Low Temperature Chemical Post-Treatment of Inkjet Printed Nano-Particle Silver Inks

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Abstract

Inkjet printing of electrically conducting tracks often faces the requirement to restrict the temperature/time range of post-processes in order to allow a wider choice of substrate materials. Thermal oven curing of inkjet printed silver nano-particle inks typically requires temperatures well above 100 ºC for several minutes, while laser-based thermal treatment uses higher peak temperatures during shorter periods.

A 'chemical' post-treatment with halogenides in a 'semi-dry' process, which was performed at room temperature or slightly elevated temperatures, was demonstrated to efficiently cure inkjet printed silver nano-particle inks, and to produce electrical conductivities close to that of pure silver. The tests were conducted on several coated paper grades and the chemical post-treatment appears especially well suited for applications in paper-based packaging, where electrical devices add functionality or 'intelligence' to packaging.

Introduction

Printing electrically conducting tracks with silver nano-particles inks is under investigation or already in use in various applications. Such printed conducting tracks are employed in simple passive electrical devices as well as in sophisticated electrical circuits [1]. While high volume applications use conventional printing techniques like flexographic- and gravure printing the high flexibility of digital printing like, inkjet printing, enables economic printing of electrical devices even in low volume applications [2]. This allows to spread printing of electronics into a large number of diversified application areas.

Several manufacturers provide a variety of water-based and solvent-based silver nano-particle inks. A characteristic feature of these inks is the polymeric shell coating each individual silver particle, which prevents coalescence and agglomeration of the silver particles in the ink, and thus allows to produce stable suspensions as needed for printing. The polymeric shells, however, prevent direct contact of the silver particles even after printing, and thus have to be removed in order to provide electrical conductivity throughout the printed pattern. Therefore suitable post-treatment processes are required to remove the polymeric shells around the silver particles. Such curing process is typically performed by thermal treatment either in an oven, by laser irradiation, or IR-treatment [3, 4]. Due to their small size the nano-particles melt at considerably lower temperatures than bulk silver [5]. Thermal post-treatment has therefore been possible at temperatures down to 150 ºC, though requiring long process times of up to one hour [6].

One specific application area for printing of electrically conducting tracks is the paper-based packaging industry, where 'smart packages' are to be equipped with functionality or intelligence. Whenever these functional devices require conducting tracks of low resistance, the use of silver nano-particle inks is mandatory. The usage of paper-based substrates places an additional requirement on the post-treatment process, which should be restricted to temperatures below 100 ºC

The 'chemical' post-treatment technique presented here can be operated even at room temperature. It furthermore was demonstrated to provide electrical conductivity very well comparable with thermal curing techniques, and it therefore appears as a candidate specifically for paper-based applications.

In the following the 'chemical' post-treatment will be introduced and its efficiency in producing high electrical conductivity will be presented in comparison with thermal post-treatments based on oven curing and laser-based rapid thermal processing. Further the different morphologies obtained with the three post-treatments will be shown. The work focused on inkjet printed pattern of silver inks specifically on paper-substrates.

The finding: Curing With a 'Chemical' Post-Treatment

Several manufacturers have produced stable suspensions of nano-sized silver particles in solvent-based or water-based carrier fluids. Agglomeration of the silver nano particles in these inks is efficiently minimized by coating the individual particles with polymeric shells, which prevent contact between the particles by either Coulomb or steric forces. While these silver inks tend to sediment strongly due to the high specific weight of silver, a short agitation of the ink by shaking the container will restore a homogeneous suspension thus proving the absence of agglomeration.

It was found by the authors that a small amount of NaCl salt added to the silver ink resulted in immediate flocculation of the fluid. Even strong agitation could not restore a homogeneous suspension. This was interpreted in the fashion that the addition of the salt had annihilated the repulsive forces of the polymeric shells around the silver nano particles and thereby had caused strong agglomeration. One could conclude from this observation that the salt had either removed or broken up the polymeric shells, and allowed direct, i.e. metallic, contact between the individual silver nano particles. Based on this hypothesis one could apply salt after inkjet printing to the printed and dried pattern, and obtain metallic contact between neighbouring particles and thus electrical conductivity. As will be described in the following it could be experimentally verified that such 'chemical' post-treatment indeed produced highly conductive inkjet printed silver pattern at slightly elevated temperatures and even at room temperature.

