

Inkjet-printed silver tracks: low temperature curing and thermal stability investigation

By Jolke Perelaer,^a Antonius W. M. de Laat,^b Chris E. Hendriks^a and Ulrich S. Schubert^{*ac}

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In this contribution the curing behavior and conductivity development of several commercially available silver inks is discussed. In addition, the preparation and characterization of a silver particle ink that shows a curing temperature as low as 80 °C is described. Good to excellent conductivity values of 5 to 56% of bulk silver have been reached by using a very small amount of organic additives without any strong adsorbing groups such as amines, amides or mercapto groups. This low curing temperature opens new routes to produce conductive features on polymeric foils that have a low T_g , like PET. Furthermore, the temperature stability of silver tracks, prepared by inkjet printing different colloidal silver suspensions, was investigated. Hereto, the resistance was on-line measured during heating of the silver tracks, from room temperature to 650 °C.

Introduction

Drop-on-demand technology in the form of inkjet printing has demonstrated a rapid growth during the last two decades.^{1–3} It has been shown that inkjet printing of conductive materials – either as suspensions or in solution – is a relatively cheap alternative for the production of electronic devices^{4–8} when compared to other micro- and nanopatterning techniques, like (photo)lithography⁹ or laser patterning.^{10,11} In particular, direct inkjet printing of conductive silver tracks onto flexible substrates has gained interest in many applications, such as radio frequency identification (RFID) tags.^{12–14} With direct inkjet printing, patterns of conductive features can be realized in one step, and therefore reduce the time, space and – also importantly – waste consumed in production. Moreover, this technique introduces the possibility for the production of flexible electronics, also in a roll-to-roll (R2R) manner.

A major concern with printed electronics involves not only the control of the morphology of the tracks,^{5,12–16} but also the stability and adhesion of the obtained conductive tracks, although scarcely investigated.¹⁷ The main focus though in plastic electronics lies in the low curing temperature of the conductive ink. Although conductive inks based on a solution rather than a suspension are described in the literature,⁵ we discuss here only the particle-based conductive inks. For particle-based inks, the curing temperature is defined as the temperature where particles lose their organic shell and start showing conductance by direct physical contact. Whereas – although often misused for the curing temperature – sintering takes place at a higher temperature where all organic material has burned off

and the conductive particles start showing a neck formation between particles. The typical temperature where (nano)particles make first physical contact and start forming a percolating pathway, *i.e.* showing conductance, needs to be below the softening temperature of the polymeric foil. A low curing temperature of a nanoparticle ink is important in plastics electronic applications where low T_g foils, like PET, are used.

The lowest temperature at which printed features become conductive is mainly determined by the organic additives in the ink.¹⁸ Several types of organic additives can be found in inkjet inks. Firstly, a dispersant is needed to prevent agglomeration of the particles in the ink. In non-polar solvents usually long alkyl chains with a polar head, like thiols or carboxylic acids, are used to stabilize the nanoparticles.^{19–21} These molecules provide a larger steric hindrance and the adsorption of the polar group on the surface of the nanoparticle reduces the surface energy.¹⁸ Secondly, organic binders are often used to assure mechanical integrity and adhesion to the substrate after drying.

Evaporation of the solvent, after printing, forces the particles close together. Conductivity will arise when metallic contact between the particles is present, and a continuous percolating network is formed throughout the printed feature. However, a residual organic layer between the silver particles as thin as a few nanometres is enough to prevent electrons from moving from one particle to the other.^{22,23} The adsorbed dispersant will very likely stay on the surface of the particles and will have to be removed by an increase in the temperature.

Besides the low curing temperature, a second important property is to obtain the lowest possible resistance of the printed features at the lowest possible temperature.

To achieve this, sintering of the particles is required to transform the initial very small contact areas to thicker necks and, eventually, to a dense layer. During this process, the conductivity will gradually increase as the contacts between the particles become larger. Note that we distinguish between the critical curing temperature at which initial metallic contacts arise and the sintering process in which the contacts are gradually intensified.

