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Ink Jet Printed Silver Lines Formed in Microchannels Exhibit Lower Resistance Than Their Unstructured Counterparts

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Abstract. *This article reports on a number of experiments that have been performed, which show that the resistance of silver lines formed in channels is lower than silver lines that have been formed on unstructured surfaces. Channels were formed either by being cut into polyimide (Kapton) using a laser or by hot embossing a polycarbonate blend (Bayfol). An ink jet printer was used to dispense silver-containing ink over the embossed channels, the laser cut channels and over unstructured Kapton and Bayfol to allow comparison. Two types of silver-containing ink were used, one was a nanoparticle (NP) ink and the other was a metallo-organic decomposition (MOD) ink. For the NP ink, a decrease in resistance was seen for the lines formed in hot embossed channels. For the MOD ink, the resistance decrease was seen for lines formed in both the embossed and the laser cut channels. © 2011 Society for Imaging Science and Technology.*
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INTRODUCTION

The combination of hot embossing with ink jet printing to produce lines with controlled morphologies and widths that ranged from 5 to 15 μm has recently been reported.[1] The experiments discussed in that report are concerned with a silver nanoparticle suspension (Harima Nanopaste) that was ink jet printed over hot embossed channels. It is found that the conductivity of the embossed tracks is 17–20% the value of bulk silver. This value of conductivity is found to be higher than the values of 5% that are obtained from freestanding tracks, which are formed by ink jet printing the same ink onto unstructured substrates. It is postulated that the higher conductivity might have resulted from an increased level of crystal packing, which in turn might have resulted from the tracks forming due to capillary filling. This contribution expands upon that observation and presents a more detailed investigation into the use of embossed channels.

The use of hot embossing in combination with ink jet printing arose as a solution to ink jet printing's limited resolution. As a general rule, droplets produced by an ink jet printer tend to have diameters that are similar to the nozzle that ejected them,[2] although some size tailoring can be achieved by varying the jetting voltage.[3–5] Moreover, once ejected the final print quality is determined by the interactions that droplets have with the substrate.[6] For a printed liquid line to be stable on a surface there should be some form of contact line pinning to resist the tendency for an unconstrained liquid deposit to seek a minimum surface shape.[7] A number of phenomena such as bulging on low-surface energy substrates[7] and coffee staining on high-energy substrates[8] can occur, resulting in irregular morphologies. Although a number of strategies are available that are correct for these phenomena,[9] their occurrence tend to inhibit the use of ink jet printing in mass production.

Hot embossing consists of a master, bearing the negative impression of the feature to be formed, being pressed

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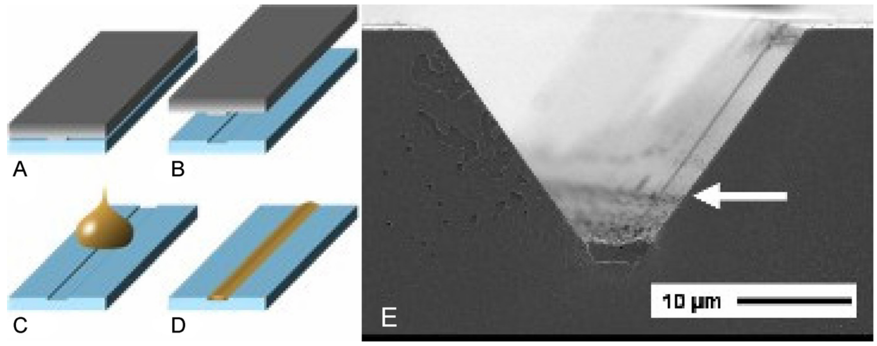

Figure 1. The scheme to the left shows the steps involved in producing silver lines in embossed channels. First, a polymer is heated above T_g, before a master is pushed into it (a). After removal of the master (b), an ink jet printed droplet is accurately placed on the formed channel (c), which is then filled through capillary forces (d). The SEM image on the right shows a single track (e) made by dispensing a nanoparticle ink into a hot embossed polystyrene structure prepared from a 42 μm pitch line master. (From Ref. 1.)

