Fabrication and characterization of entirely inkjet-printed polysilicon thin film transistors

Mao Ito and Arvind Kamath
Thin Film Electronics, Inc., 2581 Junction Avenue, San Jose, CA CA95134, United States of America
E-mail: Mao.Ito@thinfilmsystems.com and Arvind.Kamath@thinfilmsystems.com

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Abstract
In this paper, the fabrication and material innovation involved in the first and only entirely inkjet-printed polysilicon thin film transistors (TFTs) are described. To form TFT layers, five inkjet printing inks were developed with the goal of fabricating TFTs by using purely additive processing without vacuum deposition or conventional lithography. A silicon ink was developed to form both the channels and polysilicon gates, and boron and phosphorus dopant inks were developed for N+ and P+ doping. In addition, a silver nanoparticle (NP) ink was developed to form interconnect traces, and a palladium chloride ink was formulated to create palladium silicide for the ohmic contacts between the source and the drain. The first N-type metal-oxide-semiconductor (MOS) polysilicon TFT was fabricated with a top-gate self-alignment scheme. This exhibited a mobility of approximately 80 cm² V⁻¹ s⁻¹. Next, P-type MOS transistors as well as complementary MOS devices were also successfully fabricated.

1. Introduction
Wearable or disposable devices for the internet of things need low cost, thin structures, and durable, flexible logic circuits that are capable of radio frequency (RF) communication. Over the last several years, printed electronics has gained attention as a method for realizing this. Organic semiconductor solutions [1, 2] such as dinaphtho[2,3-b:2’,3’-f]thieno[3,2-b]thiophene (DNTT), metal-oxide-semiconductor (MOS) solutions [3, 4] such as indium gallium zinc oxide (IGZO) or carbon nano tube (CNT) ink [5, 6] have also been examined to realize transistors with alternative materials. Though those materials have the advantage of being able to be handled in air or to be fabricated at low temperature on plastic film, they have not demonstrated the ability to fabricate high frequency circuits, for example for RF communications. Few semiconductor materials with well-established track records have proved better than silicon when it comes to forming stable, high-performance logic circuits in volume manufacturing. Therefore, using liquid-phase precursors of silicon to form thin film transistors (TFTs) via printing has received considerable interest. One approach is to use silicon nanoparticles (NPs) as a precursor, as proposed by Weis [7]. Another approach used cyclodihydrosilane or polyhydrosilane compounds in solvents which was first reported by Shimoda et al as the first TFT using poly-cyclopentasilane (CPS) [8]. They also gave considerable attention to the associated research topics, such as polysilicon TFTs, on a paper substrate, as reported by Trifunovic [9, 10]. In addition, Ishihara [11] proposed the use of single grain TFTs, and Masuda [12] proposed the use of doped silicon.

We demonstrated an entirely inkjet-printed polysilicon TFT as a proof of concept. Five inkjet printing inks were developed with the objective of fabricating TFTs by only using printing processes eliminating the need for expensive vacuum deposition, ion implantation or lithography.

Silicon ink was developed to form channels and polysilicon gates. The polymerization process of monomer silanes, such as CPS, n-tetrasilane, and n-trisilane, was developed using proprietary heterogeneous catalysts synthesized in-house. This process does not require UV [7] or ultrasonic [13] excitation (figures 1(a) and (b)). A high-quality amorphous silicon film could be fabricated from formulated inks with either CPS, poly-CPS, or poly-tetrasilane. In this work, poly-CPS was used as a precursor of
the amorphous silicon film. Poly-CPS still has a low molecular weight and a liquid form that can be dissolved in solvents and flows due to a thermal induced Marangoni effect [14] during the heating process. For this reason, the wet film of poly-CPS needs to be irradiated with UV to increase its molecular weight and stabilize it before the thermal curing step. UV polymerization of silane compounds is used for photochemical vapor deposition (CVD) applications [15]. The UV irradiation with wavelength of 254 nm causes a decomposition by photoreaction which leads to a radical reaction that polymerizes the silanes. Mercury lamps, UV-LEDs or excimer laser can be used to polymerize silane [10]. The required solvent for formulating ink needs to be transparent and inert with UV irradiation, and should not have an oxygen constituent, as any peroxide or oxygen radicals can react with the silane. To evaluate the ink purity and to optimize process parameters, amorphous silicon films formed by spin coating were first studied extensively. A spin coating recipe with simultaneous UV irradiation was developed to directly form films from the poly-CPS. A low-pressure mercury grid lamp and exhaust were integrated with the spin coater. Also, cyclooctane, which is cycloalkane (boiling point \( \sim 150^\circ C \)) was chosen as the solvent for the spin coating, and cis-decalin (boiling point 190 \( ^\circ C \)) was chosen for the inkjet ink. The solvents were purified to remove oxidizing compounds that can potentially contaminate the silicon film and reduce carrier mobility.

