Standard Operating Procedure of Optomec Aerosol Jet Printer in Pneumatic Atomizer Mode and Characterization of Printed PEDOT:PSS Lines

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Executive Summary

The objective of this project is to delineate a standard operating procedure for the Optomec Aerosol Jet 300 Series System in the pneumatic atomizer mode and to understand its printing capabilities. The Optomec printer has a steep learning curve and the existing user-manual lacks critical details in areas such as tool operation, troubleshooting, handling of parts, and print optimization. To alleviate this problem for future Optomec users, we present herein a user-friendly, step-by-step standard operating procedure that incorporates cautions and cleaning protocols, the basic printing optimization approaches for a given ink, and how to troubleshoot the commonly encountered issues during Optomec operation.

The first part of this report provides a summary of current printing technologies, with particular emphasis on drop-on-demand printing. We explain the basic working principles of aerosol and inkjet printing, which represent the two main types of drop-on-demand printing. A comparison of the two in the context of the SNF is also given.

The second part of this report details the standard operating procedure of the Optomec in pneumatic atomizer mode, including the generation of print patterns, assembly of parts, initialization of the printer, troubleshooting common issues, printing and cleaning. This section is meant to complement the official Optomec user manual, which lacks details on machine operation.

The third section of this report describes our attempts at aerosol jet printing PEDOT:PSS, a conductive polymer. We present our results and a general framework for optimization. Though we varied ink and printing factors such as the substrate treatment, gantry speed, ink concentration and the carrier gas flows, we were unable to determine optimal conditions for print resolution and uniformity. We did however achieve printed films with a conductivity of 14 ± 10 S/cm, which is comparable to values found in literature.

The fourth and last section of this report concludes the report and offers suggestions for improving the presence of the Optomec within the SNF.

Contents

1 Introduction

The nascent field of printed electronics represents a transformative platform with broad technological implications. Printed devices such as bendable displays and wearable sensors offer attributes complementary to conventional electronics, including large-areas, low-cost, mechanical flexibility, and compatibility with roll-to-roll processes. Drop-on-demand techniques such as inkjet and aerosol jet are especially important for this field, as they offer additive, non-contact, digital patterning capabilities.

Recent years have seen immense progress in inkjet printed electronics, with demonstrations of displays, circuits, and biological sensors, among others. Aerosol jet printing, on the other hand, has only been explored by a handful of research groups in spite of its high versatility. In this section we describe the working principles behind aerosol jet printing, as well as its advantages and disadvantages compared to inkjet printing and more traditional patterning techniques.

1.1 Comparison of Drop-on-Demand Printing Techniques to Other Patterning Techniques

Drop-on-demand printing, as its name suggests, involves the dispensing of ink on demand onto the substrate. This is in contrast with contact printing techniques such as gravure or screen printing, wherein the ink is deposited all at once, and then patterned with masks or doctor blades. Patterning by drop-on-demand printing thus utilizes the movement of the printhead in combination with selective jetting of the ink. As such, drop-on-demand printing techniques tend to be digital, additive as well as non-contact, making them ideal for lab-scale prototyping.

The two main drop-on-demand printing techniques are inkjet and aerosol-jet printing. The working principles of both of these technologies are detailed in the following sections.

1.2 Working Principles of Aerosol Jet Printing (Optomec)

The aerosol jet printing process utilizes aerodynamic focusing of atomized ink microdroplets to enable patterning of fine, conformal features on non-planar substrates. This process is shown schematically in Figure 1. For the Optomec, an ink is first atomized into droplets of 1∼5 µm diameter in size [1,3], either in pneumatic or ultrasonic mode. These aerosol droplets are entrained in a gas stream (typically clean, dry N_2 or compressed air) and directed to the printhead [1,3]. A sheath gas annularly compresses the aerosol stream to a beam as small as 10 µm in diameter at the printhead such that the aerosol stream is tightly focused inside the sheath gas during acceleration through the printhead to be impinged on the substrate [1,3]. The sheath gas also prevents the direct contact between the aerosol beam and the nozzle from which the aerosol beam exits to minimize clogging [3].

Optomec claims that noncontact and conformal patterning of feature sizes as small as 10 µm can be achieved on various substrates (e.g. silicon, quartz, silicone) using various inks (e.g. metallic, polymeric, biological) [1,3]. Optomec also claims that the maximum printing distance for which this resolution is maintained is 5 mm, enabling consistent deposition of ink on 3D substrates [1,3]. Adding more printing axes and enabling rotational motion of the substrate stage can expand Optomec's 3D printing capability.

1.3 Working Principles of Inkjet Printing (Dimatix)

Inkjet printing involves an ink cartridge and a printhead with controlled XY motion. Selective jetting occurs via a piezoelectric or thermal actuator. A Dimatix piezoelectric inkjet printer is available in the SNF. Thermally-actuated inkjet printheads are typically low-cost, but limited in utility as the ink must be able to withstand high temperatures. Piezoelectric printheads are much more versatile as they rely on mechanical compression of the ink, though they are more expensive.
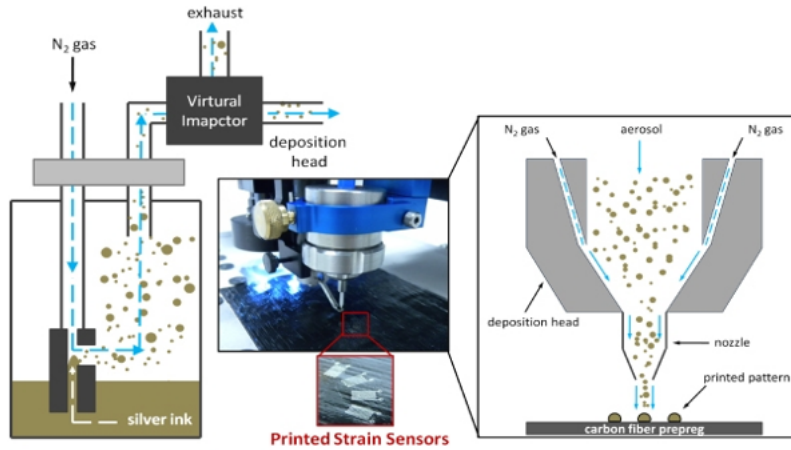
Figure 1: Schematic depiction of the aerosol jet printing process. Ink is aerosolized and enters the printhead, where it is focused by the sheath gas to produce a narrow aerosol jet [7].

Figure 2 and Figure 3 depict thermal and piezoelectric [4] printheads during operation.

Controlling jetting behavior is critical to achieving high quality prints. Desirable jetting characteristics include spherical drops, no satellite drops, and uniform jetting speed across all jets. Jetting behavior can be modified by tuning the ink rheology, such as adding co-solvents or changing ink concentration. Both printing mechanisms also offer a certain degree of control over jetting behavior. For thermal inkjet, the temperature profile can be adjusted, which affects gas bubble formation and jetting behavior by extension. However, gas bubbles cannot be formed in inks with too high of a surface tension, so ink tuning is often necessary. For piezoelectric inkjet, jetting behavior can be modified by adjusting the waveform of the applied voltage. Piezoelectric inkjet typically offers much higher control over the jetting behavior than thermal inkjet, so less ink tuning is needed.