^aLaboratory of Macromolecular Chemistry and Nanoscience, Eindhoven University of Technology and Dutch Polymer Institute (DPI), PO Box 513, 5600 MB Eindhoven, The Netherlands. E-mail: u.s.schubert@tue.nl

^bPhilips Applied Technologies, High Tech Campus 7-3A-022-3, 5656 AE Eindhoven, The Netherlands

^cLaboratory of Organic and Macromolecular Chemistry, Friedrich-Schiller-University Jena, Humboldtstr. 10, 07743 Jena, Germany

Sintering at lower temperatures can be realized by using very small particles. The driving force for sintering is the release of the high surface energy related to the small radius of curvature of the surface. This is also reflected in the decrease of the melting temperature with decreasing particle size. From the literature it is known that metal nanoparticles smaller than 5 nm show a sharp decrease in their melting temperature.^{24,25} Often high temperatures – typically up to 300 °C – are required to burn-off the organic additives and stimulate the sintering process to realize a more densely packed silver layer and a lower resistivity.^{4–8} Lee *et al.* describe an aqueous silver colloid suspension that is prepared mainly with environmentally friendly chemicals, like water and diethylene glycol.⁸ Although short times of only 10 minutes are necessary, high temperatures (200–300 °C) are required to obtain good conducting lines. It is therefore of utmost importance for further progress to identify an optimum between the time, temperature and the obtained conductivity.

However, in other applications, for example, in the manufacturing of plasma display panels²⁶ the conductivity needs to be maintained at temperatures as high as 600 °C. At this temperature, the separately produced back and front panels are joined together. To the best of our knowledge, no studies have been published in the open literature that investigate the thermal stability of printed (silver)nanoparticles features at temperatures higher than 350 °C; only a study of the pressure stability of silver nanoparticles was reported.²⁷

In view of these important applications, we firstly report on the curing properties and conductivity development of several commercially available inks and on our results in formulating an ink with a low curing temperature. We then describe the effect of higher temperatures on the conductivity and the stability of printed conductive features. Four different types of silver-based inks were investigated on their electrical resistance behavior during heating up to 650 °C.

Experimental

Ink formulation

Four types of silver inks were used. Two inks were obtained commercially: an aqueous Nippon SVW102 silver (Nippon Paint Co. Ltd, Osaka, Japan), with a particle size of 10.8 ± 6.7 nm (discussed in the text as Nippon ink), and an ethylene glycol–ethanol-based silver ink from Cabot (Cabot Printing Electronics and Displays, Albuquerque, USA), with a particle size of 52.4 ± 11.0 nm (referred to as Cabot ink). The two other inks were prepared in-house. The ink with Mitsui particles was prepared in the following way: 13 g of Ag powder (SPQ03S, Mitsui-Kinzoku, Japan) was dispersed in 13 grams of an aqueous medium containing 0.85 wt% polyvinyl alcohol with a molecular weight of $108\,000 \text{ g mol}^{-1}$ (Gohsenol, Nippon Gohsei, Japan) and 5 wt% dipropylene glycol. The average particle size of the Ag powder is 0.52 μm , according to manufacturer information. Full dispersion is realized by milling on a rollerbench for 24 hours with the aid of 0.8 mm glass beads. After milling, the beads are separated and the ink is passed over a 10 μm sieve to remove any large particles left. A second ink with Merck nanoparticles with a particle diameter of 8.9 ± 4.5 nm, and therefore referred to as Merck ink, was prepared in the following way: 4 g of Ag powder (Merck,

Germany) are mixed with 9.6 grams of an aqueous solution having 2 wt% polyethylene oxide ($20\,000 \text{ g mol}^{-1}$) and 5 wt% dipropylene glycol. Milling is not needed as this powder disperses spontaneously. The ink is passed over a filter (Slap, Millipore) to remove any large residue. Both inks are degassed by passing through He gas for one minute or by treatment in an ultrasonic bath just before use.

Inkjet printing

Inkjet printing was carried out with a piezoelectric Autodrop printer from Microdrop Technologies GmbH equipped with a 68 μm nozzle diameter MD-K-140 dispenser. The following printing parameters for voltage and pulse width were used: Cabot (172 V, 120 μs), Nippon (134 V, 82 μs), Merck (174 V, 40 μs) and Mitsui (140 V, 45 μs), respectively. All samples were printed at room temperature.

Boron silicate glass substrates were cleaned by firstly washing with acetone, followed by rinsing with isopropanol before being dried under a flow of air.

