6-2019

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Publication Information

Grubb, Peter Mack; Koushyar, Farzad Mokhtari; Lenz, Travis; Asghari, Aref; Gan, Gongwen; Xia, Wenlong; . . . and Chen, Ray T. (2019). "High Speed Roll-to-Roll Printable Transistor Enabled by a Pulsed Light Curable CNT Ink". *Journal of Manufacturing and Materials Processing, 3*(2), 33-1 - 33-10. https://dx.doi.org/10.3390/jmmp3020033

Journal of
Manufacturing and
Materials Processing

Article

High Speed Roll-to-Roll Printable Transistor Enabled by a Pulsed Light Curable CNT Ink

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Received: 25 March 2019; Accepted: 10 April 2019; Published: 16 April 2019

Abstract: This paper reports the first high speed roll-to-roll printable transistor using a carbon nanotube (CNT) semiconducting layer. The transistor is made possible through the development of a pulsed light curable CNT ink compatible with typical drop on demand inkjet cartridges. This CNT ink uses a xylene based solvent with methanol, glycerin, and Triton X-100 modifiers to create an evaporable solution with appropriate absorption spectra for a mercury or xenon flash lamp with strong energy transmission in the UVB to mid visible light range, allowing the solution to absorb the energy from the flash lamp and evaporate. Transistor dimensions were defined by the capabilities of a typical roll-to-roll drop on demand cartridge. The final device demonstrated an on/off ratio of 10^4, representing performance similar to gravure printed devices. This represents the first CNT ink which can be used in high speed production methods without long thermal curing steps in the workflow.

Keywords: flexible printed circuits; carbon nanotubes; semiconductor nanotubes; production engineering

1. Introduction

Inkjet-printed electronics use additive manufacturing (AM) methods combined with inkjet deposition technology developed for home printers to deposit various materials to form devices [1]. These methods provide many avenues for driving down the cost of antenna systems and allow for flexible form factors which are very consumer friendly. As such, the technique is targeted as a critical enabler of the Internet of things (IOT) [2], disposable sensors, and other flexible antenna systems.

Typical inkjet electronics printers are small scale, printing a single sheet usually on the order of 10 × 15″. However, several techniques have been used to apply these materials at production scale. One such method is roll-to-roll (R2R) printing, which uses a constantly moving substrate with inkjet deposition print heads suspended above the substrate to rapidly apply a digitally defined design to the substrate [3]. While roll to roll printing shows great promise and has been used in original equipment manufacturer (OEM) applications, electronics design for such a system is non-trivial. Materials need to be cured within seconds in order to maintain a high enough speed to still qualify as production scale.

One critical component for producing antenna systems on these flexible substrates is an inkjet-printed transistor. These can be used for both beamforming control and for simple data processing [4]. Past papers have explored using these roll to roll systems in order to print CNT transistors, typically using a gravure system which employs a lithographically defined stamp to produce the pattern [5,6]. This method loses one of the main advantages of inkjet printing, in that the stamp must be retooled for each design, whereas R2R drop on demand print heads do not have such a limitation. Moreover, even the few efforts which were able to print transistors using inkjet deposition were forced to use thermal curing due to the nature of the CNT inks used [5,7]. A typical CNT ink consists of a CNT high weight percentage dispersed in n-cyclohexyl-2-pyrrolidone (CHP), which requires 15 min to cure at 210 °C. This negates the high-speed capability of R2R, and puts the design out of reach for true production scale electronics.

In this paper, a UV curable CNT ink is described which can be annealed in 7 s using a Xenon high intensity UV curing system. This ink is then used in concert with a commercially available UV curable nanocomposite silver ink, and a UV curable hybrid organic/inorganic dielectric ink made by Paru Co. (Suncheon, South Korea) to build transistors with R2R compatible dimensions. The result is the first CNT transistor which can be produced using high throughput R2R technology.

2. Materials and Methods

Building a high-speed curable transistor required two different steps in the development process. First, the ink had to be developed with the correct properties to enable the design. This involved developing a solution for carrying the CNTs which absorbed UV light, allowing the solution to be evaporated using high intensity UV light. Once the CNT ink was developed, the transistor design could be created. This involved designing a device around the rigid drop spacing and layering requirements of an R2R printing apparatus. The end result was the high speed R2R processable transistor design.

