Displays are Here to Stay: Method Development for High Throughput Process Manufacturing Methods Employing R&D Scale Ink Jet Printing

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Abstract

The manufacturing of displays has increased dramatically since the introduction of household televisions. Efficient production methods are paramount to meeting market demands required for this field. Ink jet printing offers an easy, low cost alternative to current display manufacturing. However, fluid development and proper printing parameters at the research level are required for manufacturing processing. The Dimatix Materials Printer (DMP) addresses this prerequisite. In this paper, operating parameters and fluid characterization have been developed through processing both organic and inorganic conductive fluids. Because of the inherent versatility, uniformity and scalability of this system, established operating parameters coupled with proper fluid characterization will ultimately be translatable to production line systems.

Introduction

It is no surprise that the advent of electronic displays has found such attractiveness to the general population. The impact of high resolution visual displays has been apparent as computer screens of all sizes beckon attention and compete with displays on TVs, cell phones, MP3 players, dashboards, exercise equipment, advertising media and cameras. Displays are here to stay, and new methods that facilitate high throughput production of these visually stimulating devices are dominating production lines. Ink jet printing is inherently compatible to high throughput. [1] Already, interesting technological phenomena have spawned from the patterning of structurally and functionally different materials including high performance ceramics. [2] For this reason, drop-on-demand ink jet printing, a simple fabrication process, has become a prominent player in materials processing for display components. The ink jet printable electronics market for these types of products is expected to reach over $30 billion by 2013. [3] However, it is a big step for display developers to jump into robust in-line manufacturing production systems. [4] This type of equipment requires a sizable financial investment plus sufficient experience so that manufacturing specifications and in-house knowledge can be established. Thus, a low cost, easy-to-use laboratory scale system is required for preliminary experimentation. This strategy then allows both substrate evaluation and on-site development and manufacturing of specific jettable fluids to occur simultaneously. FUJIFILM Dimatix has addressed this need for an R&D tool with the DMP that offers printhead maintenance, substrate alignment, nozzle inspection and drop analysis, and its ease-of-use for a variety of fluids has been demonstrated. [5]

New Wave Manufacturing: Low Cost Ink Jet Printing

Just push print, the most common command for the desktop printer, can now be used in the laboratory or in manufacturing lines. Ink jet printing is a simple and cost effective technique with applications in the fields of electronics and biomedicine and has been shown to have specific applications in these industries. [6-8] In contrast to other multi-step production methods, ink jet printing is an additive process that precisely deposits metered quantities of fluid onto a variety of substrates including glass, silicon, plastics, organic thinfilms, and metals based on a user generated pattern. The resolution of the printed pattern is determined by a number of factors, including substrate/fluid contact angle, nozzle size, and lateral resolution of the printhead. [9] Ink jet printers can dispense fluid drops with volumes in the picoliter (pL) to microliter (mL) range, and an integral step in bringing this processing technique from the laboratory to manufacturing systems is the development of jettable fluids. The chemical properties of the fluid, including density, surface tension and viscosity, determine its jettability in a printer. [2] During drop formation, energy is distributed between the fluid's viscous flow, surface tension, and kinetic energy. [10] The deposited fluid volume is directly proportional to nozzle size. This flexibility enables microscopic patterned thinfilms of functional materials at a variety of resolutions. The physical properties of the patterned thinfilms (film thickness and pixel values) are dependent on the fluids coupled with the drive electronics of the printing device. In general, 2D drawings, pictures or structures, formatted as a bitmap image, can be translated into X and Y print coordinates for materials deposition (drop-on-demand). Each individual nozzle ejects a drop with a ligament. The ligament and the drop coalesce during flight to make a volumetric sphere and upon contact with the substrate, the sphere alters its three dimensional structure to become columnar. A resulting printed image is a compilation of drops where the third dimension is equal to film thickness, a physical property that is dependent on particle loading, drop spacing and drop spread. Once this critical but iterative R&D phase of process and material evaluation is complete to allow sustainable ink jet printing, the fluids are scaleable for production use.

