Laser Sintering of Printed Conductive Traces on Flexible Substrates Using Laser Cutter Tool

Final Report ENGR241 Spring 2022

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Introduction

Motivation

Metal traces printed on flexible substrates suffer from higher resistance when compared to traditionally fabricated rigid counterparts. Thermally annealing printed metal traces after printing results in improved resistance, but is limited by the low thermal resistance of the flexible substrates (typically PET or Polyimide) to achieve the temperatures necessary to completely evaporate solvents that make up the particle suspensions solution or ink. Metallization also remains unachievable at temperatures below 250C recommended by ink manufacturers to anneal inks and reduce reflow. Laser sintering presents a localized heating solution where the photonically sensitive metal traces are heated much faster than the nearby transparent polymer substrates due to light energy reflectance as opposed to absorption. Laser sintering tools typically utilize a chamber that is subjected to the entire laser dosage, but do not allow for specific areas to be targeted in time with fabrication. Laser cutters utilize often the same laser wavelength, albeit at much higher powers, to cut through a substrate in a raster pattern. Using a laser cutter tool at a much lower than typical power with specific frequency and speed settings is expected to result in laser sintering conditions necessary to evaporate trace solvent solution and potentially induce metallization effects in printed metal traces on flexible substrates. Using a laser cutter to laser sinter printed metal traces on flexible substrates will allow for a targeted localized sintering condition that can be utilized in improving the resistance of printed metal thin films.

Benefits to SNF

This project explores laser cutter as a cheap alternative for laser sintering conductive metal traces on flexible substrates, which allows fast sintering on localized areas. It also investigates the effects of each laser parameter on the efficiency of sintering and tests the behaviors of different substrates. Furthermore, it investigates the ink jet printing process for depositing nanoparticle metal inks onto flexible polymeric substrates. A detailed user-friendly operation process is involved which will benefit the SNF society in flexible hybrid electronics production.

Design of Experiment

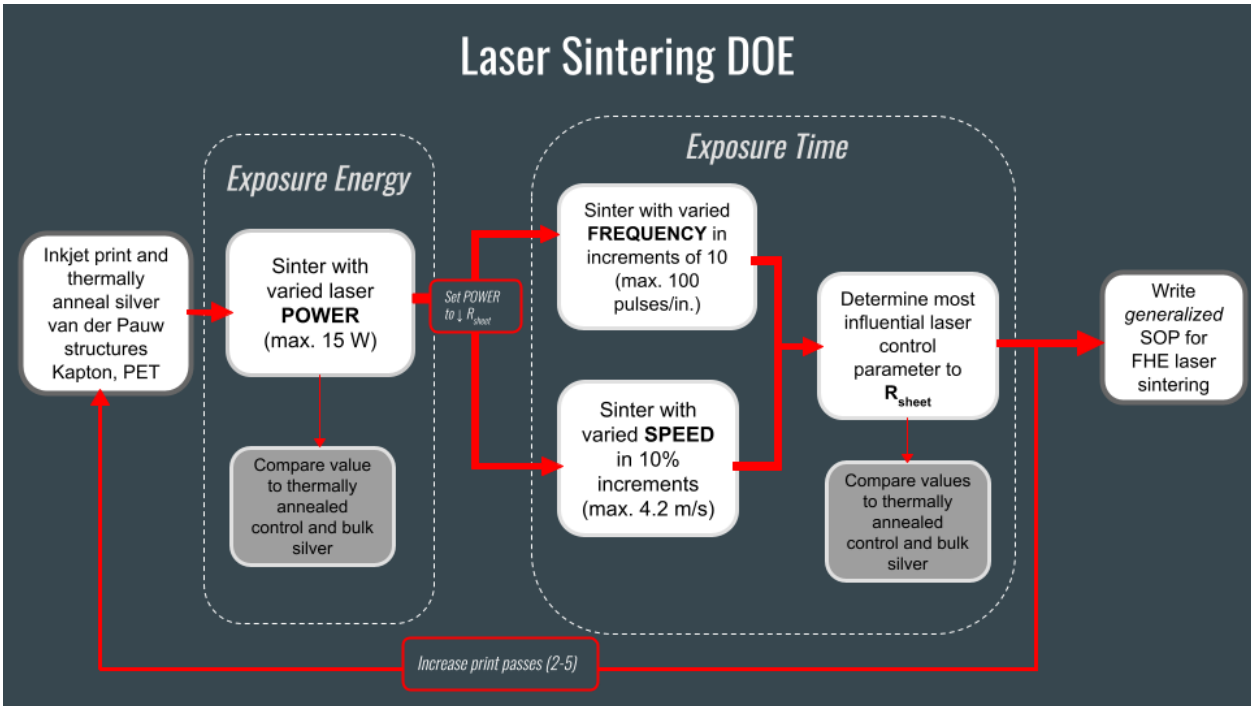


Figure 1*: Diagram detailing the planned design of experiment for laser sintering of the thermally annealed samples.*

Sample Preparation and Fabrication

In order to test that the laser cutter tool is capable of sintering conditions, metal traces were deposited on flexible substrates using printing in order to mimic a likely user sample in SNF. Inkjet printing was selected over Voltera micro dispense printing and Optomec Aerosol jet printing due to the very high variance in samples made with the Voltera and the increased training and fabrication time needed to obtain samples using the Optomec. The Dimatix printer provides low sample variance with higher volume fabrication possible in a reasonable amount of time. Roughly 150 samples were printed on both Polyimide (Kapton) and PET (Mylar) substrates to make multiple sintering experiments possible in line with the Design of Experiment.

van der Pauw Structures

In order to verify the effectiveness of the laser sintering, resistance of the annealed traces was measured. Van der Pauw structures provide a measurement technique that gives insight to sheet resistance of metal thin films regardless of geometry. This is suited to this project as the overall goal is to utilize the laser cutter as a sintering tool regardless of device structure. On flexible substrates, there are many studies highlighting van der Pauw's structures that differ from those used in silicon devices [1]. Printed electronics are less robust and particle migration at bottlenecks of designs is a greater concern during testing. Using structures with both narrow traces and blanket coverage is important to verify that particle migration is not a significant issue. The pads for probing are also greatly increased in size due to the proportions of the overall structure and idealities in printing resolution conditions. Figure 2 shows the van der Pauw structures used for this project. The overall size of each structure is 20 mm which is expected to be greater than the average inkjet printed device in SNF but is ideal for sheet resistance testing and measurement.

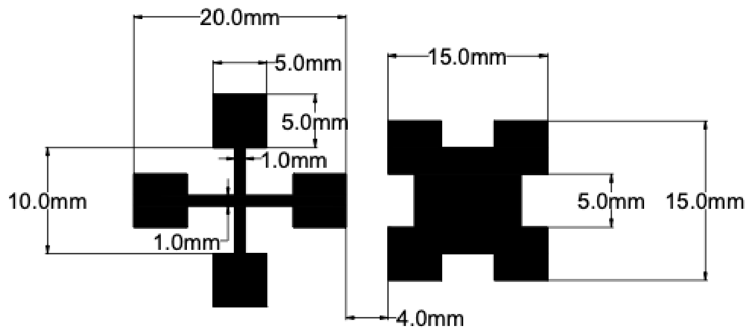


Figure 2*: CAD design of sample structures. The "cross" structure is shown (left) next to the "square" structure (right) in a single design tile that was specified to repeat to produce multiple samples in a single print job.*

Inkjet Printing

Inkjet printing was selected to create the samples for sintering in the laser cutter due to its low processing time and increased sample reliability when compared to the performance of the aerosol jet and micro dispense printers available in SNF respectively. The Fujifilm Dimatix inkjet printer, known as the nanoinkjet in SNF, was utilized with Samba cartridges purchased from Fujifilm. Figure 3 includes a schematic detailing the nanoinkjet tool similar to how it is configured in Ex-Fab. The inkjet printer in ExFab is placed inside a fume hood that cannot have its flow disabled nor the hood lifted, so it presents non-ideal printing conditions for this tool. The high air flow results in drying of prints during printing and unpredictable jetting on the substrate. Future work should include finding a new appropriate placement for this tool in SNF that would greatly improve its performance and most likely its popularity to SNF users.

Exact printing set-ups can be found in the nanoinkjet SOP written by SNF staff. For a short summary in this report, ~2 mL of commercial ink was loaded into the cartridge using the provided cartridge needle. This is over the listed reservoir capacity from Dimatix, but user/group member experience has demonstrated that the cartridge reservoirs can easily hold up 2.5 mL with no issues. The cartridge was then loaded into the printer and then the IPA rinsed/wiped substrate was placed in the platen and secured with tape for extra security. During printing, all 12 samba jets were used due to the relatively large size of the desired sample pattern and thus cleaning cycles happened roughly every 5-10 minutes of printing. Batch printing of samples took between 3-7 hours depending on the number of samples and necessary cleaning cycles to ensure consistent jetting for that printing days conditions. Prints were inspected after printing for major flaws that are visible to the eye, as well as any major overspray that could cause shorting under the print head camera at higher magnification.

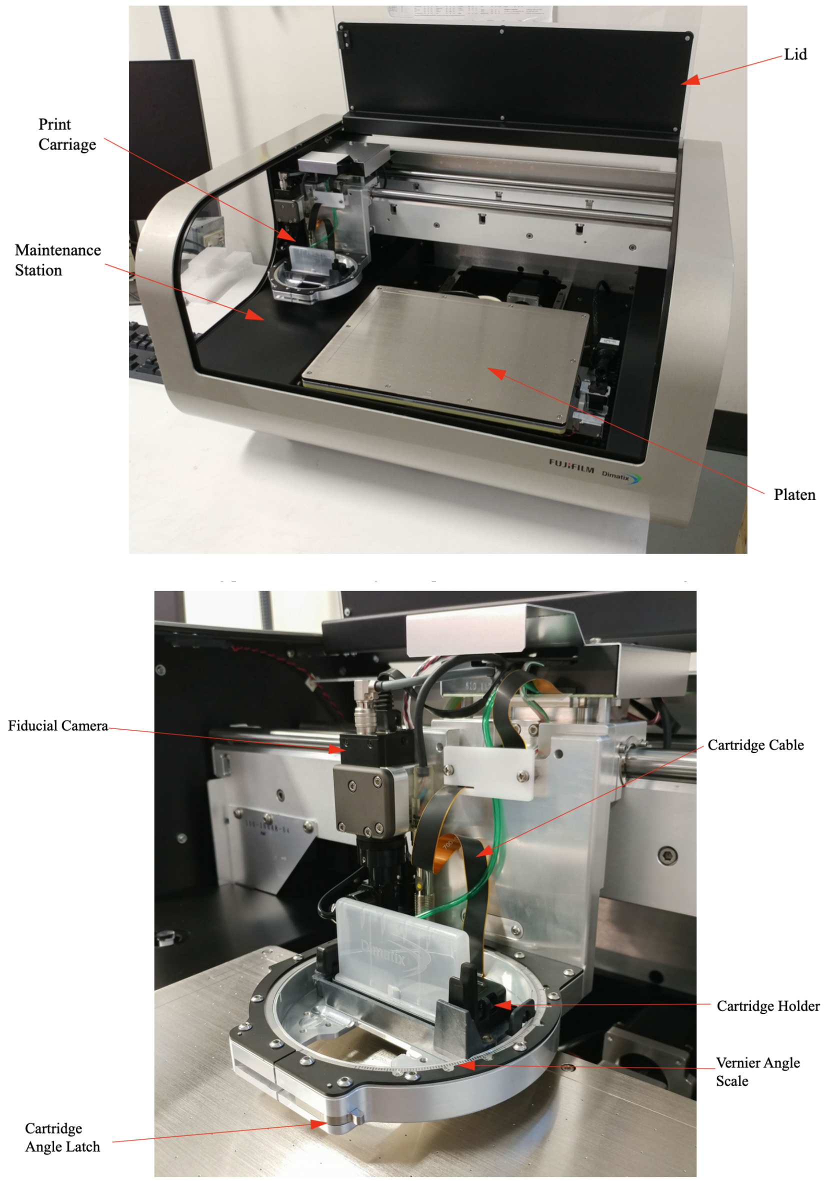


Figure 3: *Dimatix printer (top) and close-up view of print carriage (bottom) [2].*

CAD drawings were converted to 1-bit bitmap using OpenDraw. Depending on the substrate size, 15-30 samples were printed in a single print session reducing variance in experimental batches. Originally 1-5 print passes were meant to be explored, but after optimization of the silver ink in the Samba cartridges 2 passes were found to be relatively porous and give the minimum measurable results. 3 print passes were found to be ideal to create uniform gap free metal traces in the desired pattern. 4 passes were attempted, but after consideration of realistic applications of inkjet printed devices, it was determined that focusing on exploring 3 print pass samples would be more beneficial to SNF in the limited time period this project was able to be completed. The meniscus level of the ink in the print settings was increased from the ink manufacturer's recommendation of 1-2 to 4-5 on account of the differing humidity levels where the manufacturer had developed the ink and the printing conditions in SNF.