For an ink to be compatible with an inkjet deposition system, the two most critical properties are the surface tension and the viscosity of the ink solution. Typical values of 32–42 dynes/cm for surface tension, and 10–12 cPs for the viscosity have been recorded as good targets for an ink in a Fujifilm Dimatix cartridge [8]. While some R2R systems have varying targets for these values, they provide a good benchmark target for developing a new ink.

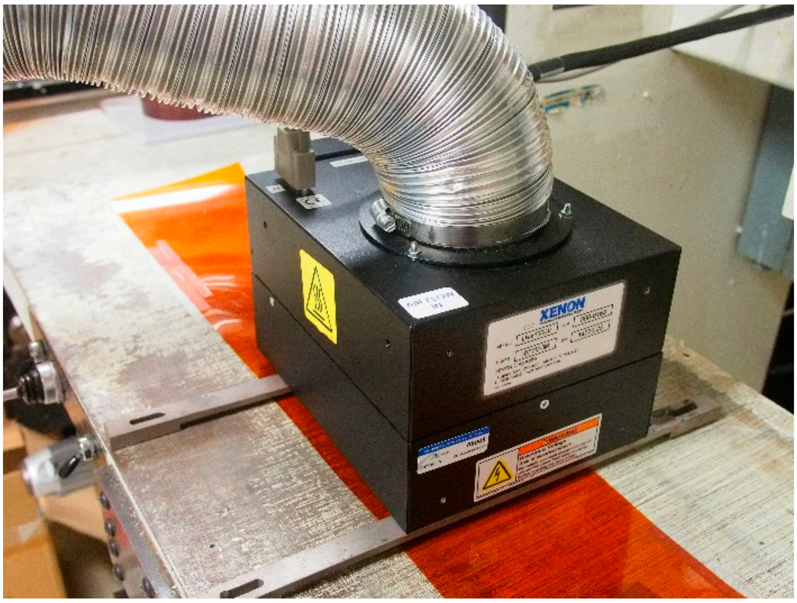


Figure 1. Xenon Sinteron-2000 system used for developing the CNT ink.

In addition to these solvent properties, the other key property for this CNT ink would be the absorption spectra. Typically, a high speed R2R system uses some sort of flash lamp system to emit high energy UV and visible light pulses. The ink can then absorb the energy from these flashes, annealing the ink. The development of this CNT ink targeted the Xenon Sinteron-2000 system pictured in Figure 1, which features a high intensity pulsed xenon lamp that provides a broadband spectrum from 240 nm to 1000 nm with an adjustable pulse energy up to 1500 Joules/pulse [9], which corresponds to UVB up to the Green/Yellow split of the visible light bands.

The typical solvent used for CNT inks is n-cyclohexyl-2-pyrrolidone or CHP. This compound is used due to its excellent viscosity (~11.5 cPs) and the fact that the boiling point is low enough to be easily curable on many substrates at 154 °C [10]. Typically the final ink compound is cured at a higher temperature if the substrate allows it in order more quickly cure the solution. This particular ink is formed by dissolving a CNT thick film into CHP at a concentration of 10% by weight [11]. Unique to the CNT ink vs. polymer or conductive traces, the end goal is to completely remove any solvents used as carriers for the ink. Thus, the ink is annealed at the boiling point of CHP, leaving only the CNTs on the substrate. However, CHP is also characterized by having most of its absorption spectra in the IR range [12]. This makes it unsuitable for UV curing applications, such as those proposed in this paper. Thus, an alternative base solvent was sought for the ink.

One class of solvents with absorption in the right range for usage with the Xenon Sinteron-2000 are the aromatic hydrocarbon liquids including both Xylene and Toluene. These solvents typically have a strong absorption peak at 400 nm in the UVA range, with a second peak between 575 nm and 750 nm depending on the exact compounds in solution [13]. Consequently, these materials are well suited to absorbing the energy from the Sinteron, allowing them to be evaporated using only the high intensity light pulses.