The laser used in this study was XeCl based (308 nm). Any resulting protuberances must be managed or eliminated as this can lead to gate oxide coverage and reliability issues over the active silicon.

For N+ and P+ doping, boron and phosphorous dopant inks were developed. Spin-on dopants [16] from Honeywell Inc. were used as the base materials for formulating the dopant inks. P-8545 was used to formulate the N+ ink with phosphorous, and B-30 was used to formulate the P+ ink with boron. The primary solvent for these materials is ethanol, which is not suitable for ink-jetting as it quickly evaporates and tends to clog inkjet nozzles. Also, ethanol’s lower surface tension and higher volatility creates a self-induced Marangoni effect [17], which causes the ink to rush out of the region where the droplets fall on the oxide surface. To formulate the ink for the inkjet printing in well-defined areas, the ethanol was replaced with a low volatility solvent and a silicone polymer surfactant was added to suppress the Marangoni flow [18]. Boron oxide in the printed P+ ink is hydroscopic and reacts with moisture in air at room temperatures to form boric acid (boiling point 300 \( ^\circ C \)). Due to the volatile nature of boric acid at the diffusion temperature, duration of the exposure to ambient air reduces the ability of the printed boron dopant to activate silicon [19]. For a reproducible P+ doped silicon, all moisture trapping phenomenon should be avoided. Print and soft cure of P+ ink need to be operated in nitrogen or dry air and duration of the exposure to ambient air before thermal annealing should be minimized.

As the contact between the silver and doped polysilicon is relatively poor [20], ink to form silicide as an ohmic contact between the silver interconnect and doped polysilicon was developed. There are metals such as nickel, cobalt, titanium, tungsten, and palladium that can form a silicide [21]. In this experiment palladium was chosen because palladium can diffuse through native oxide (SiO\(_2\)) in reducing ambient which eliminates pre-sputter etch for ohmic contact formation [22, 23]. A process to form palladium silicide (Pd\(_2\)Si) was developed. Since palladium chloride is reduced to palladium metal by annealing in forming gas (N\(_2\)/5% H\(_2\)), the palladium chloride ink was formulated as a precursor of the palladium metal, which was then used to form palladium silicide with doped polysilicon.

Silver NP ink was developed to form the interconnect layer. Silver NPs were synthesized using a liquid phase reaction.
2. Methods

2.1. Ink preparation

2.1.1. Silicon ink

The CPS was synthesized using a modified Hengges’ scheme [24], which was then purified and polymerized to semiconductor grade levels. The resulting polymer was analyzed using gel permeation chromatography, a capillary viscosity meter and an inductively coupled plasma mass spectrometry (ICPMS) to measure the molecular weight, viscosity, and the trace metal content. Both the cyclooctane (>99%, Sigma-Aldrich) and the cis-decalin (>99%, Sigma-Aldrich) were washed using sulfuric acid and then distilled over sodium to remove any remaining traces of water. A 15 vol% poly-CPS solution in cyclooctane was prepared for the spin coating. This was dispensed using a glass syringe through a 0.2 µm polytetrafluoroethylene (PTFE) disk filter on a SiO$_2$ coated silicon wafer. After a short spreading rotation at 500 revolutions per minute (rpm); the high-speed rotation at 1000 rpm, UV irradiation, and exhaust were simultaneously enabled. The coated silicon film was baked in a muffle oven under a nitrogen gas flow via a gas purifier at 400 °C for 20 min to reduce the hydrogen. The incorporated impurity levels were determined using ICPMS analyses and secondary ion mass spectrometry (SIMS) of the spin-coated film. All the operations were performed in a glove box that maintained the oxygen and moisture levels below 5 ppm. Following this, a 15 vol% poly-CPS solution in cis-decalin was prepared for the inkjet printing, and the ink was dispensed into a cartridge through a 0.2 µm PTFE disk filter.