Besides requirements on surface tension and ink viscosity, inkjet printing is also limited to printing micron-scale materials. This is due to the fact that inkjet printheads generally have a predefined nozzle resolution in the range of tens of microns. Printing materials larger than the nozzle size will lead to clogging and failure of the printhead. For the Dimatix DMP-2800 series, the nozzle diameter is 20 μm, and each drop is nominally 10 pL.
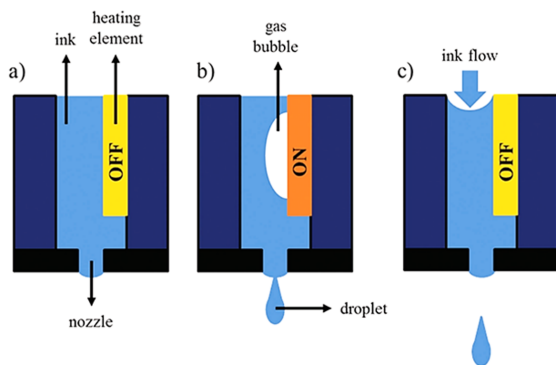

Figure 2: Schematic of thermal inkjet during operation. a-c) shows the printhead in its idle, printing, and refilling states respectively.
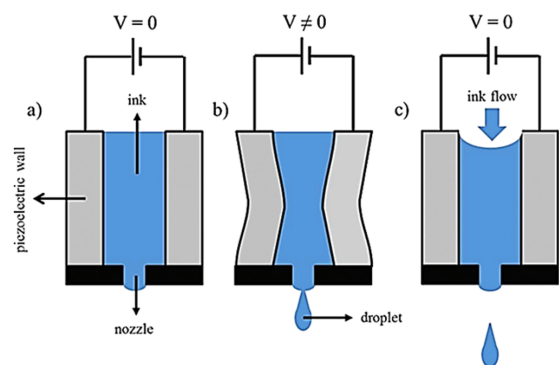

Figure 3: Schematic of piezoelectric printhead during operation. a-c) shows the printhead in its idle, printing, and refilling states respectively.

1.4 Comparison of Inkjet and Aerosol Jet Printing

In principle, aerosol jet printing has far more versatility than inkjet printing. Aerosol jet printing is compatible with inks up to 5000 cP, and due to the wide selection of nozzle types, clogging is typically not an issue. Additionally, aerosol jet printing can be performed at an angle, and also on 3D surfaces. Furthermore, by changing the nozzle size and shape, aerosol jet printing can print over large areas as well as small areas. Inkjet printing, however, is limited to low viscosity inks, printing small features, and can only print on flat surfaces. The maximal resolution is around 10 µm for both techniques.

Due to the mechanical design of the systems, however, inkjet printing is well-suited for lab-scale prototyping, while aerosol jet printing is far more involved. To switch materials on an inkjet printer, one only needs to swap or clean the printhead and printing cartridge, while to switch materials on the aerosol jet printer, everything from the atomizing assembly to the nozzle must be switched or cleaned. As we will describe later, this cleaning process is fairly involved. In facilities such as the SNF, aerosol jet printing is therefore more suitable for single-material printing, while the inkjet printer is more suitable for multi-material prototyping.

2 Standard Operating Procedure for Pneumatic Atomizer Mode

This section covers topics related to Optomec standard operating procedure under the pneumatic atomizer mode with a fine feature printhead, including generation of Optomec-compatible print pattern files, Optomec system assembly and initialization, troubleshooting of common hardware and software issues, disassembly, and the minimum cleaning protocol.

2.1 Generating Print Pattern Files Using VMTools (Overview) [2]

SNF offers computers with VMTools-installed AutoCAD software, and this section intends to provide an overview on how to generate Optomec-compatible print pattern files. A user may refer to VMTools Manual and AutoCAD Manual for details on specific software functions.

1. Create print patterns using AutoCAD and save it as a drawing file in .DXF which is the only format supported by VMTools. Since Optomec interprets the CAD data in millimeters, it is recommended to set the AutoCAD dimensions in millimeters when designing the print patterns.

2. Click on the VMTools icon 🔲 on the AutoCAD Draw menu to start VMTools. This will open a VMTools utility window with several tabs, including Import, Generic Fill, Images, Export, Tool Config, etc.

3. Make sure to save any changes to the print pattern .DXF file before importing to VMTools. In the "Import" tab of VMTools utility window, click the "Import Dxf" button to import the currently open .DXF file in AutoCAD.

4. In the "Generic Fill" tab, the user can specify the filling method (serpentine, perimeter, circle) for various closed shapes. Before proceeding to specifying the filling method, first adjust the items under "General Setting" box which is also presented in the Generic Fill tab. The item "Trace Width" specifies how wide the line of deposited ink will be. The default value is 0.1 mm but it can be adjusted to any value in the range of $0.005 \sim 5$ mm. The input trace width value is what VMTools and Optomec believe the printed line width will be, and therefore it is not necessarily representative of the actual printed line width. Therefore, this parameter should be adjusted after measurement of printed lines for a specific ink, substrate, and set of printing conditions. If the ink, substrate or printing conditions are changed, the user must print and measure again to determine the actual input trace width.

5. Click the filling method button of choice, and then select the shape entities to be filled in AutoCAD.
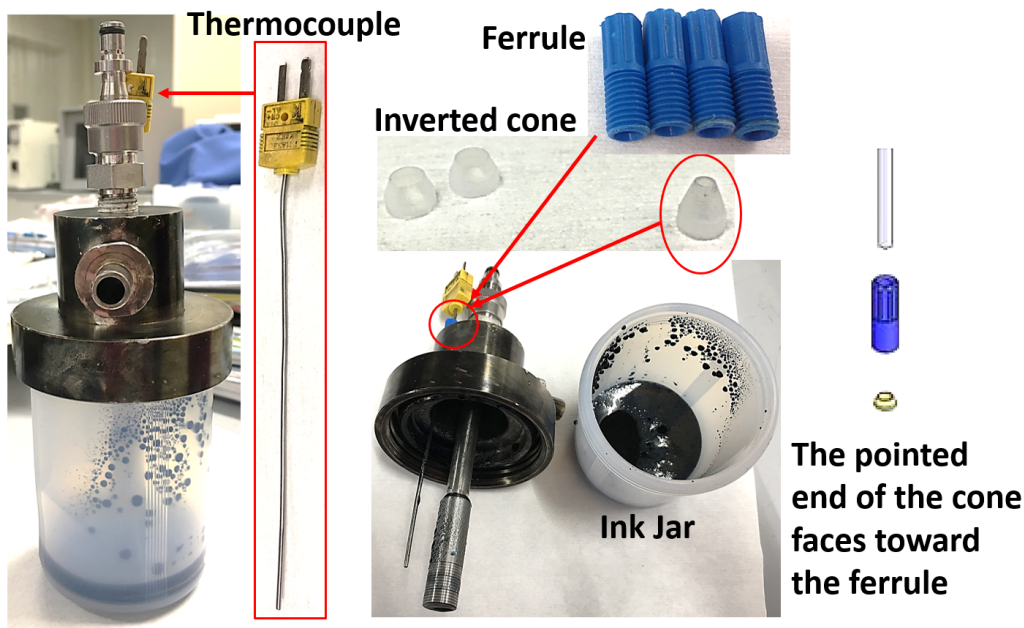
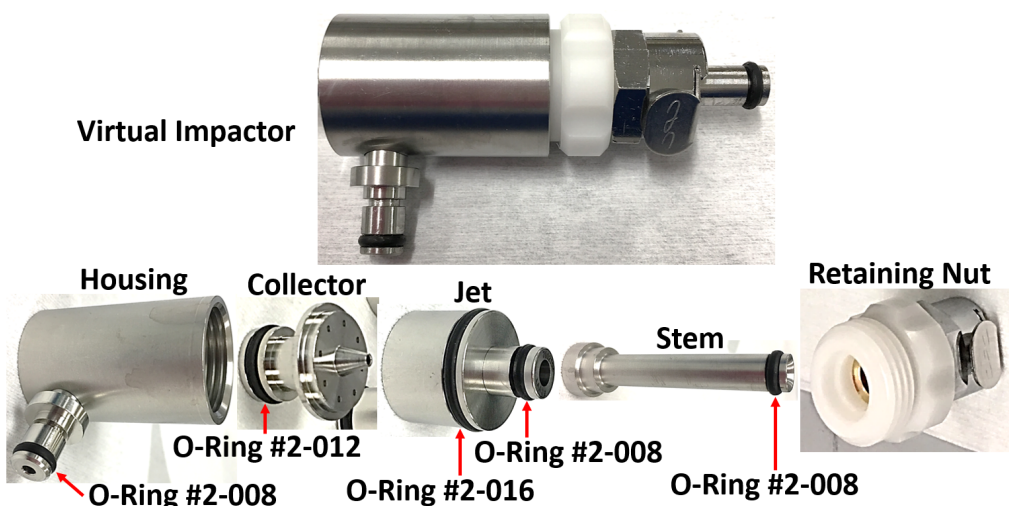Figure 4: Pneumatic atomizer assembly with PEDOT:PSS in the ink jar.