Characterization

Conductivity measurements were performed using a custom-built steel sample holder, wherein the substrate was placed in a fixed position. *Via* small holes in the steel device, high temperature stable wires were guided from the multimeter (Voltcraft Digital Multimeter VC-960, Conrad Electronics GmbH, Germany) to the substrate and then attached to the ends of a single silver line with a length of 1 cm using Leitsilber L100 conductive silver paste (Kemo-electronic, Germany), which has a resistance of 0.02 to 0.1 $\Omega \text{ cm}^{-2}$. A thermocouple equipped with a K-type sensor (Voltcraft Data-Logger Thermometer 306, Conrad Electronics GmbH, Germany) was used to measure the exact temperature. The multimeter and thermocouple were connected to a computer that recorded the resistance and the temperature every two seconds. The samples were heated in an oven at a rate of 10 °C min^{-1} .

Thermogravimetric analyses (TGA) were performed with a TG 209 F1 Iris by Netzsch under ambient atmosphere in the range from 25 to 650 °C with a heating rate of 10 °C min^{-1} . Samples of 5 to 10 mg were analyzed and prior to measuring all samples were heated to 50 °C for 20 minutes to evaporate volatile solvents. Scanning electron microscopy (SEM) images were taken using a FEG E-SEM XL30 (Philips, The Netherlands). Surface topography, thickness and cross-sectional areas of printed silver tracks were measured with an optical profilometer (Fogale Zoomsurf, white light, magnification 5 \times). Transmission electron microscopy (TEM) images were recorded on a Technai G² Sphera (FEI) electron microscope using an acceleration voltage of 200 kV.

Results and discussion

Conductivity development of inkjet-printed silver lines

We have investigated the critical curing temperatures of several silver inks, either made in our laboratories or obtained commercially. The critical curing temperature is defined in this case as the temperature at which the sample becomes conductive,

i.e. having a resistance lower than 40 M Ω , which is the upper measuring limit of the multimeter used. Note that effects in the range above 40 M Ω cannot be detected in our experimental set-up.

Single lines of the specific ink were inkjet printed onto boron silicate glass and subsequently heated to 650 °C in an oven at a heating rate of 10 °C min⁻¹. During heating the resistance was measured on-line in a semi-continuous way, by measuring every 2 seconds. Using this dynamic scan approach, differences between the various inks can be determined easily.

Firstly we investigated two types of commercially available silver inks: Cabot and Nippon. Table 1 summarizes the composition of each ink investigated. Typical resistance results for these inks are shown in Fig. 1a and Fig. 2a, respectively. The resistance of the lines with both inks decreases rapidly when heated above the critical curing temperature. The critical curing temperature for Cabot silver ink is 75 °C lower than that of Nippon ink (194 and 269 °C, respectively), while their particles sizes are 52.4 ± 11.0 nm and 10.8 ± 6.7 nm, respectively (see Fig. 1b and Fig. 2b). According to the size difference it was expected that the smallest particle sizes sinter at the lowest temperature, because of their higher sintering activity.^{24,25} This would indicate that the organic additives in the ink strongly affect the critical curing temperature. Unfortunately, the nature of the organic additives in these commercially available inks is not disclosed. To elaborate on this we have also investigated the mass decrease upon heating by means of thermogravimetric analysis (TGA). It should be mentioned that all inks have been dried prior to measuring by heating to 50 °C for 20 minutes, which removes volatile solvents. The TGA curve for Cabot silver ink shows a decrease of 72 wt%, which is not only the organic binder that is around each nanoparticle but also the non-volatile solvent ethylene glycol present in the ink (Fig. 1a). The critical curing temperature corresponds to a temperature at which the initial sharp weight loss slows down. The first step in the removal of the organic materials has ended at this temperature. The steep decrease in resistance relates to the temperature range in which the last of the organics are burnt-off. Apparently, all organics have to be removed before the sintering of the Ag particles can proceed in a fast way. This is indicative of an additive that is adsorbed strongly on the surface of the silver particles.

The lines printed with the Nippon ink reveal a critical curing temperature and a fast decrease in resistance when only about 15% of the organic additives are removed (Fig. 2a). Obviously, these particles can make metallic contact long before all organics are gone. In addition, sintering proceeds very fast due to the small particle size. At the temperature where the burning-off of

Table 1 Detailed information on the composition of the silver inks used. Each column shows the silver content, the amount of binding material per gram of silver and the solvent content of the inks, respectively

Ink	Ag content (wt%)	Ratio of binding agent to silver (g/g)	Solvent (wt%)
Cabot	20	0.50	~70
Nippon	30	0.25	~60
Merck	30	0.12	66.5
Mitsui	50	0.0085	49.6

