Progress in Organic Coatings 49 (2004) 324–335

PROGRESS IN ORGANIC COATINGS

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Gravure offset printing of polymer inks for conductors

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Received 24 June 2003; accepted 26 September 2003

Abstract

A gravure offset printing process has been developed for Ag-filled polymer conductor ink. Pad printing and roller type printing have been used. Curing and electrical properties have been studied. A roller type of gravure offset printing has been used to evaluate the printing process and pad printing to print on the non-planar substrates. Based on differential scanning calorimetry (DSC) and resistivity measurements during ink curing, it was found that the ink had an optimum curing temperature of 140 °C. Square resistance of 300 and 150 μm wide lines can be as low as 20 and 28 mΩ/sq., respectively, for 7–8.5 μm thick line. The minimum line width was 70 μm. This minimum line width can be reduced with different ink solvents, but in this case the line thickness suffers and the square resistance increases, respectively.

Keywords: Gravure offset printing; Intaglio printing; Conductive ink; Square resistance; Isotropically conductive adhesive

1. Introduction

Polymer 'inks' containing metal particles are used to make conductors and to fill vias in printed circuitry board (PCB) [1], attach die-components in the form of isotropically conductive adhesive (ICA) [2,3] and anisotropically conductive adhesive (ACA) [4,5], as solder replacement [6,7] and to create conductors on solar cells [8], silicone polymer [9] or on polyimide [10]. Different metal particles have been used, such as silver (Ag), gold (Au), nickel (Ni) copper (Cu) [3], and platinum (Pt) [11,12]. Carbon has been used in resistors [13] and for electrode applications [14–16]. Polymer inks are cured between 90 and 220 °C for few minutes to hours. Curing time and temperature are dependent; long cure times enable lower cure temperatures [17]. Major challenges for polymer inks are also the mechanical and electrical strength in conditions of changing temperature and increased humidity [18]. Polymer inks are often chosen to be printed on inexpensive substrates, plastics as polyethyleneimide (PEI), which cannot be cured or fired in high temperatures. Usually printing has been done with stencils to fill vias or to create dots of inks [19]. Other methods used are screen printing [20], dispensing [21,22], flexography [23], doctoring with a blade [18,24], ink jet printing [25] and lithography [26–28]. However, the used form of gravure offset, pad printing, offers the possibility to print on non-planar surfaces and at the same time resolution can be increased [23]. Alternatively, roller type gravure offset printing offers high manufacturing speeds with controlled parameters.

In gravure offset printing, the ink is first doctored in the gravure grooves. Then a silicone polymer or rubber roller or pad picks up the ink from the gravure grooves by rotating over the gravure or by pressing on it, respectively. Then the roller or pad with the ink on it transfers the ink to the substrate with a similar movement. Pad printing differs from a roller type gravure offset in its ability to print non-planar surfaces and in the movement of the pad to the desired place on the substrate.

In order to determine an optimum ink curing time and temperature and properties, related commonly used methods have been applied [29]. Due to the epoxy components in polymer inks, differential scanning calorimeter (DSC) has been found beneficial [17,30], because chemical reactions give a higher change in calorimetric measurement than in mass. Silver powder manufacturers have been reported to use organic coatings for fine silver particles in order to prevent particle agglomeration. Silver particle coating affects such properties as curing, rheology and conductivity [31]. Low melting point metals have also been used as coatings. This kind of coating melts and forms inter-metallic bonding during curing [7].

One goal with conductive polymer ink development is to decrease the resistance of conductor lines. When the concentration of silver particles in ACA is increased, it becomes

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doi:10.1016/j.porgcoat.2003.09.013

eventually conductive ICA [5]. Further increases of solid particle concentration will decrease resistivity and increase usable current density, but it will also weaken printing properties. Previously, polymer inks have been used in low cost power applications, printed on a PCB and flexible substrates. The obtained square resistance was 15 mΩ/(sq. mils) and the curing temperatures used were 150–200 °C [32].

In this work experimental polymer inks containing metallic particles have been studied for printing of conductor lines with gravure offset methods. Ink solvent mixtures and printing environment have been developed and optimised. Cured ink properties have been studied. Inks have been studied with a goal to print on planar 96% alumina substrates and later on non-planar organic substrates.

2. Experimental

Commercial polymer conductor inks are available for screen printing, but they need to be further developed for gravure offset printing purposes. This is required for printing of fine-lines. Solvent or oil mixtures of Ag-filled polymer inks were used in this study, to print on planar 96% alumina substrates.

The experimental inks were ink A (in most cases) and ink B, having only a higher solid particle content, from Coates Electrographics. In order to estimate the printing parameters, roller type gravure offset printer was used. Pressures (compression thickness), speeds, delay times, additional solvent and blankets have all been varied. A computer was installed to record timing between the printing stages. A systematic approach was used in evaluations of the printing parameters, of $v1$ (the doctoring speed), $t1$ (the time from doctoring to pickup), $v2$ (the pickup speed), $t2$ (the time from pickup to lay down) and $v3$ (the lay down speed). The roller type gravure offset printer was used to print all samples studied in this paper. It allowed variation of compression thickness over the gravure (pg) and substrate (ps), while these parameters vary over printed area with a pad printer. Pressures given in this paper are in micrometers of average compression of the blanket, and actual pressure related to these values are correlating exponentially (defined experimentally for a home-made blanket with $R^2 = 0.998$ for 0.030–0.580 mm compression):

$$\text{pressure (g/mm}^2) = 269 \times (\text{compression in mm})^{3.10} \quad (1)$$

The commercial blanket and the home-made blanket were 1.76 mm thick. The home-made blanket was made from silicone polymers moulded to be the surface of the thin (<1 mm), non-elastic back fabric. The silicone polymer thickness was about 0.6–0.7 mm and the back fabric 1 mm in both cases. The blankets were tightened over round rollers having a diameter of 60.80 mm and the hardness of the blankets was 65–75 Shore A.

A computer controlled XYZ-printing machine with 10 μm placing accuracy was used to study pad-printability of the inks [33]. Doctoring of ink to the gravure grooves was done by a hand-held doctor blade, which did not enable recording or adjustment of the doctoring parameters (v_1, t_1). Pickup and lay-down speeds were constant. Time on the pad was adjusted with delays. Pressures at gravure and at substrate were not uniform, due to the shape of the pad. The pad shape was a hemisphere, hardness was 18 Shore A, supplied by TampoPrint.

