Development, Characterization, and Processing of Thin and Thick Inkjet-Printed Dielectric Films

Bijan K. Tehrani^a, Chiara Mariotti^b, Benjamin S. Cook^c, Luca Roselli^b, Manos M. Tentzeris^a

^a*School of Electrical and Computer Engineering, Georgia Institute of Technology, Atlanta, USA*

^b*Department of Engineering, University of Perugia, Perugia, Italy*

^c*Kilby Labs, Texas Instruments, Dallas, USA*

Abstract

This work introduces the material and electrical characterization of two dielectric inks for use with inkjet printing fabrication and the realization of fully-printed multilayer electronic structures. The dielectric inks are categorized by the thickness of their per-layer profiles, where SU-8 polymer and poly(4-vinylphenol)-based solutions are utilized to realize thick ($> 4 \mu\text{m}$) and thin ($< 400 \text{ nm}$) inkjet-printed dielectric films, respectively. The material formulations for each ink are outlined in detail in order to achieve the desired viscosity and surface tension for optimal printing with a Dimatix inkjet printing system. Once printability and processing techniques are tuned and established, various material and electrical characterizations are performed, including printed profile measurement, multilayer profile tendencies, thermal reflow processing, UV-ozone surface energy modification, relative permittivity extraction, leakage current density, and dielectric breakdown voltage. Finally, the demonstration of fully-printed post-processed on-chip capacitors utilizing both thin and thick dielectric inks in conjunction with a silver nanoparticle-based metallic ink is presented and compared with other inkjet-printed capacitors.

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1. Introduction

Throughout the past decade, inkjet printing has gained extensive popularity as a fabrication method for electronic devices throughout a multitude of research and industrial fields. Using a drop-on-demand material system deposition, inkjet printing allows for a fully additive processing technique of electronic materials. This additive process contrasts typical methods of electronic fabrication, eliminating the need for cleanroom environments, hazardous chemical waste, and expensive photolithography masks and techniques [1].

Recent progress in the development of inkjet-printable ink formulations has led beyond purely conductive inks to the realization of printable dielectric films. Dielectric films are essential to the fabrication of any printed electronic device and are generally divided into two classifications based on their deposited thicknesses: thin ($< 1 \mu\text{m}$) and thick ($> 1 \mu\text{m}$). Thin

dielectric films are essential for printed low-profile components such as metal-insulator-metal (MIM) capacitors and organic transistor gates [2, 3, 4], while thick dielectric films are used to process selectively-patterned dielectric substrates [5, 6, 7]. Combining conductive nano-particle based inks with dielectric inks, inkjet printing can be used to fabricate complex bottom-up electronic structures in a purely additive process [8].

The deposition of thick dielectric films is integral to a wide variety of electronic applications. The inkjet-printing of thick dielectric structures allows for the realization of multilayer device fabrication through selective patterning and deposition. Dielectric spacers find use in such multilayer structures as antennas, substrate integrated waveguides (SIWs), and via interconnects [9]. With large-scale thick film deposition, electrically diverse dielectric substrates can be printed on virtually any host substrate.

Commonly used as a photoresist in integrated circuit (IC) fabrication, SU-8 is a long-chain polymer that is deposited through spin-coating onto silicon wafers for high-aspect-ratio electronic device patterning [10, 11, 12]. Recent advances in inkjet printing have allowed for this polymer to go beyond its

Email addresses: btehrani3@gatech.edu (Bijan K. Tehrani), chiara.mariotti@studenti.unipg.it (Chiara Mariotti), benjamin.cook@ti.com (Benjamin S. Cook), luca.roselli@unipg.it (Luca Roselli), etentze@ece.gatech.edu (Manos M. Tentzeris)

typical cleanroom use as a sacrificial mask towards a thick film dielectric for microwave devices. SU-8 polymer chains have two advantages for inkjet printing fabrication: low-temperature UV cross-linking (compared for instance to dielectric ceramics [13]) and high polymer content by weight while still maintaining a relatively low viscosity. Low-temperature processing is useful when dealing with organic substrates typically used with inkjet printing that may deteriorate at higher temperatures, such as paper and liquid crystal polymer (LCP). High polymer content allows for a thicker, more uniform layer-by-layer deposition, while low viscosity is required to be compatible with industrial inkjet printer cartridges.