Figure 5: The O-ring sizes and exploded view of the virtual impactor.

6. In the "Export" tab, the user may define the shutter delay time (msec). By clicking "Export" button, the user can create the toolpath file in .prg format which contains motion and shutter commands for Optomec to print the designed patterns.

7. The exported .prg file can be opened with notepad. We observed that the exported .prg file presents a syntax error - there is a line comment that is missing its corresponding comment character maker ('!'). To solve this issue, add "!" in front of the line that starts with "REAL CRangle;..." to comment the line and fix the syntax error. Other command lines can also be suppressed by adding "!" in front.

8. If the user is familiar with programming, such as MATLAB, the user may create a custom toolpath generation/optimization code, as there can be unexpected errors with VMTools (e.g. random creation of extra command lines).

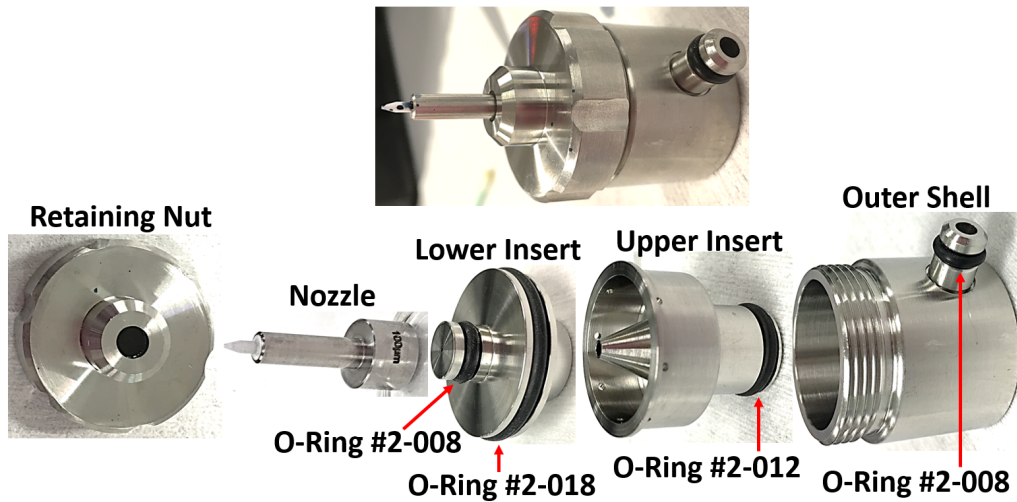Figure 6: The O-ring sizes and exploded view of the printhead for the fine feature nozzle.
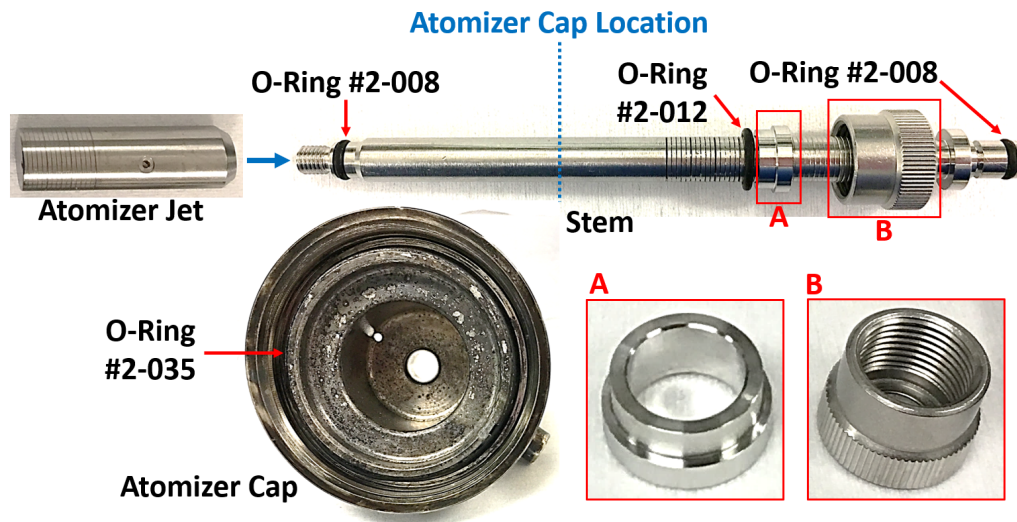

Figure 7: The O-ring sizes, atomizer cap components, and exploded view of the pneumatic atomizer stem.
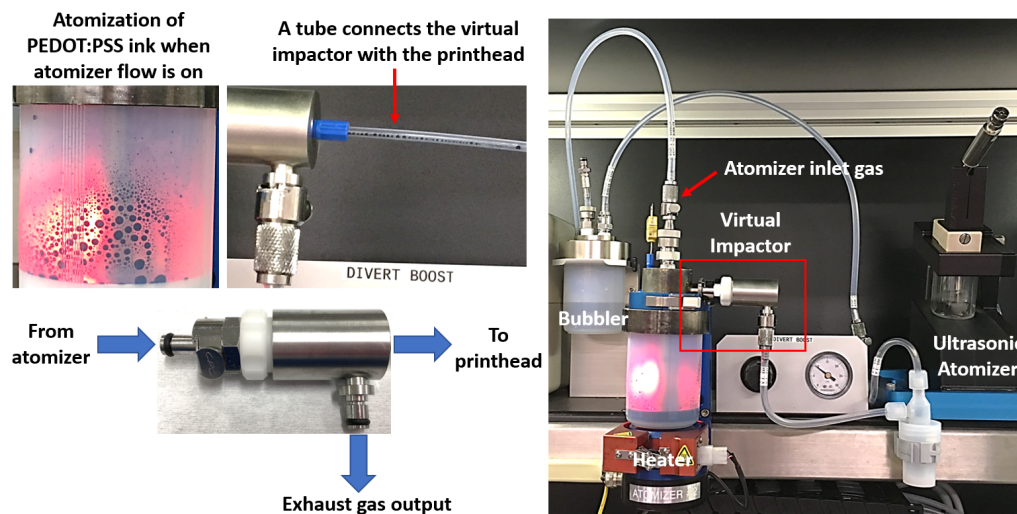