Ink

Commercial silver nanoparticle ink was utilized to reduce the necessary optimization of printing and ink properties needed to develop an inkjet nanoparticle compatible ink. Commercial conductive inks have become widely available, specifically produced and optimized to be utilized in Dimatix inkjet printers. Novacentrix JS-A211 Metalon Silver Nanoparticle Ink was selected for this project due to its clear prominence in publications concerning printed conductive traces as well as its high silver weight % inclusion with increased conductivity expectations. This ink was formulated to be compatible with Fujifilm Samba cartridges and the jetting waveform was provided by Novacentrix. No filtration or mixing was done to the ink before filling cartridges in line with recommendations from the ink manufacturer. An SOP detailing loading and usage of this ink with the inkjet printer in SNF is included in the appendix.

Thermal Annealing

Thermal annealing is necessary to practically handle the printed test structures once the ink has been deposited on the substrates. The ink remains wet at room temperature after being jetted into the desired pattern, so thermal evaporation of the solvents suspending the particles in solution is necessary. Thermal annealing does not completely remove all of the suspending solution but instead reduces it enough to completely stop reflow. Figure 4 demonstrates the visual change in the printed ink color as thermal annealing is applied and the solution is evaporated from the substrate.

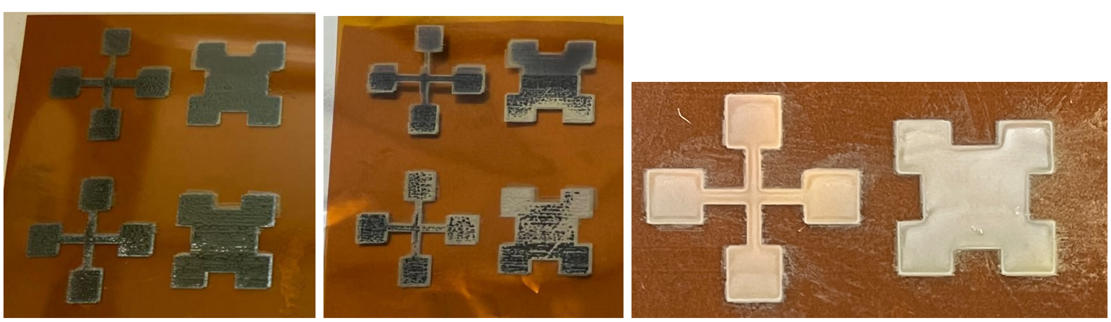


Figure 4: *Printed samples before annealing (left), immediately after contacting hot plate (middle), and close-up after full annealing time for one sample tile (right).*

The recommended thermal annealing parameters provided by the ink manufacturer were developed for the annealing of samples in an enclosed oven and not a hot plate placed in a fume hood like what is currently provided in SNF for printed electronics processing. Because of the lack of uniform heating achievable on a hot plate and the uneven air flow provided by a fume hood, the thermal annealing required increasing the time recommended by the ink manufacturer. Furthermore, increased print passes results in thicker silver deposition and thus increasing annealing time is necessary to lower the initial resistance of the test structures. Table 1 includes the parameters used to anneal the various types of printed samples. The PET substrate samples were thermally annealed at much lower temperatures due to lower thermal robustness of PET.

Table 1: *Parameters of thermal annealing process using hot plate for various samples using JS-A211 silver ink.*

	2 Pass	***3 Pass***
Kapton	250°C : 20 Min.	250°C: 30 Min.
PET	140°C : 30 Min.	140°C: 45 Min.

Sintering Experimental Methods

Laser Cutter User Parameters

Sintering is the heat treatment process in which the material is heated to a temperature below its melting point. As a result, the porosity of the material is reduced and certain properties can be enhanced, including electrical conductivity. Laser has been used as a novel heat source for sintering due to its high energy, accuracy, and fast process time. In this project, an Epilog Fusion M2 laser cutter (located in SNF 155A) was used for sintering the printed conductive traces on Kapton and PET.

Figure 5 shows the Epilog Fusion's control panel and below is a brief introduction of the parameters that can be adjusted on the user end [3]:

Laser source: The Epilog laser cutter is coupled with dual laser sources, including a CO_2 laser source (wavelength 10.6 um), and a fiber laser source (wavelength 1060nm). Since the conductive traces are silver, for which the fiber source is more readily absorbed, we used the fiber laser source for all our experiments.

Job Type:The laser cutter can perform the cutting task, the engraving task, or a combination of both. The vector job type is useful when making cuts through materials, and the raster job type is used for engraving marks at the surface of the material without cutting through. We used the raster job type for sintering.

Power: The maximum power of the fiber laser is 50W. The power for a specific job can be adjusted on the scale of 0-100%, in increments of 1%. It determines the amount of laser energy output and is a crucial factor for achieving good sintering.

Speed: The speed can be adjusted in 1% increments from 0 to 100% for how fast the carriage travels. The maximum speed is 4.2 m/s. Slower speed results in higher temperature on the conductive traces. It is also an important factor that needs to be determined to achieve optimal sintering.

Frequency: Frequency is the number of laser pulses per inch of travel. The maximum frequency is 100 pulses/inch and it can be adjusted in 1% increments on the scale of 100%. The frequency value also affects the amount of heat being delivered to the material. High frequency value means more pulses, therefore more heat output.

Dithering mode: This is a setting unique to raster jobs. It defines the dot patterns for engraving images. While the default setting for dithering is ‘Standard’, it didn’t work well for our preliminary sintering tests. Due to time constraints, we didn’t explore the dithering effects on sintering. We chose ‘Stucki’ dithering and used this mode throughout.

Resolution: Resolution defines the print quality of the laser cutter, and it also affects the print time. It can be adjusted on the user end from 75 to 1200 DPI. The higher the resolution, the finer the details, and the longer the print time. The default resolution is 600 DPI. Since there is good overlap between the dots under 600 DPI, we kept this default throughout our sintering tests.

Engraving direction: The user can adjust the engraving direction to be either top-down or bottom-up. While bottom-up engraving is good for keeping the newly engraved area free of debris, it is not suitable in our sintering experiments. On the other hand, the freshly sintered area has a higher risk of peeling off in the bottom-up engraving direction since the air flows from the bottom to the top inside the laser cutter. Therefore, we used the top-down (default) setting for all experiments.

Note: The exact speed and frequency of the laser pulse in correlation with rastering time is not provided by Epilog. Because of this, the exact dosage the samples are subjected to is not reported as this would be a misrepresentation of the tool performance as well as inaccurate at best. Rastering time was externally measured during testing, but a linear change in time in accordance with linear speed or frequency changes was not observed, most likely due to manufacturer programmed “hang times” in order to conserve servo motor lifetimes and tool part performance. For this project, outcomes are reported in the laser cutter tool terminology, but nonetheless the context used results in an overall understanding of how to apply the laser to a sample to reduce material resistance from the exact same data trends that would result in representing data as laser dosage or energy exposure.

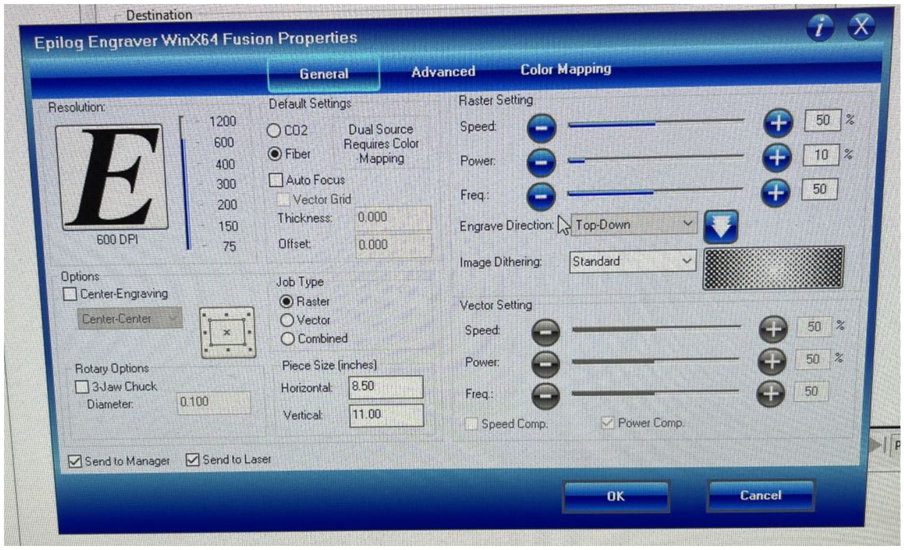


Figure 5: *Control panel for Epilog Fusion laser cutter.*

Sample preparation for sintering

The same van der pauw patterns are drawn in CorelDraw x8 and sent to the laser cutter for prints. Prior to sintering inside the laser cutter, a reference mark is engraved on a piece of ceramic tile for alignment. Since the air flow inside the laser cutter is too strong for the flexible substrates, they are secured to the tile using kapton tapes. A typical sample ready for sintering is shown in Figure 6.