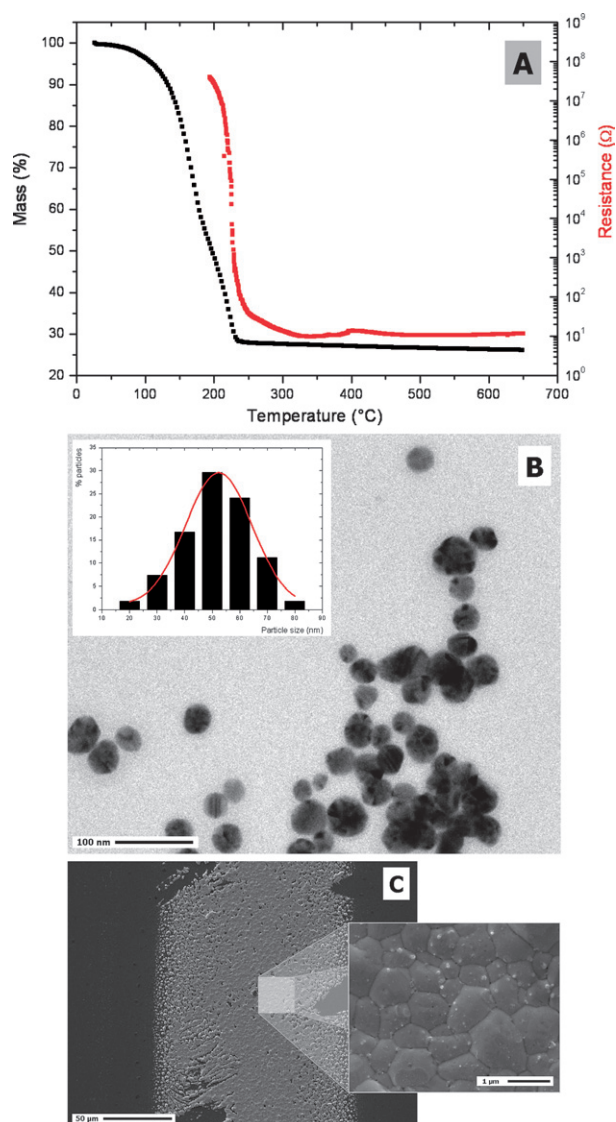


Fig. 1 Resistance as a function of temperature and thermogravimetric analysis (TGA) of Cabot silver ink (a). Transmission electron microscopy (TEM) image and particle size distribution of Cabot silver nanoparticles (b). Scanning electron microscopy (SEM) image of sintered Cabot silver nanoparticles at a temperature of 650 °C (c).

the organics is completed only a small additional decrease in resistance occurs. In this ink, only a minor part of the organic additives interferes with the sintering process but it does shift the critical curing temperature to a higher value. For both inks, however, the resistance value levels off at a certain temperature. At this temperature all of the organics are burnt-off and, apparently, the sintering process has ended and a silver layer with a final density and morphology has formed.

Fig. 1c shows a scanning electron microscopy (SEM) image of a Cabot silver track that has been heated to 650 °C. As can be seen the particles have sintered to a dense continuous line. The electrical resistivity ρ of the inkjet-printed lines was calculated after heating to 650 °C from the resistance R , the length l , and the cross-sectional area A of the line, using $\rho = RAl/l$ and compared to the value of bulk silver ($1.59 \times 10^{-8} \Omega \text{ m}$).²⁸ The resistivity was

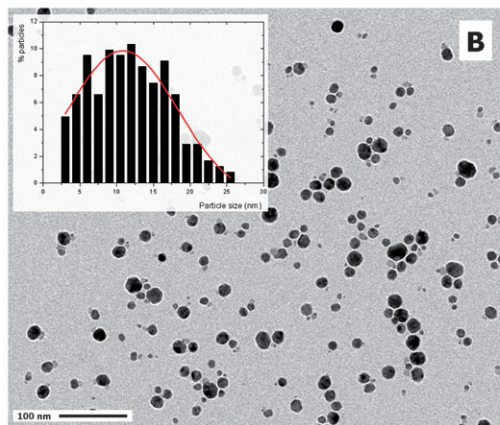
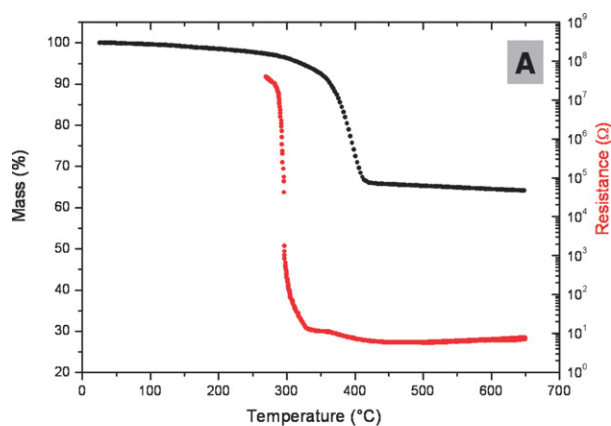