Figure 8: Pneumatic aerosol virtual impactor connections. The pneumatic atomizer bubbler and the ultrasonic atomizer (not connected in the Optomec setup in the picture) are also shown for reference.
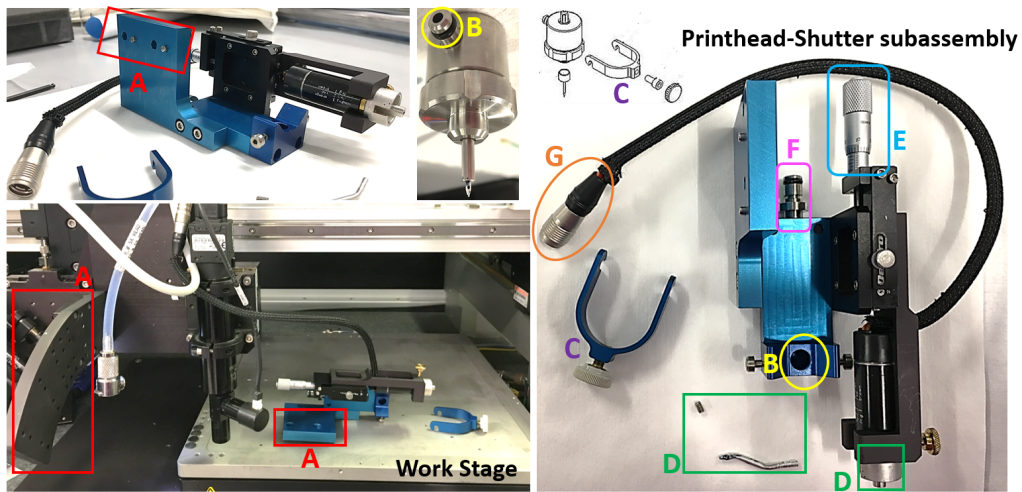
Figure 9: Fine feature printhead connections. The printhead-shutter subassembly is done by (1) making the B-B connection which is then fixed by part C (mounting the printhead) and (2) making the D-D connection (mounting the shutter using a tiny hex screw). The printhead-shutter subassembly is then physically mounted to the Optomec system by making the A-A connection which can be done at various angles (i.e. the direction of the nozzle tip with respect to the work stage can be perpendicular or at some angle). Connect part G of the printhead-shutter subassembly to the Optomec system to make electrical connection. The knob E allows the z-axis height adjustment of the shutter with respect to the substrate. While the printhead is connected to the virtual impactor by cut tubing as described above, a separate tube from the Optomec setup connects to the inlet F of the printhead-shutter subassembly as the sheath gas input.

Figure 10: Nozzle tip and shutter height (z-axis) calibration with respect to the substrate using 1mm-thick microscope glass slides and the camera setup around the work stage.

2.2 Assembly (Pneumatic Atomizer Mode, Fine Feature Printhead)

The Optomec setup begins with assembling various parts. The exploded views of the three main subassemblies illustrate how individual parts fit together with the O-rings: (1) pneumatic atomizer (Figure 4), (2) virtual impactor (Figure 5), and (3) printhead (Figure 6).
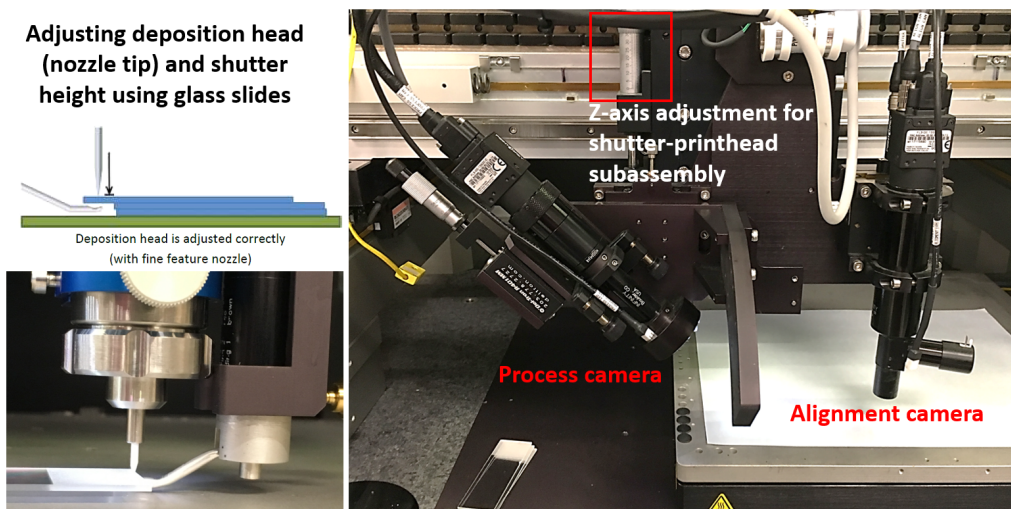
Thorough visual inspection of the O-rings is imperative; any defective O-rings (e.g. cracks, deep scratches, improper fit) must be replaced with new, good O-rings as the Optomec part assembly proceeds. Apply sufficient lubricant (Apiezon L Grease) to the O-rings using cotton swabs.

Using the tube cutter, cut a section of 1/8-in PFA tubing (Swagelok, P/N: PFA-T2-030-100, OD 0.125'', Wall 0.030'') into a length sufficient for connecting the printhead with the virtual impactor. Make sure that there is extra length to accommodate the movement of the printhead, but not enough to result in slacking or coiling of the tube, which can cause agglomeration of the ink aerosols inside the tube. It is important that the cut end of the tube is flat and pristine (no angled edge, no cut debris) to avoid gas leakage. Insert first the inverted cone and then the ferrule (Omni-lock inverted cone fitting nut) to the tube such that the pointed end of the cone points toward the ferrule (Figure 4).

Assemble the atomizer cap (Figure 7), then pour the ink into the jar (30mL of ink is the recommended volume, any less volume may adversely affect the aerosolization process), and ensure the tight closure of the jar with the cap. During our printing attempts, we experienced severe difficulties to successfully close and open the jar. We learned that the atomizer cap was an older model than the ink jar, and there were micrometer-scale dimensional differences between the two parts. This lead to an improper fitting between the cap and the jar, which would make closing and opening the jar a very arduous task and also a common source of leaks. As of December 2017, the ExFab has purchased a new cap to solve these difficulties.

Adjust the position of the atomizer stem by loosening the Swagelock fitting around the stem such that the bottom of the atomizer jet is close to the bottom of the jar without touching it [1]. The exact position is not critical [1]. However, it is important to ensure that the atomizer jet hole is approximately 15 mm (= 5/8 in) above the surface of the ink because any deeper immersion of the atomizer jet hole into the ink may result in the interference between the ink surface and the atomizer jet operation [1].

Once the pneumatic atomizer, virtual impactor, and printhead are assembled, install them in the Optomec and connect the parts as illustrated in Figure 8 and Figure 9.

As shown in Figure 10, Optomec offers the alignment camera, which allows the user to view the substrate, as well as the process camera, which allows the user to observe the aerosol stream during printing. They are set up around the work stage on which the substrate is placed. Camera focusing and adjustments can be performed after enabling the tool in Badger, as is described in Section 2.3.

Optomec recommends to set the height of the nozzle tip relative to the substrate surface and shutter by using the 1 mm-thick microscope glass slides as demonstrated in Figure 10. There are two knobs that allow the adjustment of the z-axis of the nozzle tip and the shutter: the z-axis knob in Figure 10 moves the entire printhead-shutter assembly and the z-axis knob (labeled E) in Figure 9 moves the shutter only. This is Optomec's recommended settings, but the distance between the nozzle tip and substrate can be modified as a printing variable. The range of allowed distances is nominally $3 \sim 5$mm.

2.3 Initialization

As the hourly fee of the Optomec is rather expensive ($35/hour for university users), we suggest that the user perform the following tasks prior to enabling the machine in Badger.

- Design the print patterns using AutoCAD and VMTools as described in Section 2.1. While print patterns can be edited manually at the printer for simple paths, we recommend using VMTools for most cases.

- Ensure that there is more than 1.5 bottles of isopropyl alcohol and 1/4 of a bottle of Branson cleaning solution underneath the hood in room 155. Depending on the printing specifics, more or less cleaning may be required — this is a general guideline for how much cleaning supplies are needed.

- Assemble and install the nozzle and atomizing assembly, as described in Section 2.2.

- Calibrate the height of the nozzle tip and shutter with respect to the printing substrate, as described in Section 2.2 (Figure 10).

- Ensure that the settings on the machine (bottom right cabinet under the Optomec) are as desired. For fine-feature nozzle printing, the switch must be set to "0.5X head". For wide nozzle printing, the switch must be set to "wide nozzle".

At this point the machine can be enabled in SNF Badger.