Ink Jet Printing Employing MEMS Devices

The required heating process for thermal ink jet printing (300°C) will damage thermally-sensitive materials, thereby limiting their use in devising functional devices. [7, 10] In contrast, using piezoelectric ink jet printing, temperature sensitive, functional materials are deposited under ambient conditions.

Piezoelectric printheads contain a lead zirconate titanate (PZT) piezoelectric ceramic, nozzles, and a fluid chamber. When a voltage is applied to the PZT, mechanical vibrations create acoustic waves that in turn force fluid out of the chamber through the nozzles. [11] Piezoelectric printheads are categorized based on the deformation mode of the PZT (e.g., squeeze mode, bend mode, push mode, or shear mode). [12] At FUJIFILM Dimatix, the MEMS fabrication method for printhead production was adopted to increase the precision and resolution of the deposited materials. [13] These silicon devices increase jet-to-jet uniformity and drop placement accuracy. The inertness of the silicon expands the operating ranges to allow higher chemical diversity and fluid throughput expanding piezoelectric ink jet printing from the ability to print graphic inks to the realm of printing functional fluids required for display manufacturing.

With regards to the technological advances incorporated into the DMP from FUJIFILM Dimatix, a unique feature of this table-top printing system is the printhead itself. For the first time, FUJIFILM Dimatix is producing high performance MEMS printheads that are intended to have a limited lifetime, filled once by the user, and then discarded. The silicon chip that comprises the disposable printhead consists of 16 individually addressable jets that generate drops. These nozzles are spaced 254 mm apart, but actual drop spacing during printing is determined by the lateral resolution with tuned head angle. The ink jet printhead is powered by a piezoelectric unimorph, which is constructed in the plane of the wafer and consists of patterned PZT bonded to a silicon diaphragm. [11] The silicon chip is bonded to a molded liquid crystal polymer frame with an electrical interface. This construct is the jetting module portion of the printhead and snaps to the fluid module to complete the FUJIFILM Dimatix disposable cartridge. The fluid module is fabricated with a flexible polypropylene reservoir and protective rigid polypropylene housing. The volume of the reservoir is small (1.5 mL) to conserve expensive fluids. Fluid flows directly from the reservoir through a small column into the device in the plane of the wafer through a silicon acoustic terminator and then into a pumping chamber. The fluid then flows down a descender and out the nozzles perpendicular to the wafer plane. The silicon nozzle/air interface is coated with a proprietary non-wetting material to reduce wetting of low surface tension fluids and to facilitate printhead maintenance. The effective diameter of the nozzle is 21.5 mm; this nozzle size is approximated to generate 10 pL drops. An important operating parameter of this device is the negligible void volume due to the direct fluid/printhead interface.

Employing the Disposable Printhead for Fluid Development

Fluid flow properties like low viscosities, low boiling points, high surface tensions and non-Newtonian behaviors are hallmarks functional materials required for patterned thinfilms for display processing and are all generally unfavorable chemical characteristics for printing. For this reason, the Dimatix Drop Manager software was created to tune jetting parameters for these liquids. This software manipulates the parameters that generate the electronic signal to drive the movement of the PZT, including its frequency, wave shape, wave duration and voltage. Directing these parameters has provided a significant advancement in printing an array of functional materials and has been one of the areas of our research.

Printing Conductive Precursors

The need for continual reiterations of circuit design gestures for new approaches away from the reductive process of masking and etching to create metal patterns [14] towards a more attractive, additive process. There are also market drivers for organic electronic materials due to their adherence to substrates, flexibility, performance and the ability to process these materials at low temperatures. [15] Ink jet printing provides the necessary technological platform to increase throughput and lower processing costs. Indeed, low capital costs, process simplicity, and flexibility have been the important attributes that make this technique practical for conductive trace patterning. Many conductive precursor fluids are being jetted using both thermal and piezoelectric ink jet printers. [6, 16-18] In fact, ink jet printing is considered one of the key technologies in defined polymer deposition. [16] Polymer structural confirmation varies with temperature [19] thus ambient processing conditions are required. At low temperatures, typically below the glass transition temperature (T_g), polymers maintain their natural, globular structure. At higher temperatures, above the T_g, they swell into open conformations, essentially breaking their entropically favorable π-π interactions. [20] With the conformational collapse, the material becomes less conductive. In order to move towards feasible ink jet manufacturing processes for either conductive polymers or metallo-organic fluids, initiating formulation, printing and post-processing techniques are required. The fluids must maintain solvent monodispersity; once printed, they must properly adhere to the surface. [21] These criteria are integral for successful printing, for even the smallest amount of discontinuity will make the material non-conductive and lower its mechanical strength.