2.1.2. Dopant ink

To formulate the N+ ink, first P-8545 was poured into a flask with a rotary evaporator. Then, an equal weight of diethylene glycol monobutylether (>98%, Sigma-Aldrich) was added. As the ethanol in the P-8545 was evaporated, it was replaced with diethylene glycol monobutylether. Then, 0.002 wt% of polydimethylsiloxane surfactant (Botanisil S-18, D-D Chemco) was added to suppress Marangoni effect. The B-30 was processed in a similar manner to formulate the P+ ink.

2.1.3. Palladium chloride ink

The palladium chloride ink was formulated with 2 wt% of palladium chloride (Sigma Aldrich) that was dissolved in deionized water/diethyl glycol butyl ether (99%, Sigma Aldrich, 15/85). Then, using a vortex mixer, the solution was mixed in a vial for 10 min, allowed to settle for 12 h, and then filtered prior to loading into the inkjet cartridge. A commercially available palladium precursor (palladium film, Emulsitone company) and palladium chloride ink formulated with diethyl glycol butyl ether were tested for comparison purposes.

2.1.4. Silver ink

The silver NPs were synthesized from silver oxide using liquid-phase reactions. The silver oxide (99%, Sigma Aldrich) was reduced using 1-decene (95%, Sigma Aldrich), 2-ethylhexanoic acid (99%, Sigma Aldrich), and the proprietary fatty acid surfactant in the solvent. This formed the silver NPs that were covered with the surfactant. Then, the reaction mixture was centrifuged with isopropyl alcohol (IPA). The decan supernatant was subsequently mixed with hexane and IPA and centrifuged again. The NP diameters were determined using transmission electron microscopy (TEM). To formulate the ink, 20 wt% of the silver NPs were dissolved in cis-decalin and dispensed into a cartridge through a 0.2 µm PTFE disk filter.

2.2. TFT fabrication

A top-gate self-aligned process flow using a poly-silicon gate and printed dopants was developed. Silicon wafers of 100 mm diameter were cleaned and thermally oxidized resulting in a 1 µm SiO$_2$ film as the field oxide. Surface cleaning and modification were further performed before printing the active islands. Surface modification required the oxide surface to be treated with hexamethyldisilazane (HMDS) and then exposed to UV/ozone with a low-pressure mercury lamp. The contact angle (CA) of the cis-decalin was measured using a drop shape analyzer after modification process.

The prepared 15 vol% poly-CPS silicon ink in cis-decalin was printed using an inkjet printer (Dimatix, 10 pl nozzle) with ink pattern dimensions of 60 µm × 1200 µm in a glove box under a nitrogen gas environment. Printed poly-CPS patterns were irradiated simultaneously with UV (50 mJ cm$^{-2}$ at 365 nm) from a spot UV curing system which was integrated with the inkjet printer. The printed wafer was immediately hard-baked at 420 °C for 20 min, and a further dehydrogenation bake was performed at 550 °C for 15 h in nitrogen ambient. The parallel and perpendicular cross-sectional profiles were measured using a profilometer (Tencor).

Since it is stable in air, the wafer with the active silicon islands was removed from the glove box after the hard baking process. The surface of the active island was oxidized (750 °C for 0.5 h, O$_2$) and then dipped in hydrofluoric acid (HF 50:1, 60 s) to pre-clean it before it was crystalized using the XeCl excimer laser irradiation (1200 mJ cm$^{-2}$).
Then, the wafer was loaded in a tube furnace and annealed at 850 °C in N2/O2 to form a 35 nm gate oxide. The silicon gate layer was then printed with silicon ink, and baked with an identical process used for the active channel layer.

