Inkjet Printing of Metal-Dielectric Crossovers

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Abstract

In recent years, there has been significant interest in the inkjet printing of electronics circuits and devices. The inkjet deposition of a single functional material on a substrate is well developed, however little attention has been paid to the sequential printing of different functional elements to generate 3D structures such as crossovers and interconnections, the passive elements in electronics. Here we discuss the issues to be addressed in the optimization process in all inkjet printing of metal/dielectric/metal interconnects and crossovers. We describe the inkjet printing of a commercial silver nanoparticle conductor on glass and onto dielectric films of various thicknesses, and discuss issues to be considered when printing multilayer interconnects and crossovers with especially emphasis on printing across interfaces and onto surfaces of different composition.

Introduction

In the last few years, there has been a growing interest in the development of all printed electronics, which are expected to give rise to low-cost, flexible displays and disposable electronics. A fabrication process that does not involve expensive processes or the use of silicon substrates is highly desirable. Here we focus on the use of inkjet as the printing methodology of choice. Direct additive printing has a number of benefits including, low material usage, additive rather than subtractive processing and in principle eliminates the need for other expensive technologies such as lithography, and etching vacuum processing. Moreover, in inkjet printing there is no need for masks and therefore, pattern designs can be altered almost instantaneously since an entire circuit pattern can be designed using computer graphics software which makes inkjet printing a powerful rapid prototyping technology. Additionally, inkjet printing is an additive fabrication process which reduces the generation of substantial amounts of chemical pollutants since materials are only deposited in the desire locations. These advantages are expected to result in a substantial reduced production costs making inkjet printing technology suitable for the fabrication of disposable consumer products.

While various groups have produced all-printed transistors [1], [2] little work has been reported regarding the inkjet printing of passive elements such as inductors, capacitors and multilayer crossovers and interconnects [3]. In response to the growing interest in inkjet printing of multilayer features on sensitive temperature substrates, an experimental investigation was conducted to determine the feasibility of drop-on-demand (DOD) technology to fabricate horizontal fine line conductors and pin-hole free dielectric films for the fabrication of multilayer interconnect features used in electronic components and devices. One of the main problems associated with inkjet printing of functional materials, whether dilute solutions, dispersions or UV curable, is that the deposited line can display a morphological phenomenon known as “coffee stain” [4] in which a significant amount of material accumulates at the edge of the droplet or track leading to very uneven surfaces. For conductor development “coffee staining” is a crucial issue since it results in the production of rough features with high resistivity (tracks) or high sheet resistance (films). Due to their roughness, these tracks and films are generally unsuitable for use in multilayer interconnects structures, since the overlaying dielectric are likely to have pin-holes due to the poor coverage of the numerous spikes and valleys.

In this paper we describe the optimization of the rheological properties of commercial solutions in order to obtain jettable inks compatible with a range of substrates. A silver nanoparticulate ink capable of producing conductive features at annealing temperatures of 150 ^{o}C was employed. The effect of surface treatment before printing on the morphology and topology of printed features is analyzed. Multilayer interconnects were printed on glass slides. Their morphology as well as their electrical properties will be discussed.

Experimental

Instrumentation

A Dimatix DMP-2800 inkjet printer (Fujifilm Dimatix, Inc., Santa Clara, USA) was used in the study using a disposable piezo "ink jet" cartridge. This printer can create and define patterns over an area of about 200 x 300 mm and handle substrates up to 25 mm thick being adjustable in the Z direction. The temperature of the vacuum platen, which secures the substrate in place, can be adjusted up to 60°C. Additionally, a waveform editor and a drop-watch camera system allows manipulation of the electronic pulses to the piezo jetting device for optimization of the drop characteristics as it is ejected from the nozzle. The nozzle plate consists of a single raw of 16 nozzles of 23 µm diameter spaced 254 µm with typical drop size of 10 pL. The most remarkable feature of the printer is the possibility of varying the distance between two consecutive droplets, the so-called dot spacing, parameter that has an effect in the continuity, width and thickness of printed features. For the purpose of this study, the cartridge temperature was varied in order to optimize the jetting conditions. The platen was kept at room temperature. Dot spacing was varied in order to modify film and track dimensions. The ink specification for the printer is 2-30 cPs and 25-40 mN/m [5].