into a thin film of a thermoplastic polymer that is held above its glass transition temperature for a few minutes. The technique has a high degree of resolution; circular features with diameters of 25 nm have been fabricated.[10] Hot embossing has a number of advantages, such as the straightforward location of the newly formed features and that the master can be reused to prepare samples, which helps to minimize processing times and allows for automated mass-production, and therefore enables costs to be reduced. Furthermore, the use of polymeric materials in hot embossing follows on from recent investigations, which produced conductive silver tracks on flexible substrates using ink jet printing.[11] Figure 1 shows how narrow silver lines can be made by combining hot embossing and ink jet printing.[1]

The appeal of ink jet printing lies in its ability to produce features with a reduction in the overall number of processing steps; with the technique being increasingly used by researchers to form conductive tracks.[12] The technique is versatile and has found good uptake in areas as diverse as proteins,[13,14] ceramics,[15] and light-emitting devices.[16] Although ink jet printing cannot be considered as the sole means of production when one considers the requirements that need to be met in the fabrication of printed electronics,[17] it is an excellent means of dispensing controlled aliquots at predetermined locations.[3,13]

Lines can be formed in hot embossed channels by dispensing droplets over them using an ink jet printer. Droplets are drawn into the channels and fill them by means of capillary action. In order for the tracks to be filled, the contact angle of the deposited ink must be sufficiently low, as has been discussed earlier.[1]

In this article, a number of experiments which have been performed are reported. First, a more thorough comparison is made between silver lines prepared on an unstructured surface with lines formed in hot embossed channels in the same substrate to determine if the previously made observation of reduced resistance can be confirmed.[1] This experiment used a commercially available nanoparticle (NP) ink. A second experiment is performed using a commercially available metallo-organic decomposition (MOD) silver ink in place of the NP ink since this type of ink is a promising alternative.[18] The use of the MOD ink also allowed the investigation of whether decreased resistance is due to denser particle stacking since the MOD ink does not contain nanoparticles. The method of forming channels was then changed, with laser ablation being used to score channels into polyimide. Both MOD and NP inks are used with the laser cut channels.

Figure 2. Schematic cross-sections of the three types of line discussed: (a) free-standing or unstructured lines, (b) lines formed in hot embossed channels, and (c) lines formed by laser ablation.

EXPERIMENTAL

The differences between the three types of lines discussed in this article can be seen in Figure 2, which is a cartoon showing the approximate cross-sections of the line types. The cross-section of the line seen in Fig. 2(a) is the usual type of ink jet printed line formed on an unstructured substrate; it is described as a freestanding or an unstructured line. The cross-sections of the lines in Figs. 2(b) and 2(c) are those in a hot embossed channel and a laser cut channel, respectively. All of the substrates were cleaned with 2-Propanol before printing.

Inks

Two types of ink were used in this study. The NP ink was a 20 wt. % suspension of silver nanoparticles, whose sizes ranged between 20 and 50 nm, dispersed in ethanol and ethylene glycol, which was purchased from Sun Chemical (U5603, Sun Chemicals, Slough, GB). The viscosity and surface tension of the ink were 11.5 mPa s and 29 mN m^{-1} at room temperature, respectively. The contact angle at room temperature formed by the NP ink was 36° on Kapton, 32° on Bayfol and 34° on glass.

The MOD ink was a solution of silver neodecanoate dissolved in xylene and was purchased from ABCR (ABCR

Gmbh. KG Karlsruhe, Germany). The viscosity and surface tension of the ink were 1.13 mPa s and 24 mN m^{-1} at room temperature, respectively. The contact angle at room temperature formed by the MOD ink was $<5°$ on Kapton, Bayfol, and on glass. (For more information on this type of ink, such as thermal gravimetric analysis, the reader is directed to the work of Dearden et al.[18])

Hot Embossing

The hot embossed channels were produced by the controlled pushing of a customised silicon calibration grating, referred to as a master, into 200 μm thick polycarbonate blend (Bayfol, Bayer Material Science, Germany; glass transition temperature, Tg = 205°C), which was heated to ~60°C above its Tg, for 3 min using a small press (Tribotrak, DACA instruments, Santa Barbara, CA). The as-produced hot embossed structures were not treated in any other way and were ready for use. The Tribotrak hot press consists of a lower stage and an upper counter-balanced stage that can be adjusted to accommodate substrates of different thicknesses; both stages are heatable. The interfacial pressure can be controlled by varying the mass applied to the upper stage, which in this case was 5 kg.