The gate oxides on the source and drain were removed using a 50:1 HF bath for 7 min. Then, a 200 µm diameter N+ ink was printed on top of the cross point of the island and the gate (figure 2). This was soft cured at 100 °C for 10 min. The printing and soft cure operations were performed in the glove box in nitrogen ambient. The wafer was then rapid thermally annealed at 850 °C for 5 min in nitrogen ambient.

After pre-cleaning the wafer using HF (500:1), the palladium chloride ink was printed outside of the printed dopant area and then soft cured at 230 °C for 2 min followed by an anneal at 650 °C for 2 h in the forming gas (5% hydrogen gas in argon gas, Plaxair). After annealing with the forming gas, x-ray diffraction (XRD) on the spun palladium chloride ink with various solvents on the spun poly-CPS film was used to determine the formation of the palladium metal and Pd2Si.

Silver NP ink was printed on the silicide area to form an interconnection pattern before being thermally cured on a hotplate at 300 °C for 10 min. The sheet resistivity was measured using four-point probes on appropriate test structures.

A diagram of the fabrication flow is illustrated in figure 2. The TFTs were characterized using an HP4155 semiconductor parameter analyzer.

| Table 1. Trace metal impurity of poly-CPS measured with ICPMS. |
|--------------------|---|---|---|---|---|---|
| Atom | Al | Ca | Mg | K | Na | Cr | Cu |
| Concentration [PPB] | 83 | <0.5 | <20 | <20 | 78 | 19 | 9.5 |

3. Result and discussion

3.1. Characterization of inks and fabricated films

The molecular weight of poly-CPS was Mw = 910 g mol⁻¹, and its viscosity was 6–8 mPa s, while that of the inkjet ink was 2–3 mPa s at 20 °C. The shelf life of the formulated ink exceeds several months with no change in appearance and viscosity when stored below −20 °C. Inks prepared with other solvents, such as alcohol, ether, ketone, and ester, demonstrated turbidity immediately or a day after formulation and were ruled out for use. Table 1 shows the trace metal impurity of poly-CPS measured with ICPMS. A typical hard-cured film was analyzed using SIMS and was shown to contain minimal quantities of carbon, oxygen, and other impurities, as shown in table 2. The learning and optimization from the spin coating process was directly applied to the inkjet formulation.

Due to the coffee ring effect which was caused by capillary flow induced by pinned edges during the evaporation process [16], the inkjet-printed active island formed a dog bone shape as shown in the microscope image (figure 3(a)). The parallel and perpendicular cross-sectional profiles were shown in figures 3(b) and (3), and the thickness at the center...
Table 2. Concentration of the negative and positive species in a hard-cured poly-CPS ink measured using SIMS.

| Atom | C  | O  | N   | Cl | P  | Na | Mg | Al | Cr | Cu |
|------|----|----|-----|----|----|----|----|----|----|----|
| Concentration [PPM] | 400 | 100 | 70.10 | 64.50 | 0.88 | 0.02 | 0.02 | 0.04 | 0.02 | 0.04 |

Figure 3. (a) Optical microscope top-view image of the printed island, (b) cross-sectional profile of the cured island measured using a Tencor profilometer, and (c) profile along the length of the cured island.

Figure 4. Cross-section of the excimer laser-annealed, spin-cast CPS film showing (a) an SEM image without the dehydrogenation bake, (b) an SEM image with the dehydrogenation bake.

Table 3. SIMS analysis of the silicon film for the post dehydrogenation bake.

| Deposition method | Hard bake | Dehydrogenation bake | C [atm%] | H [atm%] | O [atm%] | N [ppm] |
|-------------------|-----------|----------------------|----------|----------|----------|---------|
| Spin coat         | 420 °C 20 min in N₂ | NA | 0.03 | 8.0 | 0.02 | 1 |
| Spin coat         | in N₂ gas | 550 °C 15 h in N₂ gas | 0.01 | 0.5 | 0.02 | 7 |
| Ink-jet           | 420 °C 20 min in N₂ | NA | 0.04 | 4.4 | 0.38 | 2 |
| Ink-jet           | in N₂ gas | 550 °C 15 h in N₂ gas | 0.01 | 0.82 | 0.02 | 3 |