Early attempts at ink jet printing silver metallo-organic fluids capitalized on its advantageous annealing temperature post-printing (200°C). The resultant silver conductive traces on a variety of materials including flexible substrates and substrates are left with a low thermal budget. [18] The direct writing of silver ink onto a grid pattern of solar cells has been previously done using a self-built printer and a Siemens ink jet printhead. [17] In these experiments, Teng and colleagues modified their printhead by machining restrictive nozzle plates that varied drop size. The printer was run between 100 and 200 Hz which resulted in a printing speed of a few cmsec-1. This single laboratory technique was a slow throughput process and required multiple printing cycles for effective deposition, so although it is not agreeable to manufacturing protocols, it was an important proof of concept step.

The choice of additional organic material in the starting fluid greatly influences the obtained conductivity. [21] Once printed, the silver in the fluid must be annealed to convert the nanoparticles to a bulk silver thinfilm so that the resistance values can closely mimic bulk silver. The resulting amount of silver per volume of fluid is controlled by the annealing temperature cycle controls, and the effect of the organic decomposition into the gas phase during annealing determines the porosity of the printed material, which affects its continuity. [21] Additionally, the proper jetting parameters required for high performance printing is fundamental for reproducible deposition. The final feature size of the material on the substrate is determined by these parameters, and the overall conductivity is established according to the applied thermal processing. [21]

Materials and Methods

Inks

Two percent (2%) glycerol (Sigma Aldrich, St. Louis, MO) was added to a poly (3,4-ethylenedioxythiophene) poly(styrenesulfonate) (PEDOT/PSS) aqueous dispersion (H.C. Stark, Goslar, Germany). ANP Silverjet nanopaste (Advanced Nanoproducts, Chungcheonguk-do, Korea) and Cabot Inkjet Silver Conductor (Ag-Ij-G-100-S1, Albuquerque, NM) were used as packaged. Fluids were sonicated in a water bath in a Branson 1510 sonicator at room temperature using highest sonic level for 30 minutes. Fluids were degassed for 2 hours at 5 mbar pressure in a degassing chamber.

Substrates

Clean glass wafers were purchased from VWR (VWR Scientific, West Chester, PA). Both Kapton® (Dupont, Wilmington, DE) and Teslin® synthetic thinfilms (PPG Industries, Pittsburg, PA) were kept clean after purchasing and cut into 8 x 11 inch sheets using laboratory scissors that had been cleaned with 70% ethanol (Sigma-Aldrich, St. Louis, MO). Single-side polished 150 mm silicon 100 wafers were obtained from Silicon Quest International (Santa Clara, CA) and sputtered with 300 nm gold layer using an Au target and a converted TES sputterer.

Printer

The DMP-2831 (FUJIFILM Dimatix, Santa Clara, CA) was used according to packaging instructions. The DMC-11610 (10 pL) and DMC-11601 (1 pL) cartridges were removed from their storage bags and after degassing, 1.5 mL of fluid was injected into the cartridge. The cartridge was manually placed into the DMP carriage. The Drop Watcher camera system was activated utilizing the Drop Manager software. The default spit purge spit cycle was repeated until pulsating fluid could be seen at the nozzle plate. The time of flight (TOF) of the ~10 pL drops was recorded using a built-in stroboscopic broad band white light emitting diode and a charge coupled device camera with a high resolution 4x magnification lens that has a spectral response of greater than 60% between 400 and 700 nm. The camera's field of view is approximately 1.2 x 1.6 mm. The strobe frequency was matched to the firing pulse frequency (1 kHz for this application), and the motion control software's built-in variable delay and drop refresh rates were employed for visualization.