Evaluated results of the printings were the pickup mass, the printed mass on the substrate, the mass not printed, the printed line cross-section area, the printed image quality, the microscopic image quality and a square resistance. Used ink mixtures were made by first mixing pre-mixed ink thoroughly and then by further mixing with the 3-roll-mill, which gave a significantly better ink mixing result than without such mechanical mixing. Oils used were paraffin oil and Shell solvent ‘D90’. In order to dilute the inks and reduce their viscosity, other solvents such as 1-propanol can be used.

After printing, samples were cured in circulating air with an optimum curing time and temperature. In order to define the optimum curing time, electrodes were connected to printed samples to measure their resistance during curing. After curing, the resistances of all printed samples were measured via 4-point square resistance measurement patterns. A special centrifuge was build to simulate densification of printed ink on samples. An HP 3457A multimeter was used to make the square resistance measurements. Comparative DCS measurements were made using a Mettler TA 3000 DSC20 differential scanning calorimeter. The temperature variation study was made with an ADP Cryogenic HC-2 circulating helium cryostat with vacuum. Line profiles were measured with a Dektak 3D and analysed with a home-made program.

3. Results and discussion

Ink A had a high viscosity and tendency to dry very rapidly and irreversibly. This caused rapid gravure groove blocking of a gravure manufactured with the novel method [34]. The used ink doctoring container in the roller type gravure offset printing was closed after each printing. However, despite use of the standard doctoring pressure, there was a bad doctoring trace that led to ink accumulation on the non-image areas of the gravure. This ink has presumably a high rheological normal-force (normal stress), which causes a bad doctoring result [35]. In order to solve the problems of the ink drying on the non-image areas, the doctoring pressure used had to be double that used for ceramic inks [36]. But even after this, there were problems with the doctoring device built from doctoring knives, and grooves were still blocked.

The experimental ink A has obviously reactive components in the binder, due to the added HEMA (2-hydroxyethylmethacrylate) solvent. This reactivity has been found to be detrimental in the used printing environments, because it led to too rapid drying of the ink. Therefore, paraffin oil (boiling point 300–500 °C) was studied as an ink component, and also because it was known to have a very significant effect on ink transfer when present in the blanket or the pad [37,38]. An aliphatic hydrocarbon (Shell solvent 'D90', boiling point 225–275 °C) was also studied, because a similar oil was suspected to be an essential factor for 100% ink transfer from a blanket to a substrate for ceramic inks [38,40]. Both oils are non-polar.

3.1. Effect of paraffin oil in blanket

A home-made blanket with 10% of paraffin oil in silicone polymer was used. It was first expected that if the ink behaved as ceramic inks in earlier works, paraffin oil inside a blanket would decrease the time $t2$ required for 100% ink transfer from blanket to substrate by a decade [38]. That was found not to be the case with studied polymer inks, which indicate behaviour other than an adsorption mechanism. An adsorption mechanism describes ink transfer from a blanket to a substrate being affected mostly by solvent adsorption of the ink to the blanket. The blanket, containing paraffin oil, had neither a shorter ink transfer time nor better transfer percentage. It was eventually concluded that the ink printing behaviour relates partly to the traditional evaporation mechanism. Traditionally, ink transfer from a blanket to a substrate has been described by evaporation of solvent from the surface of the ink to the atmosphere, thus creating a sticky layer. A home-made blanket described later in the text has no paraffin oil.

3.2. Printing environment

Printing environment alterations were used in order to find the optimum printing parameters for the studied inks. It was noted that an optimum time $t2$ for the highest transfer percentage without solvents or oils was 7–15 s. One of the best printing ink mixtures (5% of D90) had an optimum $t2$ 2–4 s, to give one of the highest transfer percentages. A major effect on pickup mass was contributed by pickup speed, as shown in Fig. 1. These results in parameters and their dependencies differ significantly from those reported earlier, complying with the absorption ink transfer mechanism [40]. The major reasons are the different ink transfer mechanism and ink particle shape [38].

In practice, gravure and substrate pressures should be the same to avoid a pattern distortion due to blanket deformation. If, for example, the laboratory printer having a roller diameter of 6 cm used a pressure over the substrate 600 μm larger compared to that over the gravure, then the printed pattern will be about 4% longer in the printed direction, without forced rotation of the roller. Forced rotation means that the roller is made to rotate over the gravure with a rate set by gearing, not by the contact of the blanket. An example of the substrate pressure effect on the ink transfer percentage from a blanket to a substrate is shown in Fig. 2. Fig. 3 shows the response surface of both gravure and substrate pressures made with the Modde 4.0 program. Modde 4.0 is a factor design program that enables study of multivariate systems. Modde has been previously applied to ink component in screening and printing parameters optimisation [39,40].

3.3. Oils in the ink

Oils added to inks improved their rheology and printability. However, increased paraffin or D90 oil content decreases the highest pickup mass as shown in Fig. 4. From the perspective of a blanket to a substrate ink transfer percentage, the optimum amount of 6% of paraffin oil in ink, gave ink transfer increases from 83 to 92%. In this experiment there was a low pressure at the substrate and therefore less transfer from blanket to substrate. Gravure grooves are blocked significantly slower with ink containing oils. When oil was added, there was also a significant effect on ink rheology

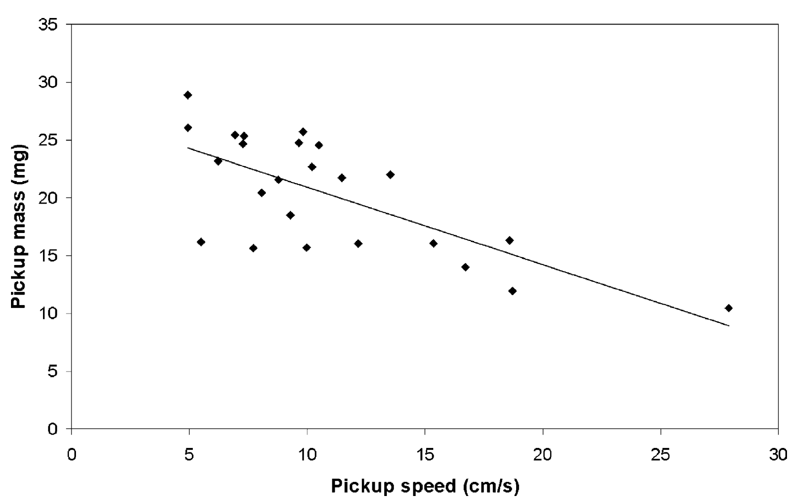

Fig. 1. Effect of pickup speed on pickup mass.