The deposition of thin dielectric films is a fundamental feature for fabrication processes as it is needed in several situations, especially in multi-layer technologies. Thus, inkjet printing of thin dielectric films is a big achievement as it allows for the prototyping of dielectric coatings, capacitive coupling, and any device in which thin insulators are needed (i.e. MIM capacitors [14]). Moreover, it is worth noting that inkjet printing is a robust fabrication method that allows the combined deposition of thin and thick dielectric films based on the application. For instance high Q inductors with electrical bridges are realized by printing thick SU-8 films, and high Q capacitors are fabricated with thin insulators. These two discrete components can be combined to realize the high Q filters and resonators used in many electronic systems.

Thin dielectric layers are realized through the utilization of two polymer inks based on poly(4-vinylphenol) (PVPh) in different ratios in order to vary the permittivity (ϵ_r) of the printed film [15, 16]. The solvent used to mix the solution is the Hexanol-6 while PVPh, which is a long-chain polymer, is cross-linked by Poly(melamine-co-formaldehyde) (PMF). It is important to underline that both PVPh and PMF have dielectric properties that contribute to the features of the final inks.

In order to pronounce the functionality of thick and thin inkjet-printable dielectric films, complete material characterization is performed in this work for two polymer-based inks. Chemical formulation is outlined in detail along with post-deposition processing techniques. Printed profiles are presented to outline surface uniformity characteristics. Surface treatment methods are investigated in order to optimize ink-to-substrate wettability for printed multilayer structures. Electrical properties such as leakage current and dielectric breakdown voltage are extracted from printed film samples for functional electrical characterization. Finally, inkjet-printed MIM capacitors utilizing both dielectric inks are demonstrated on a lossy silicon substrate as an application of the multi-layer, vertically-integrated printing process for on-chip and package-integrated post-processing.

2. Materials

2.1. Thick dielectric ink formulation

The SU-8 thick film dielectric ink is formulated using photoresists from the SU-8 2000 polymer series provided by MicroChem (MicroChem, Newton, MA, USA). The SU-8 poly-

mer is typically used as a photo-resist in cleanroom photolithographic processes with its inclusion of Triaryl salt (SbF_6) for near-UV cross-linking. A cyclopentanone solvent ($\text{C}_5\text{H}_8\text{O}$) is also included in order to improve coating and adhesion properties upon deposition while also helping to increase the viscosity of the SU-8 polymer to that of the printable range.

In order to ensure printability with the Dimatix DMP-2800 inkjet printing platform, an ink viscosity within the range of 8–16 cP is desired. A second consideration for thick dielectric printing is the solid content of the material, where a higher solid content yields a thicker per-layer deposition. This higher solid content also results in a higher fluid viscosity, creating a printability contrast between the solid content and fluid viscosity of the resist materials. In order to achieve a high polymer solid content along with a low viscosity, a study of the viscosity of the SU-8 polymer with different weight percentages within a cyclopentanone solvent is performed. Table 1 outlines the measurements of the parametric analysis using a Gilmont falling-ball viscometer (Gilmont GV-2200).

The loading of different polymer contents of the SU-8 resist within the cyclopentanone solvent effectively tunes the viscosity and solid content of the resulting ink. The parametric analysis presented in Table 1 yields an optimal SU-8 polymer weight percentage of 35% with an ink viscosity of 13 cP. Further measurements of the prepared thick film dielectric ink yield a surface tension of 30 mN/m. These material properties are within the required ranges of printability for the Dimatix system, which allows for printed profile examination.

Table 1. Viscosity versus SU-8 polymer content within cyclopentanone solvent for SU-8 ink formulation.

Polymer w/w% in $\text{C}_5\text{H}_8\text{O}$	Viscosity (cP)
0	2
29	7.5
31	8
33	9
35	13

2.2. Thin dielectric ink formulation

In order to investigate the versatility of printing thin dielectric films, two PVPh-based dielectric inks are formulated. For simplicity they are referred to as ink1 and ink2. The recipe is composed of the following: poly(4-vinylphenol) (PVPh), a long-chain polymer (Product No. 436216, Sigma Aldrich), poly(melamine-co-formaldehyde) (PMF), a heat activated cross-linker (Product No. 418560, Sigma Aldrich), and finally the solvent 1-Hexanol.