Contact Angle Measurements

Contact angle measurements were carried out using a VCA Optima XE (AST, Billerica, MA). 2 μL samples were manually pipetted for the measurements. The sample position between the LED backlight and the computer-interfaced camera was adjusted for optimal height and focus and then video captured. The associated software fit the silhouette and calculated the contact angle.

Annealing

After printing both ANP Silverjet nanopaste and Cabot Inkjet Silver Conductor, samples were placed in a clean Yamato DX400 gravity convection oven (Santa Clara, CA) and baked for 1 hour at 200°C.

Scanning Electron Microscopy

Scanning electron micrographs were obtained using a Philips XL30 ESEM. Resolution was obtained based on operating voltage. Operating voltage employed was 5 kV. Samples were magnified at either 20x or 80x.

Atomic Force Microscopy

Tapping mode AFM was conducted on a Digital Instruments Dimension 3100 using an etched silicon tip with a nominal radius of curvature of 10 - 20 nm. Scan sizes were varied, depending on the feature size. The scan rate was 0.1 - 0.3 Hz. The set point was set to 60 - 70% of the free-standing root mean square of the voltage of the oscillating tip.

Resistance Measurements

Resistance measurements were obtained using a Fluke 110 True RM multimeter. Anode was put at one end of silver contact on glass wafer and cathode was placed on top of other end of silver contact. Electrodes were manually held during measurements.

Results and Discussion

Jetting Parameters for PEDOT/PSS and Silver Metallo-Organic Fluids using the DMP

Due to approximate appropriate fluid formulations required for ink jet printing, glycerol was added to the stock PEDOT/PSS solution to increase the viscosity of the fluid. This modified fluid has a viscosity of 11 centipoise (cps) and a surface tension of 32.0 dynes per centimeter (dynescm^{-1}). A waveform was employed for successful PEDOT/PSS printing (maximum jetting frequency of 1.0 kHz for a pulse width of 17.0 ms). The pulse peaked at maximum voltage 2.2 ms and remained for 4.8 ms, recovered with a 5.1 ms slope to 20% maximum voltage and held for 1.5 ms. The voltage was then increased to 40% maximum voltage in 1.2 ms, held for 1.2 ms, and then dropped to zero volts in 0.3 ms and held for 2.0 ms. This waveform is a critical parameter for jetting this particular fluid.

The applied voltage was tuned specifically for each nozzle to provide uniform jetting speed to ensure reproducible drop volumes. Images of the jetting fluid were captured by light micrographs using this camera and software system (Figure 1). In panel A, the fluid is leaving the nozzle with the ligament still evident. In panel B, at 500 mm, the ligament has drawn into the drop, and the fluid is flying towards the substrate at 9.25 m/sec, 10 times faster than the homebuilt printer discussed above. [17] An optical micrograph of the resulting PEDOT/PSS on a glass wafer pattern is shown (Figure 2). While images of the deposited fluid can be captured by the DMP, additional physical measurements like contact angle measurements demonstrate the interplay between fluid development, printing and substrate interactions. The PEDOT/PSS spreads on an untreated glass wafer with a contact angle of 18° (Figure 3; panel A). In contrast, when the PEDOT is printed on Kapton®, little spreading occurred as evinced by the large contact angle (81°, Figure 3, panel B). When

PEDOT/PSS was printed on Teslin®, intermediate spreading was apparent (46.2°, Figure 3, panel C). The noticeable differences in the contact angle once the fluid has interacted with the substrate demonstrate the multilevel complexity of fluid development. In turn, these requirements highlight the necessity for small volumes and disposable printheads to increase experimental affordability during process development.

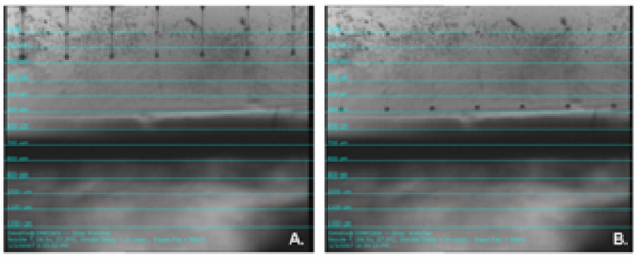

Figure 1. Light micrographs of fluid jetting from printhead nozzle and time of flights. A. Fluid leaving nozzle. B. Drop formation at 500 µm.