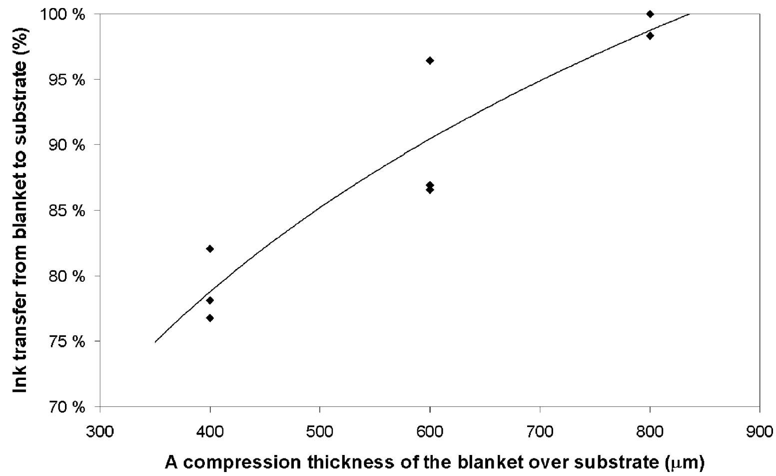

Fig. 2. The effect of blanket compression thickness (pressure) on gravure to ink transfer (with 5% of D90 oil) from a blanket to a substrate.

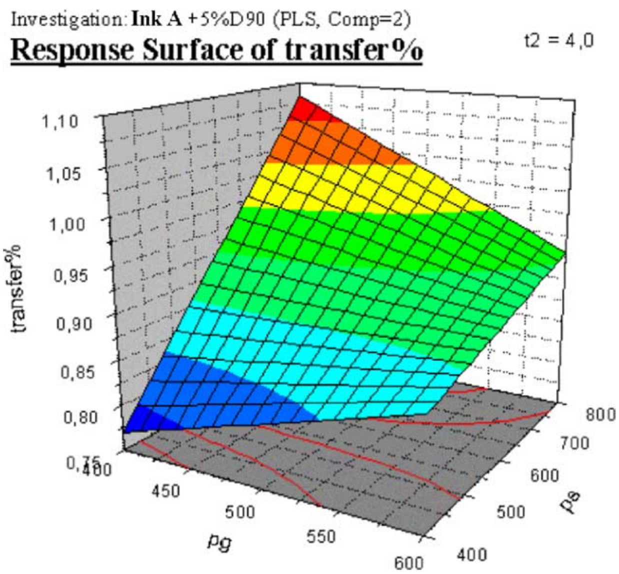

Fig. 3. A response surface for transfer percentage (1 = 100%) based on gravure and substrate pressures (μm). Figure is based on multivariate model made with Modde 4.0. $R^2 = 0.87$.

that enabled the ink to flow better down to the gravure and enable better doctoring. The effect of a paraffin oil addition on the shear rate at 10 and 100 l/s is shown in Fig. 5. The effect on viscosity of 5% of D90 is similar to 13% of paraffin oil.

From the printed results it was obvious that 13% of paraffin oil was too much, because the ink structure broke down, even after a thorough 3-roll-mill mixing. Print results without and with this ink mixture are shown in Fig. 6.

3.4. Results in optimum conditions

Good single printing traces can be produced with little or no solvent, but gravure groove blocking prevents such a series of printing experiments. This means that narrow lines are printed only after gravure cleaning. An example of a good printing result is shown in Fig. 7. Wide lines with high cross-section area can be printed with different kinds of inks, compared to narrow lines with high cross-section: lower viscosity and better ink flow are required for the printing of

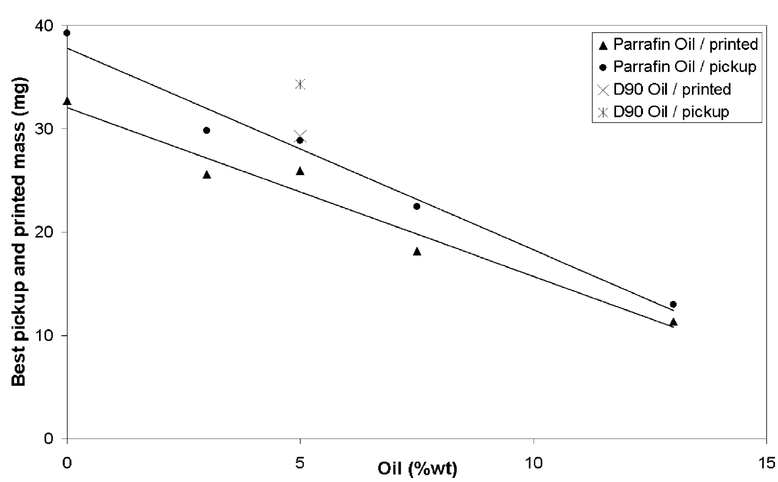

Fig. 4. Effect of paraffin and D90 oil on the highest pickup and printed mass.