Of these aromatic hydrocarbons, Xylene is one of the most common. However, its viscosity is only 0.6 cP in a multi isomer blend. This is far too low to have drop formation using a drop on demand printing system. Some sort of thickening agent is needed in order to raise the viscosity of the overall printing solution. The most common thickening agent used in inkjet formulation is glycerin, as it has an extremely high viscosity requiring very little to reach the target viscosity needed. However, glycerin is not miscible in xylene, necessitating the addition of a moderator, in this case methanol, to allow for a stable solution. The last component of the CNT ink is Triton X-100, which is an emulsifier that both keeps the CNTs in the solution and lowers the surface tension to a usable level.

Once all the solvents had been identified, the next step was to identify the proper concentrations. Experimentally, it was determined that a 1:4 ratio of methanol to xylene provided a solution which was miscible with the glycerin. Then, the Refutas equation for viscosity blending was used to determine the amount of glycerin needed to reach the target viscosity [14]. The Triton X-100 concentration was determined based on other ink formulations in the literature [15]. The end result yielded a formula of 0.5% glycerin, 0.1% Triton X-100, 19.4% methanol, and 80% Xylenes.

With the concentrations determined, an IsoNanotubes-s100 CNT thickfilm from Nanointegris was dissolved into the solution at a concentration of 20% by weight. These CNTs are high purity, with no polymer wrapping or other functionalization, and have been sorted to be 99.9% semiconducting CNTs. The resulting solution is shown in Figure 2a. This ink solution was then printed in a small patch on a piece of Kapton and glass as shown in Figure 2b, which was then placed under the Sinteron. The UV light was then pulsed 12 times at 1.8 kV with a 0.4 s separation between the pulses. This put sufficient energy into the solvent to evaporate the non-CNT components, as shown in Figure 2c. An SEM micrograph of the CNT field remaining on the substrate after the curing process is shown in Figure 2d.

Many papers have been written about designing CNT thin film transistors for drop on demand printing systems. Typically, these papers focus on the usage of small-scale printers such as the Dimatix DMP-2800. Part of what makes the Dimatix an attractive prototyping platform is the ability to vary the drop spacing from 15 microns up to 100 microns. This variability allows for the usage of a wide variety materials without complicated design parameters. In contrast, the drop spacing for an R2R system is often fixed. For this design, the Konica KM1024MHB printhead was targeted as the R2R system of choice. This print head has a 35 micron drop spacing in the direction perpendicular to the substrate axis of motion, and a 70 micron drop spacing in the direction parallel, with 1024 nozzles on each print head. Thus, feature sizes needed to be multiples of 35 microns in one direction, and multiples of 70 microns in the other.

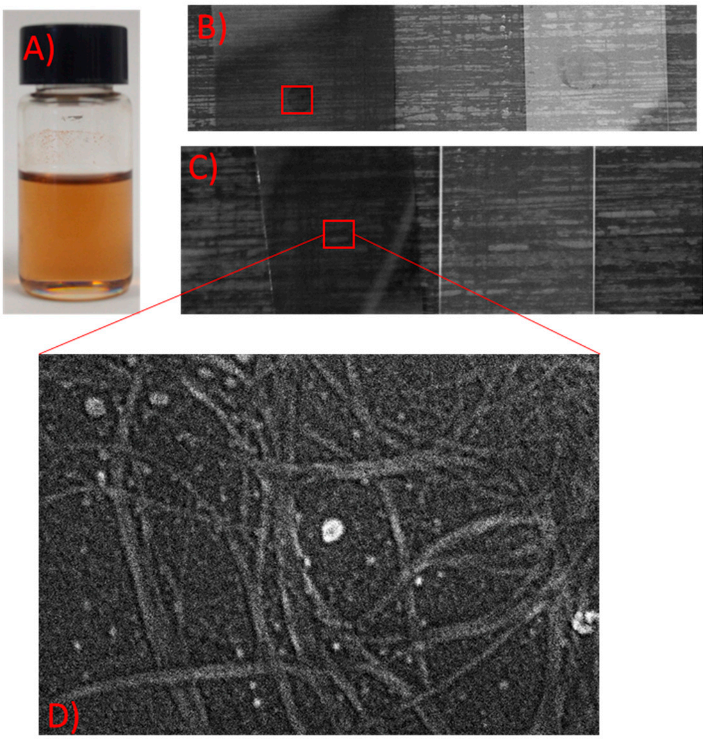


Figure 2. (**A**) The carbon nanotube (CNT) ink solution. (**B**) Printed solution before curing and (**C**) after curing, (**D**) SEM of the CNT field post curing. No disturbance of the CNT field by the high intensity pulses was observed.