Fig. 2 Resistance as a function of temperature and thermogravimetric analysis (TGA) of Nippon silver ink (a). Transmission electron microscopy (TEM) image and particle size distribution of Nippon silver nanoparticles (b).

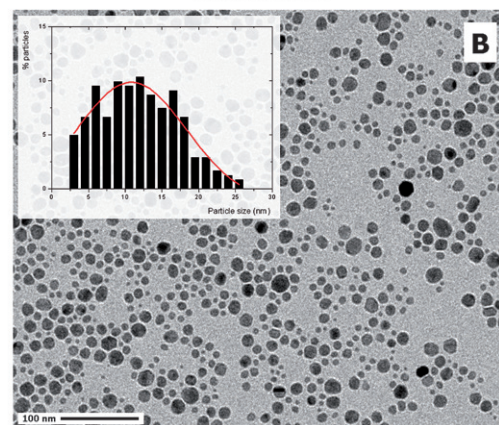
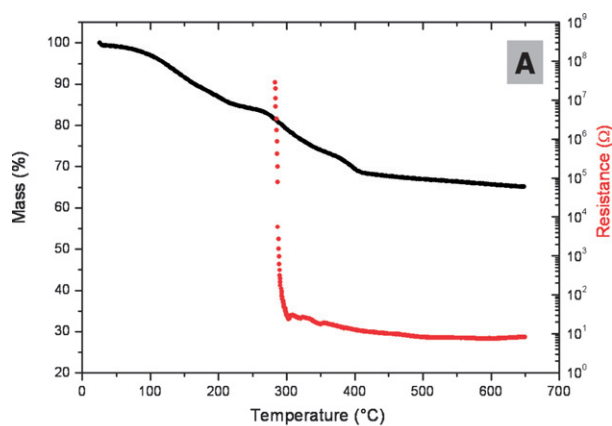


Fig. 3 Resistance as a function of temperature for a five-layer inkjet-printed line, and thermogravimetric analysis (TGA) of Merck silver ink (a). Transmission electron microscopy (TEM) image and particle size distribution of Merck silver ink (b).

calculated to be $3.10 \times 10^{-8} \Omega \text{ m}$ (51%) and $3.06 \times 10^{-8} \Omega \text{ m}$ (52%) for Cabot and Nippon, respectively. The values between the brackets indicate the percentage of conductivity of bulk silver.

Besides the commercially available silver inks, we have performed experiments with in-house prepared silver inks; see the experimental part and Table 1 for detailed information on the inks. Firstly, we have used Merck silver particles that can be easily dispersed into water by simply stirring. Thermogravimetric analysis shows a complex behavior, as shown in Fig. 3a. Clearly, the ink consists of many components, indicated by the multiple steps in the mass decrease. Fig. 3b shows the particle size distribution and the calculated average particle size, which was $8.9 \pm 4.5 \text{ nm}$. The total organic content is about 30 wt%. Furthermore, Fig. 3a displays the resistance as a function of temperature for five printed lines on top of each other (the results of single-layer inkjet-printed lines show a different sintering behavior and will be discussed in the next paragraph). The loss of mass does not immediately cause the silver nanoparticles to make contact and render the lines conductive. The behavior around the critical curing temperature of $275 \text{ }^\circ\text{C}$ is, however, the same as for the commercial inks. At this temperature approximately half of the organic material is burnt-off. The sharp decrease in resistance indicates a fast sintering process. This is due to the small particle size and the temperature that has already increased to a high

value at the moment where initial contact occurs. Both the weight loss and the resistance show a gradual decrease above $400 \text{ }^\circ\text{C}$.