Ink viscosity was measured using an Anton Parr AMVn automated micro-viscometer which is based on the rolling/falling ball principle (DIN 53015 and ISO 12058). Using a 1.8mm diameter capillary at 25 +/- 0.5 ^{o}C at an angle of 70^{o} times were determined as the average of 4 determinations (+/- 0.05 s). A KRUSS tensiometer (Drop Shape Analyzer DSA100, KRUSS GmbH, Hamburg, Germany) equipped with image analysis software was used to determine the surface tension of the inks by the pendant drop technique, and also the contact angle that a sessile drop of a certain ink formed onto the various substrates and onto layers of printed materials. Track width and thickness where determined using a Veeco Dektak 8 Stylus profilometer. It was used to asses the integrity of tracks printed across interfaces and to obtain 3D images of tracks. AFM (PSIE System, Scanwel, UK) was used to characterize the surface of printed features. In order to UV-cure the dielectric material, a high intensity UV-light mounted onto a variable speed belt system was employed (Fusion UV-curing).

Materials

The silver ink used in this study consisted of silver nanoparticles coated with a polymeric material dispersed in a mixture ethylene glycol/ethanol (Product No. AG-IJ-G-100-S1, Cabot Corporation, Albuquerque, USA). The nanoparticles have an average particle size of 30-50 nm and the dispersion had a viscosity of 14.4 cPs at 22 ^{o}C and surface tension of 31 mN/m at 25 ^{o}C. Thermogravimetric analysis was carried out on the silver nanoparticles ink and the result is shown in Fig. 1. The ink was heated at a rate of 10 ^{o}C/min. The initial mass loss observed at temperatures below 50 ^{o}C has been assigned to the evaporation of Ethanol. As the temperature increased there was another gradual loss of mass which has been assigned to the polymeric coating material. A further temperature increase implies another mass loss which has been assigned to the evaporation of ethylene glycol. The organic constituents of the nanoparticles were found to decompose at temperatures below 200 ^{o}C leaving a metallic residue, which corresponds to an initial metal content of approximately 20%. The silver nanoparticle ink was used as purchased. In order to sinter the nanoparticles ink and obtain conductive silver tracks the following procedure was followed: after printing, samples were placed on a hot plate at 150 ^{o}C for 10 min. This temperature was chosen because ultimately the aim of further research is to print crossovers and interconnects on temperature sensitive substrates and conductivity good enough for using these tracks as conductors is obtained.

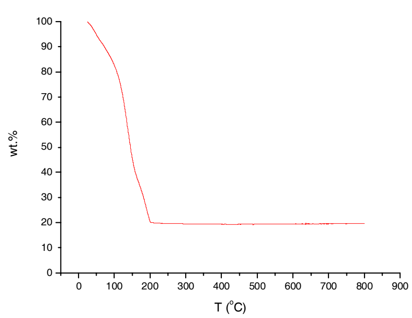

Figure 1. Thermogravimetrical analysis of Cabot silver nanoparticles ink

An UV-curable photoresist solution consisting of monomeric species, a photoinitiator and cyclopentanone as solvent (SU-8 MicroChem, Newton, USA) with an approximate solid content of 45 wt. % was employed as the insulating dielectric material. The viscosity of this solution at 25 ^{o}C was 48.1 cPs which is out of the Dimatix printer tolerable range (2-30 cPs). In order to obtain a robust jetting solution the commercial material was diluted with ethyl lactate to 15 wt.% solids solution (viscosity: 4.6 cPs at 25 ^{o}C, surface tension: 29 mN/m.).

In order to cure the inkjet printed dielectric tracks and films the following procedure was followed: (i) soft-bake step where the sample was placed in a convection oven at 90 ^{o}C for 5 min in order to evaporate the solvent; (ii) UV-cured (three passes under the UV-light at belt speed of 11 m/min) to allow the material to cross-link; (iii) hard-bake step where the sample is placed in a convection oven at 95 ^{o}C for 10 min in order to promote substrate adhesion.

Conventional microscope glass slides of 2.54x7.62 cm (Sailing Boat, Cat. No. 7101, China) and PEN (300403-523, DuPont Teijin Films, UK) were used as substrates. All substrates were ultra-sonicated for 10 minutes in an acetone bath. They were then rinsed with deionised water and placed in a convection oven at 60 ^{o}C for 10 min.

Substrates were made more hydrophilic by treatment with UV-ozone plasma (PSD-UV Ultra-violet/Ozone probe and surface cleaner, Novascan Technologies, USA) to ensure the removal of any organic contamination. The plasma treatment period varied from 5 to 20 minutes.