Manual Tests With 'Chemical' Post-Treatment

A sequence of experiments was carried out to check the feasibility of the 'chemical' post-treatment. These tests were carried out with the silver inks IJAG-4 from Nippon Paint (water-based; 30% silver weight content), and silver ink AG-IJ-G-100-S1 from Cabot (solvent-based; 20% silver weight content). These inks were inkjet printed onto Ensogloss paper from StoraEnso and dried either at room temperature or at 60ºC on a hot plate or in an oven. Solid pads of dimension 10 mm x 20 mm and thickness of the order 0.5 to 1 µm were inkjet printed to serve as measurement sites of the ohmic resistance of the printed silver layers. The ohmic resistance was measured with a Fluke Multimeter at 2 mm distance between the two probe tips. As expected the silver layers when inkjet printed and when dried did not show any conductivity. The Multimeter resistance reading was off scale, i.e. > 40 MΩ.

'Chemical' post-treatment was performed in a 'manual' fashion in these first experiments. Aqueous solutions of different salts were produced with three different concentrations, namely 0.01 mol/l, 0.08 mol/l, and saturated solutions. For each post-treatment a new cleanroom cloth was folded to produce a stamp, and this stamp was dipped into the aqueous solution. The moistened cloth was thereafter gently pressed with a finger onto the inkjet printed silver pattern for about one second. Since the cloth was not wet but only moist the treated sample was dry again after only a couple of seconds after the treatment (we therefore could call this treatment a 'semi-dry' process). In order to remove remaining salt from the samples a second treatment was conducted, this time with a stamp from a new cleanroom cloth dipped in deionised water. Again the samples dried after a couple of seconds. It is important to note that this manual process of chemical treatment was conducted at room temperature.

Measurements of ohmic resistance performed after the chemical post-treatment revealed different resistance readings depending on the type of salt and the concentration, and showed that high electrical conductivity was obtained with the appropriate salt and at a sufficient concentration. Results from these resistance measurements with a Fluke Multimeter are compiled in table 1. Pads inkjet printed with Nippon Paint silver ink IJAG_4 and Cabot silver ink AG-IJ-G-100-S1 on coated paper of type Ensogloss (from StoraEnso) were post-treated with a variety of salts and reference fluids, namely with NaCl, KCl, $MgCl_2$, NaBr, and NaI, as well as with Na_2SO_4, $MgSO_4$, $NaNO_3$, and NaH_2PO_4. Aqueous solutions of 0.01 mol/l, 0.08 mol/l and saturated solutions were used in the tests. Efficient chemical post-treatment was obtained only with the salts NaCl, KCl, and $MgCl_2$. The ohmic resistance of the respective samples is shown in table 1 for the case of the measurement directly after the chemical post-treatment and for the repeated measurement three days after (Nippon Paint ink IJAG-4) or one day after (Cabot ink AG-IJ-G-100-S1).

It was obvious that a certain salt concentration was required to reduce the resistance of the samples. At low salt concentrations of 0.01 mol/l the resistance of both types of silver ink layers remained at the level of tens of MΩ or beyond the detection limit of 40 MΩ for all three salts. Post-treatment at concentrations of 0.08 mol/l, however, had a most significant effect in reducing the resistance by up to more than 7 orders of magnitude. Specifically the silver ink IJAG-4 from Nippon Paint proved to be well suited for this

		Nippon Silver Ink IJAG - 4		Cabot AG-IJ-G-100S1	
Salt	**Concentration [mol/l]**	**R_{im} [Ω]**	**R_{3days} [Ω]**	**R_{im} [Ω]**	**R_{1day} [Ω]**
KCl	0.01	10M	40M	40M	40M
	0.08	3.0	1.5	30.0	20.0
	0.26 (saturated)	1.0	0.5	2.7	2.0
$MgCl_2$	0.01	20M	40M	14M	30M
	0.08	1.0	3.0	5M	55k
	0.36 (saturated)			2.0	2.2
NaCl	0.01	17M	20M	10M	40M
	0.08	10.0	5.0	6M	3M
	0.27 (saturated)			2.5	1.7