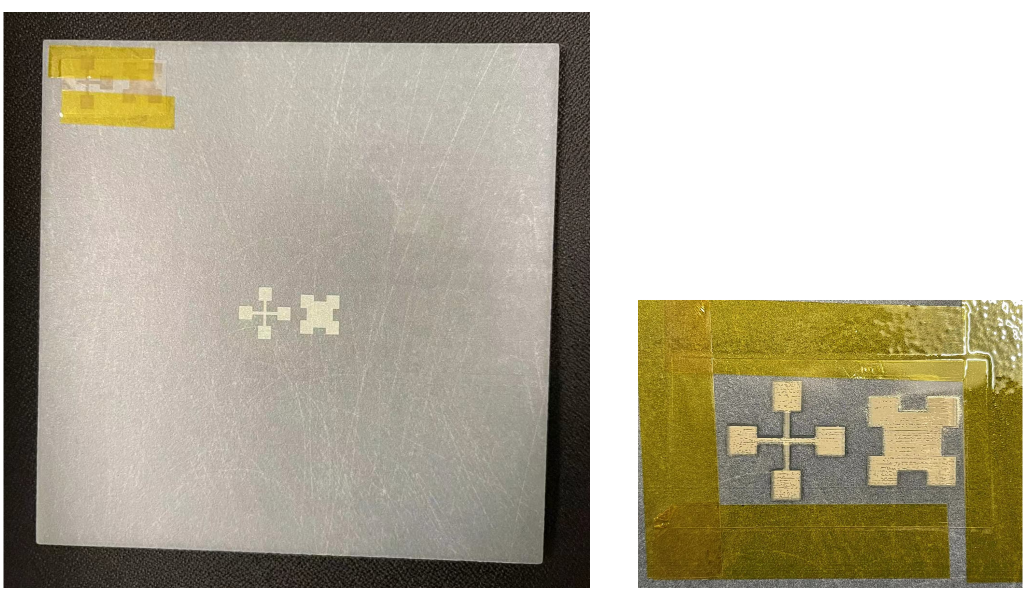
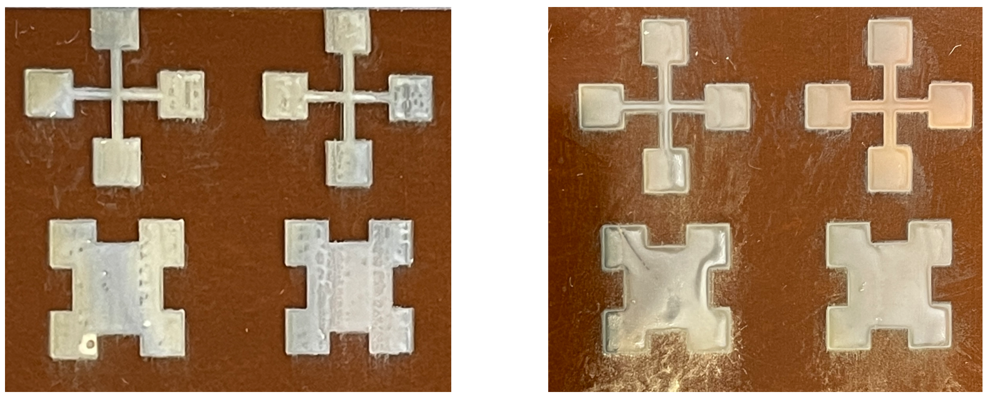


Figure 6: *Sample preparation before sintering in laser cutter. On the left is the alignment mark printed on a piece of ceramic tile for alignment. The right picture demonstrates the aligned sample secured by Kapton tapes.*

After starting the air assist and the laser cutter, the prepared sample (aligned and taped) is put onto the laser bed. The focus is manually adjusted using the fiber laser gauge and the joystick until the sample just touches the bottom tip of the gauge. The focus should be +0.069'' ± 0.001''.

Number of Printed Passes

The inkjet printing method is utilized to get thin film structures for the conductive traces, therefore, we started with 2-pass prints for sintering experiments. However, there was too much porosity in the 2-pass samples which led to peel-off issues during sintering and high resistance after sintering. Therefore, we increased the print pass to 3 passes which reduced the porosity, as shown in Figure 7, and also improved the peeling-off of the printed traces as well as the conductivity.



2-pass 3-pass

Figure 7: *Comparison of 2-pass prints (left) and 3-pass prints (right).*

Laser Parameters Experiment

Since the power, speed, and frequency are the three major factors affecting the heat delivery to the material, we decided to focus on adjusting these three parameters in this project, starting with varying the power settings. Table 2 summarizes some successful laser sintering experiments with the corresponding laser wavelengths and maximum powers.

Table 2: *Successful laser sintering of silver traces in literature*

Substrate/Materials	***Laser Wavelength***	***Max Power***
PET/Silver [4]	532 nm	5 W
PET/Silver [5]	532 nm	5 W
Rogers RT5870/Silver [6]	1064 nm	4 MW
Kapton/Silver [7]	1080 nm	20 W

A series of power tests was first conducted on the substrates, both Kapton and PET, to determine the maximum power each substrate can withstand. It was observed that visible damages began to appear when the power reached 15W, as shown in Figure 8.

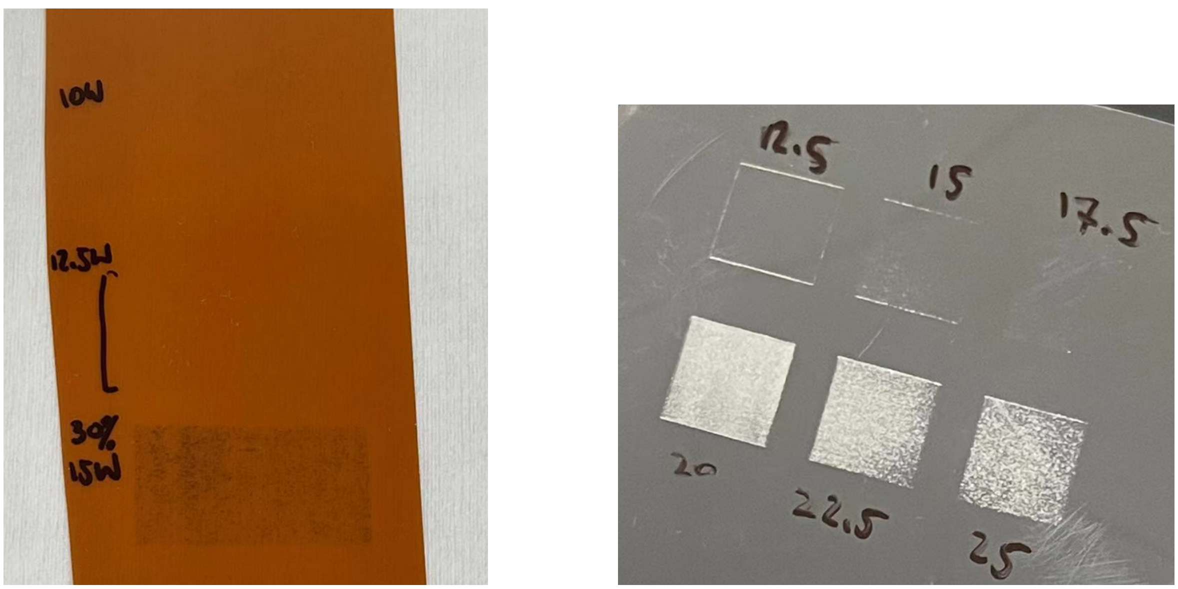


Figure 8: *Visible damages on the substrates were observed when the laser power reached 15W (i.e. 30%).*

Based on the literature and our experiments with the substrates, we decided to start experimenting with the power settings in the lower 1-7W range, while keeping the speed and frequency settings at default (50% for both). On the PET substrate, the peel-off issue was observed when the power reached 7W. However, due to the limitation of the characterization equipment, we couldn't obtain sensible results for sheet resistance.

On the Kapton substrate, peeling off of the printed traces were observed when the power went beyond 4W, shown in Figure 9. Therefore, we sintered the Kapton samples at 1-4W (i.e. 2%-8% input) and characterized the sheet resistances to find the best power setting.

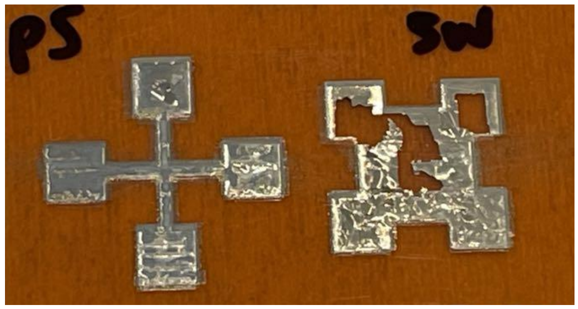


Figure 9: *The printed traces start to peel off from the Kapton substrates when sintering power goes beyond 4W (i.e. >8%).*

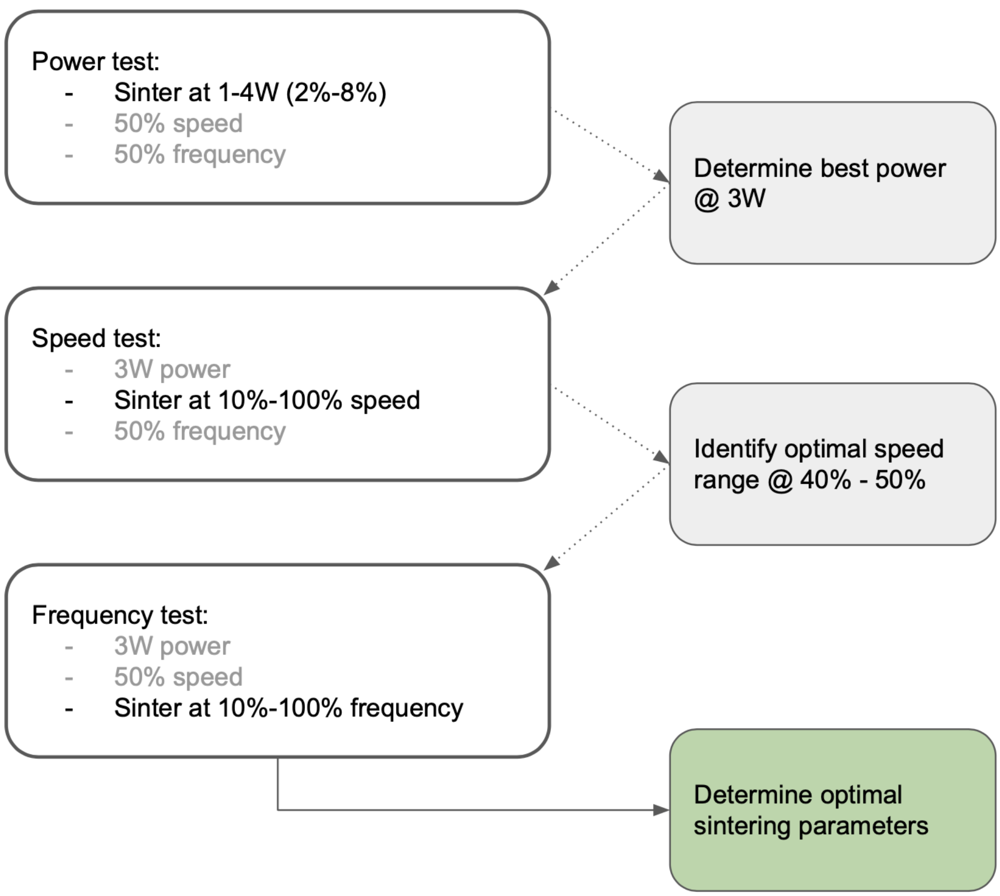


Figure 10: *Laser parameters experiments.*

After the optimal power setting was determined, we fixed the power setting and varied the speed settings, keeping the 50% default frequency. The speed was varied from 10% to 100%, first in 10% increments. After determining the speed range that gave relatively low sheet resistance, we fine tuned the speed in 5% to obtain an optimal range for the speed setting. The frequency was then varied from 10% to 100%, in 10% increments, while keeping the power and speed at their optimal settings. A flow chart of the experiments on laser parameters is shown in Figure 10. A detailed SOP of sintering with the laser cutter can be found in the appendix.

Characterization Technique

The sintered films were characterized by measuring the sheet resistance, using a four-point probe method on the Micromanipulator. The standard test setup for four-point measurements is under ‘Structures’ -> ‘Van der Pauw Square’ in the Keysight B1500A Semiconductor Device Analyzer.

The standard needle probes on the Micromanipulator were observed to pierce the thin films and substrate. Consequently, flat-ended copper wires were taped to the probe as shown in Figure 11. This provided more uniform contact without damaging the specimen. Figure 12 shows a typical sheet resistance curve for 2 pass Kapton sintered at 3W. The linear increase in sheet resistance for lowest current ranges is present in all tests using copper wires, regardless of the sample or current sweep conditions. This changing sheet resistance value can be attributed to the copper oxidation and initial voltage rise time to overcome it and report the more constant sheet resistance of the actual sample silver deposit. The relatively high sheet resistance values measured using this setup reflect the high contact resistance achieved using naked copper wire in air. Average sheet resistance obtained with this setup is of the order of a few hundred ohms, compared to a few milliohms presented in literature. This is most likely due to oxide formation at the contact.