Results and Discussion

In order to build effective interconnects and crossovers, the dielectric insulating layer needs to satisfy various requirements. First of all, it must be pin-hole free in order to avoid cross-talking between the upper and the lower layers or tracks of conductive material. Secondly, it must be as thin as possible but thick enough to accomplish the latter requirement. The conductive silver tracks must be uniform and continuous along the whole track in order to be conductive since their role is to transmit the electrical current between two active electronic components.

Dielectric

In order to produce linear structures, the individual droplets have to be printed in such a way that consecutive droplets partially overlap. The degree of overlap in addition to the contact angle of the material on the surface and the roughness and surface energy of the surface dictates the shape, width and height of the printed track [6]. Printed tracks consisted of a single-pass line of consecutive droplets. The dot spacing was varied to study the width and thickness (height) of the obtained tracks. Microscope slides were employed as substrate. In a set of experiments, slides were cleaned only with acetone while in a second set of experiments were additionally treated with UV-ozone plasma for 20 min before printing. Profilometry analysis employed to obtain the cross-sectional profiles of dielectric printed tracks showed that the larger the dot spacing (more spaced droplets) the narrower and less thick (high) the track is. In order to obtain well-defined lines the largest suitable dot spacing was found to be 20 μm. Larger dot spacing let to “wavy” tracks. The smallest well-defined line obtained corresponded to a single-droplet line printed at 20 μm dot spacing with a width of 45 μm and height of 0.8 μm. The variation of track width with dot spacing has been reported by [6] explained the fact that track width correlates well with contact angle and printing variables such as drop spacing and adjacent dot spacing. Since, the contact angle of the dielectric ink on glass (15 deg) and droplet size (27 μm) are kept constant in these experiments (same solution, substrate, substrate treatment and jetting parameters are being used) the variation in track width are purely due to the dot spacing. In order to produce a wider track or film a series of lines whose inter-line spacing corresponds to the dot spacing were printed. Firstly, a series of tracks were printed. Without substrate UV-ozone treatment, the track height increased as the dot spacing decreased due to the greater degree of droplet overlap. Well define lines of thicknesses (heights) in the range of 2.5 μm (10 μm dot spacing) to 0.9 μm (20 μm dot spacing) were obtained. The most remarkable feature of these tracks was the absence of the dimple in the middle of the track ("coffee stain") seen when printing a cyclopentanone solution of the dielectric material. Instead, when a cyclopentanone/ethyl lactate dielectric solution was printed onto glass slides, tracks showed a rounded profile. When UV-ozone plasma cleaned glass slides were employed as substrates, as expected track height increased as the dot spacing decreased. However, track height was found to be lower than for tracks printed onto none UV-ozone substrates. Since This height decrease is primarily due to the decrease of contact angle when printing on plasma treated substrates (10 deg instead of 15 deg). Track thicknesses varied between 1.7 μm, for tracks printed at 10 μm dot spacing and 0.5 μm for tracks printed at 20 μm dot spacing. Moreover, profilometry analysis of tracks printed onto plasma treated slides revealed the typical "coffee stain" pattern for samples printed at 20 μm dot spacing. However, tracks printed at 10 μm dot spacing did not show coffee stain pattern. Homogeneous films of various thicknesses were obtained by changing the dot spacing with thicker films being obtained for greater drop overlap.

Ultimately, thin films are required for interconnects and crossovers. Therefore, based on the latter observations, substrate surface treatment was employed as a method of decreasing the contact angle of the dielectric ink on glass which should then lead to a decrease in film thickness. Acetone cleaned glass slides were additionally treated with UV-ozone plasma for 20 minutes before printing. Following the same procedure described previously, single-pass films of 3x3 mm were printed. When printing at 20 μm dot spacing films of 1.04 μm thickness were obtained. However, these films were not completely pin-hole free and they were also uneven. When printing at 15 and 10 μm dot spacing, the unevenness was much more pronounced. Profilometry analysis showed films with most of the material accumulated in the edges of the film ("coffee stain"). For instance, the thickness of the film printed at 15 μm dot spacing was 1.4 μm in the centre and 3.10 μm thick at the edges. Therefore, UV-ozone substrate treatment had the same effect as a fast drying treatment inducing migration of material from the centre to the edges of the film. On the other hand, Figure 2 compares the thickness of tracks and films printed at the same dot spacing. It can be seen that film thickness is always greater that track thickness printed at the same dot spacing. This is possibly due to the fact that for single-droplet printed track, droplet overlap only happens in the x direction (direction of printing). However, for a printed film droplet overlap happens in two directions: *x* and *y* and therefore, the resulting pattern is thicker that the one for a single-droplet track. For the building of crossovers and interconnects, dielectric thickness is not as important as it is when building TFT's. The crucial issue in both applications is the absence of pin-holes. Therefore, in the crossovers work here presented the dielectric ink printing was done at 15 μm dot spacing to ensure obtaining pin-hole free films. Moreover, UV-ozone plasma treatment was avoided unless necessary.