Ink1 and ink2 have a different ratio by weight of PVPh and PMF, while the ratio between the solvent and the polymers is kept constant to 17 : 1 w/w%. In particular, ink1 is characterized by 1 : 10 w/w% ratio while ink2 is formulated with 1 : 1 w/w% ratio, between PVPh and PMF. Both PVPh and PMF are

Table 2. Recipes (ratio by weight) for the PVPh-based ink variations.

Ink	PVP	PMF	Hexanol
1	5%	0.5%	94.5%
2	2.75%	2.75%	94.5%

polymers with dielectric properties, so changing their ratio allows for the tuning of the permittivity (ϵ) and the loss tangent ($\tan\delta$) of the printed thin-film layer. In Table 2 the recipes (ratio by weight) of ink1 and ink2 are reported.

As previously stated, several tests are needed in order to verify the inks' printability with the desired Dimatix DMP-2800 printing platform. First, viscosity and the surface tension are measured for both PVPh inks. Viscosity is measured as 8.93 cP and 13 cP for ink1 and ink2, respectively. Surface tension is measured as 39 mN/m and 29.3 mN/m for ink1 and ink2, respectively. The requirements for the DMP-2800 are thus satisfied for both the viscosity and the surface energy and the inks are ready for profile tests.

3. Experimental

3.1. Thick dielectric ink processing

With the printability of the SU-8 ink established, 25 mm² square sample films of the SU-8 dielectric ink are printed directly onto a glass slide. Printing is performed with the platen at room temperature and a drop spacing of 20 μ m. Once a layer is deposited, the sample is steadily heated from 60 to 120 °C over a time of 10 minutes. Following the pre-exposure bake, the sample is exposed to 170 mJ/cm² of 365 nm ultraviolet (UV) light using a UVP CL-1000 Ultraviolet Crosslinker (UVP LLC, Upland, CA, USA). This UV dosage is derived from the recommended exposure for the deposited SU-8 thickness on a glass substrate. Finally, the sample is given a post-exposure bake at 100 °C for 7 minutes.

3.2. Thin dielectric ink processing

Once the PVP inks' printability is verified, 25 mm² square sample films of both PVP inks are printed on a glass slide to perform profile measurements in order to determine the film thickness and roughness, as well as optimize the process settings for printing. It has been verified that for both ink1 and ink2 the printer platen temperature has to be set to 50 °C, and the drop spacing should be 20–25 μ m. The curing of the printed film is performed by heating it up to 180 °C for 10 minutes on a hotplate.

3.3. Profile analysis

When fabrication is complete, the thickness profiles of the printed SU-8 and PVP film squares are measured with an Alpha-Step D-100 surface profilometer (KLA Tencor, Milpitas, CA, USA).

3.4. Total free surface energy analysis

Considering the vertically-integrated inkjet printing fabrication process, there is another parametric study that is needed in order to optimize this multi-layer technique [8]. Prior work has demonstrated that good printability is achieved when there is a difference of approximately 10 mN/m between the surface energy of the ink to be deposited and that of the substrate or latest deposited printed film [17]. UV-Ozone exposure is a pre-treatment method that increases the surface energy of the last deposited film, which is why it is important to analyze the surface energy of the film versus the UV-ozone exposure time.

A parametric sweep of UV-ozone exposure is performed with a UVO Cleaner Model No. 42 provided by Jelight (Jelight Company Inc., Irvine, CA, USA) on printed thick and thin dielectric films. The contact angle and resulting surface energy of the printed film are measured with a TL101 optical light tensiometer provided by Biolin Scientific (Biolin Scientific AB, Stockholm, Sweden) and recorded for UV-ozone exposure times up to 240 seconds.