Table 1. *The table compiles the resistance of inkjet printed pads on Ensogloss coated paper (from StoraEnso) as measured with a Fluke Multimeter (2 mm distance between the tips), both immediately (Rim) after post-treatment as well as 1 day (CABOT silver ink) and 3 days (Nippon Paint silver ink) afterwards.*

chemical post-treatment.All three salts in table 1 resulted in very similar and low resistance already at 0.08 mol/l. Post-treatment with a saturated aqueous solution of KCl showed a further but small improvement, which indicated that the curing of the silver ink had about reached its low resistance limit. When repeating the resistance measurements three days later the original results were essentially confirmed. The slight decrease in resistance might on the one hand indicate that the curing process had not yet terminated directly after the chemical post-treatment, but on the other hand the small decrease could be within the data scatter. The samples with Cabot silver ink AG-IJ-G-100S1 did not cure as efficiently. Post-treatment with aqueous solutions at 0.08 mol/l did not result in resistance as low as with Nippon Paint ink IJAG-4, and furthermore the differences in resistance were high for the different salts. While almost no reduction of resistance was obtained with 0,08 mol/l NaCl solution, the post-treatment with 0.08 mol/l $MgCl_2$ solution resulted in a reduction of resistance by more than 10^3 and that one with KCl in a reduction of resistance by more than 10^6. The relatively low resistance for $MgCl_2$ was obtained only after one day after the chemical post-treatment, which indicated that the curing process was not complete at the first measurement right after the post-treatment. When post-treating the Cabot silver ink samples with aqueous solutions of saturated salt concentration the resulting ohmic resistance data were very low and very similar for all three salts. This was interpreted as an indication that the curing process was complete, and that the samples had reached their lowest possible ohmic resistance. The measurements were repeated one day after post-treatment and the original measurements were confirmed.

Specifically the tests with the Cabot silver ink showed that KCl salt was most efficient, followed by $MgCl_2$ and NaCl. The aqueous solutions of NaI and NaBr resulted in resistance readings 10^2 and 10^4 times higher than with NaCl, respectively. Post-treatment with aqueous solutions of Na_2SO_4, $MgSO_4$, $NaNO_3$, and

NaH_2PO_4 did not result in any reduction of resistivity. Apparently the halides are key to efficient curing and of those specifically the chlorine salts, which produce the halogen ions with the smallest size, as compared with bromine and iodine. It was assumed that these small size chlorine ions can best diffuse through the polymeric shell and attach to the silver particles to form insoluble silver salts and thus displace the polymer shell. The larger size of the bromine and iodine ions, and specifically those large anions of the sulfates, nitrates and phosphates would impede diffusion through the polymeric shell, thus leaving the polymeric shell intact. This would be in accordance with the experimental finding that the latter agents did not produce any electrical conductivity at all.

Table 1 does not provide bulk resistivity data, but is sufficient to indicate which salts and which aqueous concentrations were required to produce high electrical conductivity in the inkjet printed silver layers.

The conclusions drawn from these first sets of measurements were that inkjet printed silver pattern could be chemically post-treated by salts like KCl, $MgCl_2$ or NaCl to produce highly conductive layers, and that this process could be carried out at room temperature. In the following chapters the efficiency of the chemical post-treatment is compared with the standard thermal post-treatment techniques for curing of inkjet printed silver layers.

Producing Inkjet Printed Test Samples for Sheet Resistance Measurements

The comparison of the three post-treatment techniques was conducted with IJAG-4 silver ink from Nippon Paint, printed on three different coated paper grades, Epson Premium Photopaper, as well as Ensogloss and Printocart, both from StoraEnso.

The IJAG-4 silver ink was jetted with a Xaar760 GS3 printhead. Rheological parameters of this ink were 2.34 mPas viscosity and 32 mN/m surface tension at a temperature of 25ºC. The viscosity of this ink was actually lower than the recommended viscosity range for Xaar760 GS3 printheads, but well controlled greyscale inkjet printing was possible although only in a limited frequency range up to 3 kHz. Figure 1 shows stable drop formation for all four dpd ('drop per dot') greyscale levels at a firing frequency of 1 kHz. Drop volumes of all four dpd-levels scaled well with increasing dpd-level as shown in table 2. Drop velocities of 1 dpd and 2 dpd levels were low, calling for a fine tuning of the rheological parameters of the ink. However, at the low paper feed rates of 5 and 10 mm/s high quality pattern could be inkjet printed.

