniaturized devices without affecting their performance also became possible. An example for such an advancement is the innovative paradigm of printed electronics that produces flexible and wearable electronic devices. Printed electronics propounds versatile, cost-efficient and flexible devices that are already being manufactured and put in use in a variety of areas depending on the application [2]. The flexible electronics market is expected to make billions of dollars as the years go by and provide an efficient and economical alternative to technology to continue miniaturization. Demand for electronic devices which are compact and flexible like Radio Frequency Identification Tags, super capacitors, wearable sensors can be met using printed technology.

1.3 Printed Electronics

Printed electronics that is emerging rapidly is capable of making electronic components that are durable, lightweight, compact and also flexible. With printed electronics both conventional and novel printing techniques can be used to build electronic devices on rigid or flexible substrate using different types of conductive inks and polymers for various low-cost applications. Printing is a simple additive process in which a layer of ink is printed without any etching involved to remove the unwanted material, unlike the traditional method of photolithography. This helps in saving a lot of material, hence reducing the overall cost for fabricating a device. The lithography process is subtractive with several steps involved for fabricating the device. In addition, printing is a more versatile approach for fabrication as we can print using different techniques depending on the application. Different inks and substrates (flexible or rigid) can be used and it is even
possible to make 3-D (multi-layered) devices which has a great potential in the future. Printed electronics can achieve a higher level of production, low cost and a higher throughput compared to the conventional semiconductor fabrication techniques. The key to printed electronics is to have access to conductive and insulating inks and the ability to control ink-surface interaction (viscosity, particle size etc.) which plays a very important role while printing.

Silicon technology has existed in the market for a long time now, and the cost and fabrication method for silicon technology faces the challenge of continued improvement. Miniaturization of silicon has its own constraints and is an expensive task. Silicon fabrication involves multiple steps from deposition, exposing the mask to UV light followed by etching and resist removal to get the final pattern. These steps take several days to even weeks to complete. Also, the devices are rigid and brittle and confined to smaller areas and the use is not very flexible. Though research is still on to make the substrates thinner and compact, it leads to an increase in cost and complexity in the fabrication steps. With flexibility in printed electronics, miniaturization is possible especially for low-cost and low-functionality found in wearable electronic devices.

1.4 Printing Methods

Additive device micro fabrication is not just confined to electronic components and is fast becoming a complete eco-system of solutions in RF devices, antennas, photonics, fluidic and MEMS devices. As a result, it is relevant for many applications. The market drivers for printed electronics today are sensors, RFID tags, memory devices, thin film transistors, displays and photovoltaics [3]. Different printing techniques are used depending on the application to make efficient devices.

As seen from the Figure 2, different applications require different modes of printing depending on the type of device. Traditional methods of printing involved contact printing like nanoimprinting, liquid transfer or gravure printing [4] [5]. In these techniques, the pre-pattern image is pressed against the required substrate and the ink is transferred to get the required pattern onto the substrate. Screen and inkjet printing are two most common non-contact methods of printing that has been used extensively in the industry today to make sensors and transistors. Ink jet printing involves depositing ink in a drop-by-drop manner
onto the substrate to form the pattern and has two different styles. The *continuous* ink jet printing has ink droplets continuously ejected out of the nozzle and is aligned on the substrate using an electric field. Since the droplets are continuously ejecting even when it is not required, a waste well is present which collects the unwanted ink. *Drop-on-demand* inkjet uses acoustic frequencies (1-20 KHz) to eject the ink out of the nozzle when required. The drop on demand inkjet printing is further classified into three types (thermal, electrostatic, piezoelectric) as shown in Figure 1 depending on the excitation source for the ink [7]. In thermal drop on demand inkjet printer, the thin film heater is heated by passing current through it. When the supply of current is suddenly removed, the heat transfer leads to break in the vapor bubble pushing it out of the nozzle. In the piezoelectric inkjet printing technique, expansion and compression of the piezoelectric actuator results in creating a pressure pulse for the ink to be ejected [7].

![Figure 1. (a) Thermal Inkjet Printing (b) Piezoelectric Inkjet Printing [7](a)](image)

Piezoelectric ink jet printing is the most common method used today, which employs a piezoelectric material (PZT) to eject out liquid from the nozzle. The size of the droplet depends on the amount of voltage applied and the frequency of operation. However using inkjet printing methods the feature size is typically limited to 30-100 µm [8]. As electronic components are getting smaller, it will be a challenge to print micro and nano circuits which necessitates different printing technologies to be developed. Screen printing
is the other non-contact method where a stencil or a mask of the required shape is placed over a mesh through which the ink is pushed out. The pressure applied to the squeegee and the contact made by the ink via the silk screen determines the resolution of printing.

![Figure 2: Different Printing Methods](image)

The micro-plotting technique, the main approach explored in this thesis, can be used to get a finer resolution of printing, make smaller area and compact devices. The Sonoplot® micro-plotter is a non-contact type of printing tool which works like fountain-pen where the ink comes out of micron-scale glass capillary continuously to print straight lines, arcs, and filled patterns. The Sonoplot micro-plotter consists of a dispenser which serves as the core of the micro-plotting device. A hollow tapered glass tip with a user-defined diameter is attached to a Lead Zirconate Titanate (PZT) driver plate which is a piezoelectric material. This material vibrates at ultra-sonic frequencies by which the ink is pushed out of the glass tip onto the substrate. With a miniscule volume (picolitres) of ink being ejected out of the glass tip, patterns with a resolution as small as 5µm can be printed using this printer. Unlike inkjet printers that are limited to low-viscosity (5-20cP), higher viscosity inks (up to 450cP) can be used to print with ease and precision [9]. This method
of printing keeps only the ink in contact with the substrate and the glass tip does not have
to come in contact with the surface. Printing can be done several microns above the sample
using a surface-tracking sensor based-on ultrasonic detection. The complete working of the
micro plotting printer is explained in the Appendix section of this thesis.

1.5 Printed Electronics Market

Printing technology has emerged in the market today primarily because of potential
of roll-to-roll (R2R) processing. With R2R processing large area electronic devices are
printed on continuous flexible substrates and large-scale production is possible. This
technology is used to print newspapers and other industrial productions can also be used
for flexible displays and screens. The flexible substrates are mounted and kept under
tension via large continuously rolling cylinders. Hence, printing can be done at very high
speeds, enabling large production and economic manufacturing. With R2R processing,
mass-production becomes energy efficient, and electronic device fabrication can
potentially have a significantly less environmental impact. Energy savings are higher since
the manufacturing tools use less energy while they also produce a markedly large number
of electronic devices. According to research, 76 rolls uses lesser energy per unit area
relative to 77 conventional manufacturing processes [10]. The fabrication using printed
technology is less than (1/10)th the cost of silicon. Different types of nano inks (silver,
copper, polymers) which are much cheaper than silicon can be used to print on extremely
thin flexible substrates. The resolution of printed electronics using the latest tools coming
to market [11] can go as low 1µm. These advantages of printed electronics make it an
attractive technology for the present and future low-cost electronics. Although it is known
that printed electronics will not substitute the conventional silicon technology, it is an
emerging technology that opens a new world of innovations and printed circuits for
different applications including, toys, perishable electronics, low-cost consumer
electronics, sensors, healthcare, antennas, smart integrated systems, low-cost RFID,
memory devices and wearable electronic devices. Some examples of printed devices and
systems are shown in Fig.3
The idea of flexible smart devices is an attractive technology which has increased to more than 50 billion US$ in less than 10 years. The printed electronics market is on the verge of another revolution increasing to approximately 100 billion US$ in the next decade as shown in Figure 3.

![Figure 3. Printed Electronic Device and System Examples [12]](image)

This growth in the market is driven by increasing applications of printed smart systems in healthcare, automobile, commercial and industrial devices. The majority of the development in printed electronic devices has been due to flexible displays, OLEDs and sensors. The OLED and flexible electronics markets has had a huge impact in the printed electronics industry increasing to 30 billion dollars by 2018 [12]. The printed RFID market has advantage over silicon based tags due to the cost and hence increasing to almost 60%

![Figure 4. Printed Electronics Market [12]](image)
of the printed electronics market by 2017 [13]. With emerging conductive inks and flexible substrates, printed electronics has a huge potential for the expansion of the electronics industry to a multi million dollar market. Though printed electronics cannot be replaced with silicon, innovative devices could be made which would not be possible with conventional techniques.

1.6 Different Inks and Substrates for Printing

1.6.1 Conducting Inks

The conducting inks used for printing is an integral criterion for the working of the device and proper care must be taken in selecting them in conjunction with the type of printing technique to be used. Different printing techniques behave in different ways for a particular type of ink. Parameters such as viscosity, surface tension, temperature while printing and compatibility of the ink with the substrate affect the way the printing process takes place. If the ink has a very high or very low viscosity, then printing is more complex as the ink may not be delivered or spreads uncontrollably on the surface ruining accuracy. These parameters differ with every printer and depending on the application the device needs to be printed accordingly with a suitable printing technology. Conductive inks can be of three times: metals, metal oxides and organic polymers [14]. Some of the most common metallic inks used for printing is Silver (Ag) as it has a very good conductivity of $6.7 \times 10^7$ S/m [3]. Since the whole goal of printed electronics is lower cost, cheaper metallic inks such as copper is also used. However, there are complications: Copper nanoparticles are prone to oxidation and annealing must be done either using laser/photo sintering or at prohibitively high temperatures.

Other types of inks that can be used is organic materials or polymers that also exhibit conductivity. These types of organic polymers that conduct are called as intrinsically conducting polymers (ICP’s) whose chemical structure may be complex (hence expensive to synthesize) and heat-tolerance is low. Also, typically organic polymers do not have a conductivity as high as metals but can be in the semiconductor range. To limit the cost (complexity) of the ink, compromise has to be made in the conductivity of the ink. Some of the commonly used polymers today are PEDOT, polyacetylene,
polyaniline, etc. [3]. But due to the low conductivity of organic polymers, high-performance transistors or fast sensors made with these inks are still a challenge. Polymer insulators such as PMMA, are also used for dielectric materials in printed capacitor and sensors. Another type of materials that is still at its developing stages is using nanocomposites. Nanocomposites are materials in which a metallic nanoparticles (d<100nm) is dispersed in a matrix polymer[18] such as poly dimethyloxane (PDMS). The nanofillers that are used to make the nanocomposites need not only be metallic nanoparticles, they can also be materials like carbon nanotube and graphene with high electrical conductivity and high current carrying density as well as semiconductor quantum dots that can be engineered for a specific optical/magnetic/thermal response. The behavior of the nanocomposite depends on the proper dispersion of the nanofillers in the polymers, if they are not properly dispersed then the nanoparticles are lumped together or coagulated which affects the uniformity in printing. Due to the unique properties of nanocomposites, carbon nanotubes and graphene are widely used today for flexible electronic devices.

Table 1: Types of Conductive Inks

<table>
<thead>
<tr>
<th>Ink</th>
<th>Conductivity</th>
<th>Advantages</th>
<th>Disadvantages</th>
<th>Curing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silver</td>
<td>Excellent: 6.7*10^7 S/m</td>
<td>Highly conductive</td>
<td>Higher Cost</td>
<td>Low Temperatures between 200-250°C</td>
</tr>
<tr>
<td>Copper</td>
<td>Good: 5.8*10^7 S/m</td>
<td>Good Conductivity, slightly lower than Silver.</td>
<td>Oxidizes and looses conductivity.</td>
<td>Extremely high temperatures required. Curing is usually done using flash annealing method.</td>
</tr>
<tr>
<td>Carbon nanotube</td>
<td>Excellent:</td>
<td>Flexible and transparent</td>
<td>Low viscosity makes it harder to print</td>
<td>Low temperatures between 120-150°C [15], [16].</td>
</tr>
<tr>
<td>Polymer Based</td>
<td>Average</td>
<td>Transparent, flexible</td>
<td>Lower conductivity than nonmetallic inks</td>
<td>Low Temperatures of 100-150°C or UV radiations [17].</td>
</tr>
</tbody>
</table>
In this work two types of metallic nanosilver inks, polymers, copper oxide sol gel and copper oxide metallic ink were printed. The nanosilver ink with different percentage of metallic loading and copper oxide inks used were from Novacentrix. JS-B40G is a conductive ink with 40% Ag that can be used to print on porous and non-porous substrates and this was used to print on glass and PET. For printing on paper JS-B25HV with lower Ag content of 25% was used. The viscosity of both the inks was 8-12 cP. Curing temperatures of the ink for different substrates was different. Glass was cured on the hot plate for 2 hours at 220°C. Since the flexible substrate like paper and PET had a tendency to fold at such high temperatures, they were places in the vacuum oven for 2 hours at 65°-70°C.

1.6.2 Substrates

The type of substrate that is used for printed electronics is a very important criterion for efficient working of the device. Rigid substrates like glass or flexible substrates like PET, paper and Kapton all have different properties with respect to conductivity and adhesion of the ink onto the surface that is being used. Each of the substrates have their own pros and cons and effect the performance characteristics of the device. With the micro plotter, the tip that is used for printing is very fragile and tends to break if the substrate is not smooth enough or has undulations. The tip tends to chip off while printing on rougher substrates or when the height of the surfaces is uneven at different points and hence care has to be taken to make sure that the surface is clean and smooth with proper calibrations performed to prevent the tips from breaking.

Rigid substrates such as glass was used for the optimization of the printer. For flexible substrates different types of papers, PET and Kapton was used. Printing on glass has different characteristics with the use of different printing parameters which will be discussed in section 4.1

The challenge with flexible substrates was that the surface is not uniform enough as there is a difference in height of 10µm to 15µm at different points in the surface. Due to the roughness in the surface the tips would chip while printing and the difference in height of the substrate at different points resulted in the printed patterns to be missing at certain areas as it would start printing in air. To smoothen the surface, a vacuum chuck was used
to hold the substrates tight down onto the surface and minimize the height difference and undulations at certain points. Another criterion for paper was that the surface was rough causing the tip to break on approaching the surface. To overcome this, we printed on different papers and the Cannon glossy photo paper had minimum roughness and printing could be efficiently done. The patterns could be printed without the tips breaking and with good accuracy. PET had a comparatively smooth surface and printing with JS-B25HV gave fine lines without too much spreading of the ink. Also, PET is more resistant to tearing than paper and had better adhesion of the ink to the surface. Plastic substrates like PET offers better flexibility, durability and is resistant to moisture and other chemicals.

1.7 Thesis Goal

The primary goal of this thesis is optimization and characterization of microplotting printing technique based on the Sonoplot Microplotter II tool used for the fabrication of printed devices and sensors. Specifically, exploring the limits of this novel tool and development of a viable and truly ‘flexible’ printing process is a main goal of this thesis work. To address this goal, a broad range of device geometries and substrates with different printing parameters and inks are investigated to find the optimum conditions required for efficient printing. A secondary objective is to develop a reliable process and set of conditions for pulling capillary glass tips that are essential to the success of the ink delivery process as well as establishment of procedures and know-how to mount and maintain tips so cost of ownership can be minimize. Another goal is to illustrate examples of passive (L, C, R) electrical components and simple sensors using different types of inks and different tip diameters on different substrates, to establish resolution and performance limit of this printer. The passive devices were checked for repeatability, reproducibility and reliability under optimum writing conditions.