The dimensions of the hot embossed channels formed Bayfol were measured by a Keyence VHX-100K Digital Microscope. The depth was found to be 20 μm, the width at the bottom of the channel was 5 μm, and the width at the top of the channel was 33 μm. The distance between each channel was determined as 9 μm, and the approximate cross-sectional area of the channel was calculated to be 380 μm^2.

Laser Ablation

The laser-ablated, or laser cut, channels were fabricated by scoring channels into 125 μm thick polyimide (500 HN, Krempel, Vaihingen/Enz, Germany), which has a glass transition temperature of 385°C. A Trumpf TruMark 5000 laser cutter was used.

The measured dimensions, using the Keyence VHX-100K Digital Microscope, of a channel formed by laser cutting were ~40 μm depth and 42 μm width at the top, which gave an approximate cross-sectional area of ~1,680 μm^2.

Ink Jet Printing

The unstructured lines and the lines in the channels were formed using ink jet printing. For both inks, the Dimatix DMP 2831 (Dimatix-Fujifilm Inc., Santa Clara, CA) was used. This printer was equipped with a piezoelectric printhead cartridge (DMCLCP-11610) that dispensed droplets with a nominal volume of 10 pl. Only one nozzle was used for the printed structures that are reported here. The printing voltage for the NP ink was 22 V, and the dot spacing was 25 μm, which was the optimized setting for the continuous lines on the unstructured surface. For the MOD ink, the printing voltage was set at 35 V and a dot spacing of 40 μm, which was the optimized value for continuous lines on an unstructured surface, was used. Both the platen and the print cartridge were kept at room temperature.

Table I. Overview of the experiments performed.

Expt. number	Substrate	Ink	Line type
1	Kapton	NP	Laser cut
2	Kapton	NP	Unstructured
3	Bayfol	NP	Hot embossed
4	Bayfol	NP	Unstructured
5	Kapton	MOD	Laser cut
6	Kapton	MOD	Unstructured
7	Bayfol	MOD	Hot embossed
8	Bayfol	MOD	Unstructured
9	Glass	NP	Unstructured
10	Glass	MOD	Unstructured

Characterization

The wetting angles that the inks made with polycarbonate and polyimide were determined using a contact angle measuring system (EasyDrop, Krüss, Hamburg, Germany). The formed tracks were imaged and evaluated using light interferometry (3D Zoomsurf, Fogale, France), scanning electron microscopy (Quanta 3D FEG, FEI, Eindhoven, Netherlands) and optical microscopy (VHX-100K, Keyence, Neusenburg, D). The electrical behavior was characterized using a multimeter (ESCORT 3146A, Agilent, Böblingen, Germany).

Experiments

Table I shows the experiments that were performed in the course of this investigation. In order to make a valid comparison all of the lines were printed and processed using the same conditions. However, the final printed lines exhibit different widths due to the different substrate morphologies and the difference in contact angle formed by the two inks. For example, the lines printed on the unstructured substrates (Fig. 2(a)) have a greater surface area available for solvent evaporation.

Two final experiments were performed using glass microscope slides as the substrate. These two experiments used either the MOD or the NP ink and were intended to serve as a benchmark for the overall resistance of the ink. Naturally, the same sintering conditions were used. The sintering conditions for all of the experiments involved the samples being laid upon a hotplate for 280 min at 150°C. In all experiments, except five and six, single layered lines were used. The reason why experiments five and six differ is given in the section Results and Discussion.

RESULTS AND DISCUSSION

Resistance

All of the lines were sintered using the same conditions in order to make a valid comparison. Moreover, the reference lines, those printed on unstructured polymer, were printed using the same printing conditions, such as dot spacing in order to make as valid a comparison as possible. The resistances, given in terms of Ohm/meter are given in Table II.