1. Enable the machine (nSIL>patterning>Optomec-printer).

2. If the computer is locked, the password is **Optomec1**.

3. On the computer desktop, double click the KEWA gadget shortcut to open the KEWA gadget interface. Press "start all" to initialize all KEWA applications. For correct monitoring of the tool status and operation, we suggest arranging the following KEWA applications in the PC monitor in such a way they are constantly visible:

   (a) KEWA Process Control
   (b) KEWA Motion Manager
   (c) KEWA Vision Manager

4. In KEWA Process Control, select the device controls tab for the atomizer type you will be using. In case of this SOP, select the 'Pneumatic' tab.

Before successful atomization, the processes described in the following sections must be completed.

2.3.1 Focus the process and alignment cameras

Once the tool has been enabled, it is possible to focus the process and alignment cameras. To focus both cameras, on the operator panel, turn on the alignment and process camera lights. Go to KEWA Vision Manager application and select the tab corresponding to the desired camera to focus. A live video image of the selected camera will be shown in the application. Both cameras have a micromanipulator knob to adjust the position of the camera with respect to the substrate. Use the camera knob to gently adjust the focus until you see a better resolved image in the KEWA Vision Manager application.

2.3.2 Check for gas leaks and a clogged nozzle tip

For optimum performance, all system leaks or clogging must be eliminated before atomization of the ink. This step is of utmost importance — the user should not proceed with atomization if there are any leaks or clogging present in the system, as this could damage components of the tool and/or lead to chemical contamination of tool parts and the user's samples.

Monitoring of the sheath gas flow will indicate if there are any leaks or clogging present in the system. The recommended sheath gas flow for optimum performance of the Optomec will depend on the nozzle tip size utilized (Table 1). Under the pneumatic tab in KEWA Process Control, enter the correct sheath flow value in the Sheath_MFC box and press enter. The pressure reading

Nozzle tip size (µm)	Sheath gas flow (sccm)	Expected pressure (psi)
100	50	1.69 to 2.06
150	150	2.46 to 3.00
200	150	0.88 to 1.07
250	150	0.67 to 0.82
300	200	0.39 to 0.47

Table 1: To determine if there are any leaks or clogging in the system, at sea altitude (i.e. Stanford University), the Optomec User Manual recommends applying the following sheath gas flows (middle column) according to the nozzle tip installed (left column). The measured pressure in the system should correspond to a value within the expected pressure range (right column).

will be displayed in the graph in the KEWA Process Control application, as well as next to the Sheath_MFC box. Wait until the pressure stabilizes.

A reading below the values displayed in Table 1 indicates that there is a leak in the system. A reading above these values indicates that there is a clog in the system. For troubleshooting of leaks and clogging, go to Sections 2.4.2 and Section 2.4.3, respectively.

2.4 Troubleshooting

This section describes common hardware and software issues that we experienced while using the Optomec, as well as strategies for overcoming them.

2.4.1 Optomec Operating Software (KEWA) Issues

Common software issues we experienced include:

- Software not starting fully or freezing during operation.

- Software loading print pattern file but not executing printing procedure.

- Software not measuring applied sheath, atomizer, or exhaust flow values.

Software complications are generally fixed by re-initializing the software or performing a hard reboot of the system. We propose the following procedure:

1. Software permitting, set to zero all the gas flows in the following order: **atomizer** $\rightarrow$ wait 10s $\rightarrow$ **exhaust** $\rightarrow$ wait 60s $\rightarrow$ **sheath**.

2. Close all applications of KEWA software and re-initialize all KEWA software instances.

3. If (2) fails, then turn off the PC, disable the tool using Badger, re-enable the tool, and re-start the KEWA gadget interface.

After re-initialization, turn on the sheath flow, exhaust flow and atomizer flow following the procedure stated in Section 2.5. Run again the homing routine and perform a test print to ensure that the printer is working properly.

2.4.2 Troubleshooting a Leak During Initialization

A reduced sheath flow during tool startup indicates that there is one or multiple components leaking in the system. Leaks can come from various elements, and thorough inspection of assembled components in the tool (as described in Section 2.2) and connecting elements is imperative.

Leaks most commonly occur at the locations where the tubes connect to the hardware and at the O-rings in the print head, virtual impactor, and atomizer. We have also observed on occasion

that the leaks originate from the PFA tubing, despite using freshly cut PFA tubing. While troubleshooting leaks, the user should ensure that the sheath gas flow is set to zero before carefully inspecting one item at a time and making any repairs the user deems necessary.

Given that some elements of the tool are more prone to produce leaks in the system, we suggest inspection of the tool items in the following order. After performing any repairs in the system, the user should evaluate if the repair eliminated the leak source by applying the recommended sheath gas flow pressure and reading the pressure in the system. Only after the expected pressure is in the range displayed in Table 1 the user can confirm that all leak sources have been eliminated.

1. *Connection of the PFA tube to the printhead:* Remove the tube from the printhead and inspect the quality of the cut at the tube's end. If the cut is not flat, use the tube cutter to refine the cut, since any misalignment can produce an improper connection of the tube with the printhead. Attach the tube back to the printhead by pushing the tube all the way into the printhead and screwing the ferrule. To ensure proper connection, attempt to pull the tube from the printhead. If the tube wiggles or moves inside the printhead, the tube was not correctly connected.

2. *Connection of the PFA tube to the virtual impactor:* Follow instructions stated in (1) for the connection between the PFA tube and the virtual impactor.

3. *Printhead (1st), virtual impactor (2nd), and atomizer (3rd):* For each of the components (1-3), remove it from the Optomec and disassemble it following the inverse procedure described in Section 2.2. Inspect all O-rings for any imperfections and ensure that the installed O-rings are the right size (see printhead: Figure 6, virtual impactor: Figure 5, atomizer: Figure 4). In addition, all installed O-rings must be properly greased.

   Visually inspect the part every time a sub-component is re-assembled (see Section 2.2). The user should take special attention to ensure all subcomponents are aligned and that during the re-assembly process no O-rings were damaged.

   For the re-assembly of the atomizer, ensure that the cap is completely closed. As mentioned in Section 2.2, the improper fitting between the cap and the jar makes closing and opening the jar a very arduous task that requires quite a lot of physical effort.

4. *Replacing the PFA tubing:* Cut a new length of tubing and replace the old tubing. Ensure the cuts in both ends of the tube are flat and that the connections of the tube with the printhead and virtual impactor are correct (see 1 and 2).

5. *Replacing the nozzle:* In a few situations we found that the nozzle had been damaged and was the source of leakage. If troubleshooting all the other components of the Optomec does not fix leaking, then replace the nozzle. Please inform the staff about your issues with the nozzle and label the faulty nozzle.

2.4.3 Clogged Nozzle Tip

A buildup of pressure indicates that one or more of the microfluidic components of the Optomec are clogged. Most commonly, clogging will occur at the nozzle tip, but it can also occur at the printhead, virtual impactor, or atomizer.