Since Sonoplot Microplotter is a relatively new tool that is little explored for printing on a variety of inks and substrates, this work and above goals are expected to be useful for many researchers that are planning to build printed electronic sensors and systems.
CHAPTER 2: BACKGROUND

2.1 History

Printed electronics has existed for almost two decades with applications in different areas. Printed electronics goes back to 1900 where Thomas Edison first came up with the idea of printing on linen paper [19]. Research in printed electronic for making printed devices, conductive inks and printed passive components has been there since 1960’s [20]. Printing methods were already being developed since the 1940’s by the National Department in Printed Circuits. In the analysis done by the National Department in Printed circuits twenty six different printing methods were mentioned [21]. Printing using silver conductive ink and organic inks was an idea that existed since years before [21] and is widely used today to make printed electronic devices at a large scale. Printed electronics has existed since a nascent age and started developing for full-fledged production of electronic devices since a decade for commercial and industrial applications. The first printed transistor was developed in 2003 [2]. Over the last five years printed electronics industry has explored, developed and predicted a great future making millions of dollars in the years to come. From the time printed electronics has evolved, this technology has wide range of applications in the industry for printed circuit boards, membrane switches, RFID tags, photovoltaics, batteries flexible displays, transistors, electroluminescent technologies, sensors and healthcare.

Printed electronics is gaining popularity over the conventional electronic devices because it is efficient cost effective and reliable. It is a rapidly changing field that can be used to devise innovative ideas to create novel devices. The printed electronics industry is currently growing in commercial and healthcare sector and expected to find application in automotive in the years to come. Offset, flexographic, gravure printing technologies have existed since a long time and they are conventional methods that were used for printing. However, the processes involved for printing using these techniques were complex. To account for this digital printing using inkjet technique was developed to manufacture electronic devices. Inkjet printing is one of the most commonly used method today for printed electronic device as the material required is less, no cumbersome preprocess involved and cost of fabrication is significantly minimized.
Sensor manufacturing has existed since a long time and have applications in almost every area. Printed sensor fabrication was initially implemented using screen printing technique. However today inkjet printing is the most commonly used method for electronic devices. Using printing techniques sensors have been made flexible, versatile, eco-friendly and wearable. Printing technology has been revolutionizing the way sensors are made today. Different types of sensors are used using different printing techniques for healthcare, biosensors, capacitive sensors and piezo resistive sensors. Sensors have a simple structure compared to displays, photovoltaics, super capacitors and hence are emerging at a very fast rate.

2.2 Theory

2.2.1 Capillary Action

Unlike the inkjet printers where ink is filled using a cartridge, the sonoplot Microplotter uses the capillary action to fill in the ink into the tip that is used for printing. Capillary action is an integral part of the system without which printing is not possible when using the Microplotter. Capillary action is the phenomenon due to which liquid flows into a confined space without any external forces such as gravity due to cohesion, adhesion and surface tension [22]. Cohesion causes similar particles to stick to each other and adhesion is the attraction between different particles. During capillary action different particles are attracted to each other or the adhesive forces are stronger than the cohesive forces resulting in the liquid to move in an upward direction into a narrow space with a meniscus in the upward direction or having a concave shape [23]. For wider spaces the capillary action is not significant as the cohesive forces are stronger resulting in similar particles in the liquid sticking to each other. Inks with water based solvents have good capillary action as the adhesive forces between water and glass is high resulting the ink to be filled in a narrow gap easily. However, some liquids have a higher cohesive force and the particles are attracted to themselves causing the meniscus to be in the downward direction or a convex shape where the particles are not attracted to the surrounding medium when filled into a narrow micropipette as shown in the Figure 5 below. The height to which the liquid flows is given by [49].
h = 2\gamma \cos \theta \\
\rho g r

\gamma - surface tension
\theta - contact angle
\rho - density of the liquid
g - acceleration due to gravity
r - radius of the micropipette

From this it can be seen that the height the liquid rises inside the micropipette depends on the surface tension and the radius of the micropipette. The radius of the tube is inversely proportional to the height of the rise in the liquid.

2.2.2 Conductivity of Nanoinks

The introduction of conductive nano inks has helped in the development of printed electronics to yield products which are flexible, reducing cost, adding additional functionality and developing new and innovative products. The main properties of conductive inks that are used for printing should have good conductivity, adhesion to the surface, stability, low agglomeration, particle size should be as small as possible to avoid clogging while printing and a good shelf life [25]. Conductive nano inks are metallic nanopowders mixed in a solvent along with a polymer binder or a surfactant to obtain a
homogenized solution. The surfactant or binder is required to avoid the silver nano particles from agglomeration or sedimentation [26]. The surfactant is adsorbed on the surface thus forming a protective layer preventing the particles from clustering together [26]. These are typically very important when the metal loading of the ink is more for higher conductivity of the printed patterns. As the metallic loading is increased, the ionic strength between the particles increases and the thickness of the electrical double layer formed between the interacting particles and solvent decreases resulting in the particles coming together and clustering. To avoid that surfactants or polymers which are non-ionic type are surrounded by these particles forming a shield preventing further agglomeration of the particles [25].

Silver is most commonly used for conductive inks as it has a very high conductivity of $6.3 \times 10^7 \, \Omega^{-1}\text{m}^{-1}$. Copper ink is the next higher conductivity ink that be used for printing with a conductivity of $5.96 \times 10^7 \, \Omega^{-1}\cdot\text{m}^{-1}$. However, copper forms oxides on the printed surface decreasing the conductivity and also requires high temperatures for annealing. The ink is printed on the required substrate and on annealing at an appropriate temperature the solvent evaporate leaving behind the conductive nano ink. The conductivity of the nonmetallic inks depends on the percentage of loading, annealing temperatures, the number of printed layers in the pattern.

### 2.2.3 Inkjet vs Microplotter

Unlike the ink jet printing method uses audio frequencies in the range of (1-20KHz) [7] to drop or eject out ink from the nozzle, the Sonoplot microplotting printer dispenses ink at ultrasonic frequencies (400-700Khz) [8]. In the inkjet printer due to the voltage applied to the piezoelectric material it exerts a force on the PZT which causes it to flex/expand and the ink is pushed out of the narrow channel through the tip nozzle. So basically droplets of ink are continuously jetted out at a distance from the surface to form the pattern. The amplitude of the voltage applied to the PZT controls the ink that is jetted out of the nozzle. Higher voltage amplitude will produce more ink to be released
The Microplotter has a piezoelectric material which is attached to the dispenser, the PZT vibrates along its axis when an ultrasonic frequency is applied to it causing the micropipette attached to the dispenser to also vibrate causing the ink in the tip to wick onto the surface. Unlike an ink jet printer continuous lines and arcs can be made using this technique without any break compared to droplets of ink using the inkjet printing technique. The position of the dispenser over the substrate can be controlled using the Sonoguide software. Similar to the ink jet printer the amplitude of voltage determines the vibration of the micropipette to push the ink out. The printer works like a pen plotter directly dispensing droplets in contact with the surface [8]. Once the Microplotter finds the surface, and the dispense action is initiated the ink is wicked out of the nozzle to start printing during which the tip is retracted several microns so that only the droplet of ink is in contact with the substrate and the printing action takes place. Once the printing is completed, the ink is sprayed out of the nozzle into the well at a voltage which is higher than what was used for dispensing of the ink [8]. Since vibrations cause the inks to come out only a small quantity of ink is required to print a pattern. Inkjet printer pushes one drop at a time to print and therefore more amount of ink is required compared to a Microplotter. The diameter of the tip determines the width of the pattern that is to be printed. With a Microplotter lines, arcs, rectangles and circles can be printed with a resolution as small as 5µm [8]. However using inkjet printing methods the feature size is limited to 50 µm -
100 μm [8]. Also, inkjet printing method uses cartridges to fill in the ink which needs to be replaced. In a Microplotter there are wells which is filled with the required ink and capillary action occurs when the tip of the dispenser is brought in contact with the well. The size of the droplet or the ink that is jetted out not only depends on the amplitude voltage applied to the PZT material and the diameter of the tip used but also depends on the viscosity of the ink [27]. The Microplotter systems can print using highly viscous liquids like saturated salt solution, high solid content suspensions and carbon nanotube solutions with viscosities upto 450 centipoise[28]. Inkjet printers may cause clogging of the nozzle if the ink used is highly viscous. Also in inkjet printers when the droplet is jetted out of the nozzle, the fluid may break up into multiple droplets called as satellite droplets which can cause spreading of the ink but in the Sonoplot Microplotter since the ink is in contact with the surface and not ejected out in the air the problem of fluid breakup is eliminated.

Table 2: Comparison between Inkjet Printing and Microplotting Technique

<table>
<thead>
<tr>
<th></th>
<th>PIEZOELECTRIC INKJET</th>
<th>SONOPLOT MICROPLOTTER</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Operation Frequency</strong></td>
<td>10-20 KHz</td>
<td>400-700Khz</td>
</tr>
<tr>
<td><strong>Ink Deposition</strong></td>
<td>Expansion of the PZT due to the force applied on it pushes the ink out of the nozzle.</td>
<td>Vibration of the PZT at ultrasonic frequencies results in vibration of the micropipette which pushes the ink out.</td>
</tr>
<tr>
<td><strong>Ink</strong></td>
<td>Filled inside a cartridge which needs to be replaced when the ink is over.</td>
<td>Capillary action of the tip which can be cleaned for loading different ink.</td>
</tr>
<tr>
<td><strong>Feature width</strong></td>
<td>30-100μm</td>
<td>5-10μm</td>
</tr>
<tr>
<td><strong>Satellite droplets</strong></td>
<td>Causes satellite droplets as the ink is ejected at certain distance from the surface.</td>
<td>Does not cause satellite droplets as the ink is ejected when it is in contact with the surface.</td>
</tr>
<tr>
<td><strong>Viscous fluids</strong></td>
<td>Viscous fluid may clog the cartridge.</td>
<td>Viscous inks can be used as Spraying action can unblock if the tip is clogged.</td>
</tr>
</tbody>
</table>
2.2.4 Conventional Fabrication vs Printing Technology

Photolithography is the conventional technique used for patterning in the semiconductor industry. Optimized for inflexible, hard substrates and ultra-high resolution (<100nm) it requires complex manufacturing tools that are very expensive. This method involves several steps and complex procedures for fabrication. Also the subsequent deposition or annealing requires high temperatures and the etching releases a lot of toxic waste into the environment. Since the temperature for fabrication is high it cannot be used to fabricate with on organic substrates like plastics which reduces the cost. In addition, devices fabricated with conventional technology are rigid and cannot be used for wearable electronic systems. Though the current technology for fabrication gives higher resolution, printing provides an economic and efficient method for fabrication of devices on both flexible and rigid substrates. Hence, the main advantage of printed electronics over photolithography and other conventional methods of fabrication is that it is an additive process that does not involve high temperature, specialized vacuum equipment or substrates.

In photolithography, a thin layer of photosensitive material is first deposited by spin coating. A photomask of the required pattern is exposed to light which induces a chemical change in the photoresist on developing in the developer solution. A positive photoresist the exposed regions are soluble and hence removed and in negative photoresist the exposed regions are insoluble and stay on the wafer. Hence a 2D pattern of the photomask is formed on the wafer. In order to transfer the features to the sample surface, either wet or dry etching of the regions exposed are removed. The photoresist is not needed anymore and using a resist stripper the photoresist is removed. The wafer is then rinsed to remove any unwanted materials. As seen from the Figure 7 below, lithography involves several steps and this is a major limitation.

With printed technology being additive, the required pattern is drawn using a suitable software on a computer and a single layer is printed on the substrate. In order to have multiple layers in the pattern, the ink just has to be baked at a suitable temperature and another layer can be printed over it. This does not involve etching or developing and hence it is only a two-step process making is comparatively less complex.
2.2.5 LCR Meter

All the measurements were performed using Keysight E4980A/AL precision LCR meter. The E4880A/AL is used for the measurement of inductive, resistance and capacitive components, semiconductor devices over a wide range of frequencies from 20Hz to 2MHz. Up to 201 frequencies can be entered either manually or automatically using this LCR meter for different measurements. A four-terminal setup is used to measure the device under test with the signal path being as short as possible to avoid any noise. Different types of measurements can be done using the LCR meter by changing the primary and secondary parameter. Three different measurement time modes: short, medium and long helps to get accurate results. The table below shows the different parameters that were used for the work in this thesis.
Table 3: LCR Meter Measurement Parameters

<table>
<thead>
<tr>
<th>Primary Parameter</th>
<th>Secondary Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cp</td>
<td>Rp</td>
</tr>
<tr>
<td>Cs</td>
<td>Rs</td>
</tr>
<tr>
<td>Ls</td>
<td>Rs</td>
</tr>
<tr>
<td>Lp</td>
<td>Rp</td>
</tr>
<tr>
<td>Z</td>
<td>θd, θr</td>
</tr>
</tbody>
</table>

The subscript ‘p’ refers to all the measurements that were done in the parallel mode and ‘s’ refers to the measurements done in the series mode. For the measurement of small value of capacitance, since the reactance is large the parallel circuit mode is used as parallel resistance (Rp) has more significance than the series resistance (Rs). For larger values of capacitance since the reactance is small the series mode is used. A general rule of thumb that is used with the LCR meter for all measurements is any impedance higher than 10kΩ parallel mode can be used and when the impedances is less than 100 Ω the series mode is used for all measurements [29]. Conversely, in case of an inductor, the series mode is used for measurements with smaller value of inductance as the reactance is small and Rs is more significant than Rp.

2.3 Sonoplot Microplotter

This section gives the operation of the Microplotter for printing different patterns. The sonoplot Microplotter consist of the printing software called Sonoguide which is the main interface to the printer that controls the parameters required for printing. A vector based drawing software Sonodraw is used to draw the geometry of the device being printed with the Microplotter. Sonoplot Microplotter has a precision position control system which is controlled using the software either manually for the movement along the x and y axis so that the dispenser can be moved to any selected location or automatically when printing a pattern. A charge couple camera (CCD) helps in monitoring the printing action. The required ink is filled in the ink well from which it is filled into the micropipette attached to the dispenser using the capillary action. In addition to this it also has the controls for adjusting the printing parameters like dispensing strength, speed and acceleration for printing and other advanced settings for controlling the dispenser efficiently.
The head of the dispenser consists of the PZT to which the micropipette is attached. On vibrations at ultrasonic frequencies (400-700kHz) when driven by an ac current, the ink inside the micropipette tip is pushed out [8]. The main procedure for printing occurs in three steps: calibration of the dispenser and the surface, loading the micropipette with the ink and printing on the surface one feature at a time. A drawing Software called Sonodrw is used to draw the pattern which is then used to print using the control software called Sonoguide. Other designing software like AutoCAD or layout editor can be used and then converted into dxf format. A dxf importer converts the file into the format required by Sonodraw to print the patterns. The detailed procedure for the working of the Microplotter is explained in the Appendix section of this thesis.