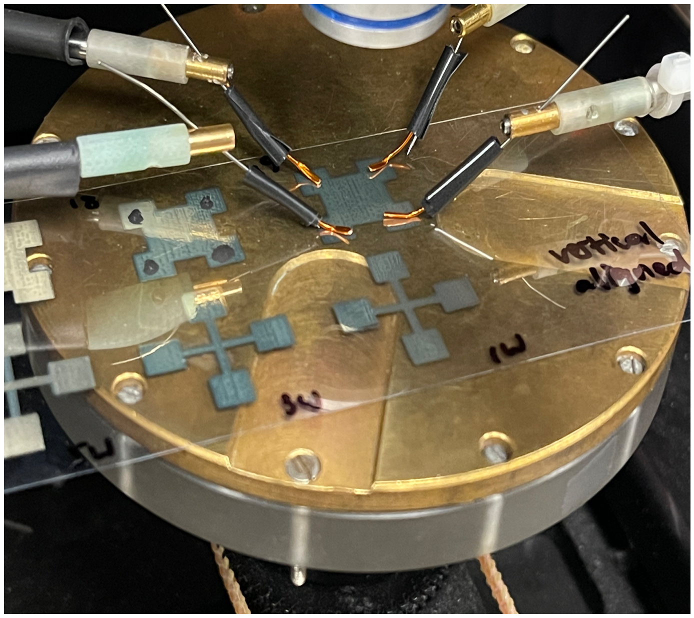


Figure 11: *Four-point resistance measurement setup using copper contacts*

Moreover, since the copper wires were taped to the probes, the contact between them was erratic after a few measurements leading to poor repeatability and frequent need to redo the setup. Subsequently, the copper wires were replaced with tinned copper wire, also called bus bar wire. Various gauges of the wire are available in the Physics Store at Varian.

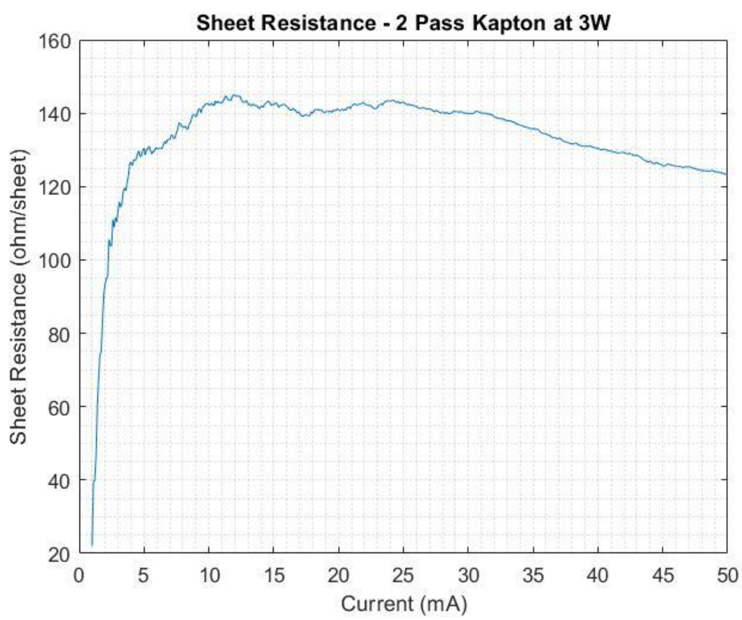


Figure 12: *Sheet resistance curve for 2 Pass Kapton sintered at 3W, with copper wire setup*

Figure 13 shows the modified setup with the tinned copper wires. The wires were soldered to the probes and taped in place to improve the reliability and repeatability of the test setup. Also, since this material has a low tendency for oxide formation, sheet resistance values were now of the order of a few milliohms. This setup minimized the lead time and ~50 measurements could be taken reliably.

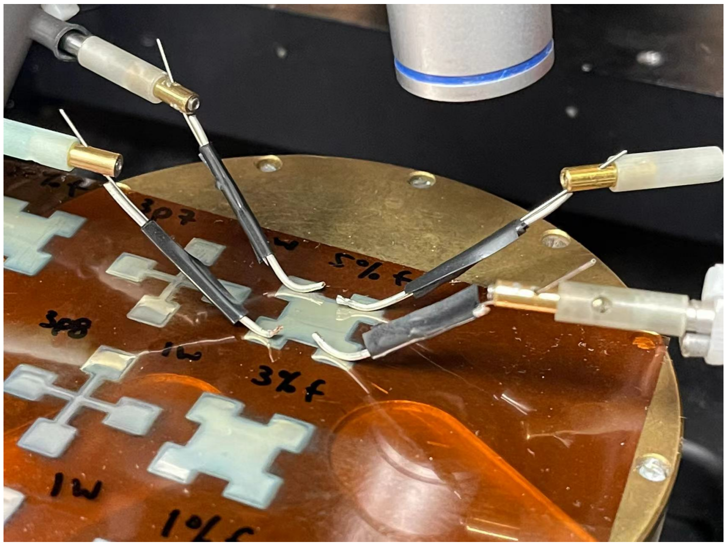


Figure 13: *Four-point resistance measurement setup using tinned copper contacts*

Results and Discussion

Kapton

First, the sheet resistance was measured for the control samples. These were the thermally annealed traces that were not subjected to sintering. When the test begins, an exponential rise in R_{sheet} was observed - of the order of a few kilo ohms, which eventually decreases to the milliohms order. This can be explained by a current limit being reached in the sample. However, since the device analyzer forces the current in the samples, the voltage across them changes, in order to accommodate those current values, leading to a lower resistance at higher current ranges. The average resistance for the cross and square control samples was observed to be 1.35 kΩ/sq. and 1.15 kΩ/sq. respectively. Next, sintered samples with varying power, speed and frequency were tested.

Power Variation: Following the DOE, the first parameter to optimize was the laser power. At the default speed and frequency value of 50%, 3 pass Kapton samples were sintered at 1-5W. Minimum sheet resistance(R_{sheet})was observed at 3W. At 4W and higher, the thin films started to peel off the substrate. Figure 14 shows the variation of average sheet resistance as a function of sintering power.

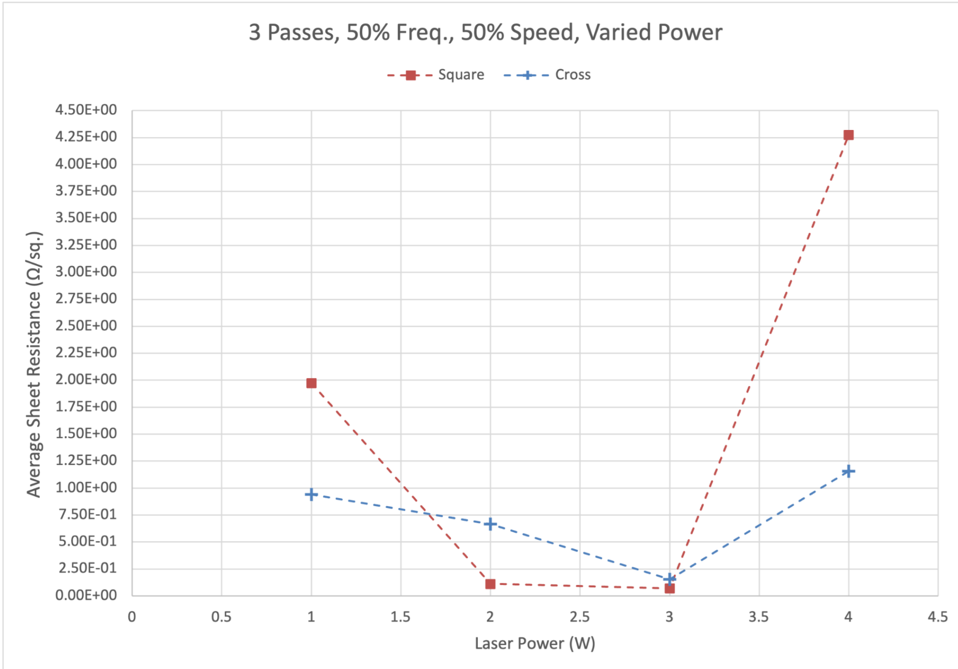


Figure 14: *Average sheet resistance as a function of sintering power at default speed and frequency*

<u>Frequency Variation:</u> With the optimal sintering power as 3W, the optimum exposure time was determined. This has two components - speed and frequency, as explained earlier. First, speed vs frequency curves were obtained. At 10%, 50% and 100% speed, laser frequency was varied at 10%, 50%, 80% and 100% of max value and vice versa. Figure 15 and Figure 16 show the speed and frequency effects curves.

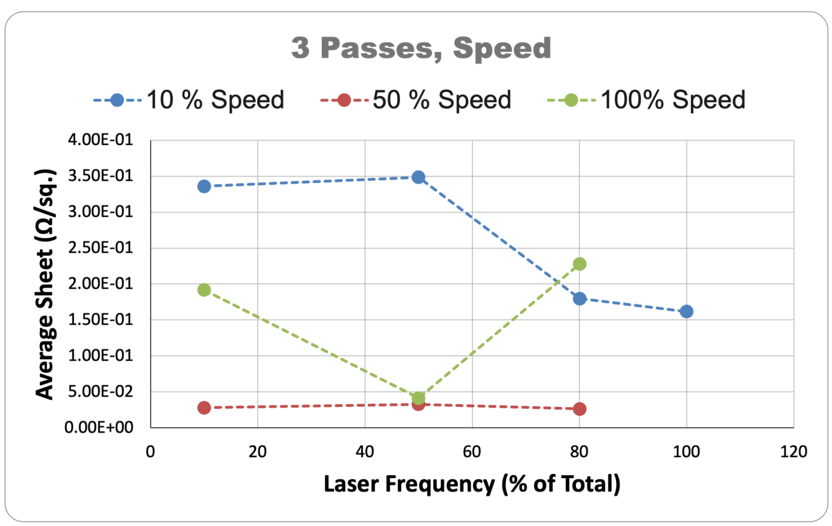


Figure 15: *Effect of speed on sheet resistance as a function of frequency.*

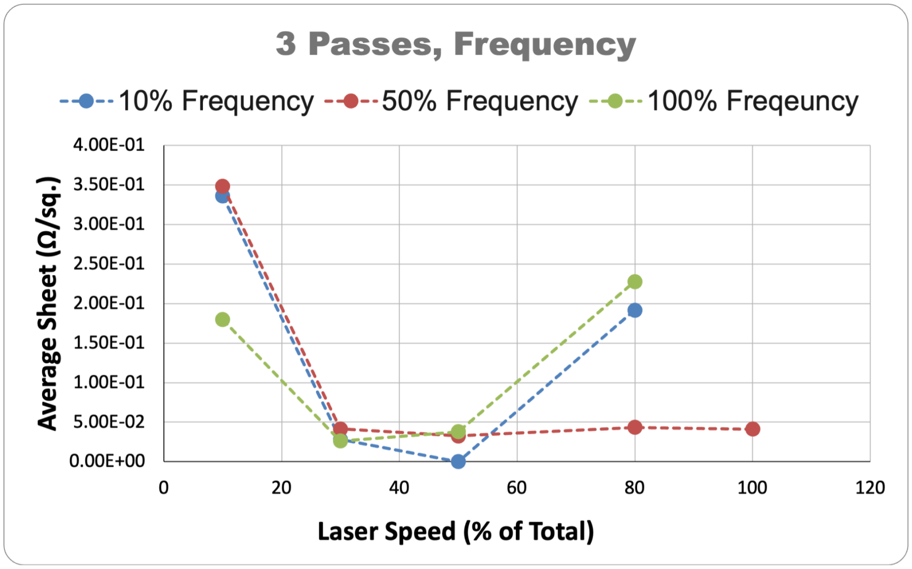


Figure 16: Effect of frequency on sheet resistance as a function of speed.