With the minimum feature sizes defined, the layout shown in Figure 3a was defined. Based on this layout, both top gate and bottom gate structures were tested. Due to the domed nature of the printed dielectric, it was not possible to consistently produce a CNT thin film on top of the dielectric material. The CNTs would invariably reflow off the dielectric, aggregating on the edges of the dielectric layer. Due to this issue, the top gate structure shown in Figure 3b was used to construct the devices. The greater adhesion between the silver nanoparticle ink and the dielectric allowed the gate to be accurately placed where needed in order to produce the device.

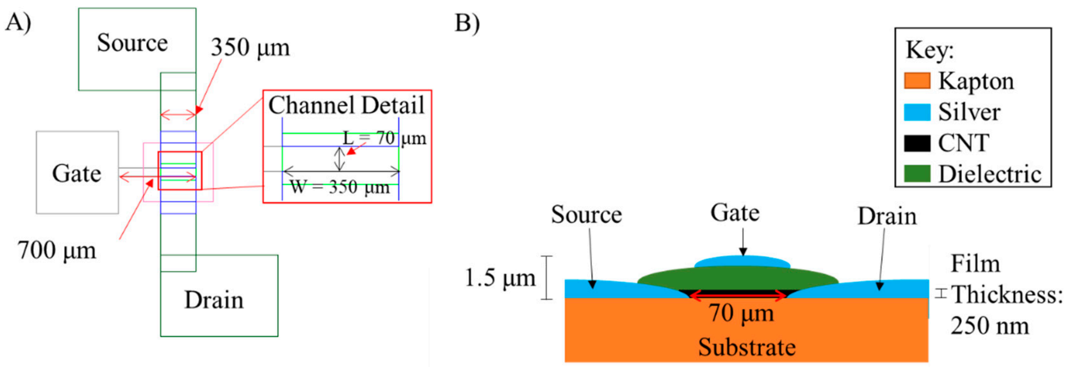


Figure 3. (**A**) The layout dimensions are all multiples of 70 microns to allow for easy roll to roll printing. (**B**) Layers are depicted as sloped surfaces to more accurately reflect the typical shape of a printed material.

Each layer was printed and then cured using the Xenon Sinteron-2000. This required careful coordination of materials, so that a subsequently printed layer would not be burned by a higher cure power on the sintering system. Based on these requirements, Novacentrix JS-B40G was selected for the conductive ink when printing the source and drain and a hybrid SU-8/BaTiO3 nanocomposite was selected for the gate insulator. Due to surface effects of the hybrid dielectric, a UTDots silver nanoparticle ink with better adhesion to SU-8 was used to print the gate. The CNT ink described above was used to create the semiconducting channel. The final device is pictured in Figure 4A, where each of the inks can be clearly seen. These devices were arrayed in batches with 22 to a cell as shown in Figure 4B.