2.3.1 Printing Patterns

Before printing, the pattern is brought close to the surface in the desired X, Y position using the “Find Surface” option. A pulse at a certain frequency and peak to peak voltage helps to find the surface which is obtained during the calibration step mentioned above. Continuous ac signal is applied to the tip and once it finds the surface, the tip is slightly pulled up while maintaining fluid contact with the surface for printing. Printing can be done while in contact with the surface or several microns up above the surface. While printing a few microns up, fluid contact has to be maintained with the surface.
without which it starts printing in air. The surface calibration that is done along the X and Y axis helps in proper Z positioning control so that if the height in the surface is uneven, since this was taken into consideration in the surface calibration step, the positioner will adjust the height as required. Since the tip is always slightly raised while printing with only the ink having contact with the surface, the glass tip is more safe and the chances of breaking can be avoided.

To start the printing process, once the dispenser and surface calibration is complete, the ink is loaded to the glass tips, the positioner is placed at the location above the surface where you want the pattern to start printing, set the required dispensing strength and patterning speeds and select the ‘Find Surface’ option. Once the tip comes in contact with the surface the print option helps to print the pattern from Sonodraw. Setting the pattern speed depends on what the shape of the pattern being printed. A very high patterning speed gives finer resolution whereas if the patterning speed is too low, the amount of ink that wicks out of the tip is more and spreading of the ink may occur. Depending on the diameter of the tip, the patterning speeds also have to be adjusted. At very slow speeds for a 60-micron tip, excessive ink is pushed out and the ink spreads. At very high speeds, the positioner is moving so fast that it does not complete the pattern feature precisely. So the exact value at which the ink flow is accurate and at the same time uniform while printing has to be properly set. This step takes a couple of attempts as it also depends on the temperature in the room being printed, humidity etc. In addition to setting the patterning speed, the dispensing strength applied to the dispenser also plays a very important role. The voltage can range from 0 volts- 20 volts. Usually the voltage is varied only between 0.1-0.5 volts even for viscous inks. The voltage that is being applied, tip diameter and the viscosity of the ink determines the thickness of the pattern that is printed. The frequency at which it prints depends on the resonant frequency of the dispenser that is obtained during the calibration process as mentioned above.

Once the printing has been complete, the remaining ink in the glass tip can be sprayed out by applying an AC pulse of higher peak to peak voltage than that was used for the dispensing action which agitates the micropipette and sprays all the ink out [9]. The glass tip is then cleaned by loading it with another solvent like DI water and then repeating
the same process of spraying again to completely clean the tip so that it can be loaded again with another ink.

![Diagram of Flexible Electronics Applications](image)

*Figure 10. Flexible Electronics Applications[30]*

### 2.4 Applications

Application of printed electronic devices are numerous including various medical and healthcare device, wearable devices, lighting and photovoltaic and memory devices. With the advent of Printed electronics, the production of innovative devices with new functionality, flexibility and higher production at lower cost could be achieved. As seen in the Figure 10 below, the flexible electronics industry is rapidly emerging with new applications in almost every part of the electronics industry.
2.4.1 Flexible Displays

Flexible displays have existed for years but it had not yet been implemented because thin film transistors on non-glass substrates was a challenge. However, with the use of polyimide plastic TFT were able to be manufactured on plastic substrates making it completely flexible. In addition to this, glass based TFT had higher chances of breaking while flexible TFT are more rugged, durable and lightweight.

![Flexible Displays and OLED](image)

*Figure 11. Examples of Flexible Displays and OLED*

Many companies have come up with bendable displays (Sony, Samsung) and also displays that are completely foldable like a newspaper (LG). The challenges associated with flexible displays is that the materials required need to be made from scratch and combined into one thin film making it a tedious process. Currently the research is on small screen flexible displays for phones and tablets, eventually the aim is to make larger screens for computer flat screens, eBooks, e-newspapers etc. Organic light emitting diodes is an LED in which printed layers of organic carbon is a emissive electroluminescent layer that produces light on exposure to electricity. OLED displays is a growing market today and is making a great future for flat displays.

2.4.2 Batteries

If flexible and wearable electronics devices, medical devices, printed identification tags have to be a successful reality an efficient portable energy storage method is required. With printed and flexible electronics emerging batteries and super capacitors have gained acclaim as when the devices are wearable and portable the power source should also be made flexible. Flexible Lithium ion batteries as a power source to power displays, RFID, wearable sensors will dominate the flexible electronics industry. Different flexible
electrolytes like organic electrolytes or polymers are used with a good ionic conductivity. Among this the gel polymer electrolytes are extensively used as they have low toxicity and good ionic conductivity [31]. Carbon nano tubes are for current carrying electrodes as they are lightweight and differ good conductivity which is the main criteria for flexible batteries [32].

Supercapacitors are known to be better than batteries as they require very less time to store energy with very low charging/discharging cycles compared to a battery[33]. Environmental friendly electronic devices that can be powered with paper based super capacitors are also being developed for lower cost and green energy to power these flexible electronic devices available today [34]. Graphene oxide combined with water is used for ink jet printing techniques to make graphene based electrodes on a Kapton substrate for the batteries is being researched[33]. Inkjet printing inks are very limited and the most commonly used is silver, however printing with silver for batteries which may be unstable in the electrolytes and hence graphene ink was made which had higher conductivity and higher mechanical properties than activated carbon which are usually used for electrodes[33]. Energy storage for flexible electronic batteries is a challenging task and to making it thinner affects the ionic conductivity and storage capacity of the battery. In addition to this, for the safe use of these batteries compact packaging is also essential to avoid any leakage or exposure to moisture which may degrade the performance of the battery [35].

2.4.3 Sensors

Sensors are used in biological, chemical, atmospheric or as multimode sensors. Printed sensors have advantages due to its conformability, scalability, and flexibility. Compared to other printed electronic devices, sensors are comparatively less complex to fabricate and hence can be used as an alternative to the conventional techniques. One of the printing techniques used is screen printing where a stencil of the required shape is made and ink is pushed through it using a squeegee. Printing usually involves the use of silver ink due to its high conductivity on the textile substrate over which a metallic ink is added for bio catalytic functionality for proper working and then an insulator layer is added for proper protection leaving the contact pads for measurements. Screen printed sensors are
used for different types of sensors like gas sensors, humidity sensors, biosensors etc. Screen printing technique was also used for making an energy efficient large size touch sensors on bendable surfaces for sensing pressure. This device was called as pyzoflex which is a four layered structure with a ferroelectric material that can exhibit both pyro and piezoelectric effects for sensing temperature and pressure [36].

Figure 12. Screen Printed PyzoFlex [36]

Other interesting applications of pressure sensors has prototypes that can be used in the field of portable digital music. However screen printing technique has a disadvantage that you need to have a mask and cannot be done on flat substrates [37] and you need a direct contact with the surface while printing [38].

To overcome this inkjet printing technique can be used without employing any masks or any other pre patterned mask. Inkjet printing of sensors is widely used today for RFID tags, antennas, biosensors and mainly wearable electronic devices. Different types of resistive, inductive and capacitive sensors have also been printed used for sensing. Using inkjet printing technique printing can also be done on flexible substrates like PET, PEN, Teflon, Kapton as they offer good flexibility, dielectrics with a good insulation property. Interdigitated capacitors are widely used for chemical and biological sensing today. A simple interdigitated capacitive sensor was used for the detection of ammonia on a PET substrate with a polymer ink PEDOT:PSS for the electrodes and a layer of polyaniline which is used to detect the presence of ammonia with a change in its conductivity[38]. Chemical sensors using silver ink are inkjet printed on PET substrates for humidity sensors [39]. Wearable sensors have become very popular for healthcare to check respiration rates,
skin temperature, blood pressure etc. Wearable capacitive sensor for respiration monitoring have been implemented. These sensors have a respiration belt that can be worn on the body which can measure the respiratory rate and other lung function parameters in the body[40]. There are several prototypes of wearable sensing devices are still in its prototype stage which can monitor the working of different parameters in the body and will soon become a reality.

2.4.4 RFID and Printed Antennas

Printed antennas already exist for used for ultra-low cost tags for different consumer goods. RFID tags have an antenna attached to it which uses electromagnetic field to interact with the reader and transmit information stored in its memory. Most of the RFID that were used before were manufactured using the etching process which was subtractive and hence the cost was more, to achieve a low cost efficient device with a high throughput printed RFID is a viable option as material is printed/deposited only in the required areas[41]. Printed RFID has simple steps wherein the antenna pattern is printed using a suitable ink, curing the ink to evaporate the solvent and integrated with the RFID chip also called as the transponder with the antenna. There are two main types of RFID tags: Passive and Active. The most important part of passive RFID is the antenna used to power the chip. The transceiver generates a signal which is transmitted from the antenna to the RFID tag. The signal that is transmitted to activate the tag is in the form of energy that can be used to power the transponder in the tag which displays any information that is stored in the tag. Active RFID tags have their own internal battery to power the circuit. The battery helps to transmit the signals to the reader and can be used over long distances. The printing technique chosen, ink used, temperature for curing and frequency ranges needs to go hand in hand for overall operation of the printed RFID. Some of the commonly printed RFID tags were done using screen printed method with a dipole antenna pattern using silver nano paste [43]. RFID tags that have been printed use silver ink because of its high conductivity on different substrates [41] [42]. Reconfiguration of antennas using “origami” help to adapt to the changing requirements useful for flexible electronics and biomedicai applications. Origami enabled antennas help to reconfigure the radiators operating characteristics, frequency of operation and radiation pattern[43]. As shown in the figure
below a flexible planar spiral antenna was modified into a conical antenna, increasing the gain of the antenna. The operating frequency could be changed by changing the height of the origami based helical antenna [43].

*Figure 13.* (a)(b) Origami Enabled Helical antenna, (c)(d) Flexible Planar Spiral Antenna converted to a Helical Antenna [43].
CHAPTER 3: MICROPIPETTE FABRICATION

An integral part of the printing process is the glass tips or micropipettes that are attached to the dispenser that pushes the ink onto the substrate. The diameter of the glass tips affects the width of the patterns that are being printed. The glass tips are very fragile and tend to break if it overshoots the surface, sudden movements while printing, dispensing strength is too high or the spraying voltage is very high. In addition to this, with repeated use the tips may get clogged and they have to be changed to continue printing efficiently. The glass tips do not have a very long life and they have to be regularly changed so that it does not hamper the printing. Since the glass tips have to be regularly changed, a glass puller was used to pull the glass with the required diameter for printing. Boron silicate glass with an inner diameter 0.86mm and an outer diameter of 1.5mm was used using the Sutter P-97 glass puller. The tips made by Sonoplot had a smallest diameter of 10 μm. With the stutter glass puller, even smaller tips of diameter of 1-2 microns could be pulled. However, with the Sonoplot Microplotting printer for extremely small tip diameter the capillary action to take the ink is not possible and hence cannot be used for printing. The smallest diameter tip that could be used where the capillary action takes place was a 3μm tip. Also the tips were fire polished to reduce the diameter of the tips and get the required diameter which will be discussed in the next section. Fire polishing is heating the pulled tips again decreasing the inner diameter and rounding the bottom of the outer diameter to give a well tapered glass tip. Fire polished tips strengthens the glass and was observed that even on hitting the surface or overshooting the surface by a few hundred microns, the tips did not break when fire polished. This technique of glass pulling helped to print with different types of tip diameters which are stronger and are more sturdy than the glass tips used by Sonoplot. The non-fire polished tips have a larger diameter of approximately 20-25 microns and the ends of the tips are more flat as a result of which they tend to break easily. To get tip smaller tip diameters they have to be fire polished multiple times.

3.1 Glass Puller

The stutter P-97 glass puller has a display which displays the program parameters that you enter. Upto 100 stored programs with different parameters can be stored in the puller controlled by a Z80 microprocessor. This makes it convenient to select the program
that needs to be run depending on the tip diameter required. The glass puller has a filament which heats up depending on the temperature of the glass which needs to be set in the program. The program can have a maximum of 8 lines in length that can be read where different values for heat, pull, velocity and delay/time values are entered depending on the diameter of the tips that are required.

Figure 14. Sutter P-97 Glass Puller [44]

The following parameters are set in the program to pull the tips:
1. Heat – The heat value that is set to heat up the filament to pull the glass depends on the type of glass that is being used. This is initially set by doing a ramp test. This test needs to be done whenever a new glass is used or when the filament is changed to obtain the glass transition temperature. The heat value that is set while pulling the glass is relative to the ramp test. The glass that is going to be pulled is placed inside the filament as shown in the Figure 14. The pull action is initiated during which the puller increments the heat at 650 milliamps per second. As the glass begins to melt the puller bars which hold the glass together starts moving apart on either sides. When a certain velocity is reached the heat value is turned off. This value is the melting temperature of the glass and needs to be used in pulling the tips.
2. Pull rate - This parameter controls the rate at which the hard pull occurs and the micropipettes snap and move away from each other to give two equal diameter tips. The stronger the pull, the smaller the tip diameter with a larger taper length can be achieved. If the pull rate is low larger diameter tips can be achieved with a shorter tapered length. Larger diameter tips can be further fire polished to make them stronger and to obtain a smaller tip diameter.

3. Velocity - The velocity of the puller bars at which the hard pull is initiated depends on the viscosity of the glass micropipette which in turn depends on the temperature at which the glass softens. Hence, the velocity parameter is the glass temperature at which the heat is turned off and the hard pull is initiated. Higher the velocity means longer is the pulling time resulting in a larger taper length with a smaller diameter tip. With lower velocities the time the hard pull is initiated is quick resulting in a shorter taper length with a larger diameter tip.

4. Time and delay - Time and delay are the two modes of cooling available. Setting a value for time sets the length of time for which cool air is passed. To control the delay time between when the heat is turned off and the hard pull is initiated the delay cooling mode is used. Higher the delay before the hard pull, larger is the tip diameter. However, this parameter has to be adjusted and does not have a definite value. As the filament heats up when you keep pulling the tips the delay and velocity have to be accordingly adjusted.

3.1.1 Heating Assembly

The main components of the glass puller include the filament, filament block assembly and the chamber. The filament is placed inside the chamber which protect the glass inside during the pull action. This can be adjusted or unscrewed when the filament has to be changed or when the glass has to be held in position. The filament block assembly has the upper and lower jaws from where the current is carried to the filament to heat it up. These jaws can also be moved and unscrewed but if they are not screwed properly then current flow to the filament is reduced and the filament does not heat up as expected. The glass puller has clamps which can be tightened by screws to hold the glass pipette in place before the hard pull is initiated and they are pulled apart. Once the hard pull is initiated the air cooling system supplies cool air to the filament. A reservoir tank is filled with drierite.
to remove any additional moisture in the atmosphere during pulling which affects the pull action. A piece of solenoid is present in the air cooling system that passes the cool air from the air reservoir tank to the nozzle located 2 to 3 millimeters below the filament. The nozzle is adjustable and can be moved if required using screws if the cool air is not supplied properly to the filament area. The nozzle is responsible to pass the cool air from the solenoid to the filament after the hard pull is initiated. A detailed fabrication procedure is explained in Appendix B of this thesis.

3.2 Mounting Tips

Once the glass micropipettes have been pulled to the required diameter, they need to be mounted onto the dispenser which can be used for printing. Mounting the glass tip to the dispenser has to be done very carefully to get the best results while printing. The dispenser is unscrewed and the RJ-11 and piezoelectric assembly is taken out. In order to remove the previous tip that is attached to the dispenser, it is dipped in acetone till the super glue holding the micropipette dissolves.