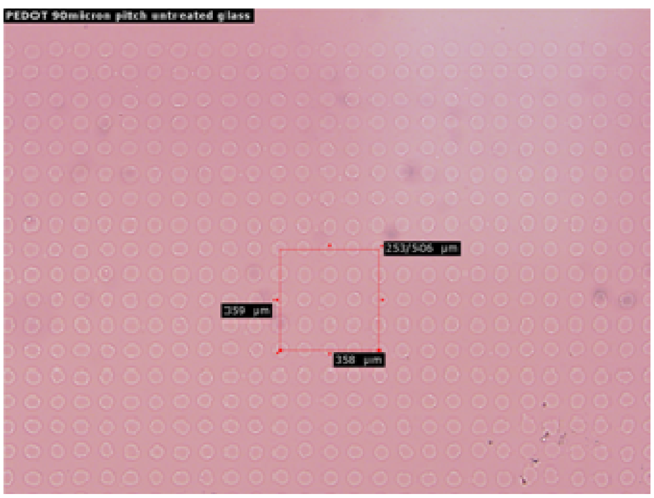

Figure 2. Single drops of PEDOT/PSS printed on glass wafer.

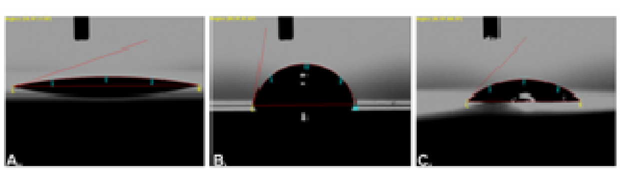

Figure 3. Contact angle measurements of PEDOT/PSS on three substrates. A. Glass wafer. B. Kapton®. C. Teslin®.

Silver Metallo-Organic Fluids

Reliable printing procedures for two commercially available conductive silver precursors have been examined. Both fluids have ideal fluid flow properties for ink jet printing and have higher than 50% silver nanoparticle load. The viscosity and surface tension values of the ANP Silverjet nanopaste are 9 cps and 26.5 $dynescm^{-1}$ respectively. This fluid jetted at a maximum frequency of 5.0 kHz with a pulse width of 13.2 ms. The pulse peaks at maximum voltage after 4.9 ms and is held for 4.3 ms. It then recovers with a sharp 0.3 ms slope to 25% maximum voltage. This voltage is held very briefly for 0.3 ms and is followed by an increase in voltage to 65% maximum voltage in 0.3 ms and held for 3.2 ms. The wavelength then sharply decreases to zero volts in 0.9 ms and is held for 3.7 ms. Directly comparing this jetting waveform to the jetting waveform for PEDOT/PSS, the overall pulse width is shorter and a longer recovery stroke is required for optimal jetting of the ink. Thus, while the physical properties of each fluid dictate the resulting parameters of the waveform, voltage and frequency, these parameters are rapidly optimized for the DMP.

Printing was accomplished on the same substrates using these nanoparticle silver fluids. Contact angle measurements were also taken. Comparing the contact angle measurements of PEDOT/PSS to the measurements of ANP Silverjet nanopaste (Figure 4) demonstrates the interplay between the fluid and the substrate. While PEDOT had a very high contact angle on Kapton® (80.95°), ANP Silverjet nanopaste spreads on the Kapton® very well (14.9°; Figure 4, panel B) although they have similar fluid properties. The ANP Silverjet nanopaste also spreads on the glass wafer (9.5°, Figure 4, panel A) and Teslin® (40.25°, Figure 4, panel C). These contact angles have established resulting feature resolution.