Table II. Measured resistances for the various types of line, the figure in parenthesis shows how many measurements were made.

	Laser cut (kΩ/m)	Hot embossed (kΩ/m)	Unstructured (kΩ/m)
NP on glass	—	—	5.2
NP Kapton	22.4 ± 2.3 (37)	—	21.2 ± 4.3 (37)
NP Bayfol	—	21.4 ± 1.7 (12)	39.2 ± 4.0 (12)
MOD on glass	—	—	55.4
MOD Kapton	11.2 ± 1.2 (21)	—	118.4 ± 85 (17)
MOD Bayfol	—	35.6 ± 5.3 (12)	582.0 ± 354.6 (13)

Table III. Calculated resistivities for the various types of line (bulk silver = 1.59×10^{-8} Ω m)

	Laser cut (Ω M)	Hot embossed (Ω M)	Unstructured (Ω M)
NP on glass	—	—	0.55×10^{-7}
NP Kapton	10.53×10^{-7}	—	2.18×10^{-7}
NP Bayfol	—	1.97×10^{-7}	3.14×10^{-7}
MOD on glass	—	—	2.27×10^{-7}
MOD Kapton	1.98×10^{-7}	—	9.71×10^{-7}
MOD Bayfol	—	4.38×10^{-7}	54.71×10^{-7}

It can be seen that for the NP ink, the resistance of the line formed in the hot embossed channel is lower than that formed on unstructured Bayfol. Whereas there is little difference between the two types of line formed using Kapton as the substrate. The value of the NP ink on glass is considerably lower, which is likely due to glass being a better thermal conductor than either polymer.[6,19] It is worth noting again that the aim of these experiments was not to achieve the lowest resistance but to determine the influence of channels formed by hot embossing and laser ablation. The lower resistances seen on unstructured Kapton compared to unstructured Bayfol are thought to be due to Kapton being about 50% thinner.

The data gathered for the MOD ink show that both structuring methods have a beneficial effect in terms of lowering the resistance. In fact, the data for the structured lines are lower than for the reference printed on glass. Obviously, as a solution instead of a suspension of stabilized nanoparticles the drying kinetics of the MOD ink is different. The data for the MOD Kapton experiment involved double-layered lines, as the scatter in the single-layered experiments was too wide for the unstructured lines. As more layers are added resistance decreases as is well known[4,20]; in order to ensure a valid comparison a second layer was also added to the lines formed in the laser cut channel.

In three of the four cases, there is a marked decrease in resistance when silver ink has been jetted into formed channels. Moreover, it can be seen that there is less variation when channels are used for all four experiments. This can be explained by the fact that the channels help to control line morphology by restricting line spreading and compensating for occasional variations in surface energy on unstructured substrates. Due to the geometry of the ink in the channels, the drying time of the ink during sintering is increased. Visual inspection of the lines during sintering showed that the lines in channels stayed "wetter" in appearance longer. As the ink is remaining liquid longer in the channels, there is an increased chance for imperfections to be smoothed over. In all cases, the resistance can be further lowered by printing more layers, as can be seen in Table III and as described by van Osch et al.[21] and Meier et al.[5] Van Osch et al. also showed the resistance decreases when smaller dot spacing is used for low layer numbers. The decrease in resistance can also be explained by the improved aspect ratios of the tracks that have formed in the channels.

Resistivity

In order to develop an increased understanding of the reasons behind the decrease in resistance, SEM and light interferometry were used to obtain cross-sectional profiles. These profiles were then used to calculate resistivity using the equation $\rho = R.A/l$ (where R is resistance, A is cross-sectional area and l is length). Some of the data obtained can be seen in Figures 3 and 4. A number of cross-sections were taken for each type of line, and the average was used in the resistivity calculations, which are shown in Table III.