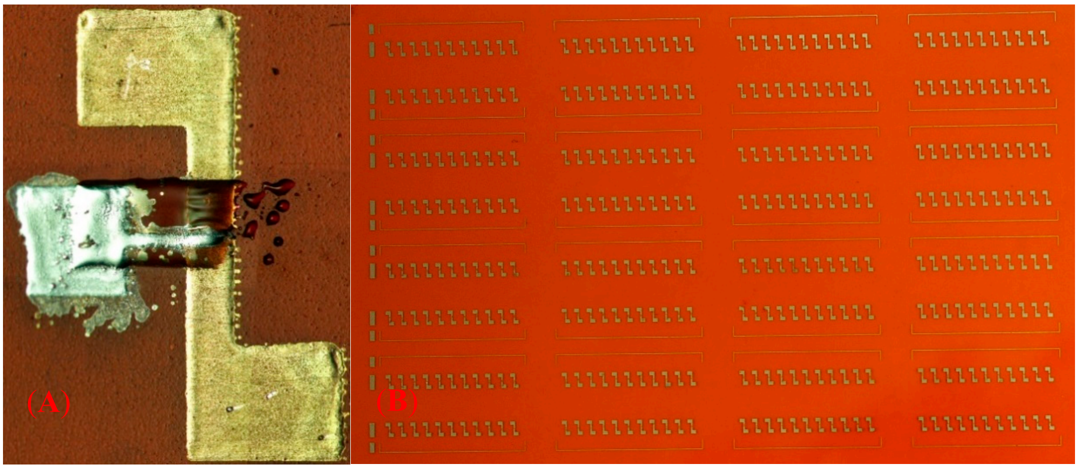


Figure 4. (**A**) A composite image of the final device produced. Multiple images were taken and assembled in order to create a higher resolution image. (**B**) An array of transistor devices in production.

3. Results

There were two broad areas that needed to be assessed in terms of the performance of the methodology described in this paper. The first involved the CNT ink, in terms of the purity of the deposited CNTs compared to the initial bulk powder. The second was the DC performance of the produced transistor acting as a proof of concept for what can be accomplished using this new UV curable CNT ink.

In order to assess the purity of the deposited CNT ink, Raman spectroscopy was used. First, as a baseline a reading was taken of the bulk CNT thick film provided by Nanointegris. CNTs were then applied to a Silicon substrate and cured using the Xenon Sinteron, and a reading was taken from the resulting thin film. It is worth noting that readings were attempted on a Kapton thin film, as this is what is typically used for producing R2R transistors. However, the Kapton substrate was too transparent for the Raman apparatus used to gather the data. The resulting data from the two readings is shown in Figure 5.

The other criterion of evaluation was the DC performance of the devices. Once produced, the devices were tested using an Agilent B1500A transistor analyzer. Different ranges were tested to find the limits of the thin film transistor before the current melted either the dielectric layer or the substrate. As is typical for printed TFTs, driving voltages for the gate were quite high due to the relatively thick gate dielectrics required to have contiguous layers without shorts into the CNT semiconducting layer. Of the 352 devices printed for this paper, 142 showed functioning transistor characteristics. However, most of the failures occurred during the dielectric application. Over 90% of the devices had contiguous CNT thin films with resistances on the order of 10 megaohms. Problems with the dielectric layer included both pinholing, and overly thick layers preventing switching. The resulting DC performance curves of a functioning device are shown in Figure 6.