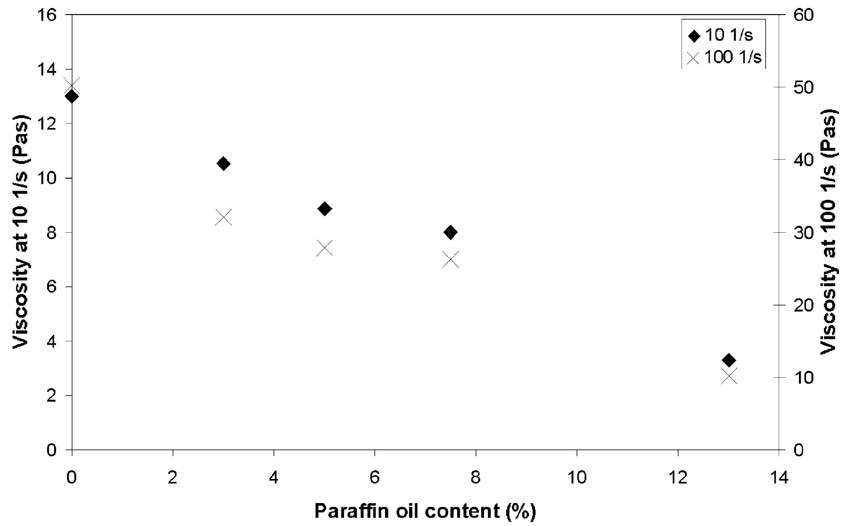

Fig. 5. Effect of paraffin oil addition on ink rheology at two different shear rates.

narrow lines, as for inks for ceramics [41]. An example of a printed pattern having a high cross-section profile and good image quality is shown in Fig. 8, which was printed with ink containing 5% of paraffin oil. Fig. 8 shows traces of lines which have designed line widths down to 25 μm. The 70 μm wide lines shown are conductive in the measurement patterns. When ink is screen printed, the ink's thixotropic property causes lines to take up a more uniform form. Also, when solder paste is melted, it seeks the minimum surface area forming thus drop-like surfaces. Since printed polymer inks have a high yield stress and because electrically conductive adhesives generally do not have self-alignment properties in die-attachment (flow during curing) as solder metals do, the line shape does not become more level or semi-spherical [42].

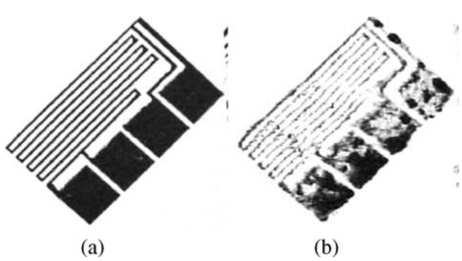

Fig. 6. 4-Point measurement pattern for 150 μm wide lines printed (a) good printed result without addition of oil and (b) ink containing 13% of paraffin oil, where structure of ink has broken down.

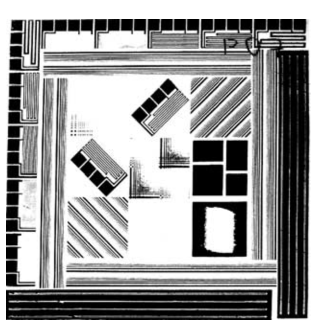

Fig. 7. Printed pattern with ink with 5% of D90. Three widest line 4-point measurement patterns with line widths of 300, 150 and 75 μm are successfully printed. Pattern contains also line, grid and dot patterns.

Almost a 100% ink transfer from a blanket to a substrate was found to be possible with the right solvent mixture and printing pressures and timings. That is, no detectable amount of ink remained on a blanket after transfer. There were only visual observations of sharp silver flakes, which had penetrated the silicone polymer surface and could not be transferred. The referred print was done with a mixture of D90 or *n*-pentane, with the parameters shown in Table 1. It was noted that *n*-pentane prevents gravure groove blocking and its mixture with ink gives the best transfer percentages and highest printed mass. The obtained minimum square resistance in the 4-point test pattern was 20 mΩ/sq. for 300 μm and 28 mΩ/sq. for 150 μm wide lines for 7–8.5 μm thick line (calculated as a rectangular shape).

The basic mechanisms of the ink transfer are very similar on both planar and on curved surfaces. Therefore printing tests were made mainly on planar surfaces with a roller type gravure offset printer. This allowed better control over the printing parameters. The measured ink transfer from a blanket to a substrate and conclusions about printing parameter *t*2, *p*s and volatile solvent amount have been summarised in Fig. 9a and b. For the studied inks there is an optimum *t*2 which can be adjusted with the concentration of volatile solvent and minimum *p*s for complete ink transfer.

Selection of the optimum ink is dependent on the required resolution, line shape and thickness. Although ink B did not show good printing properties even with addition of solvent, its printing results appear as more rectangular lines compared to ink A as shown in Fig. 10. Outer lines show the maximum and inner lines the average of variation in

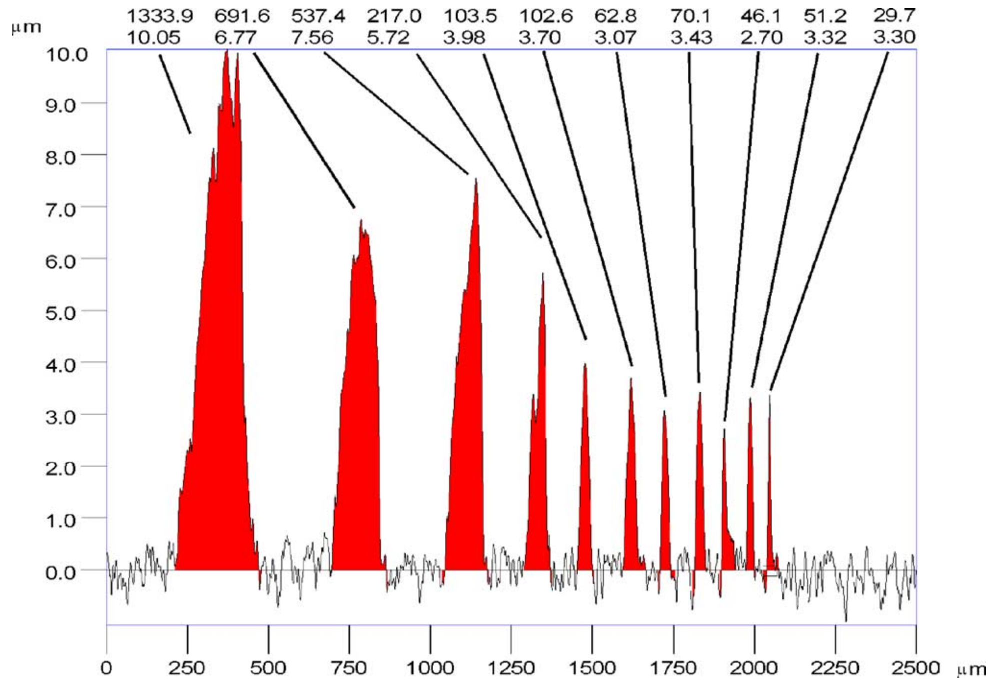

Fig. 8. A cross-section profile of printed lines using ink with 5% paraffin oil, printed on alumina. Designed line widths of visible line cross-sections are 280, 200, 140, 100, 70, 70, 50, 50, 35, 35 and 25 μm. Numbers above peaks show cross-section area in μm^2 and maximum height in μm.