To investigate the possibilities for obtaining a material with a low critical curing temperature we formulated an ink with a very low amount of organic additives without any strong adsorbing groups such as amines, amides or mercapto groups. Instead, materials that only weakly bond towards the silver particles, like poly(ethylene oxide) or poly(vinyl alcohol), have been used. We prepared a silver ink that is based on an unmodified silver powder from Mitsui with a broad range of particle sizes and shapes (further details are given in the experimental section).

After heating the inkjet-printed lines to a temperature of $650 \text{ }^\circ\text{C}$, while measuring the resistance over the lines, the obtained results were compared to those of the commercial inks; the results are shown in Fig. 4. The low content of organic additives is reflected in the TGA curve. It can be seen that this ink reveals a much lower critical curing temperature than the other silver inks: at a temperature as low as $80 \text{ }^\circ\text{C}$, the silver lines can be rendered conductive, as depicted in Fig. 4a. Apparently, the particles make metallic contact after removal of only a small part of the organic material, most probably the dipropylene glycol (DPG). Initially, the resistance decreases slowly with increasing temperature, but above $140 \text{ }^\circ\text{C}$ where most organics

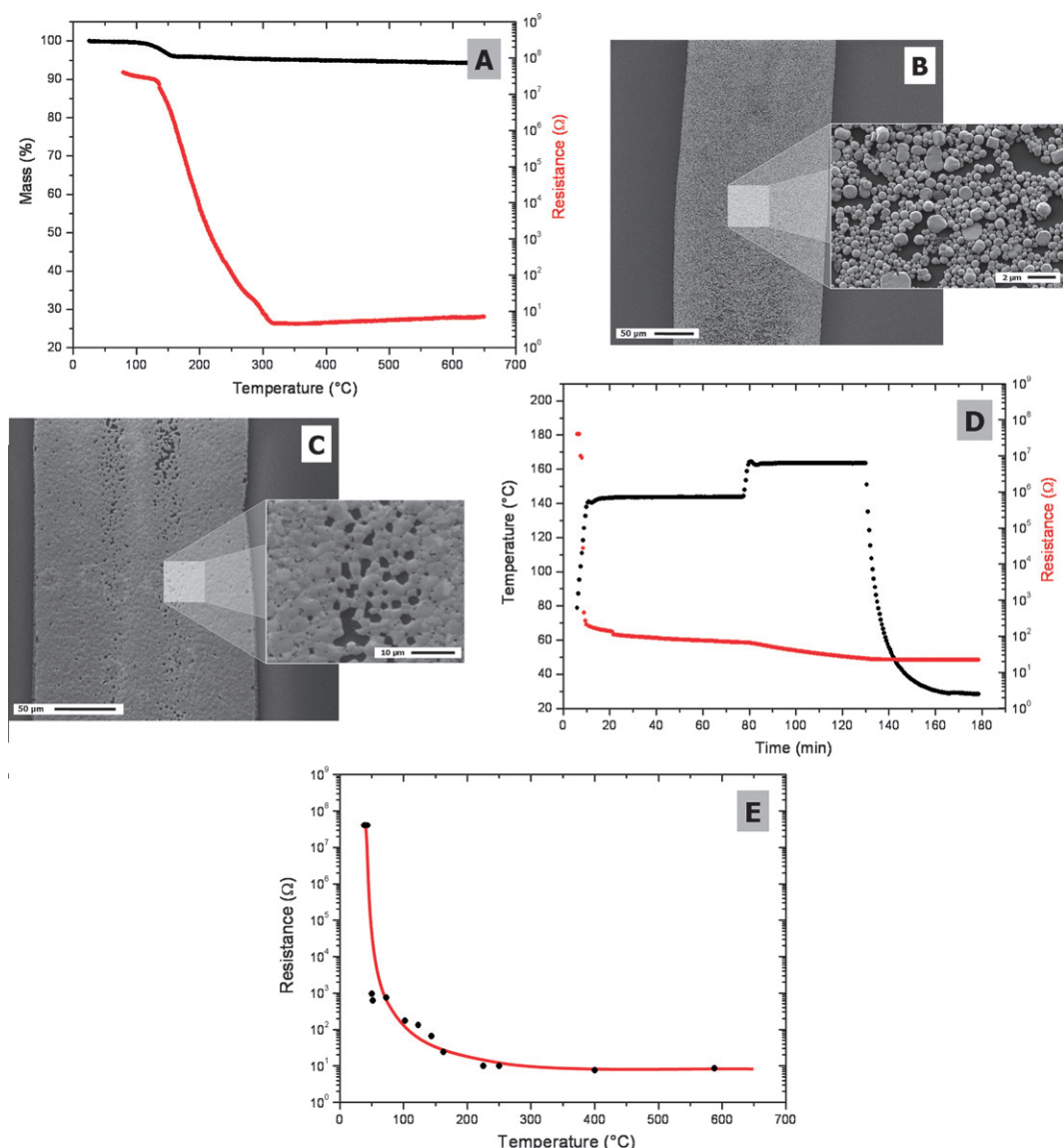


Fig. 4 Resistance as a function of temperature and thermogravimetric analysis (TGA) of Mitsui silver ink (a). Scanning electron microscopy (SEM) image of inkjet-printed Mitsui silver particles at room temperature (b) and after heating to 650 °C (c). Isothermal conductivity development of Mitsui silver ink, for a temperature of 140 and 160 °C (d). Resistance as a function of temperature for different Mitsui silver lines; each point represents a single line that has been heated for 60 minutes at one temperature (e).

are removed, the decrease is faster and reaches a minimum value of $5 \Omega \text{ cm}^{-1}$ at a temperature slightly above 300 °C. This is calculated to be 56% of the conductivity of bulk silver. The relatively slow decrease of the resistance with increasing temperature above 160 °C, as compared to the other inks, is expected to be due to the low sinter activity of the larger silver particles.