Solid pad pattern of 20 mm x 20 mm size were inkjet printed with IJAG-4 silver ink with 4 dpd greyscale printing at 5 mm/s and 720 dpi linear resolution. Two interleaved scans at the printhead's inherent 360 dpi resolution resulted in a total print resolution of 720 x 720 dpi. The thickness d_{ag} of such printed silver layers on the paper surfaces could not be measured and was thus calculated as 380 nm for one single solid layer printed at 720 x 720 dpi.

The evaluation of sheet resistance and bulk resistivity of the inkjet printed silver samples was performed with a four-point probe. The printed test pattern of 20 mm x 20 mm size were sufficiently large as compared with the 2 mm probe tip spacing s, and the layer thickness d_{ag} was very small compared with the tip spacing s so that the sheet resistance R_{sheet} could thus be calculated as R_{sheet} = 4.53 V / I, with the voltage V and current I at the four-point probe [7]. The bulk resistivity Rho of the electrically conducting material follows as Rho = $R_{sheet} \times d_{ag}$. Bulk resistivity data for the different post-treatment techniques are compiled and discussed below.

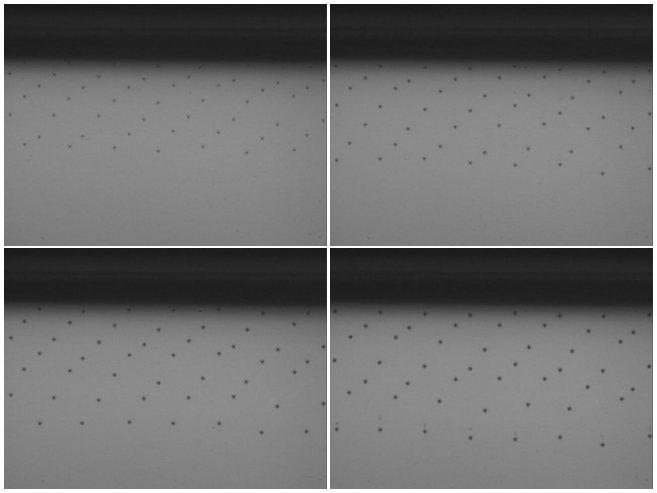

Figure 1. *stroboscope images showing all four dpd-levels operating a Xaar 3pl greyscale printhead with Nippon Paint IJAG-4 silver ink at 1 kHz firing frequency*

Grey Level	1 dpd	2 dpd	3 dpd	4 dpd
Drop speed [m/s]	4.7 ±0.2	5.0 ±0.2	5.7 ±0.2	6.2 ±0.2
Drop volume [pl]	4.7 ±0.5	8.2 ±0.6	13.8 ±0.9	15.6 ±0.9

Table 2. *Drop speed and drop volumes for the different greyscale dpd-levels as measured for inkjet printing Nippon Paint IJAG-4 silver ink with Xaar 3 pl greyscale printhead*

Thermal Post-Treatment in an Oven

The conventional method of curing inkjet printed silver pattern is thermal post-treatment in an oven or on a hotplate. Paper substrates should not be exposed to high temperatures since they dry up and deform already at temperatures beyond 80ºC. Since this is too low a temperature for thermal curing of silver ink the thermal post-treatment was carried out at a temperature of 150ºC for the three coated papers. The results of the four-point probe measurements are compiled in table 3, and discussed below.

Post-Treatment With Laser Radiation

Instead of long term thermal curing at temperatures of the order 150ºC a post-treatment at very high temperatures but very short time duration can be used. Such 'rapid thermal processing' was tested with a diode pumped cw Ytterbium fiber laser (LFC10; 1072nm) Different laser power levels, spot sizes, scan line distances and scan speeds were evaluated to identify the optimum operation regime for each paper grade. At typical spot sizes of 50µm and a laser scan speed of 650 mm/s the cw laser irradiation resembled a pulsed laser irradiation with 77µs pulse length impinging onto the silver ink pattern. During this short interaction time temperatures well in excess of 1000ºC are expected in the

silver ink layer. As shown in table 3 this rapid thermal laser process proved to be an efficient post-treatment and resulted in very low resistance data for all three paper grades.