To determine if a component is clogged, remove the part from the Optomec and disassemble following the inverse procedure described in Section 2.2. Visually inspect the subcomponents of the part in a well-lit area. If the subcomponents present ink material agglomerates, they must be cleaned prior to use. For cleaning clogged components, follow the instructions in Section 2.7.
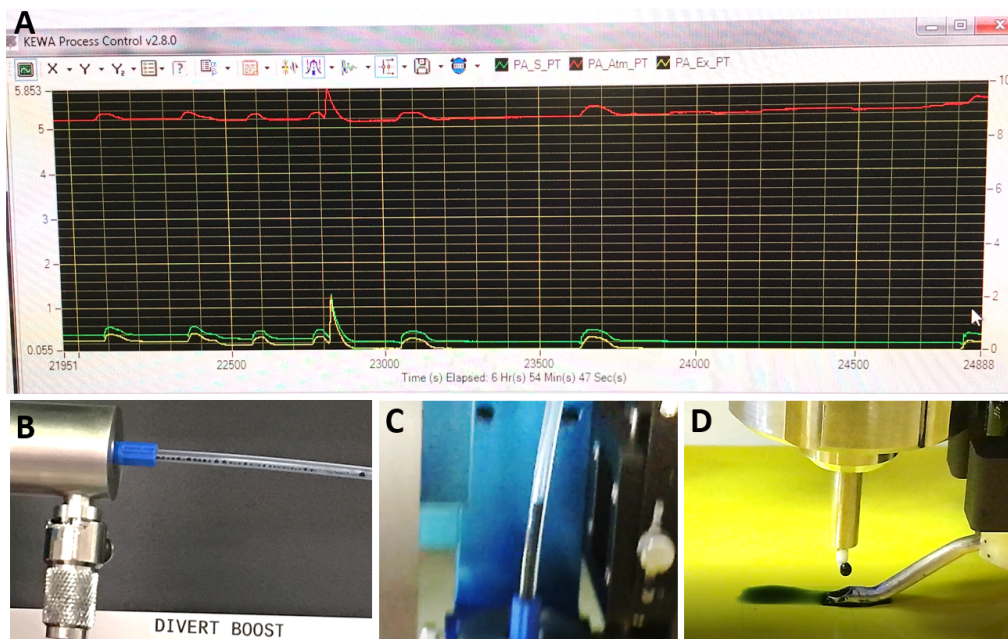
Figure 11: (A) The screenshot of KEWA Process Control window that shows spikes in the gas flows, (B) beads of PEDOT:PSS ink in the tubing that connects the virtual impactor and printhead, (C) the large droplet of PEDOT:PSS ink flowing down the tubing and entering the printhead, (D) the large droplet of PEDOT:PSS ink splashing onto the substrate.
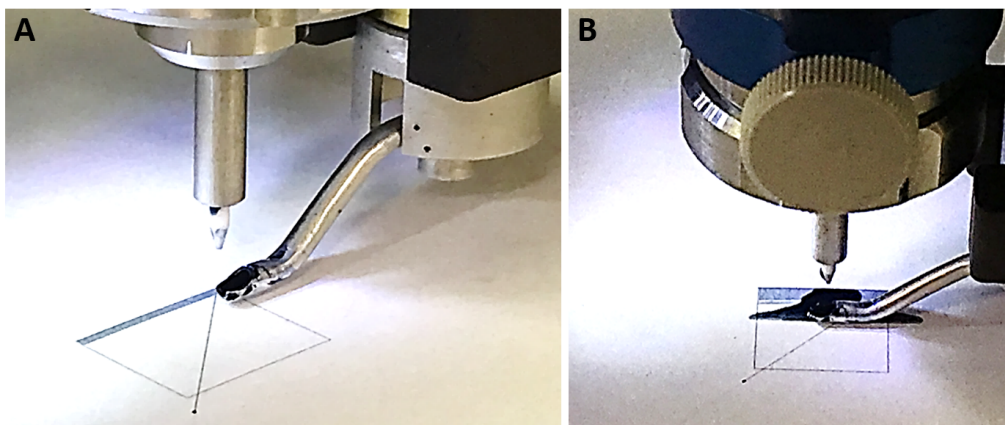

Figure 12: (a) PEDOT:PSS printing on paper with good jetting, and (b) PEDOT:PSS printing on paper with bad and excessive jetting when pressure spikes on the three gas flows were observed.

Nozzle size (µm)	Sheath flow (sccm)	Atomizer flow (sccm)	Exhaust flow (sccm)
150	55 - 60	~ 600	~ 570
200	80 - 90	~ 600	~ 570
250	100 - 110	~ 700	$\sim 650\text{-}660$
300	115 - 130	~ 800	$\sim 740\text{-}750$

Table 2: Pneumatic fine feature deposition starting parameters.

2.4.4 Excessive and Intermittent Jetting of Ink

Excessive and intermittent jetting of ink (Figure 12) may occur during printing and may worsen over the course of a printing run. This was a common problem we faced when attempting to print PEDOT:PSS. We observed formation of excessive ink blobs around the nozzle tip which had to be cleaned using a cotton swab between prints to prevent the blob from falling onto the substrate. Additionally, we observed satellite drops which caused our printed patterns to have poor spatial definition.

We also observed beads of ink droplets in the tubing that connects the virtual impactor and the printhead (Figure 11(B)), which eventually agglomerated together to form a large ink droplet. This droplet then fell down the tubing, went through the printhead, and finally splashed onto the substrate (Figure 11(C)-(D)). Such events can be correlated with spikes in the exhaust/sheath/atomizer flow (Figure 11(A)), although the spikes were not periodic. We have yet to pinpoint the cause of this issue.

Possible solutions include: (1) adjusting ink rheology such as diluting the ink to reduce its viscosity, (2) cleaning the virtual impactor and printhead, (3) replacing tubing, and (4) adjusting the "push" which is defined as the difference between the exhaust flow and atomizer flow. The user should take note that the push does not respond immediately, and that there can be residual pressure in the atomizer that exacerbates this delay.

2.5 Start Atomizing

Once it is confirmed that there is no leaks or clogging, start atomizing the ink. To avoid clogging, always turn on the gas flows in the following order [1]:

1. Sheath flow

2. Atomizer flow

3. Exhaust flow

The amount of sheath, atomizer, and exhaust flow required for optimal printing depends on the ink. If these values are not available for the ink to be used, follow the recommended starting parameters in the Optomec manual, which we include in Table 2 for the pneumatic fine feature nozzles.

If atomization occurs, the user should see droplets of ink splattering on the wall of the jar, as illustrated in Figure 4(left). If no atomization is occurring, slowly increase the atomizer and exhaust gas flow in the proper proportion [1]. Once you see good atomization of the ink, take note of the applied atomizer and exhaust flows. Vary the sheath flow to optimize print quality.

2.6 Disassembly

When printing is finished, Optomec parts must be disassembled properly as described below:

1. Turn off the three gas flows in the following order: **atomizer** $\rightarrow$ wait 10s $\rightarrow$ **exhaust** $\rightarrow$ wait 60s $\rightarrow$ **sheath**

2. Remove assembled parts from the Optomec: atomizer, virtual impactor, and printhead.

Figure 13: Optomec parts that require thorough cleaning. Critical parts: 4, 6, 7, 8, 10, 11, 12. Critical parts that cannot be cleaned with Branson: 18.

Cleaning Solution	Non-critical parts	Critical Parts	Critical Parts (no Branson)
1) Water	10min x 2 times	10min x 2 times	20min x 3 times (or 10min x 5 times)*
2) Branson	10min x 2 times	10min x 3 times	–
3) Isopropyl alcohol	10min x 2 times	10min x 3 times	10min x 3 times
4) Rinsing with isopropyl alcohol and blow dry with N_2 gun			

Table 3: Minimum cleaning procedure of Optomec parts.* The choice of suggested cleaning recipe depends on the part condition and ink.

3. Disassemble the printhead shutter subassembly

4. Dispose of tubing

5. Clean up the work stage

2.7 Cleaning Procedure

Thorough cleaning of Optomec parts after printing is the most crucial procedure of Optomec operation:

1. Disassemble each part (atomizer, virtual impactor, and printhead) into its multiple subcomponents, following the inverse procedure described in Section 2.2. Figure 13 depicts all the subcomponents.

2. Transfer disassembled parts to room 155. Place individual subcomponents in a beaker. As a general rule, each subcomponent should be placed in an individual beaker - exceptions are some non-critical parts, such as ferrules and cones, if there is not enough space available in the sonicator to place all subcomponents in separate beakers.

3. Fill beakers with cleaning solution and sonicate for 10 to 15 minutes (Figure 14). Ice cubes should be added to the sonication water bath to prevent overheating.