![Dispenser along with the RJ-11 Assembly](image)

*Figure 15. Dispenser along with the RJ-11 Assembly [45]*

In this step, only the long side of the piezoelectric should be dipped in the piezoelectric assembly and not the whole piezoelectric piece. Once the glue dissolves the glass micropipette is gently pulled out. The dried glue stuck to the piezoelectric is cleaned by using a blade to get all the dried bits out till a smooth shiny surface is obtained where the new tip can be attached. The long side of piezoelectric material has to be cleaned
properly before attaching a new tip. A clamp with one end holding the glass tip and the other end having the RJ-11 and piezoelectric assembly was used for mounting. The length of the glass micropipette is shortened at the wide end to reduce excessive length such that not more than 1mm protrudes from the top of the piezoelectric element. If not mounting to the dispenser is not possible as it would touch the ends of the dispenser. While cutting the wide end of the glass care needs to be taken to ensure that no tiny shards go inside the micropipette. If this happens then the ink is clogged with the tiny shards and it is not possible to print. Also, if the tiny shards go inside, then the glass micropipette cannot be used anymore and has to be changed. The tapered end is attached to the long edge of the piezoelectric material using super glue(cyanoacrylate) as shown in the Figure 16 below such that the bottom of the tip is 7-8 mm from the bottom of the piezoelectric element. The glue that is used to attach the glass micropipette needs to be thin quick dry glue and not in the gel form. also, while adding the glue care needs to be taken that the glue does not spread to either face of the piezoelectric element. A little glue at the long edge of the piezo is enough to hold the glass micropipette, if excessive glue is added then it causes a problem with the ultra-sonic vibrations of the printer and printing patterns is not effective. On the other hand, if less amount of glue is used, the micropipette does not stick to the piezoelectric element. Hence this is a very important step and the glue needs to added in precision for the proper transmission of ultra-sonic vibrations. Once the glue dries up the RJ-11 and piezoelectric assembly is placed inside the dispenser and held together with the screws. If the calibration curve of the dispenser is proper with a sudden low to high peak at 440 KHZ and the tip does not over shoot the surface when using the “find surface” option, then the dispenser is functioning correctly. In the whole process the tip of the micropipette is very delicate and should not come in contact with anything as it can shatter the tip.
Figure 16. Mounting the Fabricated Micropipettes to the PZT

3.3 Fabricated Micropipettes

The Figures below show different tip diameters that were pulled using the Sutter P-97 glass puller. The tip diameter after pulling the tip using the recipe mentioned in Appendix B was 20 \( \mu m \) and reduced to 10 \( \mu m \) after fire polishing as shown in Figure 17.

![Figure 17. (a) Non-Fire Polished 20 \( \mu m \) Tip (b) Fire Polished 10 \( \mu m \) Tip](image)

The 25\( \mu m \) tips that was pulled was pricked onto an elastic material or a Kleenex which chipped the tip in an uneven manner of the pulled micropipette. This increased the diameter of the tip to 50 \( \mu m \) as shown in the Figure 18. After this the tip was fire polished several times before the edges of the tips rounded and the diameter decreased as required. This method was used to pull larger diameter tips greater that 30 \( \mu m \) since the glass puller could only pull smaller diameter tips. As observed, the inner diameter decreased when fire polished to get the required diameter. Decreasing the ‘delay’ time while fire polishing can help in getting higher diameter tips. However, the tip will not be well tapered, which is
required for efficient printing. The delay option while fire polishing needs to be carefully adjusted for accurate diameters. The main challenge when fire polishing multiple times is the reposition of the micropipette after checking the tip diameter into the filament the same way it had been before.

Figure 18. (a) Non-Fire Polished 50 μm Tip (b) Fire Polished 15 μm Tip

Fire polishing to get extremely small tips less than 5 μm required precision. The tips are fire polished multiple times adjusting the delay time till the required diameter is obtained. While polishing to get smaller tip diameters, the tip might close if the delay time is set too high as it heats the tip for a longer interval of time and the tip closes. Hence they need to be constantly checked after every time is fire polished. The finger below shows the tip diameter of 10 μm after fire polishing the first time and then further reduced to 5 μm when fire polished again.

Figure 19. (a) Fire Polished Tip with a Diameter of 10 μm (b) Diameter Decreased to 5 μm on Fire Polishing again
CHAPTER 4: PRINTING OPTIMIZATION

This chapter covers the printing process, optimization and characterization of the Sonoplot Microplotter II for printed sensors on various substrates and using different inks. To optimize print efficiency, the important process and tools parameters were systematically studied and the quality of the printed patterns were examined. The process parameters altered includes the ink used and the substrates on which they are printed.

4.1 Optimization of Printer Parameters

Among the Sonoplot tools parameters, the dispensing strength as well as the speed and acceleration of the tip are the most important parameters that affects the printing. The dispensing strength, or the amplitude of voltage that is applied to the piezoelectric element, is the core of the Microplotter that uses ultrasonic vibrations to eject the ink out of the tip. This can be varied from 0.1 volts up to 20 volts. As common water-based nanoinks that are being used are not highly viscous, a voltage between 0.1 and 0.2 Volts are found to be ideal for printing. Going beyond 1.0 Volts can result in dispensing of the ink in an uncontrolled manner and the pattern is not well defined. Higher the applied voltage, more ink volume is dispensed out of the tip. The dispensing action occurs at the resonant frequency of the dispenser, which is set during the calibration process. The dispensing strength can also be set to 0 volts where minimum pulse is applied to the dispenser and dispensing action basically occurs because of surface tension, which depends on the tip diameter and the ink used. However, this can be used only for tips with larger diameters d>>10μm where the outflow of ink is relatively easily.

The motor speeds used for printing can be adjusted manually to explore the optimum printing conditions. This starts with the manual positioning of the dispenser assembly that achieves faster movement along the x, y and z axes as compared to auto placement. To move a larger distance, one can increase the speed and acceleration of the dispenser assembly that align the positioner above the surface. While approaching to the surface, the speed and acceleration must be decreased to prevent the tip from overshooting and shattering. Writing speeds can be adjusted at an appropriate level where the movement along the x, y and z axis is acceptable. The speed along the x, y and z axis plays an important role in the way the patterns are being printed. At very high speeds, the tips tend
to chip and break whereas at very low speeds, the ink jetted out is very high. An optimum speed must be selected for printing depending on the resolution and geometry of the pattern.

The optimization of the printer was done by changing the dispensing strength, patterning speed and also the tip retraction height during printing. While printing the tip comes in contact with surface and retracts several microns such that only the ink is in contact with the surface and then proceeds with the printing action. Changing these parameters helps to analyze the performance of Sonoplot Microplotter in different writing conditions. In doing so, all the patterns were printed using Novacentrix silver nanoink JS-B40G with 10 $\mu m$ tip diameter. This viscosity of the ink used for 8cP and was used due to its high conductivity and ease of printing. The standard writing conditions for this optimization study were drive voltage of 0.1V, Retraction height of 1$\mu$m and patterning speed of 3000 $\mu$m/s unless otherwise noted.

### 4.1.1 Variation in Dispensing Strength

The change in the dispensing strength is a critical measure on how to control the ink dispensing action. From 0V to 0.6V there was not much difference in the dispensing strength that was applied. The width of the line remained the same of approximately 60 $\mu$m but at the edges of the line as seen the ink start blotting or there is excessive ink deposition, as shown in Figure 20. When the dispensing strength was further increased to 1V, the width of the line increased by 10 $\mu$m. The ink starts spreading beyond 1.3V and the printing is no longer uniform beyond this point. Also, as seen from the same Figure 20, the ink that is dispensed is also not uniform along the entire pattern. On further increasing the voltage to 1.5 volts, there was an irregular dispensing action of the ink with a line width of 150 $\mu$m. The arcs and circular pattern is no longer defined and the printing action happens in an uncontrolled manner. Please note that each major axis on the scale corresponds to 100 $\mu$m and the smaller units are 10 $\mu$m in the figure.
Table 4. Observations on Ultrasonic Drive Voltage Change with Tip on Surface.

<table>
<thead>
<tr>
<th>Drive Voltage (V)</th>
<th>Linewidth (μm) (Average)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>60</td>
<td>Minimal jetting and limited continuity</td>
</tr>
<tr>
<td>0.1</td>
<td>60</td>
<td>Good print with uniform ink dispensing</td>
</tr>
<tr>
<td>0.2</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>0.3</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>0.4</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>0.5</td>
<td>60</td>
<td>Prints generally fine but blotting at the line edges as too much ink drops when the tip retracts</td>
</tr>
<tr>
<td>0.6</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>1.0</td>
<td>70</td>
<td>Excessive jetting &amp; ink spreads at parts.</td>
</tr>
<tr>
<td>1.1</td>
<td>75</td>
<td></td>
</tr>
<tr>
<td>1.2</td>
<td>85</td>
<td></td>
</tr>
<tr>
<td>≥1.3</td>
<td>140</td>
<td>Highly non-uniform linewidth</td>
</tr>
</tbody>
</table>

![Figure 20](image1.png)  
(a) Patterns Printed with Different Dispensing Strength (a) 0.1V (b) 1V (c) 1.3V.
The tip is on the sample surface. When the tip is raised at a certain height above the surface, even at relatively low drive voltages, a significant enhancement in the ejection of ink is observed. As a result, the dependence of drive voltage must be examined under different heights.

On retracting the tip 2 \( \mu m \) above the surface, it was observed that the printing pattern was not much different than when the tip was on the surface. The width of the line decreased approximately by 10\% at a dispensing strength of 0.1 volt, presumably with due to a slightly thicker line. As the drive voltage is increased the width of the line still increases. Even at a relatively low voltage of 0.3 V the ultrasonic derive of ink becomes very effective and flow of ink increases rapidly. Eventually at a higher voltage of 1.3 volts the printing is no longer uniform and linewidth varies largely at different positions.

Comparing these results to the case with tip on the surface, it is evident that the raised tip supplies more ink. Although the line thickness is reduced initially, none-the-less it rises faster with drive voltage. However, to keep the tip safe and prevent it from breaking when in contact with the surface, printing 2 \( \mu m \) up is a safer option.

\textit{Table 5. Observations on Ultrasonic Drive Voltage Change with Tip 2 \( \mu m \) from Surface}

<table>
<thead>
<tr>
<th>Drive Voltage (V)</th>
<th>Line Width (( \mu m )) (Average Values)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>55</td>
<td>Similar to printing on surface.</td>
</tr>
<tr>
<td>0.3</td>
<td>75</td>
<td>Excessive ink released as height increases</td>
</tr>
<tr>
<td>0.5</td>
<td>75</td>
<td></td>
</tr>
<tr>
<td>1.0</td>
<td>90</td>
<td></td>
</tr>
<tr>
<td>1.3</td>
<td>100</td>
<td>Excessive jetting &amp; ink spreads at parts.</td>
</tr>
<tr>
<td>1.5</td>
<td>165</td>
<td></td>
</tr>
</tbody>
</table>

For testing a number of different conditions, the printing height above the surface was further increased to 5 \( \mu m \). At this height, a few changes happen to the way the printing takes place compared to a height of 2 \( \mu m \) above the surface. At lower voltages, the print pattern looks the same but the controlled action of printing is not achieved for higher voltages at this height. Even with a relatively large tip diameter of 60 \( \mu m \), multiple passes
have to be done to get the entire pattern. Sometimes due to the uneven surface of the substrate it prints in air. Printing with $5 \mu m$ above the surface can be used when printing contact pads or a filled pattern. Raising the tip and printing is possible only with larger diameter tips greater than or equal to $10 \mu m$.

**Table 6. Observations on Ultrasonic Drive Voltage Change with Tip $5\mu m$ from Surface**

<table>
<thead>
<tr>
<th>Voltage (V)</th>
<th>Linewidth ($\mu m$) (Average values)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>60</td>
<td></td>
</tr>
<tr>
<td>0.3</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td>0.5</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td>1.0</td>
<td>-</td>
<td>Highly non-uniform linewidth</td>
</tr>
<tr>
<td>1.5</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

**Figure 21. Patterns Printed with Different Dispensing Strength (a) 0.1V (b) 0.5V (c) 1V**
4.1.2 Variation in Tip Height

For a more methodical comparison, the printing pattern was observed as the dispensing strength was kept constant at 0.1V at different heights. Printing raising the tip at a certain height is useful when the tips are very fragile (non-polished tips) or extremely small. As can be seen from the table below, the average linewidth is approximately the same at different heights from the surface for a tip diameter of 10 μm. However, it is necessary sometimes to print over multiple times as certain parts of the pattern maybe missing. Beyond 7 μm, practical printing ceases in air as there is no meniscus of ink formed between the tip and the surface. Also, it becomes especially difficult to print circular features at larger heights as lines becomes blurred. Hence, the main challenges when printing at a larger height above 5 μm are uniformity and blotting of ink. Based on the results from previous section, this upper limit of 5 μm becomes lower as the drive voltage is increased. Consequently, throughout this work we operate only with tips on surface or raised 1-2 μm above surface to ensure uniformity and continuity in lines.

*Table 7. Observations on retracting the Tip from the Surface*

<table>
<thead>
<tr>
<th>Height above surface (μm)</th>
<th>Linewidth (μm) (Average values)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>65</td>
<td>Good print with uniform ink dispensing</td>
</tr>
<tr>
<td>2</td>
<td>65</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>65</td>
<td>Prints have to be reprinted to get good continuity and there is blotting at the line edges as too much ink drops when the tip retracts</td>
</tr>
<tr>
<td>7</td>
<td>65</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>70</td>
<td>Does not print</td>
</tr>
</tbody>
</table>

4.1.3 Variation in Tip Writing Speed

The speed at which the pattern is printed has a significant effect on the amount of ink ejected out of the tip. Printing speed and acceleration alter the amount of ink coming out of the tip. Accordingly, the speed was varied and patterns were printed keeping the
height of the tip at 0 μm and 2 μm. At lower speeds the ink that is coming out is excessive and also leads to spreading of ink due to the vibrations. Printing is a little jerky at lower speeds and not very smooth. The voltage was kept constant at 0.1 volts for all the prints. The lowest speed that was tested for was 500 μm/s and the lines are not smooth as shown in the Fig. 22. As the speed increases the width of the lines decreases and the printing becomes more smooth and uniform with the spreading of ink decreasing. After multiple trials with different tips it was observed that a speed of 3000 μm/s is the most suitable for most of the prints with uniform straight lines. Higher speeds of 7000 to 10000 μm/s also give smaller linewidths but cannot be used for smaller diameter tips, since such high speeds can lead to breaking of the tips. Especially for circular patterns, printing at high speeds shatters the tip. An exception for printing at high speeds can be made for larger (d≥20 μm) tips to achieve smaller width lines.

Table 8. Observations on Varying the Tip Writing Speed

<table>
<thead>
<tr>
<th>Speed (μm /sec)</th>
<th>Linewidth (μm) (Average values)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>55</td>
<td>Convulsive printing with excessive ink jetting.</td>
</tr>
<tr>
<td>1000</td>
<td>55</td>
<td>Good print with uniform ink dispensing but blotting occurs at the edges.</td>
</tr>
<tr>
<td>3000</td>
<td>50</td>
<td>Ideal condition for printing with uniform ink dispensing.</td>
</tr>
<tr>
<td>5000</td>
<td>40</td>
<td></td>
</tr>
<tr>
<td>7000</td>
<td>40</td>
<td>Non-uniform linewidth and high speeds can damage the tips.</td>
</tr>
<tr>
<td>10000</td>
<td>40</td>
<td></td>
</tr>
</tbody>
</table>
Figure 22. Variation in the Operating Speed (a) 500micron/sec (b) 3000micron/sec (c) 5000micron/sec and (d) 1000micron/sec.