Chemical Post-Treatment

In order to avoid variations due to the manual process as described above a semi-automated process was used for chemical post-treatment. A cleanroom cloth was moistened in a saturated aqueous NaCl solution and placed on top of the paper substrate with the inkjet printed silver pads. These sandwiched silver ink patterns were then fed into a simple desktop laminator operating at 95ºC and a paper feed rate of 4 mm/s. Two consecutive runs were performed in the laminator with each sample.

Substrate	Post- Treatment Process	Rho [μΩ cm]	Rho / Rho $_{pure\ Ag}$
Epson Premium Photopaper	Thermal (oven), t = 10s, T = 150°C	14	9
	Thermal (Laser), P = 15.8%, v = 650 mm/s	7	4
	Chemical	**8**	**5**
Enso Gloss (StoraEnso)	Thermal (oven), t = 24h, T = 150°C	7000	4000
	Thermal (Laser), P = 20.5%, v = 500 mm/s	38	24
	Chemical	**40**	**25**
Printocart (StoraEnso)	Thermal (oven), t = 24h, T = 150°C	650	406
	Thermal (Laser), P = 20.5%, v = 500 mm/s	16	10
	Chemical	**9**	**6**
Glass (Reference)	Bar coated, d_{Ag} = 5.7 μm, t = 5min, T = 300 °C	4	3

Table 3. *Compilation of resistivity data obtained with the three post-treatment techniques investigated. The test samples were inkjet printed with Nippon Paint IJAG-4 silver ink onto three different paper grades. The right hand column shows the obtained resistivity as multiples of the bulk resistivity of pure silver, 1.6 μΩ cm.*

Comparison of the Post-Treatment Processes

To compare the curing efficiency of these three different post-treatment processes the sheet resistances of the post-treated samples were measured, and the bulk resistivities were calculated as described above, and compiled in table 3. Included in table 3 is the resistivity of Nippon Paint IJAG-4 silver ink when bar coated onto glass, and thereafter thermally post-treated for 5 minutes at 300ºC. At this high curing temperature the lowest possible resistivity should be obtained, and can thus be used as a reference. It was calculated as 4 μ cm, and is thus about 3 times the resistivity of bulk pure silver of 1.6 μ cm, as stated in the right hand column of table 3.

On Epson Premium Photopaper the three post-treatment techniques gave very similar results. Very low resistivity data were obtained, all of them within only one order of magnitude above the resistivity of bulk pure silver. This was interpreted to be due to the high surface smoothness of the Epson Premium Photopaper.

The inkjet printed silver layers on the coated paper Printocart (StoraEnso) could be very efficiently cured with the chemical post-treatment, yielding resistivities six times that of pure silver, while specifically the thermal oven treatment produced poorer results, some two orders of magnitude worse than the chemical post-treatment, while the thermal laser-based post-treatment gave similar good results as the chemical post-treatment.

Curing on the coated paper Ensogloss (StoraEnso) proved to be more difficult. Again both the chemical post-treatment and the thermal laser-based post-treatment showed results two orders of magnitude better than the thermal oven-based post-treatment, but their respective resistivity data where about a factor 3 higher than for Printocart substrates.

Summed up the results from table 3 show that curing of inkjet printed silver layers on a variety of coated papers can indeed be efficiently carried out with the chemical post-treatment, here demonstrated with IJAG-4 silver ink from Nippon Paint chemically post-treated with a saturated aqueous solution of NaCl. The curing efficiency of the chemical post-treatment compares well with a well tuned thermal laser-based process, and is superior to a thermal oven-based treatment performed at 150ºC.