3.5. Electrical characteristics

The use of these dielectric films requires the investigation of leakage current and electrical breakdown. Simple MIM capacitors are printed with 4 mm² silver square plates and dielectric spacers of SU-8 and PVP films with thicknesses of 7 μ m and 1.7 μ m, respectively. Leakage current measurements are performed with a Keithley 6487 Picoammeter/Voltage Source (Keithley Instruments, Cleveland, OH, USA). Applied voltage across the capacitive structure is stepped from 5–420 V and 0–120 V for the SU-8 and PVP films, respectively in order to observe the occurrence dielectric breakdown.

4. Results

4.1. Thick dielectric profile analysis

The profilometer scans of three printed samples are shown in Fig. 1. The printed samples yield a single-layer thickness of approximately 5 μ m. The average surface roughness of the films is measured to range from 20–40 nm, which is less than 1% of the overall film thickness. Good profile agreement is present between the three samples, which demonstrates the repeatability of the printing process with the SU-8 dielectric ink.

Multiple layers of SU-8 dielectric are printed in order to investigate the effect of multilayer, single-session printing on the profile of the film. Fig. 2 shows profilometer scans of 16 mm² square SU-8 patterns with 1, 3, 5, 7, and 9 consecutively-printed layers. When multiple layers of SU-8 are deposited, the profile of the resulting film changes with respect to the size of the pattern and the amount of layers printed. The profile of printed films with few layers exhibit heightened sidewalls, while the increase in deposited layers causes these sidewalls to become more significant and finally converge into a convex-profile film.

(a)

(b)

Figure 1. Profile measurements (a) and micrograph (b) of single-layer printed SU-8 films.

4.2. Thick dielectric thermal reflow

One issue with inkjet-printed dielectric films is the lack of profile uniformity across the deposited film. This can be witnessed in the single layer profile scan in Fig. 1 with the presence of the heightened sidewalls surrounding the uniform center. The phenomenon of profile variation is also present with deposition of multiple layers, where effects can increase with respect to the number of deposited layers. In order to improve the surface uniformity of a thick printed film, a thermal-based material reflow process is investigated for use with the SU-8 dielectric ink.

For reflow processing analysis, seven layers of SU-8 dielectric are printed in a 16 mm² square pattern with the typical processing parameters outlined in the previous section up to the end of the pre-exposure bake, before the UV cross-linking takes place. Once the temperature reaches 120 °C after 10 minutes, another thermal ramp to 150 °C takes place over 5 minutes. With the temperature of the printed film held at 150 °C, the profile of the film is observed for baking times ranging from 0–10 minutes. Fig. 3 displays profilometer scans of the printed films with different reflow processing times. Before reflow, the initial printed film (0 minutes) shows a large lack of profile uniformity, varying approximately 11 μm or 25%. After 2 minutes of reflow, the film exhibits a variation of approximately 2 μm or less than 5% across its effective thick-film area. For baking

Figure 2. Profile measurements for printed SU-8 dielectric films with increasing numbers of consecutively printed layers.

Figure 3. Profile measurements for printed SU-8 dielectric film with 0–10 min of reflow processing at 150 °C.

times greater than 2 minutes, the printed polymer begins to exhibit a bowing effect where the film profile becomes convex and uniformity is degraded. Through this analysis, a thermal, pre-cross-linking reflow processing yields a thick multilayer dielectric film with small profile variation (<5%) across its effective thick-film area. This multilayer deposition can be expanded to multiple session printing to yield uniform dielectrics with thicknesses exceeding 100 μm [9, 7], greatly improving upon previously demonstrated printed dielectrics reaching a maximum of 4–5 μm [18].

4.3. Thin dielectric profile analysis

Profile measurements of PVP films are shown in Fig. 4 for a single printed layer, printed with 20 μm and 25 μm drop spacings. Measurements of the 20 μm film yield thicknesses of 300 – 400 nm while the roughness is about 30 – 40 nm, approximately 10% of the film thickness. As expected, with a 25 μm drop spacing, the layers are thinner (i.e. 200 – 250 nm), while the roughness is 30 – 40 nm, similar to the previous drop spacing.