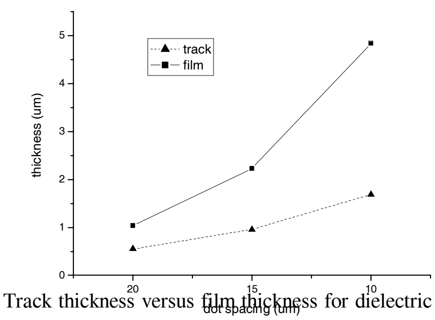

Figure 2. Track thickness versus film thickness for dielectric solution 15 wt. % solids diluted in ethyl lactate printed onto a UV-ozone plasma treated glass slide.

Conductor

Cabot silver nanoparticle ink had the appropriated dynamic viscosity and surface tension required to be used with a Dimatix printer and, therefore, was employed as purchased. Firstly, in order to study the sort of features achievable with Cabot silver nanoparticle ink a series of single-droplet lines were printed at various dot spacing onto glass slides. After printing, samples were thermally treated as stated previously. Figures 3 and 4 show the cross-sectional profile of the obtained silver tracks. As expected, the width and height of printed tracks decreased when printing at larger dot spacing.

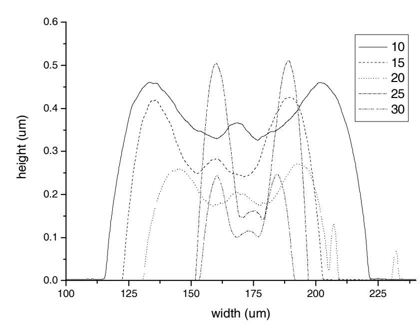

Figure 3. Cross-sectional profile of sintered silver tracks onto acetone cleaned glass slides printed at various dot spacing.

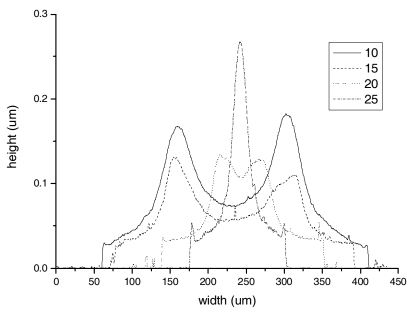

Figure 4. Cross-sectional profile of sintered silver tracks onto UV-ozone plasma treated glass slides printed at various dot spacing.

The smallest well-defined line was obtained when printing onto a none UV-ozone treated glass slide at 30 μm dot spacing with line width of 42 μm and line height of 0.15 μm. Dot spacing larger that 35 μm led to "wavy" lines and even larger dot spacing (over 45 μm) led to none-coalescent droplets. In terms of the lines profiles, all printed lines showed the typical "coffee stain" pattern due to fast drying of the tracks on the hot plate. For conductor development coffee stain is a vital issue since it results in the production of rough films with high sheet resistance. Owing to their roughness these films are usually unsuitable for use in multilayers interconnect structures, since overlaying dielectrics are likely to have pin-holes due to the poor coverage of the numerous ridges and valleys. [7] As pointed out previously, the analysis of dielectric printed tracks showed that UV-ozone plasma treatment can be used to reduce the height of printed tracks. In order to decrease the height of silver tracks ridges, glass slides were treated with UV-ozone plasma for 20 min before printing. Figure 4 shows the cross-sectional profile of silver tracks printed onto UV-ozone treated substrates. As shown in Table 1 the height of the tracks diminished when printing onto UV-ozone plasma treated substrates due to the decrease in contact angle of silver nanoparticles ink on glass from 11 deg, before plasma treatment, to < 5 deg, after plasma treatment. However, their profile still showed "coffee stain" features. It is interesting to notice that tracks printed onto acetone-only cleaned substrates showed the same profile with most of the material accumulated in the edges of the film. However, for tracks printed onto UV-ozone plasma treated substrates, the profile depends of the dot spacing with more material accumulated in the centre of the track for larger dot spacing. Moreover, track width also changed due to the better wetting (lower contact angle) of Cabot silver nanoparticle ink on plasma treated substrates. This is an important fact to consider when attempting printing very little features because surface plasma treatment compromises resolution. However, for the purpose of this work silver track resolution was not an issue and silver tracks were printed at dot spacing between 20-30 μm.