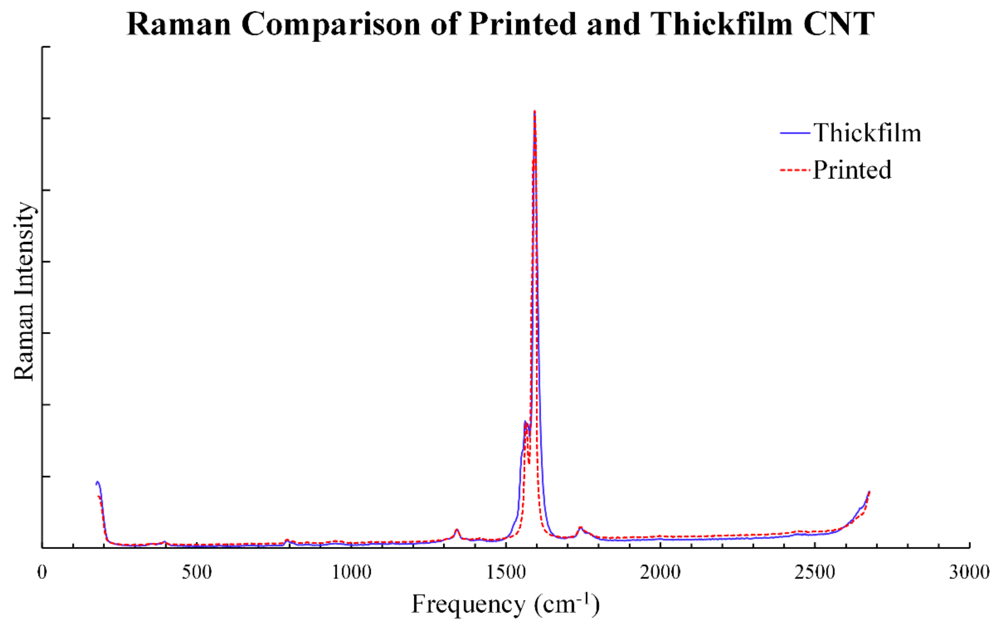
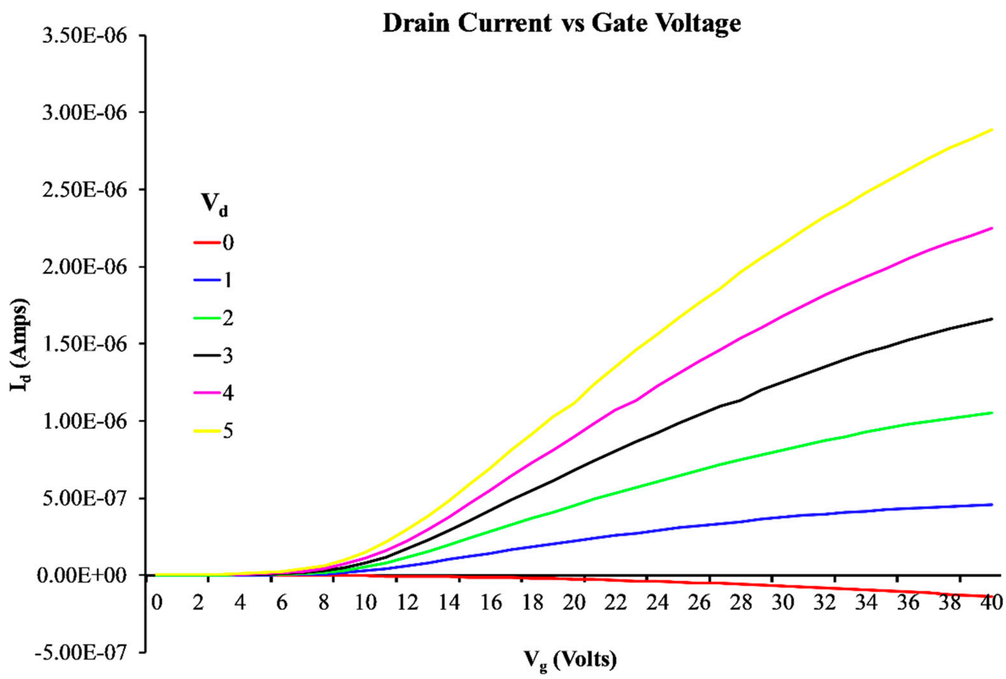
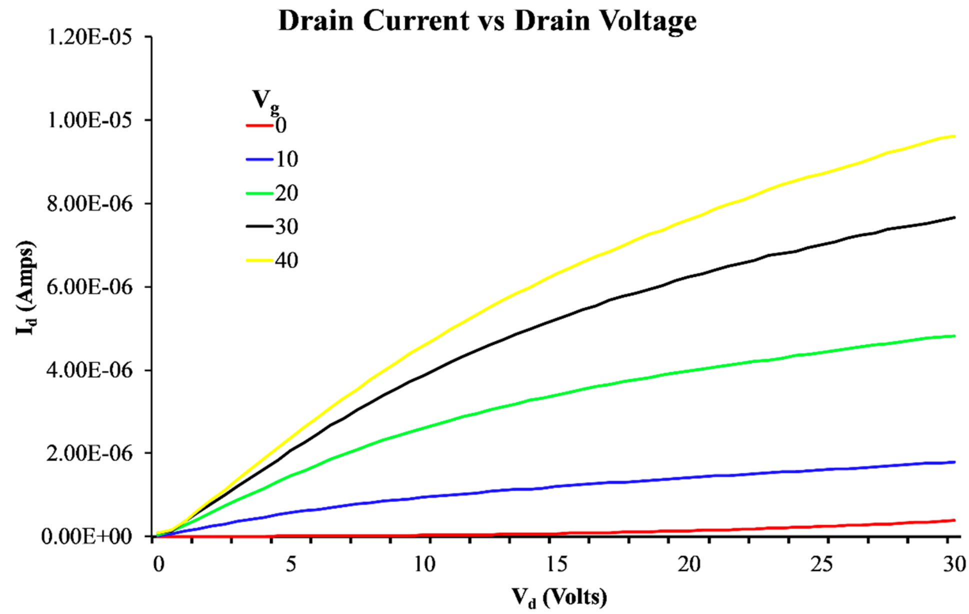