In speed effect curves of Figure 15, high sheet resistance is observed at low speed and high frequency, due to higher exposure time. This decreases to a minimum at 50% frequency, even when the speed is at the 100% maximum. A similar trend is observed in the frequency effect curves of Figure 16. High resistance is observed at low speeds, which decrease to a minimum at 50% speed and then again increases. In both the curves however, at 50% speed and frequency, sheet resistance is consistently low and the curve is flatter than the others, indicating an optimal setting around this range.

Next, the speed was set at 50% and frequency was varied in steps of 10%. Variation of sheet resistance as a function of frequency is shown in Figure 17. Beyond frequencies of 50% max value, the energy exposure is very high. This led to large variations in the sheet resistance. A minimum value of R_{sheet} = 12 mΩ/sq. was obtained at 50% frequency.

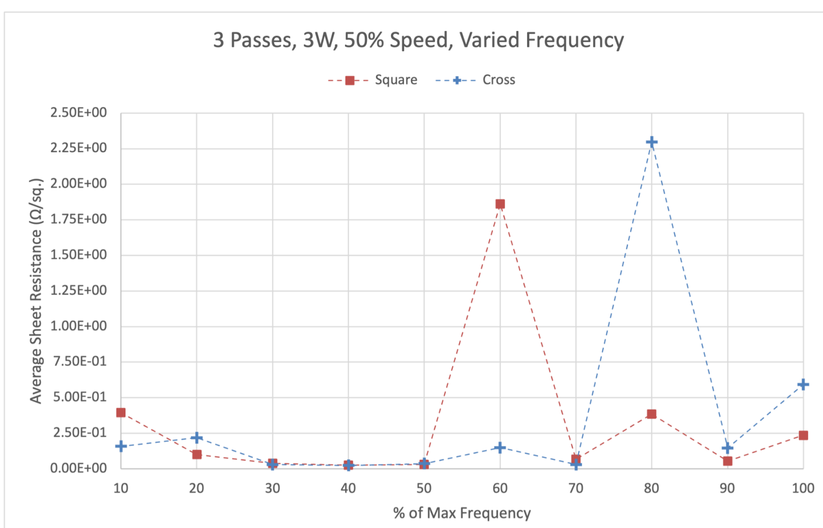


Figure 17: *Average sheet resistance as a function of frequency at 3W and 50% speed.*

Speed Variation: The final step in determining the exposure time was to determine the optimal speed. Sintering frequency and power was set at the determined optimal value of 50% and 3W respectively. Figure 18 shows the sheet resistance as a function of speed. Outlier data was observed at 25% and 30%. However, if this is ignored, R_{sheet} does not appear to be drastically affected by speed variation. Minimum sheet resistance was observed at 45% speed.

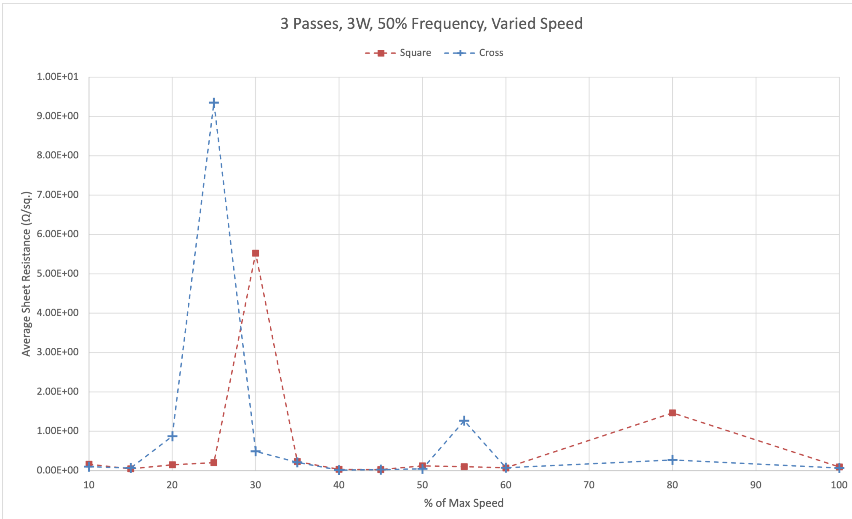


Figure 18: *Average sheet resistance as a function of speed at 3W and 50% frequency.*

Table 3 summarizes the three lowest R_{sheet} values obtained during the entire characterization process. Typical lowest sheet resistance reported in literature [8], is about 20 mΩ/sq. This corroborates well with the data obtained in our experiments.

Table 3: *Lowest Sheet Resistance values and respective sintering parameters for Kapton substrate.*

	Laser Cutter Parameters/Settings	***Measured R_{sheet}***
1	3 pass, P = 3W, f = 50% v = 50%	~12 mΩ/sq.
2	3 pass, P = 3W, f = 40% v = 50%	~23 mΩ/sq.
3	3 pass, P = 3W, f = 50%, v = 45%	~28 mΩ/sq.

PET

Initial tests on PET samples were carried out using the copper wire setup. Figure 19 shows the mean and variance of sheet resistance of 2 pass PET sintered at 1W, 3W and 5W with 50% speed and frequency. Data for cross geometry sintered at 1W is missing since those samples were damaged after multiple test runs being conducted to get the test setup working reliably. The variance of the measurement is very high. However, the trends do indicate that 3W is possibly the optimal sintering power.

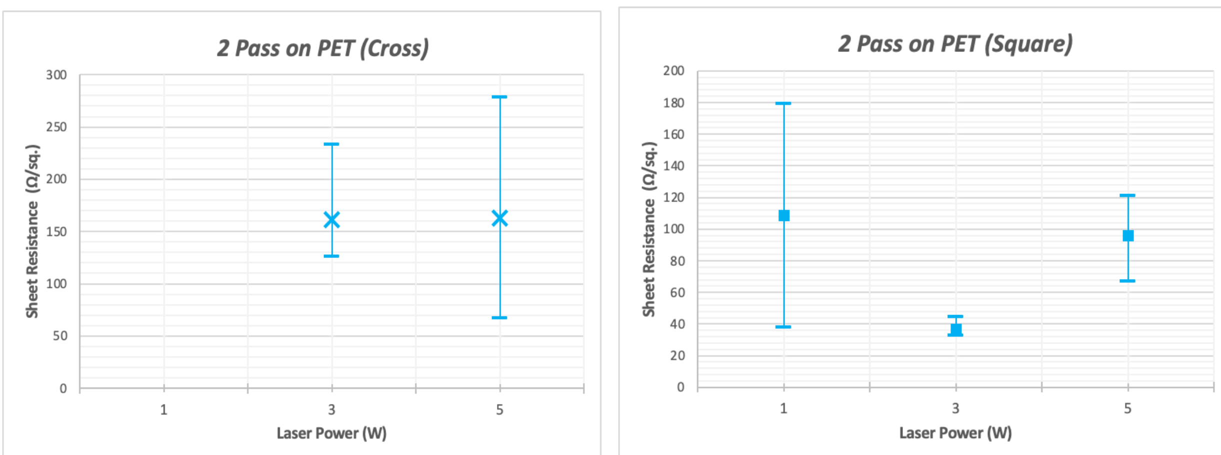


Figure 19: *Sheet Resistance with PET substrate for varying sintering power.*

Measurements with the tinner copper wire setup on the PET substrates did not result in any meaningful data. Measuring the control samples resulted in reaching the parameter analyzer voltage limits and attempting to address this by modifying the current sweep reached the allowed limit of current on the analyzer as well. This is mostly likely due to two reasons; firstly, the resistance might be too high to measure using tinned copper wire. The linear rise in resistance as observed in Figure 12 for Kapton was still observed in the PET substrates with the tinner copper wire. However, the resistance values were of the order of tens of kilo ohms. Even at 5mA current, voltage of ~6V was observed and the resistance value kept on increasing. Eventually, a voltage limit at 10V was reached (the max setting possible in the B1500A) before any stable resistance data could be recorded.

Secondly, increasing the thermal annealing warps the PET substrate leading to poor adhesion of the traces and complete loss of flexible mechanical properties. The significantly lower thermal annealing temperature limit for PET results in much higher resistance in the traces due to more suspension solvent present that cannot be evaporated off at lower temperatures. This demonstrates the unlikelihood of PET being utilized in flexible electronic applications unless this issue is addressed at the ink development stage and not sintering. The results of the Kapton experiments are only more important.

Conclusion and Future Work

In this project, we explored using the laser cutter as a sintering tool for conductive metal traces printed on flexible polymeric substrates. The samples were prepared by depositing silver nanoparticles with an inkjet printer onto flexible Kapton and PET films. A fiber laser source (1062nm) was used for sintering and the sintered samples were characterized by measuring their sheet resistance on a probe station. It was determined, for 3-pass silver patterns printed on Kapton, that sintering at 3W power, 50% speed and 50% frequency (Epilog Fusion M2) gave a lowest sheet resistance of 12 mΩ/sq, which is lower than that of bulk silver reported in literature and much lower than the initial sheet resistance of the samples by several orders of magnitude.

While we have readily obtained the sheet resistance for the samples on Kapton substrate, it was difficult to get measurements for PET substrate with the current setup as the sheet resistance went beyond the limit of the micromanipulator. However, PET will be a good candidate for flexible electronics due to its low cost and continued lack of compatibility with metal thermal requirements. Therefore, the next step is to develop a new method or to find an appropriate setup for measuring sheet resistances of conductive traces on PET substrates. In addition, the dimensions of the samples used in this project are on the centimeter scale (2 cm and 1.5 cm). Future work can explore the feasibility of using a laser cutter to sinter in more confined areas of lower minimum resolution.

References

[1] E. Gieva, G. Nikolov and B. Nikolova, "Sheet Resistance Measurement of Inkjet Printed Layers," 2019 42nd International Spring Seminar on Electronics Technology (ISSE), 2019, pp. 1-6, doi: 10.1109/ISSE.2019.8810284.
[2] *Dimatix Materials Printer DMP-2800 Series User Manual,* Fujifilm Dimatix, Inc.
[3] *Epilog Laser Fusion M2 32/40 Laser System Manual - Model 13000/14000*, Epilog Laser, 2019.
[4] M. Kim, J. Y. Hwang, H. Kang, K. Kang, S. Lee and S. Moon, "Laser sintering of the printed silver ink," 2009 IEEE International Symposium on Assembly and Manufacturing, 2009, pp. 155-158.
[5] Myong-Ki Kim et al., "Laser sintering of inkjet-printed silver nanoparticles on glass and PET substrates," 10th IEEE International Conference on Nanotechnology, 2010, pp. 520-524.
[6] E. A. Rojas-Nastrucci et al., "Characterization and Modeling of K-Band Coplanar Waveguides Digitally Manufactured Using Pulsed Picosecond Laser Machining of Thick-Film Conductive Paste," in IEEE Transactions on Microwave Theory and Techniques, 2017, vol. 65, no. 9, pp. 3180-3187.

[7] C. E. Hajjaji et al., "Optimizing the Conductivity of Ink-Jet Printed Microwave Components on Polymer Substrates by Laser Sintering," 2019 49th European Microwave Conference (EuMC), 2019, pp. 778-781.
[8] S. Merilampi, T. Laine-Ma, P. Ruuskanen,"The characterization of electrically conductive silver ink patterns on flexible substrates", Microelectronics Reliability, 2009, vol. 49, issue 7, pp. 782-790.