The printing speed was also varied at a height of 2μm from the surface. This has resulted in a broadly similar printing performance without substantial changes. At lower speeds the ink dispensed is more, especially when the tip is raised at a certain height. Accordingly, the linewidths almost doubled for a speed of 500 μm/s. For high speeds the resulting print performance were all similar and there were not significant changes with faster writing leading to thinner lines.

4.2 Printing on Flexible Substrates

Printing on flexible substrates using Sonoplot Microplotter II was a challenging task due to the fragile nature of the tips. Flexible substrates were rough, had undulations and calibration curves would have a high slope due to the difference in height along the substrates. The main problem with flexible substrates was the tip would overshoot the
surface at certain points and bend while printing resulting in the tips to break. To even out the surface and get minimum undulations a vacuum chuck was used to hold the flexible substrates tightly down. The vacuum chuck significantly improved the surface calibration, however the tips overshoot the surface at certain points bending the tip and shattering. Since the difference in height in flexible substrates was a major problem all the prints were done on the surface (not retracted during printing). On doing multiple prints it was observed when printing backwards in the y direction (vertical prints) the tip would bend and break. Printing in the forward direction along the y axis did not have any problem. This was due to the design of mounting the tips which was done along the vertical axis of the piezoelectric element which resulted in bending of the tip due to uneven surface heights of roughness in flexible substrates. Also, printing in the x direction (horizontal) was smooth without any bending. Since the software does not have control over the direction of printing, it was observed that if only one feature at a time is printed then the printing always happened in the forward direction. Even though this took longer to print the patterns, this was one of the methods to print so that the tip would not break. Printing with smaller diameter tips was not possible as the tips were too fragile and would chip as soon as the printing process was initiated. Different capacitors were printed on PET and Cannon extra glossy paper. For printing on flexible substrates Novacentrix silver nano ink JS-B25HV with a viscosity of 8cP was used. When printing contacts, the tip moves back and forth to complete the rectangular fill.

4.2.1 Poly Ethylene Terephthalate (PET)

Printing on a flexible substrate like PET was done using a vacuum chuck to hold the sheet down and reduce undulations. PET had a fairly smooth surface for printing. However, as mentioned above with PET as well, the tips would bend and chip during the printing process. Printing was done one feature at a time so that it prints in the forward direction. In addition to this, an option called ‘Find surface when printing’ was used. Usually while printing the tip retracts several microns to move to the next position after printing a feature. When this happens, with flexible substrates, sometimes the height difference is very high at certain points. Even though the surface has been calibrated, if the height difference is too high (>50 μm), it starts printing in air. To avoid this the ‘find
surface when printing’ helps to ensure that even when the tip retracts several microns before starting to print a new feature, it makes sure it ‘find the surface’ i.e. approaches as close as possible till the ink has contacted the surface before printing is continues. This happens every time the tip retracts to print a new pattern. This feature is very helpful when printing on flexible substrates due to the uneven heights along the sheet. Different capacitors and circular patterns were printed using a 50$\mu m$ tip diametr. The capacitance of three different capacitors with a gap of 500$\mu m$ and a finger length of 1000$\mu m$ is as follows.

![Figure 23. Printed Capacitor on PET](image)

**Table 9. Repeatability Test for a Capacitor on PET with a Gap of 100 $\mu m$ between Fingers**

<table>
<thead>
<tr>
<th>Number of trials</th>
<th>Capacitance (pF)</th>
<th>Phase Angle(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.545</td>
<td>-88.2</td>
</tr>
<tr>
<td>2</td>
<td>0.548</td>
<td>-88.6</td>
</tr>
<tr>
<td>3</td>
<td>0.588</td>
<td>-88.4</td>
</tr>
<tr>
<td>Mean/ Std Deviation</td>
<td>0.56/0.02</td>
<td>-88.4/0.2</td>
</tr>
</tbody>
</table>

As seen from the Fig 23 the width of the lines is 140 $\mu m$ with a spacing of the fingers. The starting and ending point of the fingers have blots of ink. When starting to print since the ‘find surface when printing option’ is used, every time the tip contacts the surface to print a new feature it leaves a blob of ink (which shows the ink made contact with the surface) and then continues to print. The blob of ink at the ends basically is present
due to the large diameter of the tip and the slow movement of the tip along the z axis. An average capacitance value of 0.56pF was obtained with a standard deviation of 0.02. The phase angle was also constant with an average value of -88.4° and a std deviation of 0.2°. The electrode fingers in the capacitors were brought closer to each other by reducing the gap between the electrodes to 200 \( \mu m \) using the same diameter tip of 20\( \mu m \).

**Table 10.** Repeatability Test for a Capacitor on PET with a Gap of 100 \( \mu m \) between Fingers

<table>
<thead>
<tr>
<th>Number of trials</th>
<th>Capacitance (pF)</th>
<th>Phase Angle(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.735</td>
<td>-88.23</td>
</tr>
<tr>
<td>2</td>
<td>0.778</td>
<td>-88.07</td>
</tr>
<tr>
<td>3</td>
<td>0.768</td>
<td>-88.21</td>
</tr>
<tr>
<td>Mean/ Std Deviation</td>
<td>0.76/0.02</td>
<td>-88.17/0.08</td>
</tr>
</tbody>
</table>

The value of the capacitance increased as the capacitor fingers were closer to each other. The width of the line is 90 \( \mu m \) with a spacing between the electrodes equal to 200 \( \mu m \). An average capacitance value of 0.76pF was obtained with a standard deviation of 0.02. The phase angle was also constant with an average value of -88.2° and a std deviation of 0.08°. Printing with tip diameter of 10\( \mu m \) resulted in chipping of the tips while printing. Gradually as the tip chipped, the diameter of the tip would increase and hence it was not possible to print patterns with smaller diameter tips for better resolution.

**4.2.2 Cannon Extra Glossy Paper**

Printing on paper is more challenging that PET as it is more rough. The tips were more fragile and would break if printing was not done carefully. The vacuum chuck was used to hold the paper tight down to reduce uneven surface difference. Again for printing on paper as well the ‘Find surface option was used ‘which resulted in blotting of ink at the starting and ending points. However, with the Cannon extra glossy paper, the ink would spread. Since a smaller diameter tip could not be used on flexible substrates due to shattering on coming in contact with the surface. just like PET capacitors with a gap of 200 \( \mu m \) was printed. Printing on paper gave a higher capacitance value of 0.96pF compared to other printed pattern on glass and PET. Three different trials were done to check for repeatability and to ensure proper working of the capacitors.
Table 11. Repeatability Test for a Capacitor on Paper with a Gap of 100 μm between Fingers

<table>
<thead>
<tr>
<th>Number of trials</th>
<th>Capacitance</th>
<th>Phase Angle(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.941</td>
<td>-88.71</td>
</tr>
<tr>
<td>2</td>
<td>0.965</td>
<td>-88.42</td>
</tr>
<tr>
<td>3</td>
<td>0.972</td>
<td>-88.21</td>
</tr>
<tr>
<td>Mean/ Std Deviation</td>
<td>0.95/0.01</td>
<td>-88.4/0.25</td>
</tr>
</tbody>
</table>

Figure 24. Printed Capacitor on Cannon Extra Glossy Paper

The capacitance value on paper was higher than when printed on glass or PET. An average capacitance value of 0.95pF was obtained with a standard deviation of 0.01. The phase angle was also constant with an average value of -88.4° and a std deviation of 0.25°.

4.3 Printing with Different Inks

The optimization of the printer was done using Novacentrix Silver nano ink JSB40G with 40% silver loading. This ink had a viscosity of 8cP. Printing with silver ink was done as the viscosity is not too high and the ink adhesion to the surface was good while printing. The compatibility and stability of this ink was good and hence used for all the comparisons. The silver nano ink JS-B25HV with 25% silver loading was used to print on flexible substrates. This ink also had a viscosity of 8cP. The spreading of ink was also less using this ink compared to different inks as mentioned below.

4.3.1 Poly Methyl Methacrylate (PMMA)

Printing with polymer solutions such as PMMA was also tested using Sonoplot Microplotter II. A solution of 5% PMMA was prepared with ethyl lactate as a solvent with
a viscosity of 8.34 cP. Due to its good dielectric properties they can be used to print as an insulating medium in electric devices. A higher diameter tip of 20\(\mu m\) was used to print patterns. Printing with PMMA led to spreading of the ink while printing. If a smaller diameter tip of 10 \(\mu m\) was used for printing, to avoid the spreading of the solution then the capillary action to take the ink into the tip of the dispenser was not possible. To efficiently print using this polymer the printing speed and voltage was adjusted. On adjusting the printing parameters, PMMA could be printed efficiently with a line width of 70 \(\mu m\) . The dispensing strength was set to minimum equal to 0.1 volt and the printing speed was increased to 10000 micron/second. Increasing the printing speed reduced the spreading of the ink when in contact with the surface.

4.3.2 Copper Oxide (CuO) Ink

CuO is another useful conductive ink that has various application as magnetic storage devices, super capacitors and sensors. Copper has a conductivity approximately 6% lower than silver with a lower cost [25]. However, the Cu nanoparticles tend to form copper oxides at ambient temperature decreasing the conductivity of copper and increasing the annealing temperature to a very high value. The metallic copper oxide ink from Novacentrix had similar characteristic and adhesion to the surface like the silver nano ink. ICI-002HV with a viscosity of 9cP was used for printing. The printing parameters required for printing with copper oxide ink was set similar to silver nano ink with a dispensing strength of 0.1 V and a printing speed of 3000microns/second. The Figure 25 below shows capacitors printed with CuO ink with a tip diameter of 15 \(\mu m\). However, the printed patterns were not annealed as CuO required high temperatures for annealing. A photonic curing process is used for CuO with Pulse Forging tools for curing. CuO ink was also tested on flexible substrates like PET and paper, showing good adhesion to the surface. It was observed that printing with CuO ink has similar characteristic to the silver nano ink.
To have a good conductivity of the printed patterns, curing of the ink at the appropriate temperature is a very crucial step. Unlike lithography which has several steps for fabrication. Printed electronics has only two steps, printing on the desired substrate with conductive ink and then curing the ink. Different inks have different curing temperatures and getting the temperature to the exact value so that the solvent is evaporated helps in improving the conductivity of the device. If the ink is not cured properly the conductivity of the device is low decreasing the efficiency of the device being printed.

Silver nano ink curing temperatures were studied with different printed patterns to get the exact range of heating the sample. To start with, first the sample was heated on a hot plate at 150 degrees for two hours but this was not sufficient and it did not cure the sample. On increasing the time to 4 hours at 150 degrees also made no difference. Hence it was observed that 150 degrees was too low and the temperature was further increased to 200 degrees. At this temperature, the resistance of the pattern was very high in the mega
ohms range and hence the conductive was still low. The sample was then placed in the oven to check for any changes and to get uniform heating. On comparison with the samples on the hot plate, the resistance was the same and did not change significantly. To improve the conductivity further the temperature was increased to 230 for two hours and this gave a considerable resistance of 95.4Ω with a phase angle of 0.01°. The Figure 26 below shows the pattern when cured on the hot plate without any pin holes or void with a good conductivity.

![Figure 26. Cured Silver Nano Ink at 230°C for Two Hours](image)

To further get a range of curing points, the resistance was increased to 300 in the oven after pre-baking the sample at 170 for 30 minutes. The sample was kept in the oven for one hour. The resistance increased to 130Ω with a phase angle of 0.02 degree. It was seen that at a few points the ink had also evaporated and left tiny pin holes or voids as seen in the Fig 27.

![Figure 27. Cured Silver Nano Ink at 300°C for One Hour](image)
The temperature was further increased to 350°C in the oven for one hour. This led to the ink being completely evaporated and hence it was concluded that this temperature was very high.

On repeated experiments, it was observed that a temperature range of 220-250 was in the considerable range with the resistances also being finite and not very high. Hence this showed that good conductivity could be obtained in this temperature range.
CHAPTER 5: PRINTED RESISTIVE, CAPACITIVE AND INDUCTIVE SENSORS

5.1 Resistor

This chapter deals with the use of Sonoplot tool and the developed process in printing passive R,L,C devices, which are the fundamental to all electronic circuits. Several example designs are considered and process issues are investigated, along with a practical example of resistive strain sensor that can be used to detect compressive and tensile strain on flexible surfaces.

Table 12. Repeatability Test of Resistor

<table>
<thead>
<tr>
<th>Number of trials</th>
<th>Resistance (Ω)</th>
<th>Phase Angle(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>19.32Ω</td>
<td>0.023</td>
</tr>
<tr>
<td>2</td>
<td>11.03 Ω</td>
<td>0.022</td>
</tr>
<tr>
<td>3</td>
<td>11.48 Ω</td>
<td>0.021</td>
</tr>
<tr>
<td>4</td>
<td>13.74 Ω</td>
<td>0.023</td>
</tr>
<tr>
<td>5</td>
<td>16.92 Ω</td>
<td>0.023</td>
</tr>
<tr>
<td>Mean/ Std Deviation</td>
<td>14.5/3.56</td>
<td>0.0224/0.00089</td>
</tr>
</tbody>
</table>

5.1.1 Straight Line Resistor

Though printing a straight line for resistors seems to be a simple task, the continuity of straight lines is sometimes an issue especially for smaller diameter tips. The conductivity of resistors changes depending on the amount of ink jetted out while printing. Overprinting needs to be done sometimes if the printing is not uniform at certain points to avoid any pin holes or breaks along the pattern. Also, prolonged line printing and the associated heating of the tip leads to uniformity and consistency of lines toward the very end of such long patterns. As expected and shown in Table 13, when the length of the line was increased the resistance also increased. Beyond 50mm lines, the uniformity in the line width decreases and they have to be over printed. From this experiment, it was observed that as the length of the line increases, there is a discoloration of ink at certain points: Darker brown spots which hamper the conductivity of the ink is observed in some instances. In places where there are brown spots, the conductivity of the ink is very low and hence the resistance increases disproportionately. This issue comes up mostly when printing larger length lines (greater than 30mm). This
observation was checked with multiple attempts of printing of lines with larger lengths. It is proposed that the appearance of dark spots is due to non-uniformity in printing especially when the tip overheats due to prolonged printing. Since the ink deposited at certain points is low and the rate of evaporation is higher when annealed, this leads to the ink being evaporated rapidly at such points causing a decrease in the conductivity.

Table 13. Variation in Resistance with increasing Length for a Straight Line Resistor

<table>
<thead>
<tr>
<th>Length of the Resistor (mm)</th>
<th>Resistance (Ω)</th>
<th>Phase angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>16.92</td>
<td>0.023</td>
</tr>
<tr>
<td>20</td>
<td>161.6</td>
<td>0.022</td>
</tr>
<tr>
<td>30</td>
<td>240.5</td>
<td>0.024</td>
</tr>
<tr>
<td>40</td>
<td>352.6</td>
<td>0.025</td>
</tr>
<tr>
<td>50</td>
<td>440.6</td>
<td>0.023</td>
</tr>
</tbody>
</table>

Figure 29. 30mm Line with Pin Holes after Curing

5.1.2 Zig-Zag Resistor

To further optimize the printed resistors, instead of straight lines, zig zag lines of different length were also printed. The zig zag angle for all the patterns were constant, changing only the length of the lines. For a zig zag angle resistor, the resistance was observed to be higher than a normal straight line resistor due to the current crowding at corners of the pattern. However, since tip spends slightly more times in forming a corner, broadening of the lines and formation of an ‘elbow’ can complicate this otherwise straightforward process.
Figure 30. (a) Zig Zag Resistors (b) Zig Zag Resistor showing the Width of the Line is 50 \( \mu m \).