Figure 5. Raman spectroscopy of the thickfilm and printed film shows no significant shift in peaks, revealing very little contamination from un-annealed solvents.



(**A**)

Figure 6. *Cont.*



(**B**)

Figure 6. The DC performance curves for the R2R compatible transistor design. (**A**) Drain Current vs. Gate Voltage; (**B**) Drain Current vs. Drain Voltage.

In order to better illustrate the On/Off ratio, the subthreshold swing is reproduced using the I_d/V_g plot on a logarithmic scale in Figure 7. The resulting DC curves demonstrate an On/OFF ratio of up to 10^4. Additionally, it is worth noting that at 0 volts V_d a small reverse biasing effect was observed at high values for the gate voltage on some devices. This is common in printed TFT devices [16–18], but was easily overcome even at small source/drain voltage levels. Significant hysteresis was not observed when testing the devices. Hysteresis in printed CNT devices with polymer dielectrics is usually caused by impurities in the deposited CNT thin film or traps formed by voids in the dielectric layer [19]. The wet application of the dielectric over the CNTs minimized the formation of traps at the CNT/dielectric interfaces, and impurities in the deposited film were minimal as shown via the Raman spectroscopy. These properties are all average for a small-scale printed CNT TFT, though they are produced by a device which is compatible with high speed R2R methods.

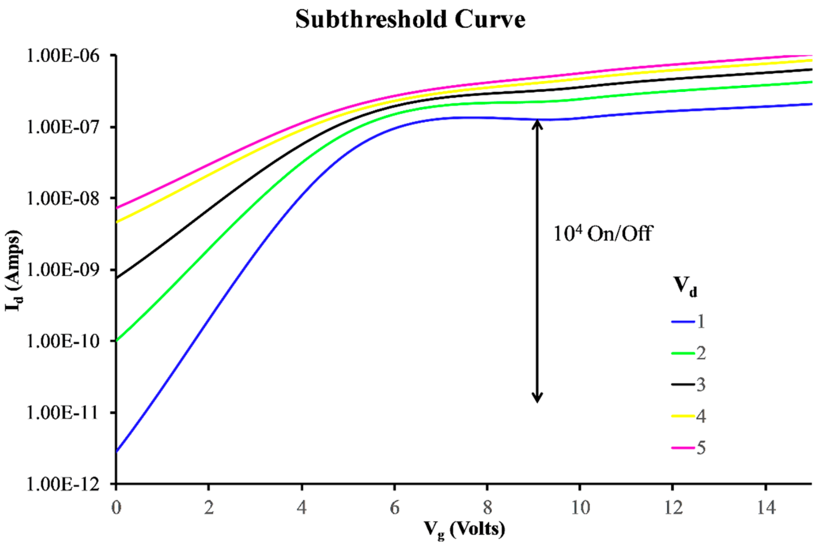


Figure 7. The subthreshold curve shows the 10^4 On/Off ratio at V_d = 1 Volt.

4. Discussion

It is important to consider the context that these transistor devices exist in. The literature is replete with examples of printed high performance transistors ranging from ion gel devices to self-aligned gates [20–23]. Most of the devices described in these papers have major hurdles relating to potential roll-to-roll applications, usually due to the small minimum feature sizes they require to achieve their significant performance increases. While this device development is important, moving to something that can actually be used in a consumer device requires moving these techniques to production scale technologies such as R2R drop on demand or gravure printing.