Fig. 4b shows scanning electron microscopy (SEM) images of the Mitsui particles. The largest particles are almost 2 μm in diameter. Generally, particles that are greater than 5% of the orifice diameter will cause at least some instability in the drop-generation behavior.²⁹ Since the print head nozzle used for these experiments had a diameter of 70 μm , particles with sizes below 3.5 μm would be printable. We have observed no problems with clogging when printing this specific aqueous ink.

Fig. 4c displays a single printed line of Mitsui particles that was heated to 650 °C and it can be seen that some voids are present in the line, which causes the conductivity to be lower than that of bulk silver.

In addition to the dynamic heating, some experiments were done at a constant temperature. A typical result is shown in Fig. 4d. Heating to a temperature of 140 °C results in resistance values of 50 to 60 Ω , which decreases only slightly when heating for a longer period of time at this temperature. However, increasing the temperature to 160 °C lowers the resistance down to 25 Ω . At this temperature all organics are removed from the particles and the sintering process is further stimulated by the higher temperature. Evidently, the final resistance value after a longer time at 160 °C is much lower than the value recorded in the dynamic experiment (Fig. 4a). To study the effect of longer

curing times on the resistance decrease, different silver lines were heated isothermally at various temperatures for 60 minutes. Fig. 4e shows that at a temperature as low as 80 °C good conductivity values were obtained. Even at these low temperatures conductivities of approximately 5% compared to bulk silver were observed.

Thermal stability of Inkjet-printed silver tracks

High temperature stability is essential in certain applications such as plasma display panels.^{26,30,31} Therefore, the resistance of the samples during heating was measured up to 650 °C. The printed lines of most of the inks discussed in the previous section are stable in resistance at high temperature, independent of their