Appendix

Budget

Materials		
Item	*Quantity*	*Subtotal ($)*
NovaCentrix silver inkjet ink (JS-A211-S)	1 (50ml bottle)	335.00
Fujifilm Dimatix Samba cartridges	1 (box of 10)	1299.00
Kapton polyimide film (0.005'' thick)	5 (12" x 12")	157.25
PET film (0.005'' thick)	1 (12" x 40")	22.34
Tinned copper wires	3	56.89
Equipment		
Tool Name	*Hours*	*Subtotal ($)*
nanoinkjet	55	548.33
lasercutter	15	549.75
micromanipulator	30	150

Total ($)	**3118.56**

Standard Operating Procedure - Inkjet Printing JS-A211 Silver Nanoparticle Ink

See Next Page.

STANFORD UNIVERSITY

STANDARD OPERATING PROCEDURE TEMPLATE
Stanford Nanofabrication Facility

#1	CONTACT INFORMATION

Procedure Title	**Inkjet Printing of Conductive Inks**
Procedure Author	Jasmine Cox
Creation/Revision Date	04/17/22
Responsible Person	***Stanford Nanofabrication Facility, Swaroop Kommera***
Location of Procedure	Allen 155, Mavericks
Approval	[PROM approval is required.]

#2	THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

☒ **Specific laboratory procedure or experiment**
[Examples: synthesis of chemiluminescent esters, folate functionalization of polymeric micelles, etc.]

☒ **Generic laboratory procedure that covers several chemicals**
[Examples: distillation, chromatography, etc.]

☐ **Generic use of specific chemical or class of chemicals with similar hazards**
[Examples: organic azides, mineral acids, etc.]

#3	PROCESS OR EXPERIMENT DESCRIPTION

This process involves the deposition of conductive metallic traces on polymer substrates using piezoelectric inkjet printing in SNF with Novacentrix Metalon JS-A211 Silver Inkjet Ink.

Frequency:	☐ one time ☒ daily ☒ weekly ☐ monthly ☒ other: As needed
Duration per Expt:	_______ minutes; or ___2____hours

#4	SAFETY LITERATURE REVIEW & HAZARD SUMMARY

1. Hazardous Substances

   Novacentrix Metalon JS-A211 Silver Inkjet Ink – SDS attached

2. Other Hazards

N/A

3. References

Dimatix Printer Operation - attached

[List all references you are using for the safe and effective design of your process or experiment, including safety literature and peer-reviewed journal articles. Safety resources are available at http://web.stanford.edu/dept/EHS/cgi-bin/lcst/creating-standard-operating-procedures/.]

#5	**STORAGE REQUIREMENTS**

Novacentrix JS-A211 should be stored away from combustible materials and high temperatures. Prints containing unannealed traces of JS-A211 should also be stored away from combustible materials and high temperatures.

#6	**STEP-BY-STEP OPERATING PROCEDURE**

[For each step's description, include any step-specific hazard, personal protective equipment, engineering controls, and designated work areas in the left hand column.

a. **Guidance on Engineering and Ventilation Controls –** Review safety literature and peer-reviewed journal articles to determine appropriate engineering and ventilation controls for your process or experiment. Guidance is available from health and safety specialists at Stanford EH&S and online in the General Use SOPs and Laboratory Safety Sheets in the Laboratory Chemical Safety Toolkit (http://chemtoolkit.stanford.edu/)

b. **Guidance on Personal Protective Equipment -** To assist with your PPE selection, refer to http://chemtoolkit.stanford.edu/LabPPE. Respiratory protection is generally not required for lab research, provided the appropriate engineering controls are employed. For additional guidance on respiratory protection, consult with EH&S, 723-0448.

c. **Designated work area(s)** - Required whenever carcinogens, highly acutely toxic materials, or reproductive toxins are used. The intent of a designated work area is to limit and minimize possible sources of exposure to these materials. The entire laboratory, a portion of the laboratory, or a laboratory fume hood or bench may be considered a designated area. See the Chemical Safety Toolkit for more information.

Describe the possible risks involved with failure to follow a step in the SOP in the right hand column.]

Step-by-Step Description of Your Process or Experiment	**Potential Risks if Step is Not Done or Done Incorrectly (if any)**
1. Don personal protective equipment. ☒ appropriate street clothing (long pants, closed-toed shoes) ☒ gloves; indicate type:Nitrile ☐ safety goggles ☒ safety glasses ☐ face shield ☒ lab coat ☐ flame-resistant lab coat ☐ other: ________	
2. Check the location/accessibility/certification of the safety equipment that serves your lab:	

ITEM	STATUS	
Laboratory Fume Hood/Glove Box or other Ventilation Control	Location: Allen 155 *Check sticker to ensure that hood was certified within last 12 months.*	
Eyewash/Safety Shower	Location: Allen 155 *Ensure that it is accessible, not blocked. Check tag that it has been tested within last month.*	
First Aid Kit	Location: Allen 155	
Chemical Spill Kit	Location: Allen 155	
Fire Extinguisher	Location: Allen 155	
Telephone	Location: Allen 155	
Fire Alarm Manual Pull Station	Location: Allen 155	

3. In the fume hood, load the appropriate amount of ink into the appropriate syringe with the provided Dimatix cartridge needle tip (Volume should be less than 1.5 mL).	More ink is loaded into syringe than Dimatix reservoir has capacity for.
4. In the fume hood, insert the syringe tip fully into the Dimatix cartridge reservoir and depress the plunger to fill the reservoir. Clean the syringe tip and dispose of the syringe as solid waste.	Reservoir is overfilled and syringe backfires.
5. Attach the cartridge print head to the cartridge reservoir and ensure that cartridge is fully seated and clipped on top of the reservoir. The white plastic clips of the reservoir should be visible in the openings at each side of the print head.	Reservoir is not sealed and ink leaks when cartridge is loaded into printer.
6. Insert the cartridge into Dimatix printer in the cartridge holder. Ensure the circular opening on the print head is aligned to the circular opening of the holder and the cartridge is clipped into the holder. Clip the cartridge holder on top of the cartridge to ensure it is placed properly.	
7. Insert the cleaning pad into the designated opening on the platen by pressing the black end of the pad into the opening until it is clipped in. Remove the cleaning pad cap.	Print head runs into unseated cleaning pad during printing.
8. Inkjet print using conductive ink.	
9. Unclip and lift cartridge from cartridge holder. Replace cap on cleaning pad and press down to unclip from printer platen. Store both items.	
10. Wipe down any ink spills and workspace that may have ink solution exposure with IPA and wipes. Dispose of wipes as solid waste.	Conductive ink may contaminate work area for future unknowing users.
11. Remove PPE and wash hands. Gloves are disposed of as solid waste.	

#7	EMERGENCY PROCEDURES

1. **Health-Threatening Emergencies**

2. **Fire, explosion, health-threatening hazardous material spill or release, compressed gas leak, or valve failure, etc.**

3. Call 911 (9-911 from a landline).
4. Alert people in the vicinity and activate the local alarm systems.
5. Evacuate the area and go to your Emergency Assembly Point (EAP) at the Circle Fountain.
6. Remain nearby to advise emergency responders.
7. Once personal safety is established, call EH&S at 725-9999 or the SNF Duty Phone at (650)521-7306.
8. Provide local notifications (Duty phone at (650)521-7306).

   Note: For compressed gas leaks, shut off gas supply only if this can be done safely, without risk to personnel.

9. **Injuries and Exposures:**

10. Remove the injured/exposed individual from the area, unless it is unsafe to do so because of the medical condition of the victim or the potential hazard to rescuers.
11. Call 911 (9-911 from a landline) if immediate medical attention is required.
12. Call 725-9999 to report the exposure to EH&S.
13. Administer first aid as appropriate.
14. Flush contamination from eyes/skin using the nearest emergency eyewash/shower for a minimum of 15 minutes. Remove any contaminated clothing.
15. Bring to the hospital copies of SDSs for all chemicals the victim was exposed to.

16. **Non-Health-Threatening Emergencies**

17. **Injuries and Exposures**

    For injuries and exposures that are not considered serious or a medical emergency, call the Occupational Health Center (OHC) at 725-5308 between 8:00 am-5:00 pm M-F at (650) 725-5308 for immediate phone triage and to schedule an appointment. For urgent conditions when SUOHC is closed, go to the Stanford University Medical Center Emergency Department.

18. **Spills**

    For hazardous material spills or releases which have impacted the environment (via the storm drain, soil, or air outside the building) or for a spill or release that cannot be cleaned up by local personnel:

19. Notify Stanford University responders by calling 725-9999. These services are available 24 hours a day, 7 days a week.
20. Provide local notifications (Duty phone at (650)521-7306).

21. **Local Cleanup of Small Spills**

    In the event of a minor spill or release that can be safely cleaned up by local personnel using readily available equipment (absorbent available from EH&S in Small Spill Kit) and laboratory PPE:

22. Notify personnel in the area and restrict access. Eliminate all sources of ignition.
23. Review the SDS for the spilled material, or use your knowledge of the hazards of the material to determine the appropriate level of protection (do not clean up spills requiring respiratory protection locally).
24. Wearing appropriate personal protective equipment, clean up spill. Collect spill cleanup materials in a tightly closed container. Manage spill cleanup debris as hazardous waste.
25. Submit online waste pickup request to EH&S.
26. Reporting Requirements: All spills cleaned up locally must be reported if they occur outside of secondary containment. A spill that occurs within secondary containment (a laboratory hood is considered secondary containment) must be reported if it is greater than 30 ml or if it takes longer than 15 minutes to clean up. To report a spill, call EH&S at 725-9999 as soon as possible.

27. Lab-Specific Procedures

[This section is for any emergency procedures different from standard responses, or for additional emergency information due to the nature of materials or task. Include information on gas leaks, chemical spills, and personal exposure/medical emergency as appropriate.]

28. Building Maintenance Emergencies

Call Facilities Operations at 723-2281 (or 721-2146 in the School of Medicine) for building maintenance emergencies (e.g., power outages, plumbing leaks).

29. Local Notifications

Contact the Duty phone at (650)521-7306 and send an email to snf-safety@lists.stanford.edu.

#8	**WASTE DISPOSAL**

Solid waste will be placed into ziploc bag and placed in designated waste disposal bins available in Allen 155. Liquid waste generated will be placed into appropriate waste container with label and stored in satellite waste accumulation in SNF until pick-up.

#9	**TRAINING REQUIREMENTS**

General Training *(check all that apply):*

☒ SNF Safety Tour

☒ Room 155 Safety Orientation

☒ Other: Dimatix Operation Training

Location Where Records Maintained:	Badger

Laboratory-specific training *(check all that apply):*

☒ Review of SDS for chemicals involved in process/experiment

☒ Review of this SOP

☐ Other: _______

Location Where Records Maintained:	Allen 155

#10	**PRIOR APPROVALS**

[You **must** seek prior approval from your principal investigator (PI) or lab supervisor if you plan to use **restricted chemicals** (dimethylmercury and toxic gases regulated by Santa Clara County).

You should also consult your PI or lab supervisor if your experiments involve **high-risk chemicals and operations,** as special safety precautions may need to be taken. High-risk chemicals and operations may involve chemicals with a high level of acute toxicity, carcinogens, reproductive toxins, and highly reactive materials. For additional guidance, see section 5.3 of the Chemical Hygiene Plan.

Your PI or lab supervisor's prior approval may be documented by his/her signature in the Approval Signature section of this document. For granting prior approval to individuals other than the procedure author, use one of the methods described at http://web.stanford.edu/dept/EHS/cgi-bin/lcst/restricted-chemicals-high-risk-procedures/.]

Prior Approval *(check if applicable):*

☐ Prior approval from the PI or lab supervisor is required for this procedure

Standard Operating Procedure - Laser Sintering with Laser Cutter

See Next Page.