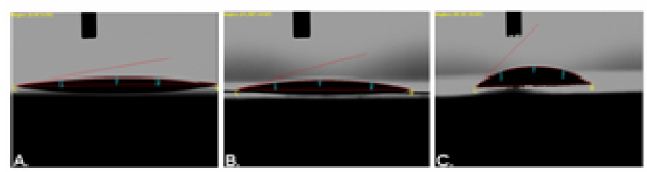

Figure 4. Contact angle measurements of ANP Silverjet nanopaste on three substrates. A. Glass wafer. B. Kapton®. C. Teslin®.

Because of its high particle load (54%) and uniform particle size as demonstrated by transmission electron microscopy (Figure 5) [22], low-temperature annealing produces a traceable conductivity in the printed material. Scanning electron micrographs were obtained of the annealed printed nanoparticles to compare the films produced (Figure 6). Panel A shows the ANP Silverjet nanopaste on Teslin® before annealing (silver on left, Teslin® on right). With single-pass printing, the fluid makes a uniform film on the Teslin® substrate in spite of the material's surface roughness (Panel A). Figure 6, panel B shows the same film on the same substrate after annealing for 1 hour at 200°C (annealed silver on left, Teslin® on right). Not only do the edges look slightly more uniform, but full coverage of the film on the substrate with single pass printing created a very thin silver full coverage film on the Teslin®. Because accurate feature measurements are difficult on flexible substrates, feature thickness was measured on gold-coated polished silicon nitride wafers (contact angle 41.8º, data not shown). We measured feature sizes using atomic force microscopy (AFM) with features printed at 20 mm drop spacing. Figure 7 shows the overall scan area of a single row of drops (Panel A). The three-dimensional rendered view in Panel B shows the overall jetting uniformity. Panel C shows the calculated feature measurements. The width of the feature was 40.6 mm, and the film thickness of 1.59 mm demonstrates the utility of producing patterned thinfilms using ink jet printing technology. Because electrical performance is often described in terms of the bulk resistivity, resistance values were measured after annealing the silver nanoparticles (Figure 8). The resistance of the ANP Silverjet nanopaste is shown in Panel A (1.1Ω), and the resistance of the Cabot Inkjet Silver Conductor is shown in Panel B (0.3Ω). The low resistance measurements in both cases were taken on equally-sized patterns on identical glass wafers. These values are in the same range as resistance values obtained by Sawhney and colleagues. [16]

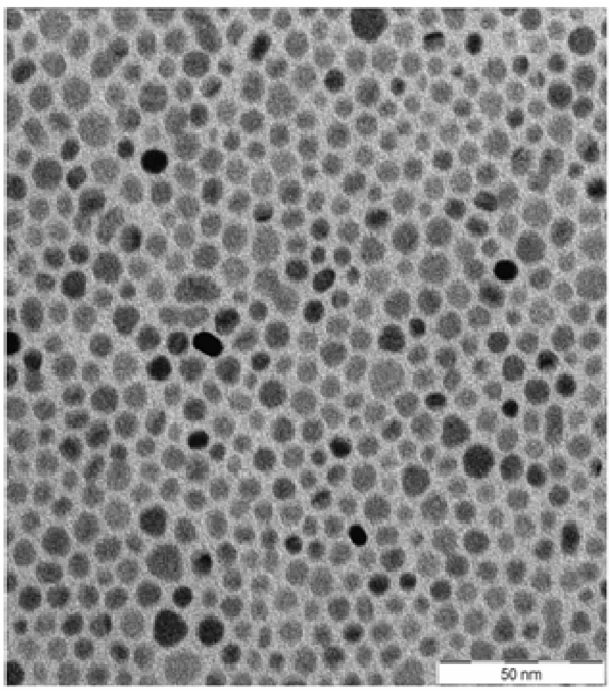

Figure 5. *Transmission electron micrograph of ANP silverjet nanopaste.*

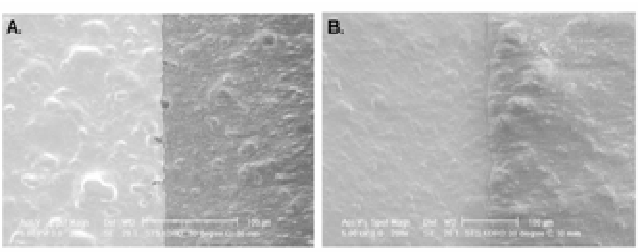

Figure 6. *Electron micrographs of ANP silverjet nanopaste on Teslin®. A. Before annealing. B. After annealing.*