It is worth noticing that, compared to the SU-8 based dielectric inkjet printed film, the thickness of the PVP-based inks are one order of magnitude lower. This significant difference in magnitude highlights the importance of having material and

(a)

(b)

(c)

(d)

Figure 4. Profile measurement (a–b) and micrographs (c–d) of the PVPh film for ink1 and ink2, respectively.

structural versatility with inkjet printing, allowing for the deposition of a variety of film thicknesses necessary for an application. In Fig. 4(c) and 4(d) the micrographs of the inkjet printed films are also reported showing good uniformity for both ink1 and ink2.

4.4. Thick dielectric total free surface energy analysis

Fig. 5 shows the total free surface energy of an SU-8 film with parametric exposure to UV-ozone. Once a 10 mN/m differential is achieved between the printed SU-8 film and the printable ink, the surface energy is considered optimal for printing. As UV-ozone exposure time increases, the surface energy of the film saturates to approximately 45 mN/m.

Figure 5. Total free surface energy measurements versus UV-ozone exposure time for SU-8 films.

Figure 6. Surface energy measurements versus UV-Ozone exposure time for PVPh-based films.

4.5. Thin dielectric total free surface energy analysis

Figure 6 shows the measured total free surface energy for the PVPh-based printed films (for both ink1 and ink2) exposed for a time ranging from 0 and 240 seconds. As expected, the surface energy increases with the exposure time and saturates after 3 minutes at values of about 55 – 60 mN/m.

4.6. Thick dielectric electrical properties

Leakage current density measurements for applied voltages up to 125 V are shown in Fig. 7. Using the printed MIM capacitors, DC capacitance was measured in order to extract the relative permittivity (ϵ_r) of the SU-8 film. A summary of all extracted electrical properties of printed SU-8 dielectric films is outlined in Table 3. Measurements show that the printed SU-8 dielectric exhibits a small leakage current for typical operating voltages while offering a high dielectric breakdown voltage.

Table 3. Summary of SU-8 dielectric film electrical properties

Relative Permittivity (ϵ_r)	Leakage Current Density (10 V)	Breakdown Voltage
3.75	1.98 pA/mm ²	420 V

Figure 7. SU-8 dielectric film leakage current versus applied DC voltage.

Figure 8. PVP dielectric film leakage current curves versus applied DC voltage.

Table 4. Summary of PVP dielectric film electrical properties

PVP Ink	Relative Permittivity (ϵ_r)	Leakage Current Density (10 V)	Breakdown Voltage
1	3	13.2 pA/mm ²	100 V
2	5.5	9.03 pA/mm ²	120 V

4.7. Thin dielectric electrical properties

The measurements in Fig. 8 report the leakage current density (J_{leak} in A/mm²) for MIM capacitors fabricated with both ink1 and ink2: the behavior is typical as reported in [19]. The curves are reported with a DC voltage up to 50 V, however the tests are performed for V_{DC} up to 120 V demonstrating that the breakdown voltages are 100 V for ink1 and 120 V for ink2. It is important to note that the tested MIM capacitors have a dielectric film thickness of 1.6 – 1.8 μ m. A summary of all extracted electrical properties of printed PVP dielectric films is outlined in Table 4.

5. Application

In order to demonstrate the importance of dielectric films for inkjet printing processes, Metal-Insulator-Metal capacitors

Figure 9. (a) Side and (b) top views of the on-silicon capacitor model. (c) Image of the capacitor testing configuration. (d) Micrograph of the fabricated capacitors under test.

Table 5. 3D-model material characteristics for capacitor design and simulation in CST Microwave Studio.

Material	t (mm)	σ (S/m)	ϵ_r	$\tan\delta$
Brass	1	$1.6e^7$	–	–
Silicon	0.38	0.1	–	–
SU-8	0.03	–	3	0.04
Silver	0.0023	$5e^6$	–	–
PVPPh	0.001	–	5	0.025

have been fabricated on a silicon substrate. Silicon is a substrate with a difficult printability as it has surface energies with poor wettability [20]. The design is characterized by 500 μ m radius circular parallel plates separated by PVPPh-based film (ink2 in the reported case). Side and top views of the capacitor models are shown in Fig. 9(a)–(b). Table 5 summarizes the materials featured in fabrication as well as their respective parameters used to model the RF characteristics of the capacitor model in CST Microwave Studio.