There was considerable variation seen, especially for the tracks prepared by laser cutting. Therefore, it is necessary to point out that the data given in Table III is provided as a rough indication. Fig. 3 shows that the NP ink did not form a clear rectangular cross-section such as been seen in earlier work;[1] and is shown in Fig. 1. The difference is thought to be due to the different NP ink that was used in this study. In the earlier study, Harima NP ink was used, which formed a much lower wetting angle to Bayfol, whereas in this study the SunTronic ink forms an equilibrium contact angle of 32°. Low angles are preferable for capillary filling, which is the mechanism thought to be in play for the tracks formed by hot embossing. At higher contact angles, the imbibition rate greatly decreases.[22] The dot spacing used may also have had an effect. In earlier work,[1] a much larger dot spacing was used which greatly facilitated capillary filling. In this study, a smaller dot spacing was used, which was necessary in order to form a valid comparison.

It can be seen from Table III, that the resistivity for both types of ink in the hot embossed channels is lower than their comparable unstructured lines. The MOD ink in the laser cut channel has lower resistivity than its unstructured counterpart unlike for the NP ink. The section Discussion discusses what may have caused these lower resistivities and what mechanisms may have had an effect during printing, drying, and sintering.

DISCUSSION

Three distinct geometries have been produced in this study. Unstructured lines were formed due to the rapid, sequential

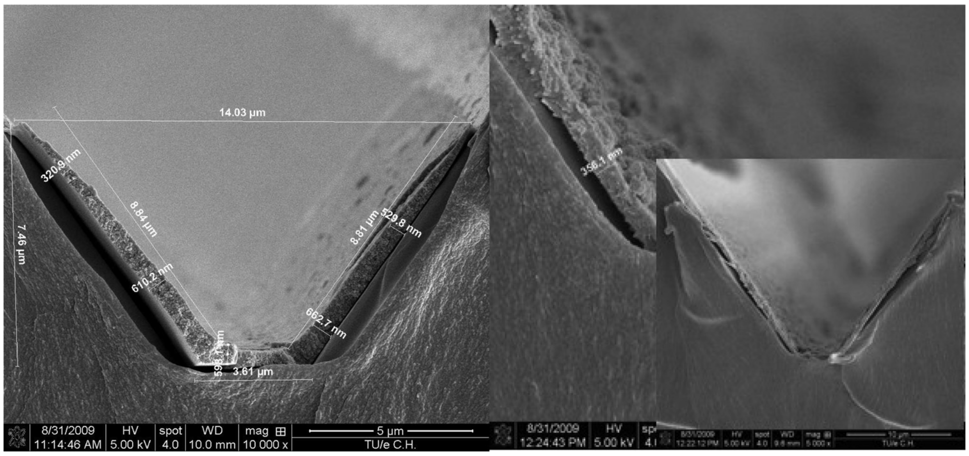

Figure 3. SEM images of the hot embossed channels in Bayfol. The image on the left (a) is of the NP ink and that on the right (b) is of the MOD ink.

placing of ejected droplets from the ink jet nozzle. The initial formed tracks can be thought of as having a flattened dome-like cross-section. The structured lines were formed in either a U-shaped channel, which was made by hot embossing, or in a V-shaped channel that was laser-ablated. All of these initial line geometries can be seen in Fig. 2. The different channel geometries for the two structured lines affect both the filling behavior of the ink. All three line types are thought to vary in terms of drying kinetics.

In all three cases, the same dot spacing was used for each ink in order to ensure that the same volume of ink per unit length was deposited. As each line type was then sintered to obtain the final conductive feature the final morphologies are expected to vary. Such a difference can be seen in Fig. 3, where the MOD ink has formed a thinner silver deposit and has covered more of the sides of the Bayfol. This difference can be explained by the much lower wetting angle formed by the MOD ink with Bayfol.

The decrease in resistance is matched by a corresponding decrease in resistivity. The NP ink in hot embossed Bayfol and the MOD ink in both the hot embossed Bayfol and laser cut Kapton have lower resistances and lower resistivities. An initial conclusion to make when confronted with lowered resistances is to assume that the cross-sectional areas are smaller. However, Table IV shows that the areas for these three experiments (NP Bayfol, MOD Bayfol and MOD Kapton) are either similar or larger.