Table 14. Variation in Resistance with Increasing Length for a Zig-Zag Resistor

<table>
<thead>
<tr>
<th>Length of the Resistor (mm)</th>
<th>Resistance (Ω)</th>
<th>Phase angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10mm</td>
<td>79.56</td>
<td>0.022</td>
</tr>
<tr>
<td>20mm</td>
<td>356.62</td>
<td>0.021</td>
</tr>
<tr>
<td>30mm</td>
<td>425.35</td>
<td>0.023</td>
</tr>
<tr>
<td>40mm</td>
<td>590.81</td>
<td>0.022</td>
</tr>
<tr>
<td>50mm</td>
<td>760.76</td>
<td>0.022</td>
</tr>
</tbody>
</table>

The next set of prints was done by keeping the length constant and then changing the angle at the turn of the zig-zag line segments. The length of each line segment was kept a constant at 10mm. It was observed that wider the zig-zag angle, larger the resistance of the printed resistors. Thus the angle of the corners had a significant effect on the resistance of the line printed. It is envisaged that small angles lead to excessive ink delivery in the corners and margining of each segment such that overall resistance is decreased.

Table 15. Variation in Resistance with Changing Angle of Peaks for a Zig-Zag Resistor

<table>
<thead>
<tr>
<th>Angle (degrees)</th>
<th>Resistance (Ω)</th>
<th>Theta (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>71.5</td>
<td>594.21</td>
<td>0.01</td>
</tr>
<tr>
<td>56.3</td>
<td>366.87</td>
<td>0.02</td>
</tr>
<tr>
<td>45.0</td>
<td>286.24</td>
<td>0.02</td>
</tr>
<tr>
<td>36.9</td>
<td>83</td>
<td>0.02</td>
</tr>
</tbody>
</table>
5.1.3 Piecewise Resistor

In drawing long (>10mm) lines, it became obvious that it useful to break the patterns into several segments for heath management and better continuity. Printing larger length lines is easier this way so that continuity can be obtained without overprinting. In some patterns with multiple arcs, curves and complex rectilinear patterns such break points are a necessity. However, when splitting lines into segments, a blob of ink is left at each joint before the next feature is printed since the ink is still dispensed or takes time to stop flowing. It was observed that this does have some effect with an observable change in the resistance. The blob or widening of linewidth in the joints is an artifact of printing. When the line is drawn with breaks on Sonodraw software, the tip retracts a few microns before starting a new print, leading to the formation of this artifact. The amount of ink that is dropped can be controlled by altering the z axis acceleration and speed. However, from a straight 5 mm straight line with different number of breaks, it was observed that there is not a significant difference in the resistance. The test was performed with a relatively large tip diameter of ~20μm. The resistance of a 5mm lines with different number of breaks is as shown in Table 16 below. It is clear that larger number of breaks on a straight line is leading to reduction in resistances, since each joint has lightly low resistance then a continuous line. The resistance was reduced by ~21% as a straight line is broken to 10 segments.

Table 16. Variation in Resistance when Printed with Breaks for a 5mm Line

<table>
<thead>
<tr>
<th>Number of breaks</th>
<th>Resistance (Ω)</th>
<th>Phase angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Continuous line</td>
<td>105</td>
<td>0.023</td>
</tr>
<tr>
<td>3 breaks</td>
<td>103</td>
<td>0.022</td>
</tr>
<tr>
<td>5 breaks</td>
<td>94.65</td>
<td>0.021</td>
</tr>
<tr>
<td>10 breaks</td>
<td>82.75</td>
<td>0.022</td>
</tr>
</tbody>
</table>

To further clarify this issue, a test was done for a longer 30 mm line as well. Again the resistance did show a 25% change as the line was divided up to 10 segments, which is significant. Hence it is important for high quality printing to reduce unnecessary splits in pattern. When impossible to avoid, the breaking points must be optimized so they do not create reliability and repeatability concerns for printed devices.
Table 17. Resistance when Printed with Breaks for a 30mm Line

<table>
<thead>
<tr>
<th>Number of breaks</th>
<th>Resistance (Ω)</th>
<th>Phase angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Continuous line</td>
<td>495</td>
<td>0.021</td>
</tr>
<tr>
<td>3 breaks</td>
<td>484</td>
<td>0.021</td>
</tr>
<tr>
<td>5 breaks</td>
<td>385</td>
<td>0.022</td>
</tr>
<tr>
<td>10 breaks</td>
<td>370</td>
<td>0.023</td>
</tr>
</tbody>
</table>

Figure 31. Patches of Ink at Different Points

5.1.4 Printing with Smaller Diameter Tips

Resistance measurements was also done with different tips to see the performance of the printer. However, for an extremely small tip of 3 μm, a 10 mm line could not be printed as they would end up with breaks and the ink delivery is not consistent to form a continuous straight line. Several attempts were made to print a straight line with a 3μm tip, but the resistor had continuity issues. The only solution was to overprint the line twice so that there would be no breaks and the resistance can be measured. For the sake fairness, the resistor pattern was overprinted twice using other tips in this study as well. The resulting linear resistors are shown in Fig.32, where the linewidth is increasing with tip diameter as expected. The measured resistance values are compared in Table.18.

Please note that each major axis on this scale corresponds to 50 μm and each minor axis corresponds to 5 μm.
It is found that the resistance goes down as the diameter is increased as expected. It is clear from Table 18 that while the measured width increases, each line also gets thicker as larger tips are used. Hence the observed large reduction of resistance is not due simply to change in the width of the resistor but also to the increasing thickness of the ink material delivered. Using the data obtained from DekTak-2a profilometer (see also Table.18) we can also calculate the resistivity of lines and the linewidth made by each tip.

The reason for a decrease in resistivity with tip diameter can be explained due to the roughens and spreading of ink with the larger diameter tips. Also with larger tip diameters, the printing is not completely uniform resulting in a variation in linewidth throughout the pattern.

### 5.2 Capacitor

To test the use of the Sonoplot solution printing/dispensing system for electronic manufacturing, we optimize the necessary printing process and provide characterization of

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**Figure 32.** Optical Image of Printed Resistor with (a) 3 \( \mu m \) Tip (b) 5 \( \mu m \) and (c) 10 \( \mu m \)

**Table 18.** Characteristics of the Lines when Printed using different Diameter Tips

<table>
<thead>
<tr>
<th>Tip Diameter (( \mu m ))</th>
<th>Line Width (( \mu m ))</th>
<th>Thickness (( \mu m ))</th>
<th>Std Deviation (( \mu m ))</th>
<th>Resistance (( \Omega ))</th>
<th>Resistivity (( \Omega ). cm)</th>
<th>Phase Angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>23</td>
<td>0.16 ± 0.0192</td>
<td>0.006</td>
<td>1100.01</td>
<td>4.02e-5</td>
<td>0.032</td>
</tr>
<tr>
<td>5</td>
<td>32</td>
<td>0.221 ± 0.0207</td>
<td>0.006</td>
<td>473.7</td>
<td>3.18e-5</td>
<td>0.031</td>
</tr>
<tr>
<td>10</td>
<td>64</td>
<td>0.303 ± 0.0249</td>
<td>0.008</td>
<td>94.1</td>
<td>1.83e-5</td>
<td>0.032</td>
</tr>
</tbody>
</table>
capacitor devices using conductive inks. An interdigitated capacitor was used throughout all the prints with a geometry as shown in the Figure 33 with air as a dielectric.

![Figure 33. Layout of the Printed Interdigitated Capacitor Finger length=1000 µm; Number of Fingers=10; Electrode Gap = 100µm](image)

Novacentrix silver Nano ink JS-B40G was used initially for printing for optimization of the printer as the conductivity of silver nano ink is high. An interdigitated capacitor design as shown in the Fig 33, drawn using Sonodraw plotting software, was printed on a glass substrate. The gap between the lateral electrode fingers is 100µm and contacts used for the electrical measurements were 3mm×3mm. The patterns were printed using a relatively large tip with a diameter of 30µm. Once the capacitor was printed they were pre baked at 170C for 30 min and then cured at 230c for 1 hour on a hot plate. The measurements were performed using the Agilent E4980A LCR meter at a frequency of 100 KHz. The capacitor gave a capacitance Cp of 0.5pF, with a phase angle of -89.88 degrees and parallel resistance Rp of 60MΩ. The Fig.34 below shows the images of the printed capacitors. Each major axis on the scale corresponds to 100 µm and the smaller units are 10 µm.
To check the repeatability of the device several more prints with the same tip diameter (20 𝜇m), printer settings (dispensing strength=0.1V and speed=3000 𝜇m/s), curing temperature (230°C) and on glass substrate was performed. Five different printed capacitors gave capacitance with approximately similar values and exhibited good conductivity. Table 19 below shows the outcome of all capacitance measurements, indicating that the process is repeatable and fabricated capacitors possess an average value of μ_{Cp}=0.56pF and standard deviation (σ_{Cp}) of 0.019pF (~3.3%) over 5 different trials. This corresponds to ~10% variation using three sigma (±3σ) metrics. The statistical analysis of the measured phase angle for five capacitor indicate even a better picture with an average angle of -88.1° and standard deviation = -0.6%.

Table 19. Capacitance Measurements

<table>
<thead>
<tr>
<th>Number of trials</th>
<th>Capacitance(pF)</th>
<th>Theta(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.561pF</td>
<td>-88.9</td>
</tr>
<tr>
<td>2</td>
<td>0.572pF</td>
<td>-88.25</td>
</tr>
<tr>
<td>3</td>
<td>0.536pF</td>
<td>-87.61</td>
</tr>
<tr>
<td>4</td>
<td>0.573pF</td>
<td>-88.2</td>
</tr>
<tr>
<td>5</td>
<td>0.586pF</td>
<td>-87.6</td>
</tr>
<tr>
<td>Mean/ Std Deviation</td>
<td>0.56pF / 0.019pF</td>
<td>-88.11 / -0.53</td>
</tr>
</tbody>
</table>
The analysis of the printed capacitance is also performed with varying frequencies from 400kHz to 2 Mhz. There is not a significant change in the value of the capacitance upto 1.5MHz after which the capacitance starts to decrease with frequency. The variation over this frequency range is limited to 0.07%.

![Impedance vs Frequency](image.png)

*Figure 35. Frequency Sweep of the Interdigitated Capacitor*

Next, the dimension of the capacitor was changed to check the performance of the printed pattern under different geometries. Different parameters like the distance between the electrode fingers, length of the electrode fingers and the number of fingers was changed.

### 5.2.1 Varying Distance between the Electrode Fingers

Using a standard capacitor design with a length of 3000\(\mu m\) and electrode gap of 100\(\mu m\), the number of electrodes were varied in this section. As before, the patterns were printed with the same 20 \(\mu m\) tip with a printing speed of 3000\(\mu m/s\) and the dispensing voltage of 0.1V.
Table 20. Change in Capacitance as the Distance between Electrode Varies

<table>
<thead>
<tr>
<th>Gap between the electrodes (μm)</th>
<th>Capacitance (pF)</th>
<th>Theta (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0.76pF</td>
<td>-88.23</td>
</tr>
<tr>
<td>300</td>
<td>0.67pF</td>
<td>-82.64</td>
</tr>
<tr>
<td>500</td>
<td>0.57pF</td>
<td>-82.78</td>
</tr>
<tr>
<td>1000</td>
<td>0.42pF</td>
<td>-80.92</td>
</tr>
<tr>
<td>1500</td>
<td>0.34pF</td>
<td>-83.81</td>
</tr>
<tr>
<td>3000</td>
<td>0.33pF</td>
<td>-82.89</td>
</tr>
</tbody>
</table>

As expected and can be seen from Table 18 above, increasing the gap between the electrodes of, the capacitor decreases as the coupling between them decreases, leading to drop in measured capacitance. As the electrode gap was varied from 100 μm to 3000 μm and a decrease in the capacitance from approximately ~0.76pF to ~0.33pF was observed, corresponding to a 50% reduction. The average phase angle theta was -83.7°, which is reasonable and has a standard deviation of 2.75° (or 3.2%). It can be seen from the images provided in Fig. 36 below that, even though the gap between the electrodes of the capacitor were set as 300 μm, due to the spreading of the ink while printing, the actual gap has reduced to 240 μm and 440 μm respectively. While this introduces a concern, the difference between the design and real devices can be explained by varying gap between the tip and the surface between the two writing sessions, which was not kept constant 2 μm.

Figure 36. (a) Gap between the Electrodes is 300 μm and (b) 500 μm
5.2.2 Number of Fingers/Electrodes in the Capacitor

Using a standard capacitor design with a length of 3000\(\mu m\), with a gap of 100 \(\mu m\) between the electrodes the number of fingers were varied in this section. All the capacitors were printed with the same tip with the printing speed of 3000\(\mu m/s\) and the applied voltage to be 0.1V. Increasing the number of electrodes 10 to 30 in this study increases the charge storing ability of the capacitors from 0.75pF to 3.61 pF with an average angle of -88.41 and standard deviation of 0.66 degrees. The growth of the capacitance is monotonic but not linear, which is indicative of imperfect printing or possible conductivity changes between the fingers.

Table 21. Change in Capacitance as the Number of the Finger Increase

<table>
<thead>
<tr>
<th>Number of Fingers</th>
<th>Capacitance (pF)</th>
<th>Theta(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.75</td>
<td>-88.26</td>
</tr>
<tr>
<td>15</td>
<td>1.02</td>
<td>-89.54</td>
</tr>
<tr>
<td>20</td>
<td>1.37</td>
<td>-88.41</td>
</tr>
<tr>
<td>25</td>
<td>2.28</td>
<td>-87.98</td>
</tr>
<tr>
<td>30</td>
<td>3.61</td>
<td>-87.88</td>
</tr>
</tbody>
</table>

Printing large number of finger patterns proved to be challenging. Initially the ink ejected out of the tip in a controlled manner. But eventually, excessive ink wicked out of the tip resulting in poor linewidth quality. On multiple trials, it was observed that typically beyond 20 fingers, excessive ink ejected out. To overcome this the tip needs to rest for a few minutes between consecutive prints, or print in several breaks if processing large patterns using larger diameter tips (>10 \(\mu m\)). This indicates that overheating is an issue at the tip and either time or temperature must be managed to succeed in writing a large pattern. In other words, temperature control may be necessary for prolonged writing sessions, especially when friction is high with larger (d>10\(\mu m\)) tips.
5.2.3 Varying the Length of the Fingers

Using a standard capacitor design with a gap of 100 \( \mu m \) between the electrodes and 10 fingers the length of the fingers was varied in this section. Increasing the length of the fingers resulted in increasing the overall area of the capacitor thereby increasing the capacitance.