of the peak was 45–65 nm. Using spin-coated films, laser crystallization was studied as a first step to realize high-mobility. The irradiation from the excimer laser can lead to the formation of bubbles due to the hydrogen in the film (figure 4(a)). However, the dehydrogenation bake [25] at 550 °C for 15 h in a nitrogen flow prevented the films from forming bubbles (figure 4(b)). Table 3 shows that the hydrogen content decreased to less than 1% after the dehydrogenation bake. This was low enough that the dehydrogenated film did not show any bubbles after the excimer laser irradiation. The same hard cure and dehydrogenation bake conditions were applied to the inkjet-printed poly-CPS layer. This also decreased the hydrogen content to less than 1 atm%. Table 3 shows SIMS analysis comparison the hydrogen content before and after the dehydrogenation anneal.

The dome shape of the printed island caused the thin edges to be overdosed by the laser, which melted and pulled them back to form a serrated pattern
(figure 5(a)). When the island forms a serrated shape, the printed gate was broken from the edge resulting in holes, as shown in figure 5(b). The cross-sectional profile of the active island was optimized to prevent formation of the serrated structure (figure 5(c)) by controlling the CA of the cis-decalin on the oxide, which was controlled \( \theta < 5^\circ \) using HMDS and UV/ozone treatment.

Figure 6(a) shows thickness at the center of 15 islands after hardbake with and without surface modification. Figure 6(b) shows cross section TEM image of a laser crystalized island.

The printed dopant inks on the cross points of the active islands and gates stayed at the printed area with good definition, as shown in figure 7(a). Since the inkjet-printed dopant lost its doping capability in air, printing and soft cure operations were performed in a glove box in nitrogen ambient. After the thermal activation, the typical sheet resistance of the P+ doped silicon was 1 kΩ cm\(^{-2}\), while that of the N+ doped silicon was 300–500 Ω cm\(^{-2}\).

The XRD spectrum of the palladium chloride inks spin coated on silicon film after the silicidation anneal is shown in figure 7(b). As evident from the phase diagram of palladium-silicon system [26], the peaks at 2 theta values of 38.1°, 41.4°, 42.4° and 48.5° can be assigned to the reflection of (201), (210), (300) and (110) planes of hexagonal Pd\(_2\)Si. The ink with ethylene glycol shows a strong palladium peak compared with that containing diethyl glycol butyl ether or commercially available palladium precursor (palladium film, Emulsitone company). Functional via chains using Pd\(_2\)Si and aluminum interconnects were similarly demonstrated with contact resistances of approximately 10\(^{-6}\) Ω m.

The approximate diameter of the synthesized silver NP was 5 nm in tight distribution as shown in figure 7(c). The formulated silver NP ink was extremely stable for day-to-day jetting compared with commercial inks, as it does not contaminate the nozzle plate. The typical resistivity of the heat-cured silver film was 10 µΩ cm.

### 3.2. TFT characterization

An optical microscope image of an entirely inkjet-printed N-type MOS (NMOS) TFT is shown in figure 8(a). Since this was an early stage study for
Figure 7. (a) Printed dopant on the active island and silicon gate, (b) XRD spectra of the spun palladium chloride ink after reduction in the forming gas, and (c) TEM image of silver NPs.

Figure 8. (a) Optical image of an entirely ink-jet printed NMOS polysilicon TFT, (b) The \( I_d - V_g \) characteristics for the PMOS TFT and (c) NMOS TFT.

The proof of concept, device sample statistics were limited.