STANFORD UNIVERSITY

STANDARD OPERATING PROCEDURE TEMPLATE
Stanford Nanofabrication Facility

#1	CONTACT INFORMATION

Procedure Title	**Laser sintering with laser cutter**
Procedure Author	Mingqi Shuai
Creation/Revision Date	06/07/22
Responsible Person	***Stanford Nanofabrication Facility, Swaroop Kommera***
Location of Procedure	Allen 155A, Venice
Approval	[Obtain prior approval, as appropriate. See section #10.]

#2	THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

☒ **Specific laboratory procedure or experiment**
[Examples: synthesis of chemiluminescent esters, folate functionalization of polymeric micelles, etc.]

☐ **Generic laboratory procedure that covers several chemicals**
[Examples: distillation, chromatography, etc.]

☐ **Generic use of specific chemical or class of chemicals with similar hazards**
[Examples: organic azides, mineral acids, etc.]

#3	PROCESS OR EXPERIMENT DESCRIPTION

This process involves sintering ready-printed conductive silver traces on Kapton film utilizing the Epilog Fusion M2 laser cutter. The laser parameters are specific for 3 passes inkjet printed silver traces (Novacentrix, JS-A211S) on 0.005'' Kapton film. For sintering other conductive traces, the laser parameters can be optimized by adjusting the power, the speed, and the frequency.

Frequency:	☐ one time ☐ daily ☐ weekly ☐ monthly ☒ other: As needed
Duration per Expt:	___90___ minutes; or _______hours

#4	SAFETY LITERATURE REVIEW & HAZARD SUMMARY

1. Hazardous Substances

N/A

2. Other Hazards

Fire hazards

3. References

Epilog Fusion M2 laser system manual - attached

[List all references you are using for the safe and effective design of your process or experiment, including safety literature and peer-reviewed journal articles. Safety resources are available at http://web.stanford.edu/dept/EHS/cgi-bin/lcst/creating-standard-operating-procedures/.]

#5	STORAGE REQUIREMENTS

N/A

#6	STEP-BY-STEP OPERATING PROCEDURE

[For each step's description, include any step-specific hazard, personal protective equipment, engineering controls, and designated work areas in the left hand column.

a. **Guidance on Engineering and Ventilation Controls –** Review safety literature and peer-reviewed journal articles to determine appropriate engineering and ventilation controls for your process or experiment. Guidance is available from health and safety specialists at Stanford EH&S and online in the General Use SOPs and Laboratory Safety Sheets in the Laboratory Chemical Safety Toolkit (http://chemtoolkit.stanford.edu/)

b. **Guidance on Personal Protective Equipment -** To assist with your PPE selection, refer to http://chemtoolkit.stanford.edu/LabPPE. Respiratory protection is generally not required for lab research, provided the appropriate engineering controls are employed. For additional guidance on respiratory protection, consult with EH&S, 723-0448.

c. **Designated work area(s)** - Required whenever carcinogens, highly acutely toxic materials, or reproductive toxins are used. The intent of a designated work area is to limit and minimize possible sources of exposure to these materials. The entire laboratory, a portion of the laboratory, or a laboratory fume hood or bench may be considered a designated area. See the Chemical Safety Toolkit for more information.

Describe the possible risks involved with failure to follow a step in the SOP in the right hand column.]

Step-by-Step Description of Your Process or Experiment	Potential Risks if Step is Not Done or Done Incorrectly (if any)
1. Don personal protective equipment. ☒ appropriate street clothing (long pants, closed-toed shoes) ☒ gloves; indicate type:Nitrile ☐ safety goggles ☒ safety glasses ☐ face shield ☒ lab coat ☐ flame-resistant lab coat ☐ other: _______	

2. Check the location/accessibility/certification of the safety equipment that serves your lab:

ITEM	STATUS
Laboratory Fume Hood/Glove Box or other Ventilation Control	Location: Allen 155A *Check sticker to ensure that hood was certified within last 12 months.*
Eyewash/Safety Shower	Location: Allen 155 *Ensure that it is accessible, not blocked. Check tag that it has been tested within last month.*
First Aid Kit	Location: Allen 155
Chemical Spill Kit	Location: Allen 155
Fire Extinguisher	Location: Allen 155A
Telephone	Location: Allen 155A
Fire Alarm Manual Pull Station	Location: Hallway outside 155A

3. Log on Badger and activate the laser cutter. Turn on filter (press the on/off button) and air pump (open the valve clamped to the table next to the laser cutter). Turn on the laser cutter using the on/off switch.	Without appropriate air filtering, the contaminated air or inhalable dust will leak out and pose health risks.
4. Turn on the computer and open CorelDraw x8.	
5. (This step can be skipped if no need for accurate alignment.) Have a known mark on your sample and draw that mark in CorelDraw in black. Engrave it onto the tile.	Inaccurate sintering of desired patterns.
6. If needed, align your sample with the alignment mark on the tile. Make sure the sample is flat and use Kapton tapes to secure the sample onto the tile. Check that the tape does not cover your sintering area.	The flexible substrates will not be stable during sintering, results in bad sintering.
7. Open the laser cutter lid. Place the prepared sample (on tile) onto the laser bed. Put the manual focus gauge on the carriage. The "V" shaped gauge should be on the left-hand side of the laser cutter. Make sure to use the silver 3'' gauge for fiber laser. On the right-hand side, use the arrow key on the control panel to select 'Focus', then use the joystick to slowly adjust the laser bed until the sample just touches the bottom of the gauge.	Out of focus will lead to inefficient sintering.
8. Draw your pattern for sintering in black in CorelDraw or import your file. Select File/Print and click the Preferences button to open up the laser dashboard. Under the 'General' tab, select **Fiber** for laser source and **Raster** for job type. Change the Piece Size (Inches) to **32** horizontal and **20** vertical. Under 'Raster Setting', change the Power to **6%** (corresponds to 3W), and leave the Speed and Frequency at 50% default values. Click the selection bar for 'Image Dithering' and select	Wrong parameter input will results in inadequate sintering. However, these settings are specific to 3 passes inkjet printed silver traces (Novacentrix, JS-A211S) on 0.005'' Kapton

Stucki. Leave the resolution at **600 DPI** and the engraving direction at **Top-Down**. Double check that the boxes for 'Send to Manager' and 'Send to Laser' are selected and no other boxes are selected. Then hit the 'OK' button on the bottom right.	film. For sintering other conductive traces, the laser parameters can be further optimized.
9. After clicking 'OK', it will automatically return to the print window. Check in the print preview that your pattern is in the correct location and do print. Make sure the lid of the laser cutter is closed. On the laser cutter control panel, make sure it's under 'Job'. Wait until the job is sent to the laser cutter and hit the green Go button.	
10. Be at the laser cutter and watch the entire sintering process.	Be alert for potential fire hazards.
11. The laser cutter will give a beep sound after once the job is finished. After the job is finished, remove your sample from the laser cutter.	
12. Close the laser cutter lid. Turn the laser cutter off. Turn off the air pump and filter. Exit CorelDraw and sign off. Deactivate the laser cutter on Badger.	
13. Remove PPE and wash hands.	

#7	**EMERGENCY PROCEDURES**

1. **Health-Threatening Emergencies**

2. **Fire, explosion, health-threatening hazardous material spill or release, compressed gas leak, or valve failure, etc.**

3. Call 911 (9-911 from a landline).
4. Alert people in the vicinity and activate the local alarm systems.
5. Evacuate the area and go to your Emergency Assembly Point (EAP) at the Circle Fountain.
6. Remain nearby to advise emergency responders.
7. Once personal safety is established, call EH&S at 725-9999 or the SNF Duty Phone at (650)521-7306.
8. Provide local notifications (Duty phone at (650)521-7306).

   Note: For compressed gas leaks, shut off gas supply only if this can be done safely, without risk to personnel.

9. **Injuries and Exposures:**

10. Remove the injured/exposed individual from the area, unless it is unsafe to do so because of the medical condition of the victim or the potential hazard to rescuers.
11. Call 911 (9-911 from a landline) if immediate medical attention is required.
12. Call 725-9999 to report the exposure to EH&S.
13. Administer first aid as appropriate.
14. Flush contamination from eyes/skin using the nearest emergency eyewash/shower for a minimum of 15 minutes. Remove any contaminated clothing.
15. Bring to the hospital copies of SDSs for all chemicals the victim was exposed to.

16. **Non-Health-Threatening Emergencies**

17. Injuries and Exposures

For injuries and exposures that are not considered serious or a medical emergency, call the Occupational Health Center (OHC) at 725-5308 between 8:00 am-5:00 pm M-F at (650) 725-5308 for immediate phone triage and to schedule an appointment. For urgent conditions when SUOHC is closed, go to the Stanford University Medical Center Emergency Department.

18. Spills

For hazardous material spills or releases which have impacted the environment (via the storm drain, soil, or air outside the building) or for a spill or release that cannot be cleaned up by local personnel:

19. Notify Stanford University responders by calling 725-9999. These services are available 24 hours a day, 7 days a week.
20. Provide local notifications (Duty phone at (650)521-7306).

21. Local Cleanup of Small Spills

In the event of a minor spill or release that can be safely cleaned up by local personnel using readily available equipment (absorbent available from EH&S in Small Spill Kit) and laboratory PPE:

22. Notify personnel in the area and restrict access. Eliminate all sources of ignition.
23. Review the SDS for the spilled material, or use your knowledge of the hazards of the material to determine the appropriate level of protection (do not clean up spills requiring respiratory protection locally).
24. Wearing appropriate personal protective equipment, clean up spill. Collect spill cleanup materials in a tightly closed container. Manage spill cleanup debris as hazardous waste.
25. Submit online waste pickup request to EH&S.
26. Reporting Requirements: All spills cleaned up locally must be reported if they occur outside of secondary containment. A spill that occurs within secondary containment (a laboratory hood is considered secondary containment) must be reported if it is greater than 30 ml or if it takes longer than 15 minutes to clean up. To report a spill, call EH&S at 725-9999 as soon as possible.

27. Lab-Specific Procedures

[This section is for any emergency procedures different from standard responses, or for additional emergency information due to the nature of materials or task. Include information on gas leaks, chemical spills, and personal exposure/medical emergency as appropriate.]

28. Building Maintenance Emergencies

Call Facilities Operations at 723-2281 (or 721-2146 in the School of Medicine) for building maintenance emergencies (e.g., power outages, plumbing leaks).

29. Local Notifications

Contact the Duty phone at (650)521-7306 and send an email to snf-safety@lists.stanford.edu.

#8	**WASTE DISPOSAL**

N/A

#9	**TRAINING REQUIREMENTS**

General Training *(check all that apply):*

☒ General Safety & Emergency Preparedness (EHS-4200)
☒ SNF Safety Tour
☒ Other: Epilog Fusion M2 laser cutter training

Location Where Records Maintained:	Badger

Laboratory-specific training *(check all that apply):*

☐ Review of SDS for chemicals involved in process/experiment
☒ Review of this SOP
☒ Other: Review of the operation manual for Epilog Fusion M2 laser cutter

Location Where Records Maintained:	Allen 155A

#10	**PRIOR APPROVALS**

[You **must** seek prior approval from your principal investigator (PI) or lab supervisor if you plan to use **restricted chemicals** (dimethylmercury and toxic gases regulated by Santa Clara County).

You should also consult your PI or lab supervisor if your experiments involve **high-risk chemicals and operations,** as special safety precautions may need to be taken. High-risk chemicals and operations may involve chemicals with a high level of acute toxicity, carcinogens, reproductive toxins, and highly reactive materials. For additional guidance, see section 5.3 of the Chemical Hygiene Plan.