Using the data obtained from profilometry analysis the dimensions of the tracks were obtained. Areas in the range of 3.8-0.6 x 10^{-11} m^2 were obtained for tracks printed onto acetone-cleaned glass slides while areas of 3.0-1.0 x 10^{-11} m^2 were obtained for tracks printed onto UV-ozone treated glass slides.

Table 1. Comparison of printed silver tracks width and height printed on glass substrates with and without UV-ozone plasma treatment.

dot spacing (μm)	width without UV-O_3 (μm)	width with UV-O_3 (μm)	height without UV-O_3 (μm)	height with UV-O_3 (μm)
10	101	352	0.32	0.087
15	80	315	0.29	0.068
20	76	212	0.24	0.063
25	54	129	0.22	0.083
30	42	83	0.15	0.041

Crossovers

The following procedure was used to print the crossovers: (i) first silver track (bottom track) printed; (ii) single-pass dielectric film deposited; (iii) finally a second silver track (top track) perpendicular to the first one was printed on top of the dielectric film. The silver tracks must be conductive from end to end in order to act as interconnections. The dielectric must be pin-hole free in order to act as insulating material and prevent cross-talking between the top and the bottom silver tracks. To ensure the insulation between bottom and top silver tracks, the dielectric film was design to be slightly wider (2x2 mm) than the silver tracks (16x0.5 mm) so in that way the whole width of the bottom silver track should be covered by the dielectric film. Bottom silver tracks were printed at various dot spacing (25-40 μm) onto a UV-ozone plasma treated glass slide. Tracks were thermally treated as stated previously in order to allow the nanoparticles to sinter. Once the silver track was annealed a film of dielectric was printed at 15 μm dot spacing. Profilometry analysis revealed that the dielectric film thickness was uneven with more material accumulated in the edges of the film that in the centre because of "coffee stain" phenomenon which did not occur when tracks were printed onto acetone-cleaned glass. Therefore, when printing the dielectric material onto an annealed silver track, this increases migration towards the edge of the film leading to a "donut" type of structure. Initially this phenomenon was attributed to the dielectric material adopting the shape of "coffee stain" pattern of the silver track underneath (bottom track). In order to verify the latter hypothesis, a track of dielectric material was printed onto a film of silver. The silver nanoparticles ink was printed onto an acetone cleaned glass slide at 20 μm dot spacing and annealed using on a hot plate at 150 °C for 10 min which resulted in a film thickness of approximately 300 nm. Although the film surface was slightly rough, most of the material was evenly distributed and none ridges or valleys were found across the film. The dielectric track was printed at 15 μm dot spacing onto the silver film. Figure 5 shows 2D and 3D images of the dielectric track across the interface glass-silver film. It can be seen that the dielectric tracks suffers from "coffee stain" when printed onto the silver film but *not* when printed onto glass as previously described. This is a consequence of the higher surface energy of the silver track relative to the UV-ozone plasma treated glass.

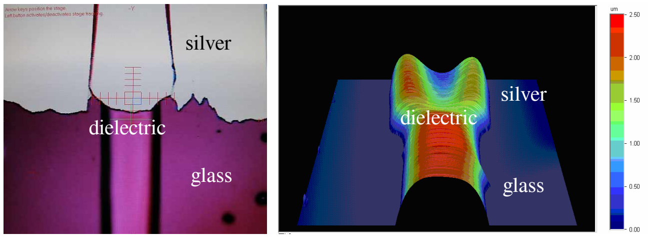

Figure 5. 2D (left) and 3D (right) images of fully cured dielectric track of a solution of 15 wt.% solids diluted in ethyl lactate printed across the interface of an acetone cleaned glass slide and an annealed silver film of 0.31 nm thick