Morphology of Inkjet Printed Silver Ink Layers Before and After Post-Treatment

SEM-evaluation of inkjet printed (Nippon silver ink IJAG-4) samples on coated paper before and after post-treatment showed different morphologies of the cured silver layers for the three post-treatment processes investigated. Figure 2 shows a top view of the silver ink sample before post-treatment. Apparently the individual particles did not connect to each other due to the polymeric shell around each individual particle. Indeed this sample had essentially no electrical conductivity.

After thermal post-treatment in an oven the individual small particles had clearly grown together to form larger grains, which built a relatively dense silver layer. The polymeric shells around the individual particles were apparently removed during the thermal curing process, and in fact this silver layer, as shown in figure 3, provided high electrical conductivity.

During the chemical post-treatment process the silver nano-particles coalesced to grains of size similar to those obtained in the thermal oven curing. These grains formed a rigid framework structure (see figure 4), less compact as compared with the silver layers cured thermally in the oven. Although being carried out at room temperature or temperatures below 100ºC the chemical post-treatment resulted in efficient coalescence of the silver particles. Since the coalescence is an irreversible process it was possible without damage to the coalesced silver framework to remove residual salt in a following process step, e.g. by stamping the sample with a cloth soaked in deionized water.

Rapid thermal post-treatment with laser radiation resulted in coalescence of the individual particles in the form of strings, which built up the very open framework shown in figure 5. This morphology of the silver layer can be explained by forcefully driving out any residual low vaporizing material during the short (<100 μs) but hot (>1000ºC) process. Cracks as seen in figure 5 are a problem with rapid thermal processing due to the large and short thermal cycling.

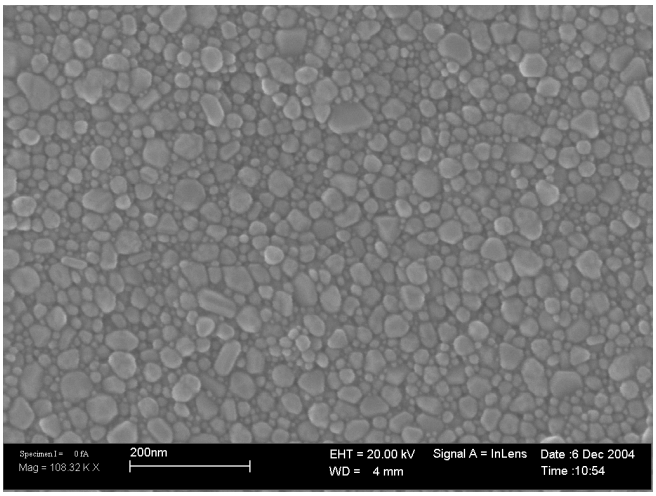

Figure 2. *Top view of the silver ink layer (IJAG-4) before post-treatment. The individual particles were still coated with a polymeric shell. They did ot touch each other and the sample had no detectable electrical conductivity. The scale indicates 200 nm.*

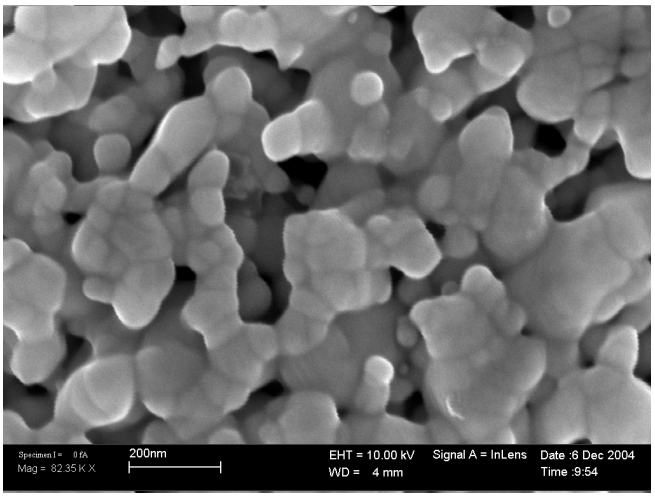

Figure 4. *Top view of a chemically post-treated silver layer. The individual particles had coalesced into large grains, which formed a silver metal framework and yielded high electrical conductivity. The scale indicates 200 nm.*