Fabrication begins with the deposition of a 30 μ m thick SU-8 film, followed by the curing profile previously outlined. In this design, the SU-8 film is used as an insulation layer between the capacitor and the silicon substrate as well as a general adhesion layer. After the SU-8 is cured, a 30-second UV-ozone exposure is performed in order to increase the surface energy of the printed SU-8 film. When the surface is prepared for subsequent printing, 5 passes of silver nanoparticle ink are printed and baked at 120° C in the oven. Two minutes of UV-ozone exposure are needed before inkjet print PVPPh. Following UV-ozone exposure, PVPPh films are printed to provide the thin dielectric for the capacitor. The samples are then cured at 180° C, followed the printing of the second and final metal capacitor

Figure 10. Graphs reporting the comparison between simulated and measured (a) capacitance and (b) quality factor versus frequency.

plate. A final baking in the oven at 180° C is then performed to complete the fabrication process.

Fig. 9(c)–(d) shows an image of the testing configuration and a micrograph of the printed capacitor prototype. The printed capacitors are measured using ACP40-GSG-500 probes from Cascade Microtech (Cascade Microtech, Beaverton, OR, USA) with an Anritsu 37369 VNA (Anritsu Company, Kanagawa, Japan). The RF characteristics of the printed capacitors, including capacitance (C) and quality factor (Q) versus frequency, are reported in Fig. 10. The reported graphs show the following results: quality factor, capacitance per unit area, and self resonant frequency (SRF). The measured quality factor of the printed capacitors is approximately 23, a 300% increase from previously reported inkjet-printed MIM capacitors in literature [21, 22, 23, 2]. Capacitance per unit area is measured to be 36 pF/mm² and matches well with simulated results. The measured SRF value is within acceptable error of the simulated 800 MHz SRF value.

The results reported in this application example thus demonstrate that being able to inkjet print dielectrics with a large scale of thicknesses can address various objectives, specifically that a passivation layer should in fact be much thicker than an insulation layer in MIM capacitors.

6. Conclusion

This work outlines the material compositions, processing profiles, and various physical and electrical characteristics of two varieties of inkjet-printed dielectric films. Ink solution formulations are outlined for both thick SU-8 polymer-based and thin PVPh-based dielectric inks compatible with industrial inkjet printing platforms. The processing profile of the reported ink formulations are then outlined to ensure the reliable patterning of these dielectric films. Printed profiles are then investigated, yielding per-layer thicknesses as low as 200 nm and as high as 5 μ m for the PVPh and SU-8 inks, respectively. The effects of thick multilayer SU-8 prints is investigated, offering a solution of thermal reflow processing to improve the profile uniformity of printed films exceeding 100 μ m in thickness. Surface energy adjustment profiles with UV-ozone exposure are outlined for the printed dielectric films, yielding tunable ranges from 30–45 mN/m and 30–60 mN/m for SU-8 and PVPh films. The electrical characteristics of these printed dielectrics are investigated yielding high breakdown voltages of 420 V (SU-8) and up to 120 V (PVPh) as well as leakage current density measurements with 10 V DC bias at 1.98 pA/mm² (SU-8) and as low as 9.03 pA/mm² (PVPh). As a final demonstration, an application of both thin PVPh and thick SU-8 dielectric films is presented with fully post-processed on-silicon RF capacitor. The measured results of the inkjet-printed capacitor on a lossy silicon substrate yield a Q value higher than other demonstrations in literature while also exhibiting excellent matching with simulation data.

The two dielectric inks presented in this work have the potential to be further integrated into the ever-expanding field of inkjet-printed electronics. Thin dielectric inks can be used to create on-chip passive components, such as decoupling capacitors, and can also offer thin insulation films for low-profile interconnectivity. Thick dielectric inks can be used to additively deposit bulk dielectric materials for such applications as on-chip antennas and IC die packaging and encapsulation. The further utilization and refinement of the dielectric inks presented in this work will open the door to the development of even more novel materials to be utilized in the purely additive, low-cost, and highly robust inkjet printing fabrication process.

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Graphical Abstract

Inkjet-Printed Films

Robust Multilayer Printed Electronics