Table 1
Optimum printing parameters for two different ink mixtures

Ink mixture	Pressure at substrate (μm)	*v*1 (cm/s)	*t*1 (s)	*v*2 (cm/s)	*t*2 (s)	*v*3 (cm/s)	Transfer (%)	Resistance of 150 μm wide lines (mΩ/sq.)
5% D90	800	18.9	3.3	10.9	2.3	6	100	42
10% *n*-pentane	800	10.1	3.4	5.4	11.9	4.5	98	28

cross-section profiles. A 2× print multiprint is sufficient to produce over 10 μm high lines, with increased yield. Therefore selection of gravure offset printing over conventional PCB method depends on the required quality, depending if high resolution is more important than printed cross-section area of wide lines. Fig. 11a and b shows comparison of qualities between these two technologies. Ink B shows a more rectangular shape with a higher cross-section area, but worse printing resolution compared to ink A.

3.5. Gravure groove blocking

Silver particle shapes and sizes have an effect on ink rheology and especially on the yield stress [43,44]. Some yield

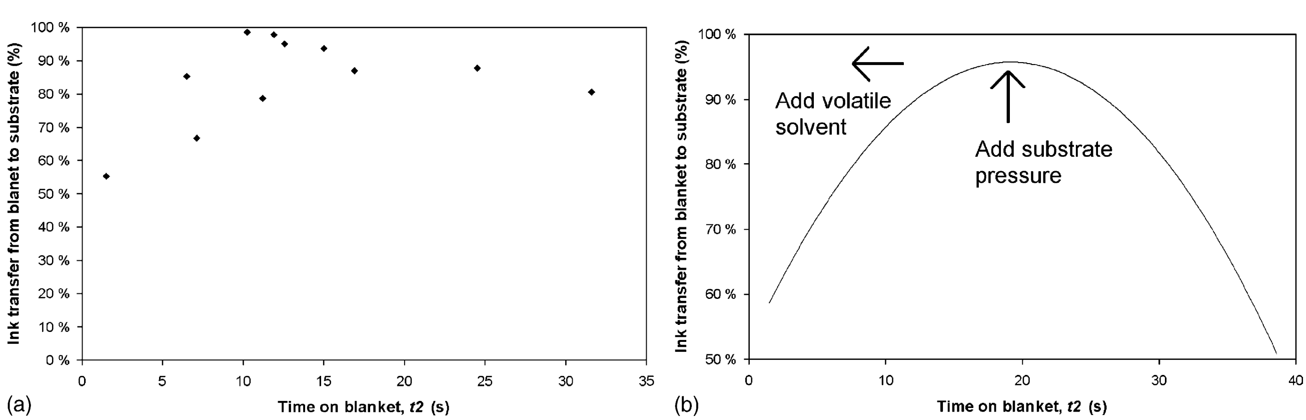

Fig. 9. Ink with 5–10% of *n*-pentane transfer percentages from a blanket to a substrate as function of t_2, (a) measured and (b) schematic summary.