4. Dispose of cleaning solution and refill beakers with new cleaning solution. Repeat sonication for 10 to 15 minutes. Repeat this step as many times as necessary - the specific cleaning solution as well as the number of times this step must be repeated will depend on the user's ink.

   Generally, each sonication step per cleaning solution must be repeated several times in newly filled cleaning solution for at least 10 minutes before proceeding to the next sonication step. The order of cleaning solutions is: water, Branson, and isopropyl alcohol. Branson is a specially formulated cleaning solution, and it is effective in removing rust and metal oxides. The Branson solution is prepared by mixing 1 part Branson concentrate to 11 parts water.

The minimum cleaning procedure is provided in Table 3, which is suitable for PEDOT:PSS ink, but more thorough cleaning may be required for other ink types. Optomec parts that directly contact the ink are considered to be critical parts and must go through more extensive cleaning
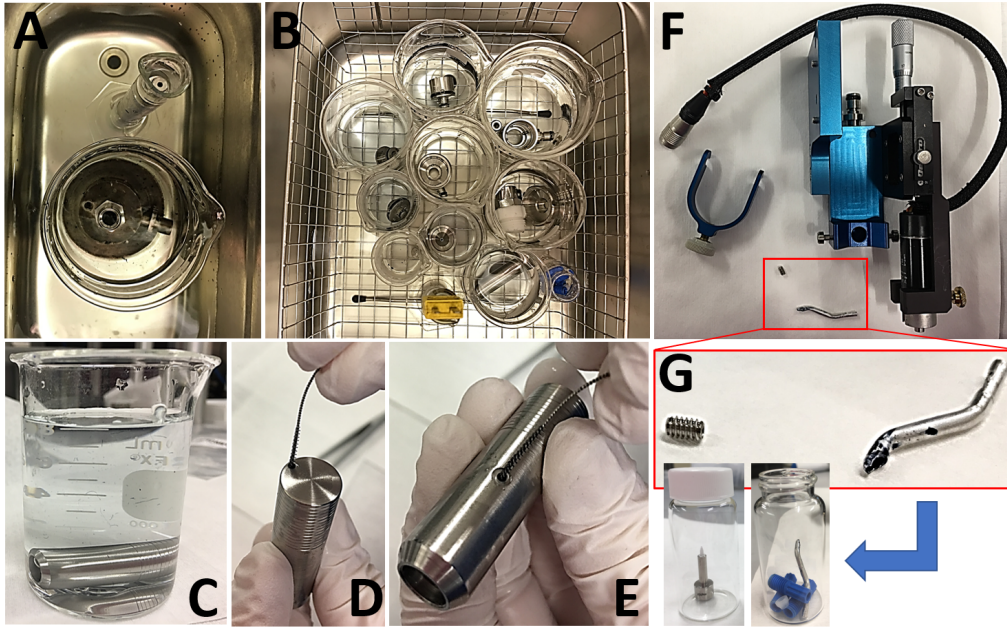
Figure 14: Optomec parts submerged in water and sonicated (A and B), a critical Optomec part submerged in water taken out from sonication (C) for thorough cleaning with a brush (D and E), Optomec shutter disassembly (F), zoom-in of shutter components and other small parts (nozzle, cones and ferrules) to be cleaned (G).

than non-critical parts. The cap of the ink jar is a critical part that cannot be cleaned with Branson. Some parts have small openings which must be cleaned with a miniature brush between some sonication steps (Figure 14(D and E)). Furthermore, it is important to make careful visual inspections of all parts at the end to ensure their cleanliness. The cleaning procedure must be repeated as many times as necessary for a part until it is completely clean. Once the part is completely clean, it should be rinsed with new isopropyl alcohol and blow-dried with N_2.

3 Aerosol Jet Printing Of PEDOT:PSS

The user must perform the optimization process for every ink and substrate combination, which can be arduous and time consuming given the wide variety of parameters to be considered. To gain insight into the optimization process, we printed and characterized lines of PEDOT:PSS ink using various printing parameters. The toolpaths for the lines were generated by AutoCAD/VMTools, with the following dimensional specifications: linewidth 10 µm, line spacing 1 mm, and line length 1 cm.

3.1 Optimization Process

There are a wide variety of parameters that must be considered when optimizing aerosol jet printing, including ink, printing, and substrate parameters. Relevant ink parameters include the solids content, solvent and rheological behavior, among others. In general, inks with low surface tension will have better wetting, and vice versa. Additionally, the rheology and composition of the ink will affect the aerosolization process. In our situation, we decided to use PEDOT:PSS PH1000 purchased from Heraeus Clevios™, which uses water as the solvent. To prevent clogging, we sonicated and filtered the ink with a 0.45 µm filter before printing.

Printing parameters can be divided into two main categories: software-controlled parameters and hardware parameters. Software-controlled parameters include sheath flow, exhaust flow, atomizer flow, and gantry speed. Sheath flows are set according to the user manual for the selected
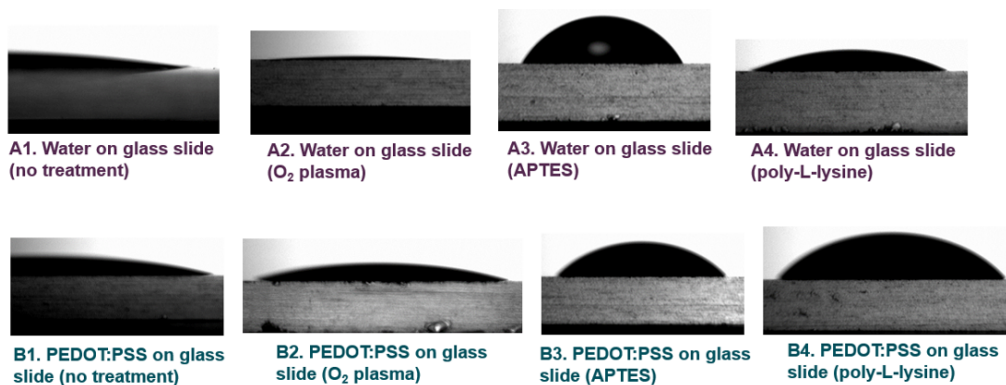
Figure 15: Contact angle measurements of water and PEDOT:PSS on a glass substrate with and without surface treatments to qualitatively analyze the effects of various surface treatments on the wetting behavior of a given ink. The contact angle measurement was done on water as a control.

nozzle size; higher sheath flows are known to focus the aerosol jet more. The exhaust flow determines the particle size distribution and the atomizer flow defines the density of aerosol that is generated and transferred to the printhead.

Hardware parameters involve physically adjusting the machine itself, and include nozzle size and printing distance. Available nozzle sizes include 100, 150, and 300 µm. A 3 mm wide-nozzle is also available for printing large areas, though it uses a different setup, and will not be covered in this guide. Printing distance refers to the distance between the nozzle tip and the substrate surface. For optimal resolution, the Optomec user manual suggests a printing distance ∼3 mm as described in Section 2.1. This represents the minimum printing distance, as the bottom of the shutter is about 3 mm from the tip of the nozzle. A larger printing distance can be used, resulting in greater spatial spread in the printed pattern. For increased consistency between prints, the same printing distance should be used each time.

The final factor that can be adjusted is the substrate, specifically its temperature and surface termination. Surface treatments are commonly used in the field of printing to promote uniform wetting of deposited materials, or in some cases, to prevent wetting on certain regions. Examples include oxygen plasma treatment, functionalization with poly-l-lysine and functionalization with (3-Aminopropyl)triethoxysilane (APTES). The effect of surface treatments on wetting can be determined from contact angle measurements of the ink as shown in Figure 15 and Table 4. Temperature can also be adjusted to prevent ink reflow on the surface, or to control the ink drying process.