As suggested earlier, one possible reason for the reduced resistances and resistivities seen when using structured substrates may be improved crystal packing,[1] such has also been seen in coffee stained droplets.[23] This may be due to a decreased evaporation rate as a consequence of the confined geometry of the channels in both the Bayfol and Kapton. The role of capillary filling in helping to order the deposited particles in the NP ink, and those that precipitate from the MOD ink, may also play a role, such as has been reported by Whitesides[24,25] for particles being packed into microchannels. Moreover, the control of evaporation imparted by the channels may well improve packing density, such as been reported for photonic crystal formation by Arpiainen et al.[26] and Velev and Gupta.[27] Although it is worth remarking that the primary influence on resistance is the sintering regime, the initial packing of the crystals due to the confined geometry may well help to deliver improved performance.

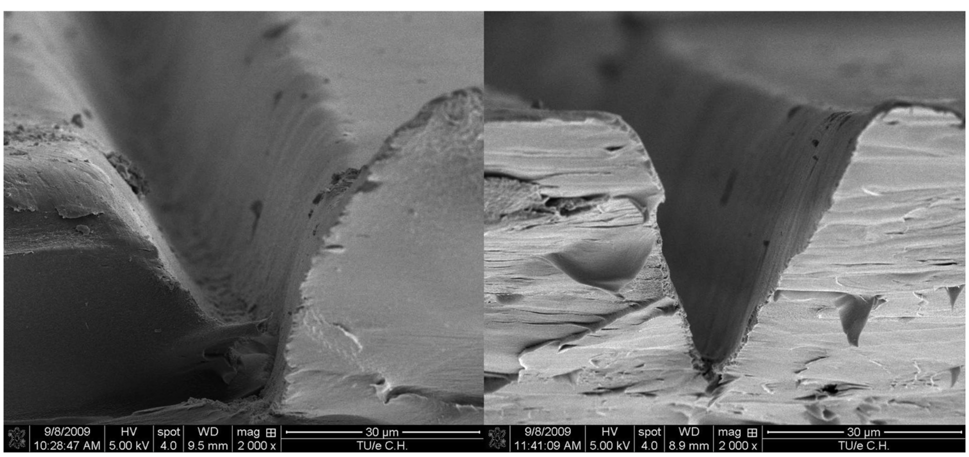

Figure 4. SEM images of (a) the MOD ink and (b) the NP ink in Kapton.

Table IV. Measured cross-sectional areas of the silver lines produced for this study.

	Laser cut (m^2)	Hot embossed (m^2)	Unstructured (m^2)
NP glass	—	—	10.6×10^{-12}
NP Kapton	47×10^{-12}	—	10.3×10^{-12}
NP Bayfol	—	9.2×10^{-12}	8.0×10^{-12}
MOD glass	—	—	4.1×10^{-12}
MOD Kapton	17.7×10^{-12}	—	8.2×10^{-12}
MOD Bayfol	—	12.3×10^{-12}	9.4×10^{-12}

Finally, the results discussed here may well be of interest to users of laser direct-write addition (LDW+) techniques too.[28] Although many of the approaches used by LDW+ deposit material onto a substrate just as with conventional ink jet printing, a variant known as "mill and fill" has been reported, which uses laser micromachining to create patterns in the surface of a substrate.[29] Here a squeegee was used to spread a paste over and into the channels with the excess being wiped away. This approach reported resistivities four times that of bulk silver.

CONCLUSIONS

A number of experiments have been performed in order to investigate if the use of embossed substrates confers an improvement in the conduction of ink jet printed silver lines. Channels are formed either by being cut into polyimide (Kapton) using a laser or in polycarbonate (Bayfol) by hot embossing. An ink jet printer is used to dispense silver-containing ink over the embossed channels, the laser cut channels and over unstructured Kapton and Bayfol to allow comparison. Two types of silver-containing ink are used, one is a nanoparticle (NP) ink and the other is a metallo-organic decomposition (MOD) ink. For the NP ink, a decrease in resistance is seen for the lines formed in hot embossed channels. For the MOD ink, the resistance decrease is seen for lines formed in both the embossed and the laser cut channels. In general, the results show that the resistance and resistivity of silver lines formed in channels are lower than silver lines that have been formed on unstructured surfaces.

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