The channel width is approximately 60 \( \mu \)m. Saturation conditions were used to extract the device parameters using a gate capacitance density equivalent to 35 nm SiO\(_2\) of approximately 98.6 nF cm\(^{-2}\) (SiO\(_2\) \( \sim \) 3.9). The field-effect mobility of NMOS TFT was determined to be 80 cm\(^2\) V s\(^{-1}\). There was no observable hysteresis though this was not recorded. Due to the early nature of development, the all printed TFTs were limited by bulk leakage through the semiconductor which was indeed higher than conventionally produced poly-Si TFTs. Gate leakage was not recorded but was verified to be significantly lower than the reverse leakage. We demonstrated that the mobility of the entirely printed polysilicon TFTs can reach the range of the mobilities of common low-temperature polysilicon (LTPS) TFTs, which are fabricated using conventional vacuum deposition and lithography processes. Complementary MOS (CMOS) TFTs were then fabricated using NMOS and P-type MOS (PMOS) TFTs. The drain current–gate voltage \( (I_d - V_g) \) curves of the best-performing TFT pairs are shown in figures 8(b) and (c). The areas and mobilities of the typical good performing NMOS TFTs were 50–110 cm\(^2\) V s\(^{-1}\), while the mobility of the best PMOS was 14 cm\(^2\) V s\(^{-1}\) (table 4). The mobility and on/off ratio of the PMOS TFTs were worse than those of the NMOS TFTs. This is attributed to the P\(^+\) dopant ink, which was more sensitive to the duration in air, as it degraded the doping capability resulting in a higher sheet resistance. For comparison table 5 shows TFT characteristics fabricated using other solution-base processes. The mobility of the entirely inkjet printed polysilicon TFTs were significantly better than any of others shown TFTs. High performance CMOS TFTs with mobility more than 100 cm\(^2\) V s\(^{-1}\) is required to fabricate logic circuit [27]. This result demonstrates the future potential of printed electronics.

| TFT Type | PMOS | NMOS |
|----------|------|------|
| Mobility (cm\(^2\) V s\(^{-1}\)) | 14 | 110 |
| \( V_t \) (V) | \(-2.3\) | 2.3 |
| Slope (V dec\(^{-1}\)) | 0.68 | 0.58 |
| On/Off \( \times 10^3 \) | \(3 \times 10^{-4}\) | \(3 \times 10^{-4}\) |

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4. Conclusion

In this study, entirely inkjet-printed polysilicon TFTs were fabricated using a unique set of newly developed inks. No ion implantation, vacuum deposition or lithography techniques were used for any part of the process flow. CMOS TFTs were successfully fabricated, and the mobility of the best-performing NMOS TFTs was between 50 and 110 cm² V⁻¹ s⁻¹, but it was 14 cm² V⁻¹ s⁻¹ for the PMOS TFTs, which was due to P+ ink sensitivity to moisture; further research could improve these results. We demonstrated that the mobility of these printed polysilicon TFTs exceeded any other fully ink-jet printed TFTs such as CNTs, organic or metal oxide and were able to reach the range of the mobilities of common LTPS TFTs, which are fabricated using conventional vacuum deposition and lithography processes.

The future for printed electronics is promising. Not only have we demonstrated that high enough mobility can be achieved for RF logic circuits but since this study was completed, high frequency enabling gate lengths down to 1–10 μm range with inkjet and other printing techniques [28, 29] have also separately been achieved. In future articles we will shift our focus forward to manufacturing issues addressing fabrication and yield, which need to be improved, and address new developments for low capital cost, low step count manufacturing enabling volume production.

Data availability statement

The data that supports the findings of this study are available within the article.

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Conflicts of interest

There are no conflicts of interest to declare.

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Table 5. TFT properties of TFTs fabricated from liquid phase materials.

| Semiconductor film | Precursor | Deposition technique | Mobility (cm² V⁻¹ s⁻¹) | Vt (V) | Slope (V dec⁻¹) | ON/OFF | Reference |
|--------------------|-----------|----------------------|------------------------|--------|-----------------|--------|----------|
| Polysilicon        | Poly-CPS  | Ink-jet/laser        | 110.0                  | 2.30   | 0.58            | 3 × 10⁻⁴ | This work |
| Polysilicon        | Poly-CPS  | Grain-filter/laser   | 597                    | 1.7    | 0.21            | 1.7 × 10⁻⁸ | [9]      |
| Organic            | DNTT      | Ink-jet              | 1.6                    | No data| No data         | 2.2 × 10⁻⁷ | [1]      |
| IGZO               | Sol-gel   | Spin                 | 13.0                   | 3.30   | No data         | 1.7 × 10⁻⁴ | [4]      |
| CNT                | CNT       | Ink-jet              | 37.7                   | No data| 0.16            | 10⁷     | [6]      |

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ORCID iD

Mao Ito †https://orcid.org/0000-0002-2776-5244
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