Optimization of aerosol jet printing is a cyclic process involving optimization of ink, printing and substrate parameters. A common approach is to first formulate different inks with the goal of finding an ink that aerosolizes well. After this ink is identified, print and substrate parameters are concurrently adjusted to optimize characteristics such as resolution (linewidth), uniformity, and the performance of printed films.

In our case, we mixed commercial PEDOT:PSS solution with a 5% volume of ethylene glycol, which has been reported in literature to improve its conductivity [5]. This was found to be printable, so we did not make further adjustments to our ink, though we did try diluting it for better uniformity. The printing parameters we adjusted were the three different gas flow rates, gantry speed and nozzle size (100 or 300 µm). A fixed printing distance of 3 mm was used. Printing was attempted on quartz with various surface treatments, paper, and Kapton foil.

3.2 Determining Print Quality

Print quality can be defined as a combination of print uniformity and resolution. These factors will in turn affect the performance of printed films, including mechanical properties such as

Material	No treatment	O$_2$ plasma	APTES	Poly-L-lysine
Water (control)	20.46° ± 0.37°	7.75° ± 0.35°	61.65° ± 1.45°	25.4° ± 0.4°
PEDOT:PSS	12.57° ± 0.52°	22.7° ± 0.7°	49.8° ± 0.6°	45.15° ± 0.5°

Table 4: Contact angle measurements of water and PEDOT:PSS on a glass substrate with and without surface treatments. Water was used as a control.
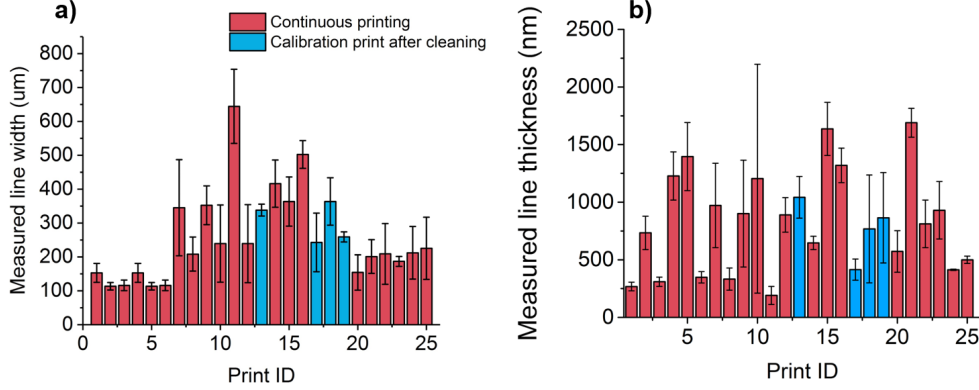

Figure 16: Measured linewidths (a) and line thicknesses (b) for multiple PEDOT:PSS lines printed while varying gantry speed (0.5 mm/s and 1 mm/s), substrate (Kapton, quartz and paper), sheath flow (130 sccm, 115 sccm and 100 sccm), and number of passes (2 or 6), concentration (as-received, 3:1 and 1:1 dilutions), and push (25 and 50 sccm).

adhesion, strength and flexibility, as well as electrical performance such as conductivity, isotropy, etc. Oftentimes there may be a trade-off between one or more of these properties, so the working definition of print quality is ultimately left to the discretion of the individual user.

In our case, we wished to maximize conductivity and resolution, without regard for other properties. Resolution was determined by measuring average linewidths using optical microscopy. Conductivity was determined by performing 4-point probe measurements to measure sheet resistance, and calculating conductivity from measured sheet resistance values with thickness values measured by profilometry.

3.3 Characterization of Aerosol Jet Printed PEDOT:PSS

As described previously, we varied a number of different ink, substrate and printing parameters. However, we were unable to correlate print resolution with any of these factors. ANOVA analysis on the data shown in Figure 16a revealed no significant factors (defined as p < 0.05). In particular, there is no dependence on substrate. This indicates that the aerosol jet printing of PEDOT:PSS is a dry process, and that there is no ink reflow on the surface, unlike inkjet printing. This effect can be in part attributed to the low boiling point of water, and we believe that higher boiling point solvents may be more sensitive to surface treatments.

We were also unable to correlate any of our parameters to measured line thicknesses, shown in Figure 16b. There are several possible reasons for this. First, the variable space we explored was limited in scope. For example, most of the gas flow rates we used were within the range suggested by the Optomec. Similarly, concentration was only explored at three values, and gantry speeds were kept within an order of magnitude of the suggested setting. Exploring the effects of these parameters at more extreme values may provide greater insight into the importance of individual parameters.

Moreover, the quality of our prints could have been adversely affected by clogging, smearing out the effects of individual parameters. The effect of clogging on print quality is illustrated in Figure 17. As shown in Figure 12, occasionally an agglomerate of aerosol particles would enter the
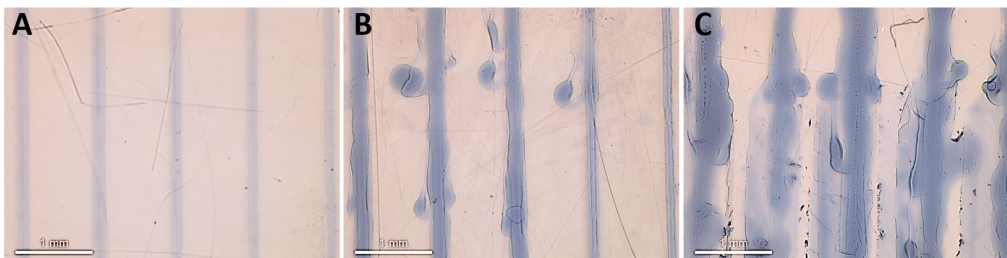
Figure 17: Optical microscopy images for printed PEDOT:PSS lines with different print qualities: (a) good resolution and uniformity (Print ID 1), (b) medium resolution and medium uniformity (Print ID 18), (c) poor resolution and poor uniformity (Print ID 8).

printhead, resulting in a large and uncontrolled drop of PEDOT:PSS depositing on our sample. To mitigate the impact of this issue, we cleaned the printhead before subsequent prints. Cleaning is indicated by the blue columns in 16. We observed that print quality appears to worsen over time, but is 'reset' by cleaning. We believe that the issue of agglomeration may be due to using push values that are not suitable for our ink. Additionally, it may be possible to avoid agglomeration by diluting the ink to lower its viscosity, or by adjusting the position of the atomizer within the atomizing jar.

Despite the immense variability in print quality, we were able to achieve conductivity values of 14 ± 10 S/cm, which is comparable to values found in the literature [6]. We attribute the uncertainty of our value to the variability in print quality. Though we were unable to achieve resolutions or print uniformities close to the values advertised by the Optomec, we believe that with proper optimization, the Optomec can be used to pattern materials in a versatile and controlled manner distinct to other equipment in the SNF.

4   Conclusions and Suggestions for Future Users

In this report, we present a user-friendly standard operating procedure for the Optomec Aerosol Jet printer in pneumatic atomizer mode and demonstrate printing PEDOT:PSS lines with the Optomec.

In the first part of this report, we briefly covered the working principles of aerosol jet printing and compared it to other printing techniques, especially inkjet printing. In the second part of this report, we provided a step-by-step standard operating procedure for future users of the pneumatic atomizer of the Optomec. This entails a description of tool assembly, troubleshooting of leaks and clogging, cleaning procedure, and printing. Finally, in the third part of this report, we demonstrated printing of PEDOT:PSS lines and described the parameters the user can optimize to improve the print quality.

Given our experience with the Optomec, we would like to offer a few suggestions that might benefit future Optomec users at the SNF:

- Develop a leak protocol to streamline the troubleshooting process.

- Purchase a new tube cutter that easily allows users to achieve flat, clean cuts for the PFA tube used during tool assembly.

- In the long term, considering to expand the solvents allowed for the Optomec to include some common organic solvents will expand the capabilities of the tool.

5 Acknowledgements

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