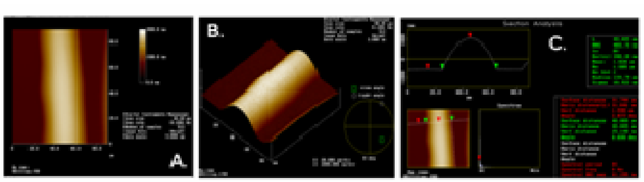

Figure 7. *Atomic force microscope images of single row of ANP silver nanopaste drops. A. Overall scan area of a single row. B. 3D rendering. C. Feature measurements.*

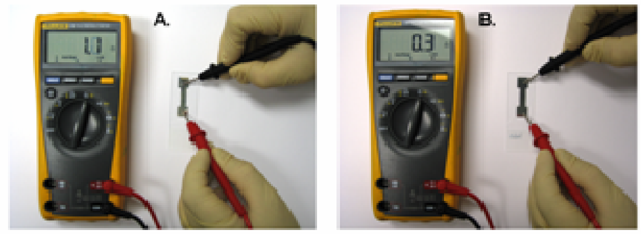

Figure 8. *Resistance values. A. ANP silverjet nanopaste. B. Cabot Inkjet Silver Conductor.*

In order to achieve even finer features in manufacturing electronic applications, drop volumes below 10 pL are required. Employing 1 pL printheads, the reduced drop volume will produce 20 µm silver fluid features on Kapton® employing the ANP Silverjet nanopaste fluid. The miniaturization of drop volumes is demonstrated by ink jet printing with the 10 pL cartridge followed by ink jet printing with a 1 pL cartridge on the same wafer substrate and performing scanning electron microscopy (Figure 9) where the radius of the 1 pL feature is more than twice as small as the radius of the 10 pL feature. Smaller drop volumes will lead to fine conductive traces required for appropriate feature sizes in photovoltaic and electronic applications [23] and directly address market demands.

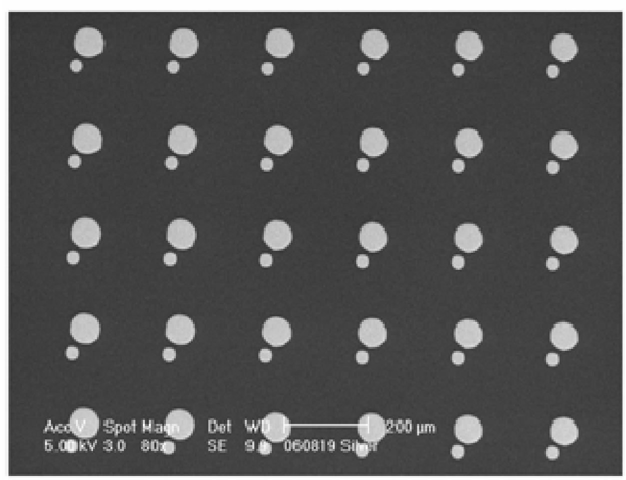

Figure 9. *10 pL versus 1 pL.*

Conclusion

The software interface and waveform tuning features make the DMP an ideal research and development ink jet printing tool that allows fluid process development. It possesses the features required to make ink jet printing of electronic materials a cost-effective manufacturing process. The printing parameters for printing PEDOT/PSS have been demonstrated, and the resulting interaction with the substrate demonstrates that ink jet printing is an iterative process where the interplay between the chemical properties of the fluid, the cartridge assembly, the machine operating procedure, the substrate and post-ink jet processing all determine whether this process is viable. In addition, the printing of two silver nanoparticle fluids have demonstrated the flexibility of the DMP, and these silver nanoparticles were successfully processed into conductive traces. Established operating parameters can now be translatable to production line systems with built in versatility, uniformity and scalability.

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