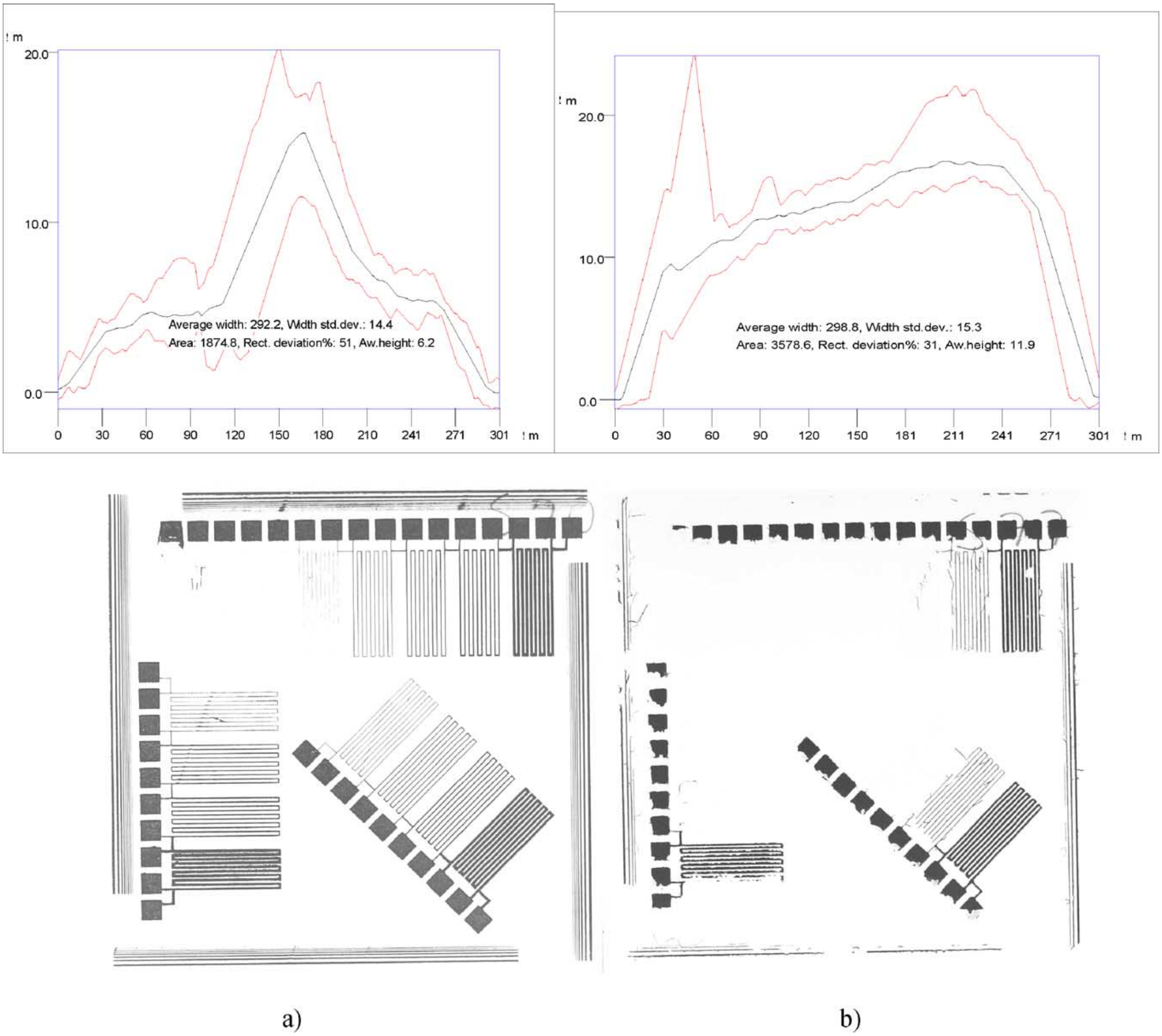

Fig. 10. Surface profiles of 270 μm wide lines (average of 10 lines). The printed samples can be seen below. (a) Highest printed cross-section is obtained when printed twice with ink A. (b) Compared to more rectangular lines printed twice with ink B with 5% of D90.

stress is needed, but too much of it together with a high viscosity prevents ink from flowing down to the gravure grooves and pickup out of them. Gravure groove blocking was observed to be a problem with the used printer set-up, due to the doctoring device that exposed the ink to the atmosphere for long times. Addition of paraffin oil or *n*-pentane to the ink decreased the viscosity of the ink and gravure groove blocking. There are problems with ink stability with these oils if sufficient mixing is not done before use. If ink is stable with an *n*-pentane-alike solvent and there is a suitable ink doctoring container available to prevent its fast drying, that might be the most practical solution for making ink work better.

Differences in ink mixtures' effect on gravure groove blocking have been studied in two ways. Firstly this effect is seen by the change of pickup mass as shown in Fig. 12. The pickup mass was reduced without cleaning of the gravure grooves, after a number of prints, most significantly in the absence of solvents or oils from the ink. Paraffin oil has the most significant effect on preventing gravure groove blocking. Unfortunately, oil content in an ink increases the resulting square resistance in printed and cured samples, exponentially in the case of paraffin oil. The square resistance is however much better with D90 mixtures compared to inks without solvent due to the higher pickup and printed mass.

The second approach to determine gravure groove blocking and repeatability of printing was to study the surface profiles of printed samples. Cross-section areas at similar places of each sample were measured. Then a cross-section area was divided by the designed line width to give a relative line height. Then the narrowest lines, that have a relative

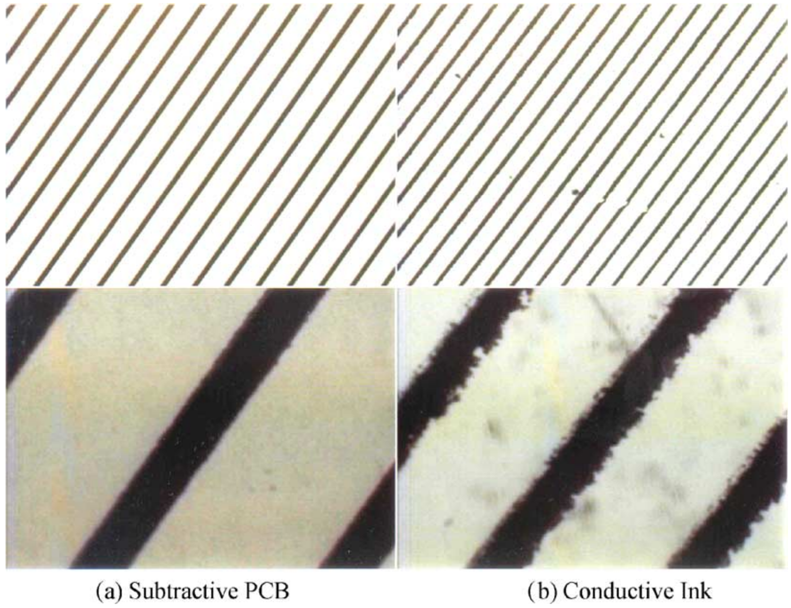

Fig. 11. Similar pattern manufactured from copper with (a) subtractive PCB technology and (b) additive printing technology with 250 μm wide lines and 570 μm wide line spaces. Larger image on conductive ink shows random printing errors. Courtesy of Thales Airborne Systems.

line height more then 1 μm, were listed. Results are presented in Fig. 13, which shows significant effect on gravure groove blocking with and without 5% of paraffin oil. More oil causes the ink to loose too much of its internal cohesion and printed results are not acceptable anymore. One solution to prevent the gravure groove blocking is the usage of a self-cleaning gravure [45].

3.6. Curing

It is typical for polymer inks to have an optimum curing temperature in a given curing time [17]. A longer curing time usually enables lower curing temperatures [30]. When the ink is being heated during curing towards the optimum temperature the square resistance decreases (calculated corresponding to the room temperature). At the optimum temperature the square resistance reached the optimum. If the ink is heated above the optimum temperature, the square resistance can start to increase again due to polymer softening and chemical reactions taking place. To determine this optimum temperature the DSC and square resistance measurements were made as functions of time with constant heating rates. Three different samples were studied with DSC: 1 = N_2 atmosphere airflow, 2 = atmospheric airflow

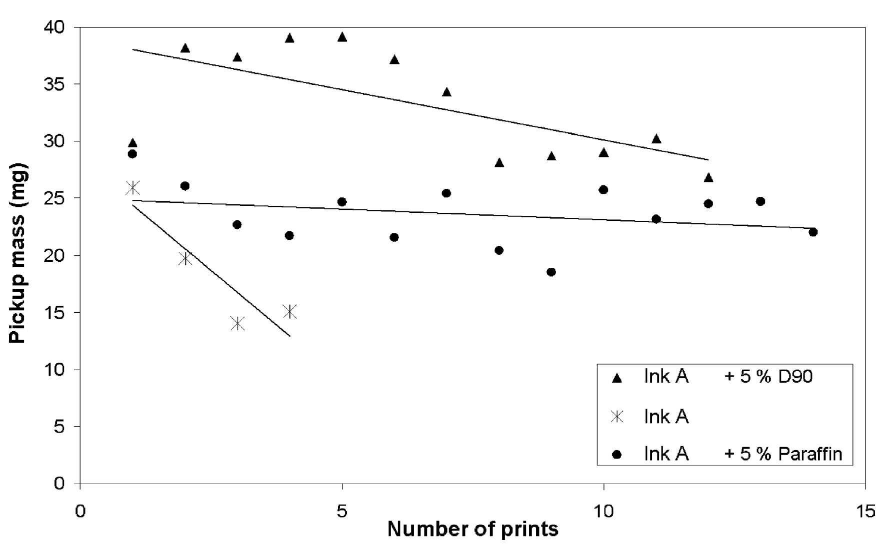

Fig. 12. Gravure groove blocking effect shown with pickup mass using home-made blanket, containing 10% of paraffin oil. Addition of oils to ink prevents gravure groove blocking based on pickup mass.