<table>
<thead>
<tr>
<th>Finger Length (( \mu m ))</th>
<th>Capacitance(pF)</th>
<th>Theta(degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>0.59</td>
<td>-86.43</td>
</tr>
<tr>
<td>3000</td>
<td>0.72</td>
<td>-78.94</td>
</tr>
<tr>
<td>5000</td>
<td>1.62</td>
<td>-76.42</td>
</tr>
<tr>
<td>7000</td>
<td>1.87</td>
<td>-68.78</td>
</tr>
<tr>
<td>10000</td>
<td>2.51</td>
<td>-58.93</td>
</tr>
</tbody>
</table>

As seen from the plot above the capacitance increases significantly from 3000 \( \mu m \) which has a capacitance of \( \sim 0.72 \)pF to \( \sim 2.5 \) pF at 10000 \( \mu m \). The phase angle had an average value \(-75^\circ\) with a std deviation of 10.5 \( ^\circ \). This happens because to print a line as long as 10000 \( \mu m \) may have continuity issues as mentioned in Section 5.1.1. The main problem with larger length lines is uniformity as the ink wicked out is different as you go along the line. This may be due to uneven points on the surface because of which when the tip is not close to the surface the amount of ink ejected out is less. This problem does not happen for larger tips (\( > 50 \mu m \)) where the flow of the ink onto the substrate is continuous and does not have any issues. This also affects the curing process as where the ink is less
the solvent evaporates quickly leaving voids whereas at points where excessive inks are present, the ink would not dry. However, for extremely small tips (3-5 microns), to get continuous lines without breaks is challenging and over printing is the best solution.

### 5.2.4 Printing with Smaller Diameter Tip

For printing with smaller tips, the biggest challenge is pushing the ink out of the tip. Also sometimes, for extremely small fire polished tips the capillary action does not happen for the ink to filled into the tips. In such cases, the tip has to be changed as printing is not possible with it. To show the characteristics of the printer, capacitors were also printed using smaller diameter tips for the best resolution. The smallest tip that was used to print was approximately 3\( \mu m \). The capillary action for such small tips is not as good as the larger tips. Very little ink is filled inside the micropipette and hence ink must be filled multiple times if a large pattern is to be printed. Also with smaller diameter tips they must be repeatedly cleaned after every print thoroughly to avoid any clogging. If not, the tip gets clogged quickly and then cannot be used for printing and must be discarded. Since such small tips are fire polished, cleaning is also not very easy as the spraying action causes the tips to clog. Cleaning of tips smaller than 5 \( \mu m \) is not possible as they get clogged once a pattern has been printed. Using a standard capacitor design with a gap of 100\( \mu m \) and 50 \( \mu m \), 10 fingers and finger length 1000 \( \mu m \), the tip diameter was changed.

### Table 23. Change in Capacitance with Varying Tip Diameter

<table>
<thead>
<tr>
<th>Tip Diameter (( \mu m ))</th>
<th>Gap Between the Fingers (( \mu m ))</th>
<th>Capacitance (pF)</th>
<th>Phase Angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>50</td>
<td>0.143</td>
<td>-84.27</td>
</tr>
<tr>
<td>3</td>
<td>100</td>
<td>0.031</td>
<td>-74.87</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>0.176</td>
<td>-87.23</td>
</tr>
<tr>
<td>5</td>
<td>100</td>
<td>0.129</td>
<td>-84.65</td>
</tr>
</tbody>
</table>
Figure 38. Printed Capacitors with Tip Diameter (a)(b) 3 μm and (c)(d) 5 μm with a gap of 100 μm between the Electrode Finger.

With the gap between the electrode as 100 μm, the capacitance was measured to be very low as the amount of silver nano particles deposited while printing was less. On increasing the diameter of the tip to 5 μm, there was an improvement in the capacitance and the phase angle of the capacitor. Also, as seen in the Figure 38 above with extremely small diameter tips, the printing is not very smooth and is a little convulsive. For a 3 μm tip, the measured value of the capacitance was very low with a value of 0.08pF, the width of the line is approximately 20 μm with a gap of 70 μm after printing. For a 5 μm tip, the capacitance increased to 0.12pF with the width of the line increasing to 30 μm with the gap between the electrodes as 60 μm. The Table 21 above shows the measured capacitance for smaller diameter tips with different gap between the electrodes.

Summarizing the characteristic of the capacitor when printed with different diameter tips when the gap between the electrodes in 100 μm with 10 interdigitated fingers is as shown above. Larger diameter tips greater than 20 μm give a higher capacitance value but the gap between the electrodes have to be increased in order to avoid shorting between the fingers.
5.3 Inductor

Apart from having the right geometry for an inductor, low resistance inductors can only be achieved by having a thick layer of conductive nanoink. A square spiral inductor was first printed with a single layer with the contacts in the center as shown in the Figure 39. The square spiral inductor has 10 turns with the spacing between them as 200 $\mu$m. It was observed that printing with this geometry made the inductor have a high resistance in the G$\Omega$ range with a higher capacitance value. The capacitance was very large and hence the phase angle was also lagging at -90 degrees. The same pattern was also printed for a circular spiral which had the same effect. The spacing between the turns was increased to reduce the capacitive effect, this also had no improvements in the working of the capacitor. The size of the contact was decreased at the center to improve the magnetic field. This had some improvement in the inductor. But even though the phase angle was at 87.4 degrees at 100 KHz, the value of the inductance was very high at 33.56H with a high resistance of 199.2k$\Omega$ for a 10 turn inductor with a spacing of 200 $\mu$m between the turns and the width of the line being 30 $\mu$m. This seemed to be unreasonable for a small printed inductor. On increasing the frequency to 2MHz, the inductance decreased to 70mH but with a high resistance value of 12.2 k$\Omega$. Hence the resistance and the inductance value that was measured was too high. The continuity in the inductor was not good which led to such high resistance and low conductivity. On carefully observing the inductors, there were tiny pin holes or voids at certain points. Even the smallest pin hole would not make the inductor work. Multiple layers were printed consecutively to increase the width of the lines significantly and also decrease the possibility of any void in the pattern.

<table>
<thead>
<tr>
<th>Tip Diameter ($\mu$m)</th>
<th>Capacitance (pF)</th>
<th>Phase angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>0.031</td>
<td>-74.88</td>
</tr>
<tr>
<td>5</td>
<td>0.128</td>
<td>-84.65</td>
</tr>
<tr>
<td>10</td>
<td>0.373</td>
<td>-88.92</td>
</tr>
<tr>
<td>15</td>
<td>0.568</td>
<td>-88.98</td>
</tr>
</tbody>
</table>

Table 24. Change in Capacitance when Printed with Different Diameter Tips
The table below shows, the change in inductance and resistance for a 5 turn inductor when the number of layers for printing increases which was measured using the LCR meter at 1kHZ. Also it was observed that, printing multiple layers (one on top of the other) led to spreading of the ink and dragging the ink at certain points which would result in the turns getting shorted. Instead the lines were printed as parallel lines next to each other to increase the thickness of each turn. As the number of layers increased, the ink spreads and eventually it is like over printing on the same line. Printing more than 5 layers led to excessive spreading of the ink without good edge definitions and hence could not be done. Since at the edges the ink deposited was more, printing more than 5 layers led to excessive spreading of the ink around the edges.

Table 25. Inductance Measurement with Different Number of Printed Layers

<table>
<thead>
<tr>
<th>Number of Printed layers</th>
<th>Inductance(Ls)</th>
<th>Resistance(Rs)</th>
<th>Phase Angle (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single Print</td>
<td>14.56H</td>
<td>186.2kΩ</td>
<td>88.7</td>
</tr>
<tr>
<td>Double layer</td>
<td>8.6H</td>
<td>120kΩ</td>
<td>84.5</td>
</tr>
<tr>
<td>Triple layers</td>
<td>20mH</td>
<td>15.65kΩ</td>
<td>66.2</td>
</tr>
<tr>
<td>5 Printed Layers</td>
<td>5.4μH</td>
<td>66Ω</td>
<td>0.02</td>
</tr>
</tbody>
</table>
5.3.1 Phase Angle

It was observed that the measurements of printed inductors using the LCR meter consistently resulted in $\theta = 0^\circ$ phase angle and a resistance of $66\Omega$ for all designs attempted, despite utmost care in fabrication and characterization. The resistance in the model is relatively high for the value of the inductance measured. This high value of resistance within the inductor can be minimized by using a higher viscosity ink to improve the conductivity within the turns of the inductor and minimizing the resistance. Also, increasing the number of turns within the inductor can help to improve the overall performance of the inductor.
5.4 Application- Bending Sensor

A low cost sensing device was fabricated using the Sonoplot Micrplotter. Two different designs were implemented to show the working of the device [46][47]. Both the sensors were printed with a 15 $\mu m$ tip for better comparison. The resistance of the device changes when subjected to compression or expansion. The strain sensor was printed on a PET substrate with a conducting silver nanoink from Novacentrix, JS-B25HV. The resistor was flexed at different radii and the measurement of the resistance was obtained using LCR meter. It was observed that bending outwards resulted in increasing the resistance of the device due to the cracks formed between individual nanoparticles when stretched while bending outwards. Whereas bending inwards, results in a decrease in the resistance as the nanoparticles in the ink cluster together increasing the conductivity of the device. The prototype of the resistor is as shown in the Figure 40 with the following dimension: Total Length 2cm, the spacing between the tracks is 200$\mu m$, width of each track is 200 $\mu m$ with 10 tracks. A 15 $\mu m$ tip diameter was used to print the device.

![Prototype of the Flexible Sensor](image)

*Figure 40. Prototype of the Flexible Sensor*

The printed pattern was cured in a vacuum oven at 100$^\circ$C for 1 hour. The initial resistance of the device before bending was 3.64k$\Omega$. Objects with different radii was used to measure the variation in resistance of the device on bending. The resistance was flexed around different circular objects with different radii to observe the change in the resistance of the device. The following results were obtained when the resistor was flexed in the outward direction.
As seen from Figure 41, the resistance decreases significantly from 7.27kΩ to 3.95kΩ on increasing the bending diameter. Decreasing the bending diameter results in flexing the sensor more hence increasing the resistance. There was an increase in resistance by 45% as the radius of the object decreased [46]. The phase angle was an average value of 0.022° with a std deviation of 0.008°.

Figure 42. Optical Image of the Printed Sensor corresponding to Fig 41.
The same resistor design was also flexed in the inward direction resulting in the decrease of resistance as the radius of the object decreased. The radius decreased from 3.51 kΩ to 2.21 kΩ resulting in a 35% decrease as shown in Fig. 43.

![Figure 43. Variation in Resistance with Bending Diameter when Bending in Inward Direction](image)

The length of the resistor in the sensor was decreased to 1 cm with 20 tracks and a reduction in the resistance by 20% was observed when bending at different radii. Since the length of the resistor was decreased and the cross-sectional area was increased the overall resistance of the device reduced. The prototype of the resistor had the following dimension: Total Length 1 cm, the spacing between the tracks is 200 μm, width of each track is 200 μm with 20 tracks. The characteristics of the device were similar to the previous sensor. Initially the resistance was 3.01 kΩ before bending the sensor. On flexing the sensor with different radii objects, an increase in resistance from 3.05 kΩ to 5.79 kΩ was obtained as the diameter of the objects decreased as shown in Fig 44. The resistance value increased by approximately 40% from the initial value [46]. The average phase angle for this resistor was also 0.022° with a std deviation of 0.023°.
The same resistor was also flexed in the inward direction resulting in a decrease in resistance as the diameter of the object decreased as shown in Fig 45. The reduction in the resistance was by 25%. Several measurements were done to check the reliability of the device with different diameter objects.

**Figure 44.** Variation in Resistance with Bending Diameter when Bending in Outward Direction

**Figure 45.** Variation in Resistance with Bending Diameter when Bending in Inward Direction.
CHAPTER 6: CONCLUSION

A novel printing approach was studied in this work for printed electronic devices. The performance of capillary delivery based microplotting approach was optimized under different conditions for efficient printing. The characteristics of passive LCR components was also analyzed on a broad range of designs, inks, printing conditions and substrates. The change in the capacitance on changing the geometry of the interdigitated capacitor and printing parameters was observed. Printing of the inductor had several issues with the measurements, which required that parasitic series resistance and parasitic parallel capacitance had to be taken into account to explain observed phase angle being 0°. A tri element model was constructed for the LCR meter and the impedance and phase angle was calculated which matched with the experimental results. The main observation on printing inductors was that there should be no voids in the pattern when printing. Hence multiple prints had to be done and cured properly for good results. Different straight line resistors and zig zag resistors were also printed under different conditions. In addition to this, a printed strain sensor was also fabricated with two alternative designs. It was observed that on expansion and compression of the device there was a change in the resistance and this could be used as a resistive sensor for different wearable applications.

The minimalist design of the resistors, capacitors and inductors had a value in the range of 15Ω, 0.5pF and 5.0µH, respectively, depending on the glass tip diameter as well as printing parameters. The value of these components can be increased by changing the geometry of their design: For instance the number/length of fingers or gap between the fingers for capacitors and number of turns for the inductor. Substrate and ink used for printing also has an impact on the process and final component value: glass, polymeric and paper surfaces are confirmed to be compatible with Sonoplot along with conductive (Silver (Ag), Copper(Cu)) and insulating (Poly methyl methacrylate (PMMA)) inks. For instance, interdigitated capacitors on PET polymer surface increased the capacitance by 20%. The printed R,C,L component values has a dependence on the way the pattern was printed such as tip diameter, tip-sample gap and curing temperature, with an average resistivity of $3.01 \times 10^{-5} \Omega \cdot \text{cm}$ for the silver nanoink. Continuity is a concern for larger (>10mm) printed patterns since the tip can get warm or deformed. Hence multiple overprints must be done
for larger patterns to ensure good conductivity or it can be written in multiple sessions with short (~1min) breaks for cooling. For example, the spiral inductor had to be printed with 5 layers to reduce parasitic series resistance below an acceptable value (~10ohms). Beyond 5 layers, the ink starts spreading and integrity of the inductor is questionable in short gaps.

The micropipettes that were used for printing was fabricated using a glass puller to achieve stronger tips than the tips provided by Sonoplot. Smaller diameter tips could be pulled using the fire polishing technique to get tips less than 5 \( \mu m \) that could be used for printing. Since the dispensers are expensive to buy, fabrication of the micropipettes helped to reduce the cost and improve the quality of the tips. Several problems were encountered while printing using this technique which will be discussed in section 5.1.

6.1 Challenges

6.1.1 Continuity

The main challenge with high-value (larger length) resistors was continuity in the patterns while using smaller diameter tips. When the length of the resistors was very large (>40mm), there could be breaks in the line or voids as it prints, if the surface traction fails. Also, the ink deposited at certain points was less which led to brown spots on curing the sample due to rapid evaporation. As a result of this, the expected conductivity of the pattern can decrease appreciably. Therefore, it is essential to have a flat and vibration free substrate and high-quality surface traction using the multi-point correction maps available in the software as an option. This is especially true when printing very small resistors that are sensitive to such momentary lapses in linewidth.