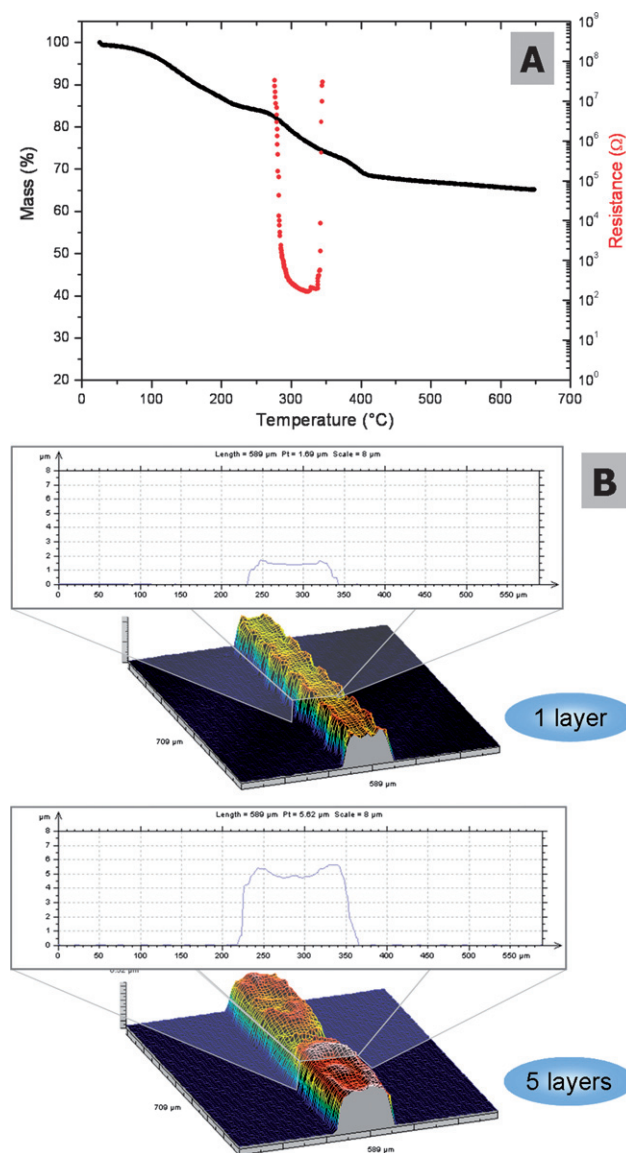


Fig. 5 Resistance as a function of temperature and thermogravimetric analysis (TGA) for a single-layer line of inkjet-printed Merck silver ink (a). The thin dashed line indicates the first derivative of the TGA. White light interferometer images of inkjet-printed silver tracks consisting of Merck silver nanoparticles: topography of a single-layer silver line and five layers printed on top of each other are shown (b).

line thickness. In contrast to this, we have observed with the Merck silver nanoparticles ink, that the inkjet-printed lines do not show a stable and low value for the resistance at high temperature. When a single line of 1.5 μm is heated towards 650 °C, the resistance decreases at a temperature of 275 °C and reaches a minimum of 160 Ω at 325 °C, as shown in Fig. 5a. When heating further, the resistance suddenly increases at 350 °C and the conductivity of the lines is lost within a temperature increase of 20 °C. With further heating the lines lose their conductivity. These results are in contrast to the findings described in the previous section: when lines with greater line thickness were sintered, no loss of conductivity was observed (Fig. 3b). Three-dimensional images of the printed lines are shown in Fig. 5b. Typically, the layer thickness for a single line is 1.5 μm and for 5 layers printed on top of each other the layer thickness is approximately 6 μm.

The reason for the sharp increase of the resistance when sintering lines with a height of 1.5 μm is most probably due to the formation of cracks in the printed tracks. Since the silver ink contains 30 wt% of organic material, the shrinkage of the lines upon heating is significant. This shrinkage creates voids in the lines, which results in a smaller amount of percolating pathways for the electrons, thus a higher resistance. The evaporation of the solvent and transport mechanism of the solute is expected to play an important role in this process.³² Besides the shrinkage, the formation of numerous large silver agglomerates also plays a role in the breaking-up of the conductive path, as was reported previously for very thin spin-coated films (<100 nm) of silver nanoparticles by Chou *et al.*³³ When thicker lines are sintered, no cracks are formed at elevated temperature in contrast to the single-layer lines. We believe that, despite the formation of cracks, there is enough material to compensate the vertical shrinkage and to fill the formed cracks. This leaves a sufficiently high amount of percolating pathways within the line, and thus a low resistance.

Conclusions

In summary, we have prepared a silver nanoparticle ink that has a critical curing temperature as low as 80 °C. Good to excellent conductivity values of 10 to 56% of bulk silver have been reached by using a very small amount of organic additives without any strong adsorbing groups such as amines, amides or mercapto groups. This low temperature opens the possibility to produce electronic devices on polymeric foils that have a low glass transition temperature, for example PET. In order to reveal first structure–property relationships and to develop the new ink, the sintering behavior of other inkjet-printed silver tracks based on commercial or home-made inks was studied in detail. Furthermore, the temperature stability of inkjet-printed silver tracks was studied in detail. Three of the four silver inks that were tested revealed a low and stable resistance up to a temperature of 650 °C. A fourth ink showed a loss of the conductance at temperatures above 350 °C when the tracks had a thickness of 1.5 μm. When increasing the layer thickness of the silver tracks, *i.e.* by using multiple inkjet-printed layers, this loss of conductance disappeared and again a low and stable resistance was measured up to a temperature of 650 °C. We believe this effect can be explained by the large amount of organic material

(30 wt%) present in the silver ink. High temperature stable inkjet-printed silver tracks can be used for applications, where high processing temperatures are necessary, for example in plasma display production.

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