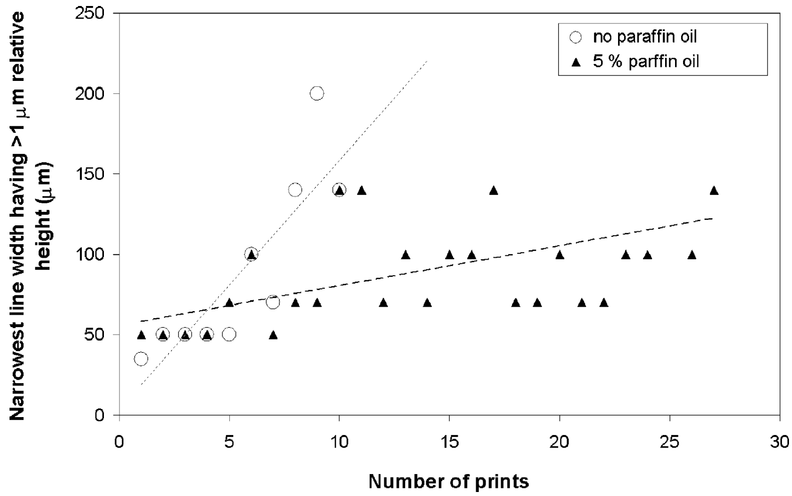

Fig. 13. Gravure groove blocking effect shown listing of narrowest lines with relative height over 1 µm as function of number of prints. An addition of 5% paraffin oil to the ink results in least gravure groove blocking.

and 3 = with D90 solvent in atmospheric airflow. N_2 atmosphere has been used for metal particles that may oxidise in air [8]. Data is shown in Fig. 14. Results indicate that: (1) the chosen atmosphere effects the optimum temperature: in N_2 atmosphere higher temperatures must be used. (2) Addition of D90 oil has a minor or no effect on this optimum temperature.

It has been suggested by the ink manufacturer, that densification of silver particles could decrease the resulting resistance. Similar results have been reported in the literature [46]. To test this presumption, four groups of samples were made. Their resistance was measured before and after treatments (densification) with curing (the last group of samples was not cured). Changes in resistances and variations in results are shown in Table 2. It was concluded that 5 min of 900 G centrifuging of samples does not have a statistically significant effect on the decrease of the resistance.

3.7. Square resistance values

Since oils in the ink caused increase resistance; the following measurements were done with samples printed without any oil. The effect of square resistance as a function of time and temperature during curing is shown in Fig. 15. Actual resistance comes a few minutes later as shown in Fig. 16 (different solvent and printed thickness compared to Fig. 15). The optimum temperature was found to be 140 °C, based on DCS analysis and resistance monitoring during curing.

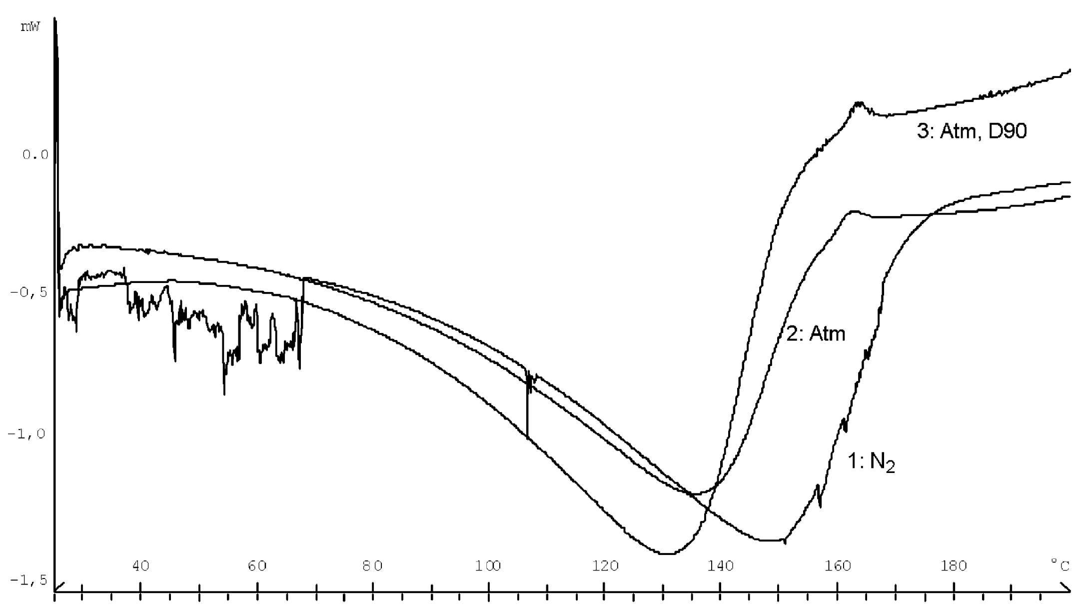

Fig. 14. DSC diagram from 1 = N_2 atmosphere airflow, 2 = atmospheric airflow and 3 = with D90 solvent in atmospheric airflow.

Table 2
The resistance of the treated samples compared to resistance immediately after printing[a]

	Uncured resistance (%)	S.D.
Directly cured	1.68	0.34
900 G and cured	2.00	0.27
Dried 4 days and cured	1.51	0.16
4 days at room temperature	40.0	2.79

[a] The first three samples have been cured after each treatment. Standard deviation is also given; 900 G centrifuging time was 5 min.