6.1.2 Micropipettes

Microplotting is a relatively new form of printing using micropipettes of different diameters. Since these tips are fragile, they tend to break in friction with the surface. The life of a tip can be short requiring regular replacement, a costly and labor intensive effort. It was shown that using the fire polished tips following glass pulling helps in overcoming this problem as the fire polished tips were stronger. However, printing with extremely small diameter tips (< 3\( \mu m \)) is a tedious task as not only is pushing the ink out of the tips an issue, but capillary action for ink loading is limited or totally absent.
6.1.3 Printing on Flexible Substrates

Calibration of the device on flexible substrates was a challenging task. Due to the undulation and/or vibration on the surface, calibration had to be done several times to get a successful writing session, so that printing could be efficiently done. In addition to this, while printing on flexible substrates it was observed that the dispensers could wobble when rastering/tracing backwards along the y direction. Hence the pattern had to be drawn such that the control software would always print in the forward direction. This was related to the nature of the ultrasonic drive head that is only held in place via electric contact. Also, smaller diameter tips (< 5 μm) could not be used on flexible substrates as they would chip due to a small difference in height or surface roughness even though a vacuum chuck was used throughout the printing process. All such effects are minimized when printing is done with optimal distance between the tip and the surface, which is in the range of 1-3 microns.

6.1.4 Printing on Large Areas

It is challenging for Sonoplot to write large solid (filled) areas such as antennas, displays and solar cells, especially for large scale manufacturing purposes. Even though it is possible to do so for R&D phases, doing this in a manufacturing environment would require multiple tips that are difficult to control together or very large tips that can be limiting in resolution. Also, the ink has to be filled multiple tips when printing a large pattern which may result clogging the tip eventually. This will result in non-uniform printing. Also with larger areas, the ink jetting out initially is controlled but as the pattern size gets larger, the printing becomes non-uniform due to excessive heating case by the vibrations. In other words, temperature control may be necessary for prolonged writing sessions, especially when friction is high with larger (d>10μm) tips.

6.2 Future Work

Future work of this research includes printing with extremely small tip diameters (1 μm) to improve the resolution of printing. Printing on different flexible substrates and polymers also has to be optimized to avoid chipping of the micropipettes while printing or bending while printing in the opposite directions. It would be especially useful to use other types of glasses for pipetting that can last longer and improve resolution. The ink surface interaction has to be improved so that different types of inks such as carbon nanotube and
graphene can be used for printing to make flexible transparent electronic devices. Thus a procedure to wet and condition the pipette/tip inner or outer walls, prior to ink loading is highly desirable. Printing with more viscous inks and inks with smaller and larger particulate matter needs to be also studied in further detail. This can be especially useful for limitations of low viscosity inks as they tend to come out with substantial ease, leading to limited controllability and spreading of the ink on the surface. Factors like surface tension, adhesion of the ink to the surface and viscosity of the ink must be controlled for efficient printing to get the best results. Atmospheric factors like temperature and humidity also affect the ink that is ejected out from the surface, which needs to be studied in further detail. Finally overheating of the tip appears to be important for quality of printing, consistency between writing sessions and longevity of the tip itself. A Thermal camera can be used to study the tip temperature to monitor and study this particular impact.

Furthermore, the capacitance can be improved by printing a layer of a dielectric medium like PMMA on the surface before printing. The working of the inductor can also be enhanced by printing a magnetic core like iron oxide layer to increase the magnetic field at the center of the device. In addition to this, different kinds of sensor fabrication, biosensors, antennas and transistors can be also be pursued using the microplotting approach for various applications.
REFERENCES


[29] K. E. a Al, “Keysight E4980A/AL Precision LCR Meter.”


APPENDIX A: OPERATION OF SONOPLOT MICROPLOTTER – II

Sonodraw

Sonodraw is a user-friendly software that can be used to draw spots, lines, arcs, rectangular areas and rectangular filled areas. For patterns which cannot be drawn using Sonodraw, other designing software like AutoCAD or layout editor can be used and then converted into dxf format. A dxf importer converts the file into the format required by Sonodraw to print the patterns. The printer is more accurate for smaller feature patterns having a smaller area for efficient printing. The Microplotter prints patterns in the micron scale with great definition and accuracy. All the pattern dimensions can be adjusted with Sonodraw drawing software except for the width. The width of the pattern depends on the diameter of the tip chosen while printing. The feature width option in Sonodraw can be used only for rectangular fills wherein lines are filled closer next to each other if the feature width is small. Basically the rectangular area is filled up with more ink when the lines are closer to each other for smaller feature widths. So for larger tips the feature widths can be made much higher so that the lines are spaced more from each other and also to minimize the usage of ink.

Sonoguide

The Sonoplot Microplotter consist of a dispenser which is the core of the microplotting device. A hollow tapered glass tip with a certain diameter is attached to lead zirconate titanate (PZT) material. To move the dispenser to the required position a high precision positioning system is used. A charge coupled device (CCD) camera is setup in the Microplotter to monitor the printing continuously. The movement of the high precision positioning system is controlled manually the user Sonoguide on the PC. The positioning systems helps to move the dispenser along the X and Y axis along a 3x30x9.6 cm volume. The speed at which the positioner moves can be controlled manually depending on whether the positioner needs to be moved fast or slow. The value that is used. The positioning speed and acceleration also effects the way the positioner moves when it is printing. Since while printing the positioner moves according to the shape of the pattern, so the values need to be carefully set. The dispenser is positioned above the substrate manually and during
printing it is automatically controlled by the pattern which is designed by the user in Sonodraw.

**Dispenser Calibration**

The dispenser must be calibrated for optimum printing in order to avoid it from any overshooting when it comes in contacts with the surface (i.e. there is precise detection of the surface before printing). This frequency at which the ink is pushed out in an ordered way is at the resonant frequency which differs with every dispenser. Hence, every time a new dispenser is used it has to be calibrated. The dispenser is clipped onto the system and connected using the RJ-11 cable. Once the dispenser has been properly set, the software detects the dispenser and will calibrate the dispenser automatically. The calibration of the dispenser is a very important step as this is used to deposit the ink in an ordered way and also to detect the physical height from the surface. Dispenser calibration has to be done time and again during the printing process, if there are any variations in the amount of ink present in the micropipette or there is a drift in the system. Usually, the dispenser is calibrated during the start of the process once it has been clipped on the system without any ink and then calibrated once more once the ink has been filled. The calibration curve has a series of drops and sudden spikes but the sharpest drop off followed by a spike occurs at around 440 KHz [9]. Minimum impedance occurs at the bottom dip before the sudden spike which is the resonant frequency of the dispenser. The stronger the drop off followed by a spike, the stronger is the resonance. Once the resonant frequency for the dispenser is obtained, the frequency is automatically assigned for smooth dispensing and spraying action while printing and cleaning respectively. The figure below shows how an ideal calibration curves looks like if the dispenser has been mounted correctly.
Surface Calibration

In addition to the dispenser calibration which is used to electronically sense when it comes in contact with the surface, Surface cant calibration helps to prevent any damage cause to the tip when they come in contact with the surface and take into account any undulation or non-uniformity in the heights of the substrate. Once the micropipette comes in contact with the surface, the surface cant calibration is done along the entire area of X and Y directions. The X and Y distances can be manually set depending upon the area of the substrate. Usually a value slightly less than the dimensions of the substrates is set so that the tip does not go beyond the dimensions of the substrate. Once the appropriate distances are set, the calibration process is initiated. The Microplotter does leveling of the surface at 5 different points along the X and Y directions and is expressed as a slope which gives an idea to the dispenser the height it needs to rise along the surface while printing.

*Figure 46. Dispenser Calibration Curve [9]*
Typically, if the surface is completely flat the slope is zero and the dispensing action is smooth. However if there are undulations in the surface then the slope varies significantly, anything between 0.0001 to 0.005 for is acceptable and the dispensing action will occur without causing any damage to the tip[9]. A high slope value may damage the tip while printing and is not the most suitable surface to print. To overcome this, in case of glass slides the surface can be cleaned and made sure it is free of any dust particles and calibrated again starting from a different position to keep the slope as minimum as possible. In case of flexible substrates, since the roughness and undulations is more a vacuum chuck was used for printing. If the surface calibration is not done accurately (minimal slope), the tips attached to the PZT overshoot the surface and shatter as they are very fragile when the printing action is initiated.

The real time diagnostics along with the CCD camera helps to understand when the tip has touched the surface. The diagnostics have a threshold (red line) which increases in a stepwise fashion and crosses the threshold as shown in the Figure 45 when the tip comes in contact with the surface. If the dispenser calibration has not properly been done, the signal is not strong enough to cross the threshold and is not able to detect the surface, in that case the dispenser is recalibrated again. When the dispenser is retracted from the surface, the signal should again drop off sharply below the threshold value. Also, mounting the tip onto the PZT is a very crucial step as if the tip is not attached to the PZT perfectly, the tip may not detect the surface and overshoot.

![Figure 47. Real-time Diagnostics when Tip Approaches Surface][9]
Capillary Action

Once the calibration processes are complete, the ink is filled into the micropipette using a capillary action. Since the diameters of the tip are usually between 5µm -100µm, when the glass tip of the dispenser is dipped in the solution, the ink is drawn inside the tip. This simple method helps to fill picolitre amount of ink and prevents any wastage of the ink as a large volume is not required. If the ink does not fill up either the diameter of the tip is too small and the ink cannot be pushed in or the tip maybe clogged with a tiny dust particle. To overcome this, there is a spray option available which will cause severe agitation in the dispenser and this should spray out any dust particles or debris present inside the glass tip. The amount of time required to load the ink into the glass tip can vary from a few seconds to a couple of minutes depending on the diameter of the tip. The smaller diameter tips take a longer time for the capillary action while the shorter diameter tips load ink in a few seconds.
APPENDIX B: GLASS MICROPIPETTE FABRICATION

Pull Cycle

Place the micropipette between the filament which is tightly held by the clamps on either side of the puller bars. The program that is stored in the glass puller can be selected for execution. The required program with is selected depending on the diameter of the tip that needs to be pulled.

- The heat is turned on as set in the program which is obtained during the ramp test depending on the glass used.
- The glass heats up and melts until it reaches the entered velocity.
- If time is initiated a hard pull is executed and cool air is passed for the time mentioned in the program.
- If delay is activated, then first cool air is activated for 300ms and then the hard pull is activated.

Pulling Parameters

On multiple trials and pulling several different tips, it was observed that the pulling cycle was not repeatable. The pulling cycle had changes depending on the temperature of the room and humidity which made pulling tips continuously with repeatability a challenging task. With the same parameters entered, the pulling would vary if the filament was at a very high temperature or the temperature was not hot enough to melt the glass resulting in uneven pulling of tips. The values of heat, pull, velocity and delay that was used to pull the tips is as shown in the table below. The heat value was set to a fixed 517 which was obtained during the ramp test for borosilicate glass from Sutter Instruments. On doing the ramp test the glass pipette heated upto 517 and the puller would turn off when a certain velocity is reached. This was the temperature at which the glass (glass transition temperature) would begin to soften and hence a fixed heat value is set in the program. A velocity between 10-12 gave the best results for a tip diameter of 10-15 microns. The delay time used also made a difference to the pulling of tips significantly. the first few tips that are pulled are usually larger as the filament is not heated sufficiently. The velocity in line 3 and 5 were adjusted between 10 and 11 to pull tips with a larger diameter. The delay in line 1 had to also be adjusted deepening on how the initial set of tips were pulled. It was
observed that decreasing the delay by 7-8 units on line 1 gave larger tips of around 20-25 microns. However, the lines of code that was used is not always consistent and the velocity and delay have to be altered on a regular basis depending on the temperature in the room. The pull rate remains 0 as already mentioned previously, higher the pull rate larger are the tips that are pulled. Also after repeated trials it was seen that after 6 lines of the code were run the pull action was initiated. However sometimes after the 6 line of the program if the pull action does not take place then the code repeats from line 1 till the hard pull is initiated and two equal diameter tips are obtained.

Table 26. Micropipette Fabrication Parameters

<table>
<thead>
<tr>
<th>Line</th>
<th>Heat</th>
<th>Pull</th>
<th>Velocity</th>
<th>Del</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>517</td>
<td>------</td>
<td>11</td>
<td>200</td>
</tr>
<tr>
<td>2</td>
<td>517</td>
<td>------</td>
<td>10</td>
<td>250</td>
</tr>
<tr>
<td>3</td>
<td>517</td>
<td>------</td>
<td>11</td>
<td>200</td>
</tr>
<tr>
<td>4</td>
<td>517</td>
<td>------</td>
<td>10</td>
<td>250</td>
</tr>
<tr>
<td>5</td>
<td>517</td>
<td>------</td>
<td>10</td>
<td>150</td>
</tr>
<tr>
<td>6</td>
<td>517</td>
<td>------</td>
<td>10</td>
<td>250</td>
</tr>
</tbody>
</table>
The pictures below give the step by step illustration of pulling glass tips.

**Figure 48. Micropipette Fabrication Procedure**

**Fire Polishing**

Once the tips are pulled they usually do not have a rounded bottom, they are flat at the bottom of the tapered part. To round the edges and make it more pointed and decrease the diameter of the tip a technique called as fire polishing is used. The tip of the pulled glass micropipette is placed inside the filament by moving the puller bars closer to the filament and then held together with a T shaped aluminum spacer with adjustable screws. So by adjusting the screw the pipette can be repositioned within the filament as required. Once the spacer is adjusted and the tip of the pulled glass is inside the filament, the heat value is turned on again with different delay times. The value of the delay time used
depends on the diameter of the tips required and varies. Sometimes multiple pulls have to be done to get the required diameter. During fire polishing the velocity and pull rate is kept 0 so the heat is on as long as whatever time/delay is set in the code. If the delay time is set too high, fire polishing results in excessive heating of the glass due to which the diameter of the tips becomes very small and eventually closes. When fire polished, since the pulled tip is exposed to heat again, the tip diameter becomes smaller and the outer edges are rounded to give a strong tapered glass tip that can be used for printing. Fire polishing of the tips make them more sturdy and it is observed that they do not break even on touching the surface from a significant height. They are not as fragile as the non-fire polished tips and have a longer life.

![Figure 49. (a) T-shaped Aluminum Spacer for Fire Polish (b) Placing the Spacer in the Grove [48].](image)

The spacer that is used for fire polishing is placed in a slot in the puller bar which can be adjusted depending on where the puller bar needs to be fixed so that just the tip is inside the filament. The difference between a fire polished and non-fire polished tips is as shown in the Figure 48. In the Figure 48 below each major axis in the scale is equal to 50\(\mu m\) and each divisions is equal to 5 \(\mu m\) for the fire polished tips. The non-fire polished tip has each major axis on the scale equal to 100 \(\mu m\) and each division equal to 10 \(\mu m\). As seen from the figures below the diameter of the tip decreased from 50 \(\mu m\) to 15 \(\mu m\) when the tip is fire polished. Also, the non fire polished tips are the commercial tips from
Sonoplot which are flat at the bottom as seen in the finger. With fire polishing a well tapered rounded bottom tip is obtained.

Since this puller is known to pull tips in the 1-3 $\mu m$ range, the largest tip that was possible to pull was approximately 25-30 $\mu m$. For more viscous inks, polymers, a tip diameter of around 50$\mu m$ was required for printing. Hence using the glass puller smaller tips as well as larger tips could be pulled to make the printing process efficient.

*Figure 50.* (a) Non-fire Polished (b) Fire Polished