This change in surface energy is responsible for the loss of line integrity when printing across the interface between glass and the silver film. When printing crossovers, "coffee stain" pattern was observed for dielectric films printed from 10 to 20 μm dot spacing. In order to reduce the height of the ridges one could think about treating the silver track surface with UV-ozone plasma before printing the dielectric layer however, the contact angle of dielectric ink onto silver film is already very low (approximately 7 deg). Moreover, it is known that UV-ozone plasma treated silver metal surfaces can tarnish due to the formation of Ag_2O, which decreases silver conductivity. [8] Therefore, for further experiments in order to build multilayer crossovers, the dielectric film was deposited at 15 μm dot spacing onto the first silver track without silver UV-ozone treatment. According to contact angle measurements carried out on fully cured dielectric films, it was seen that the contact angle of a drop of Cabot silver nanoparticle ink onto the dielectric is approximately 57 degrees indicating that Cabot silver nanoparticle ink had a poor dielectric surface wetting. This was confirmed by a parallel experiment in which a track of Cabot silver nanoparticle ink was printed across a film of fully cured dielectric. The silver track was not continuous but instead, a series of silver ink agglomerates were found onto the dielectric film. This result indicated the need of UV-ozone plasma treatment of the dielectric film before printing the top silver track of the crossover. In order to minimize the oxidizing effect of the ozone plasma treatment on the bottom silver track, the latter was covered with a glass slide. Then, the dielectric film was UV-ozone plasma treated for 5 minutes. According to contact angle measurements, the latter exposure time was enough to decrease the contact angle of Cabot silver ink onto a film of dielectric from 57 to 13 degrees. UV-ozone plasma exposures longer that 20 minutes made the dielectric turn yellow indicating over-curing. The top silver track was printed onto the dielectric at various dot spacing and finally annealed on a hot plate. According to profilometry analysis data the bottom silver track was found to be continuous across the whole track for all the dot spacing employed. However for all print conditions the top silver tracks was found not to be continuous across the whole track. Discontinuity was mainly observed in the portion of the track printed onto the dielectric film. Figure 6a shows an image of the bottom silver track printed and Figure 6b shows a the top silver track printed at 20 μm dot spacing onto the dielectric film. Profilometry images showed that there is a correlation between the degree of discontinuity (cracking) found in top silver track and the dot spacing employed for printing with larger dot spacing leading to more discontinuous tracks. The nature of this "cracking" needs further investigation but it is known to be a very common problem when building multilayer metal-dielectric features. The most probable reason is the differing thermal expansion coefficients between the dielectric and the top silver track lead to stress cracking during the annealing stage.

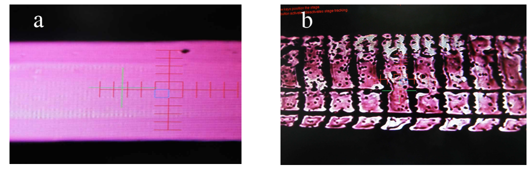

Figure 6. Image showing bottom (a) and top (b) annealed silver tracks of the crossover. Top silver track onto the dielectric film.

The resistance of the continuous bottom silver tracks of the crossovers was determined by the 4-point probes technique. The values recorded varied slightly according to the dot spacing employed to print the silver tracks with slightly higher resistance obtained for thinner tracks printed at larger dot spacing. The influence of line dimension on the resistance of metal interconnects has been reported previously. [9] Cross sectional areas were found to be in the range of 0.97-1.86 x 10^{-10} m^2, with a track length of 16 mm. Resistances varied from 28 to 116 ▯. Resistivity calculated for the sintered tracks annealed at 150 ^oC were found to be in the range 3.3-7.0 x 10^{-7} ohms·m which corresponds to approximately 2-5% the reported resistivity of bulk silver (1.6 x 10^{-8} ohms·m). This is due to the fact that the sintering temperature employed, 150 ^oC which was chosen to be subsequently compatible with plastic substrates, is too low for the silver nanoparticles to be completely annealed. This is supported by the TGA data which showed that at 150 ^oC the ink has not been converted to metallic silver and, also by AFM images which showed the granular nature of the sintered tracks due to the existence of not fully sintered nanoparticles.

Conclusions

The study highlights the difficulties encountered in the direct inkjet printing of multilayer structures comprising materials of widely differing surface wetting characteristics and thermal expansion behaviour. The definition of such processes requires fixing the fundamental electronic materials properties of each component first in order to be fit for purpose. As shown these will have serious implications towards the fabrication process, where optimization of each print step can materially alter the properties of both the previous and subsequent layers.

Acknowledgements

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