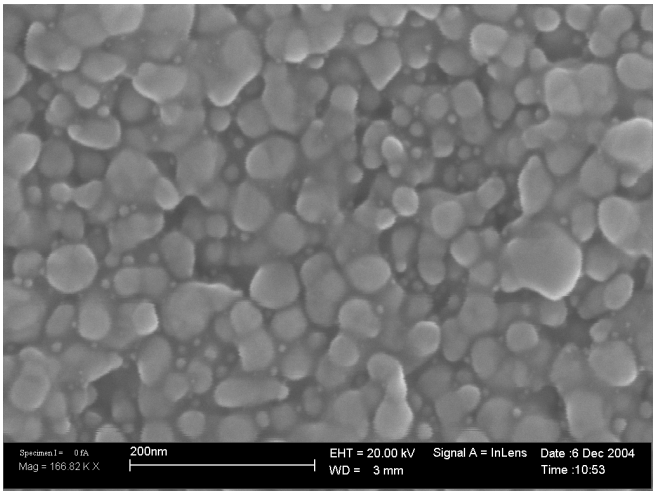

Figure 3. *Top view of the silver layer after thermal curing in an oven at 150°C. The polymeric shells around the particles were removed during thermal curing and the individual particles had grown together to form an electrically conductive structure. The scale indicates 200 nm.*

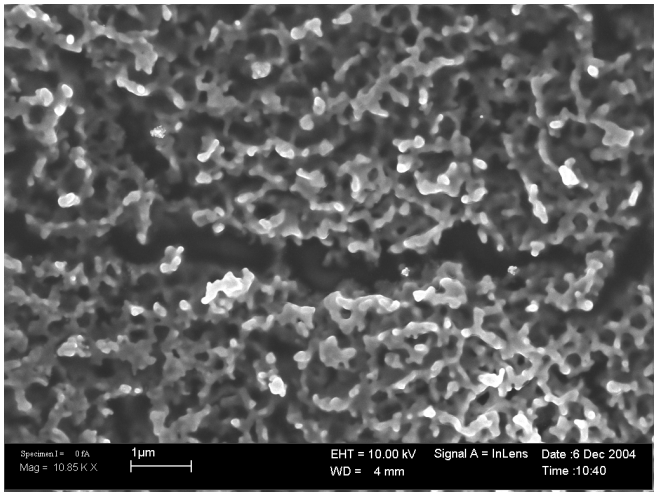

Figure 5. *Top view of a silver layer after rapid thermal post-treatment with laser radiation shows a very open framework. Observe that the scale indicates 1 µm, when comparing with figures 2 - 4.*

All three post-treatment processes are effective in removing the polymeric shell around the individual silver nano-particles. The coalescence of the particles into larger grains and macroscopic structures, however, is decisively different. As is known thermal curing at temperatures above 150ºC for periods as long as 1 hour yields compact silver layers [4], while rapid thermal processing with lasers results in very porous morphology and the possibility of cracks in the sintered layer. Chemical post-processing produced silver layers with a relatively compact framework, i.e. a morphology with porosity between that of oven-cured and laser-sintered silver layers. It is obvious that chemically post-treated silver layers can very well be used in dc- and low-frequency applications. Whether and how the skin effect affects the electrical performance at very high frequencies due to the specific silver framework morphology has to be investigated further. On the other hand the relatively open but rigid silver metal framework of chemically post-treated silver layers is expected to be more mechanically flexible than the compact silver as produced in thermal (oven, hotplate, IR) curing.

Together with the low process temperatures (room temperature or slightly elevated) such mechanical flexibility makes the chemical post-treatment an ideal choice for paper-based packaging applications where electrical devices add ‘intelligence’ to packaging.

Conclusions

The chemical post-treatment described here proved to efficiently cure inkjet printed silver pattern on paper surfaces such that the pattern having no detectable electrical conductivity after printing were converted into layers with electrical conductivity close to that of pure bulk silver. The curing efficiency compared very well with that of conventional thermal oven- or laser based post-treatment techniques. The layer morphology produced with chemical post-treatment was a rigid metal framework of coalesced silver grains.

Specific with the chemical post-treatment is the low process temperature – the technique works even at room temperature. This makes the chemical post-treatment an ideal choice for paper-based packaging applications, where electrical devices add functionality or 'intelligence' to packaging.

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