Relative square resistance has almost reached its minimum at a time corresponding to 150 °C. However, in an isothermal environment, it took about 10 min for the sample to reach the minimum resistance after stabilisation as shown in Fig. 16. It shows that 20 min should be a sufficient time.

3.8. The end-user application

Gravure offset printing of conductive polymer inks was found in preliminary printing experiments to be suitable for non-planar substrates. There are many large volume

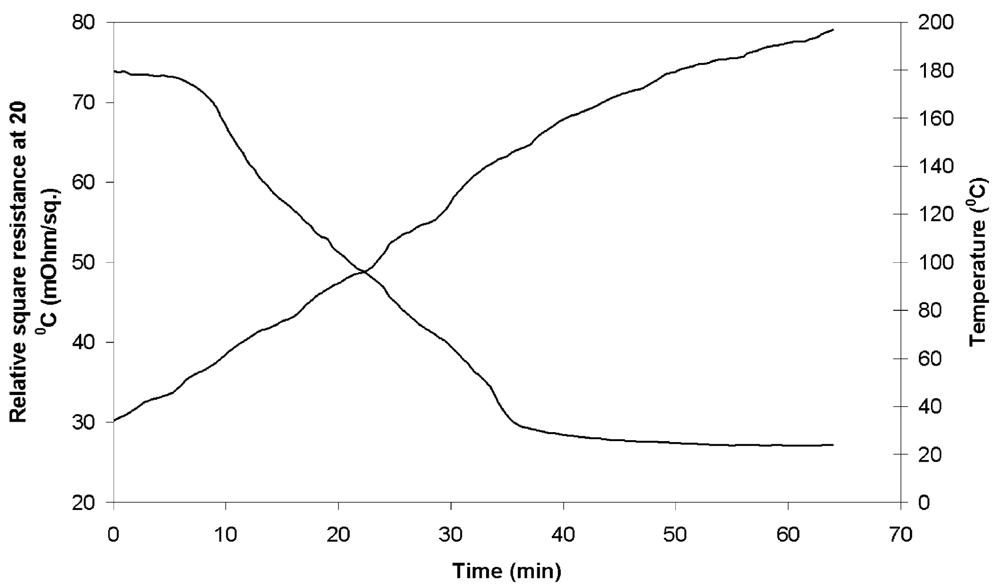

Fig. 15. Temperature (rising curve) and relative square resistance (partly downward curve) of ink in an oven during 65 min long measurement. At room temperature the final square resistance was 30 mΩ/sq. for 300 μm wide test pattern. Actual resistance comes a few minutes later as shown in Fig. 16.

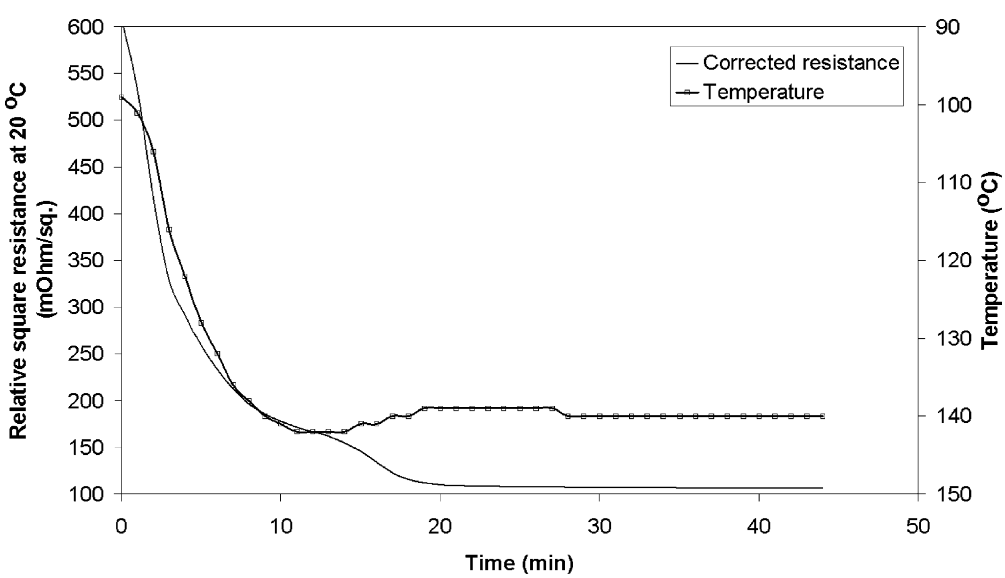

Fig. 16. Ink in isothermal environment. Temperature shown is measured by probe attached to the sample. Temperature correlated resistance (to the room temperature) is shown as a function of time. At room temperature this test pattern had square resistance of 116 mΩ/sq.

applications, that require far less than 10% of their surface area to be coated with a metal, as for example reflectors for radar components. However, such surfaces can be parabolic or hemispherical. For such surfaces, it is hard to apply well-known etching methods for PCB, but by using a pad printer these surfaces can be printed. The pad printer was successfully tested in printing of optimum ink composition with optimum observed parameters. Results were similar and allowed planar and non-planar printings.

4. Conclusion

Usage of this gravure offset method provides an environmentally friendly, economically viable method for large volume production of electrical fine-line conductors. Although there can be such problems a gravure groove blocking in production lines, in radar reflector applications such problems, shown by narrowed or even randomly broken conductor lines, do not disqualify the product. Also, printed thickness and obtained yield can be significantly increased with multiprinting, which is allowed by the developed method.

The minimum square resistance with ink B was 30 mΩ/sq. for 300 μm wide lines. With ink A for 300 and 150 μm wide lines, square resistances as low as 20 and 28 mΩ/sq., respectively, were measured with less than 15 μm high peak shapes lines. The minimum line width with an acceptable square resistance was 70 μm with ink A with a square resistance of about 50 mΩ/sq.

The purpose of this work was to study properties of the experimental ink and find modifications to make it more suitable gravure offset printing. During ink printing studies, it was found that successful printing with 100% ink transfer from a blanket to a substrate can be achieved. The minimum printed resolution of conducting lines of 70 μm and a curing temperature of 140 °C provide a large range of end-user applications, such as conductor layer for PCB additive processes.

Acknowledgements

This research was supported by Brite/Euram project ‘Development of a high-resolution printing process for cost-effective production of ultra-fine line electronic circuitry’ (BE 97-4607). Experimental inks were received from Coates Electrographics.

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