Your PI or lab supervisor's prior approval may be documented by his/her signature in the Approval Signature section of this document. For granting prior approval to individuals other than the procedure author, use one of the methods described at http://web.stanford.edu/dept/EHS/cgi-bin/lcst/restricted-chemicals-high-risk-procedures/.]

Prior Approval *(check if applicable):*

☐ Prior approval from the PI or lab supervisor is required for this procedure

Standard Operating Procedure - 4-point Probe Set-up for Flexible Substrates

See Next Page.

STANFORD UNIVERSITY

STANDARD OPERATING PROCEDURE TEMPLATE
Stanford Nanofabrication Facility

#1	CONTACT INFORMATION

Procedure Title	**Four-point Sheet Resistance Measurement on Flexible Substrate**
Procedure Author	Aditya Shah
Creation/Revision Date	06/09/22
Responsible Person	***Stanford Nanofabrication Facility, Swaroop Kommera***
Location of Procedure	Allen 151, Ocean
Approval	[Obtain prior approval, as appropriate. See section #10.]

#2	THIS STANDARD OPERATING PROCEDURE (SOP) IS FOR A:

☒ **Specific laboratory procedure or experiment**
[Examples: synthesis of chemiluminescent esters, folate functionalization of polymeric micelles, etc.]

☐ **Generic laboratory procedure that covers several chemicals**
[Examples: distillation, chromatography, etc.]

☐ **Generic use of specific chemical or class of chemicals with similar hazards**
[Examples: organic azides, mineral acids, etc.]

#3	PROCESS OR EXPERIMENT DESCRIPTION

This process involves setting up the micromanipulator6000 probe station for four-point sheet resistance measurements of printed conductive traces on flexible substrates.

Frequency:	☐ one time ☐daily ☐weekly ☐ monthly ☒ other: As needed
Duration per Expt:	___90____ minutes; or _______hours

#4	SAFETY LITERATURE REVIEW & HAZARD SUMMARY

1. Hazardous Substances

N/A

2. Other Hazards

N/A

3. References
Micromaniupulator6000 User Manual - link attached
https://snfexfab.stanford.edu/docs/operating-instructions/micromanipulator6000

#5	**STORAGE REQUIREMENTS**

N/A

#6	**STEP-BY-STEP OPERATING PROCEDURE**

[For each step's description, include any step-specific hazard, personal protective equipment, engineering controls, and designated work areas in the left hand column.

a. **Guidance on Engineering and Ventilation Controls –** Review safety literature and peer-reviewed journal articles to determine appropriate engineering and ventilation controls for your process or experiment. Guidance is available from health and safety specialists at Stanford EH&S and online in the General Use SOPs and Laboratory Safety Sheets in the Laboratory Chemical Safety Toolkit (http://chemtoolkit.stanford.edu/)

b. **Guidance on Personal Protective Equipment -** To assist with your PPE selection, refer to http://chemtoolkit.stanford.edu/LabPPE. Respiratory protection is generally not required for lab research, provided the appropriate engineering controls are employed. For additional guidance on respiratory protection, consult with EH&S, 723-0448.

c. **Designated work area(s)** - Required whenever carcinogens, highly acutely toxic materials, or reproductive toxins are used. The intent of a designated work area is to limit and minimize possible sources of exposure to these materials. The entire laboratory, a portion of the laboratory, or a laboratory fume hood or bench may be considered a designated area. See the Chemical Safety Toolkit for more information.

Describe the possible risks involved with failure to follow a step in the SOP in the right hand column.]

Step-by-Step Description of Your Process or Experiment	**Potential Risks if Step is Not Done or Done Incorrectly (if any)**
1. Don personal protective equipment. ☒ appropriate street clothing (long pants, closed-toed shoes) ☒ gloves; indicate type:Nitrile ☒ safety goggles ☒ safety glasses ☐ face shield ☐ lab coat ☐ flame-resistant lab coat ☐ other: _______	
2. Check the location/accessibility/certification of the safety equipment that serves your lab:	

<table><tr><th>ITEM</th><th>STATUS</th></tr><tr><td>Laboratory Fume Hood/Glove Box or other Ventilation Control</td><td>Location: <u>Allen 151</u> *Check sticker to ensure that hood was certified within last 12 months.*</td></tr><tr><td>Eyewash/Safety Shower</td><td>Location: <u>Allen 155</u> *Ensure that it is accessible, not blocked. Check tag that it has been tested within last month.*</td></tr><tr><td>First Aid Kit</td><td>Location: <u>Allen 151</u></td></tr><tr><td>Chemical Spill Kit</td><td>Location: <u>Allen 151</u></td></tr><tr><td>Fire Extinguisher</td><td>Location: <u>Allen 151</u></td></tr><tr><td>Telephone</td><td>Location: <u>Hallway outside 151</u></td></tr><tr><td>Fire Alarm Manual Pull Station</td><td>Location: <u>Hallway outside 151</u></td></tr></table>	
3. Take about 1 inch of tinned copper wire and flatten one of the ends using pliers. Make four such pieces.	
4. Take 4 micromanipulator needle probes. Using ethanol, clean these and the wire pieces to remove any dirt	Ensure gloves are on. Wipe off any solvent that might have spilled
5. On the solder station at the end of the room, solder each tinned copper wire piece to the needle probes, such that the flat end of the wire extends beyond the needle-end of the probes. Cover the solder with electrical insulation tape.	Needle probes get very hot, very quickly. Do not attempt to touch them with your hand.
6. Using the allen key, remove any existing probes on the station. Replace them with the soldered probes. Ensure all probes are approximately at equal level height and vertical travel screw is midway in position. This ensures you can move the probes up and down without reaching the limit.	
7. Ensure all SMU's are connected to the probes	The test will still run and you might think the contact is lose
8. Activate micromanipulator on Badger. Place the substrate on the chuck and turn on the vacuum. Lower the probes and ensure proper contact with the conductive traces. Close the lid.	Contact too close to the edge of the printed trace.
9. Create a new workspace on the B1500A. Under 'Category' choose 'Structures'. Under 'Library', select 'VanDerPauw Square' test setup. Choose the desired SMUs for current input and voltage readings respectively from the drop down menu in the circuit layout.	
10. Enter the desired current start, stop range and step value against 'I! Start', 'I1 Stop' and 'I1 Step' respectively. Select compliance voltage 'V1 Limit'.	Min I1 Start : 100 uA Max I1 Stop : 100 mA Max V1 Limit : 10V

11. Run the test and observe the resistance values. Stop if the voltage exceeds compliance values. If resistance is negative, interchange SMUs reading the voltage.	If setup is correct, the voltage measurement will be a linearly increasing curve

12.Try changing order of SMUs if the setup does not work	
13. Record and name the test results. Open the lid and put down the probes on the next set of samples.	
14. After completion of the test run, replace the soldered probes with another set of plain needle probes for the next user. Close the lid, logout out of your workspace and deactivate the micromanipulator on badger	

#7 EMERGENCY PROCEDURES

1. **Health-Threatening Emergencies**

2. **Fire, explosion, health-threatening hazardous material spill or release, compressed gas leak, or valve failure, etc.**

3. Call 911 (9-911 from a landline).
4. Alert people in the vicinity and activate the local alarm systems.
5. Evacuate the area and go to your Emergency Assembly Point (EAP) at the Circle Fountain.
6. Remain nearby to advise emergency responders.
7. Once personal safety is established, call EH&S at 725-9999 or the SNF Duty Phone at (650)521-7306.
8. Provide local notifications (Duty phone at (650)521-7306).

   Note: For compressed gas leaks, shut off gas supply only if this can be done safely, without risk to personnel.

9. **Injuries and Exposures:**

10. Remove the injured/exposed individual from the area, unless it is unsafe to do so because of the medical condition of the victim or the potential hazard to rescuers.
11. Call 911 (9-911 from a landline) if immediate medical attention is required.
12. Call 725-9999 to report the exposure to EH&S.
13. Administer first aid as appropriate.
14. Flush contamination from eyes/skin using the nearest emergency eyewash/shower for a minimum of 15 minutes. Remove any contaminated clothing.
15. Bring to the hospital copies of SDSs for all chemicals the victim was exposed to.

16. **Non-Health-Threatening Emergencies**

17. **Injuries and Exposures**

   For injuries and exposures that are not considered serious or a medical emergency, call the Occupational Health Center (OHC) at 725-5308 between 8:00 am-5:00 pm M-F at (650) 725-5308 for immediate phone triage and to schedule an appointment. For urgent conditions when SUOHC is closed, go to the Stanford University Medical Center Emergency Department.

18. Spills

For hazardous material spills or releases which have impacted the environment (via the storm drain, soil, or air outside the building) or for a spill or release that cannot be cleaned up by local personnel:

19. Notify Stanford University responders by calling 725-9999. These services are available 24 hours a day, 7 days a week.
20. Provide local notifications (Duty phone at (650)521-7306).

21. Local Cleanup of Small Spills

In the event of a minor spill or release that can be safely cleaned up by local personnel using readily available equipment (absorbent available from EH&S in Small Spill Kit) and laboratory PPE:

22. Notify personnel in the area and restrict access. Eliminate all sources of ignition.
23. Review the SDS for the spilled material, or use your knowledge of the hazards of the material to determine the appropriate level of protection (do not clean up spills requiring respiratory protection locally).
24. Wearing appropriate personal protective equipment, clean up spill. Collect spill cleanup materials in a tightly closed container. Manage spill cleanup debris as hazardous waste.
25. Submit online waste pickup request to EH&S.
26. Reporting Requirements: All spills cleaned up locally must be reported if they occur outside of secondary containment. A spill that occurs within secondary containment (a laboratory hood is considered secondary containment) must be reported if it is greater than 30 ml or if it takes longer than 15 minutes to clean up. To report a spill, call EH&S at 725-9999 as soon as possible.

27. Lab-Specific Procedures

[This section is for any emergency procedures different from standard responses, or for additional emergency information due to the nature of materials or task. Include information on gas leaks, chemical spills, and personal exposure/medical emergency as appropriate.]

28. Building Maintenance Emergencies

Call Facilities Operations at 723-2281 (or 721-2146 in the School of Medicine) for building maintenance emergencies (e.g., power outages, plumbing leaks).

29. Local Notifications

Contact the Duty phone at (650)521-7306 and send an email to snf-safety@lists.stanford.edu.

#8	**WASTE DISPOSAL**

N/A

#9	**TRAINING REQUIREMENTS**

General Training *(check all that apply):*

☒ SNF Safety Tour

☒ Room 151 Safety Orientation

☒ Other: Micromanipulator6000 training

Location Where Records Maintained:	Badger

Laboratory-specific training *(check all that apply):*

☐ Review of SDS for chemicals involved in process/experiment

☒ Review of this SOP

☐ Other: Review of the operation manual for Micromanipulator6000

Location Where Records Maintained:	Allen 151

#10	**PRIOR APPROVALS**

[You **must** seek prior approval from your principal investigator (PI) or lab supervisor if you plan to use **restricted chemicals** (dimethylmercury and toxic gases regulated by Santa Clara County).

You should also consult your PI or lab supervisor if your experiments involve **high-risk chemicals and operations,** as special safety precautions may need to be taken. High-risk chemicals and operations may involve chemicals with a high level of acute toxicity, carcinogens, reproductive toxins, and highly reactive materials. For additional guidance, see section 5.3 of the Chemical Hygiene Plan.

Your PI or lab supervisor's prior approval may be documented by his/her signature in the Approval Signature section of this document. For granting prior approval to individuals other than the procedure author, use one of the methods described at http://web.stanford.edu/dept/EHS/cgi-bin/lcst/restricted-chemicals-high-risk-procedures/.]

Prior Approval *(check if applicable):*

☐ Prior approval from the PI or lab supervisor is required for this procedure