The primary area where there has been success in mass producing printed TFTs has been using gravure printing methodologies. The transistor data shown in this paper matches previous On\Off ratios from gravure printing papers [6,24]. However, it does so without requiring any kind of gravure or stamp to be produced using lithographic means. This means the device can be produced while retaining the high re-configurability inherent to drop on demand systems. The only other reported device that can be produced using high speed gravure printing required the usage of an organic semiconductor due to lacking a UV curable CNT ink [6].

The main advantage of gravure printed devices is the thinner dielectric layers they are able to maintain, and therefore the lower on voltages required to operate the device. For a potential IOT device, this lower on voltage would correspond to significantly lower power usage. This advantage is fundamental to the physics of how the two technologies apply material to the substrate. Future efforts will need to investigate the potential for thinning the gate layer while still maintaining integrity in order to lower the on voltage of the device. These thickness issues along with the yield rates observed in the dielectric application step suggest that the need for improved dielectric materials is significant. Alternatively, it would be instructive to reformulate the UV curable CNT ink to a gravure printable version. This would allow for a high-speed roll to roll printable device with better performance in low power environments.

Raman spectroscopy also provides an important data point in terms of the purity of the CNT film that is deposited. While minor shifts are seen throughout the curve, most are not significant enough to show significant variation. The two normalized Raman datasets are over 99.99% correlated, corresponding to a low level of contamination from the solvents used to suspend the CNT ink before applying it to the substrate. By evaporating off all the solvent components, what remains on the substrate is the high purity semiconducting CNT thin film needed for the transistor device. The close correlation between the sample after curing and the reference thick film sample shows that the carrier solution does not significantly contaminate the CNT particles. This lack of contamination is critical to preventing the emergence of significant hysteresis in the final device.

Compared to thermal curing, this UV curable CNT ink shows no discernable differences in performance. This was primarily assessed via the performance of the transistor compared to past efforts [16–18]. Given the Raman spectroscopy results, this was as expected given that most CNT inks aim to minimize impurities in the final thin film. That said, the UV CNT ink does not stay in solution as long as CHP based solutions due to the interactions between the non-polar solvents and the CNTs themselves. This requires sonication or circulation of the ink prior to usage to ensure a uniform solution. Ultimately, no performance impact to the CNTs was measured when using photonic sintering.

5. Conclusions

The core innovation of this paper is a CNT ink which can be cured using high intensity pulsed light. Rather than using the typical CHP solution, this ink uses a Xylene based solution with modifiers to make it compatible with typical drop on demand printing cartridges.

This ink enables a device representing the first high speed R2R printable device using CNTs as the semiconducting layer. Moreover, it is the first such transistor design intended for production using high speed drop on demand technology as opposed to gravure printing or other stamp-based processing. Final on/off performance of the device was in line with past efforts using gravure printed

CNTs with slower processing speeds. These devices represent a critical step in the effort to have mass produced flexible antenna systems, particularly in disposable device development where cost drivers are paramount.

Future efforts will focus on thinning the dielectric layer for improved V_{ON} levels to improve performance in low voltage devices. The dielectric layer also represents the primary source of yield rate issues for this design, as maintaining a cohesive layer that is still thin enough for switching is quite difficult. Additionally, with such a device described future papers should use the described materials and transistors to implement simple printed devices such as flip-flops and amplifiers.

Author Contributions: conceptualization, P.M.G., F.M.K., H.D., H.S., R.T.C.; methodology, P.M.G., F.M.K., T.L., A.A.; validation, A.A., G.G., W.X.; formal analysis, P.M.G., F.M.K.; investigation, P.M.G., F.M.K., T.L., G.G., W.X.; resources, H.D., H.S., R.T.C.; data curation, P.M.G., F.M.K.; writing—original draft preparation, P.M.G.; writing—review and editing, P.M.G., F.M.K.; visualization, P.M.G.; supervision, H.D., H.S., R.T.C.; project administration, R.T.C.; funding acquisition, R.T.C.

Funding: This research received no external funding.

Conflicts of Interest: The authors declare no conflict of interest

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