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Laser induced forward transfer of Ag nanoparticles ink deposition and characterization

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ABSTRACT

In this work, we report on the printing of silver nanoparticles (Ag NPs) ink by means of laser-induced forward transfer (LIFT) process. The optimum conditions for printing circular shaped features using a Nd:YAG laser at 266 nm have been examined. A study of the influence of the laser fluence and the use of a pre-coated intermediate layer (sacrificial layer) on the donor substrate was performed in order to understand how these parameters affect the printed droplets morphology. We also provide a detailed discussion of the influence of the annealing temperature on the printed features morphology and on their resistivity. Based on these results, the conditions have been determined for printing uniform circular shaped droplets with a diameter as small as 25 μm and an average thickness of 150 nm. Atomic force microscopy on the cured printed droplets revealed a uniform surface morphology with no coffee ring effect. Finally, conductive features with reasonably low resistivity (approximately eleven times that of bulk silver) and at sufficiently low sintering temperatures (100–150 °C) were produced on silicon oxide on silicon and flexible polyimide substrates.

1. Introduction

In recent years, metal nanoparticles inks, due to their unique properties, have attracted great attention from not only the scientific community but the industries as well. Metal nanoparticles such as Au, Ag, and Cu have been widely studied as promising functional materials for conductive inks, as they exhibit high conductivity (~105 S/cm) and low sintering temperature [1,2], which allow the printing of conductive patterns on flexible substrates. The most common processes used for high resolution metal patterning of microelectronics are vacuum metal deposition and photolithography. An alternative to these expensive, time-consuming and labor-intensive methods are printing processes such as gravure [3], screen [4], transfer [5], ink-jet [6] and laser printing [7,8] which reduce manufacturing costs. In addition, lately, laser sintering processes have been widely studied [9–11]. This approach is a low temperature sintering process which enables a high-resolution patterning and minimizes the heat-affected zone and the thermal damage to the substrate.

Here, we present the printing of silver nanoparticles (Ag NPs) ink by means of the laser induced forward transfer (LIFT) process [12]. LIFT is capable of high spatial resolution printing of metallic nanoparticle ink droplets. In addition, LIFT can be applied to inks with a wide range of rheological properties, including both low viscosity NP inks [8] and high viscosity NP pastes [13]. On the contrary, ink-jet is limited by the clogging of nozzles when high viscosity inks are used [14]. LIFT of such inks has been used recently to print features with different shapes and sizes [13], as these parameters have become controllable [15]. Printing droplets with a well-controlled diameter and shape will be useful for future printing of more complex structures in one single printing step. For example, the laser printing of well-defined Ag lines with only 30 μm width has become possible by juxtaposing adjacent droplets [16,17].

The LIFT process consists of the laser transfer of a part of an Ag NP ink layer from a quartz donor substrate placed in close proximity to a receiving substrate, as shown in Fig. 1a. The transfer is carried out using a single pulse. In order to avoid direct exposure of the material to be transferred to the incident laser pulse, the donor substrate was pre-coated with a 40 nm titanium layer (Fig. 1b), usually referred as dynamic release layer (DRL) or sacrificial layer. The most common sacrificial layers are either metallic layers (such as Ti, Au, Pt, Cr) [18–20] or UV-sensitive photopolymers (such as triazene polymers (TP), polyimide film) [21,22]. By using metallic sacrificial layers, the incident beam reaches a very thin volume of the metal layer where

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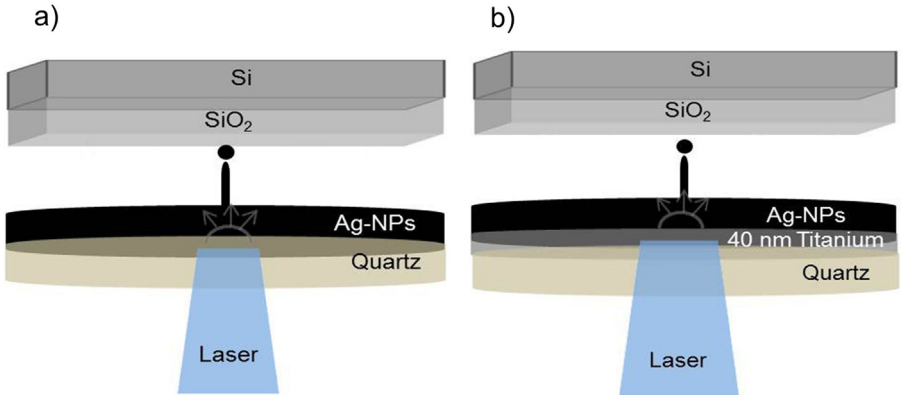

Fig. 1. Schematic view of the liquid phase LIFT process: (a) without using sacrificial layer: the laser pulse is absorbed by the Ag NPs layer resulting to the ejection of an Ag NP ink jet. (b) Using titanium sacrificial layer: Laser absorption within a titanium (Ti) film results in vaporization of the adjacent ink, producing a vapor pocket, the expansion of which provides the impulse force for the ejection of the Ag NP material.

it gets converted into heat and leads to melting and local vaporization. As a result, at the interface Ti/Ag NP ink a vapor pocket is formed, the expansion of which provides the impulse force for the ejection of an Ag-droplet. For comparison, donor substrates were also prepared without using the titanium sacrificial layer to evaluate the influence of the sacrificial layer on printing quality.

Our main objective was to accurately print patterns of Ag inks using the laser-based low temperature process with high spatial resolution. This will enable the direct printing of metallic inks on flexible substrates for organic electronic applications.

In this context, we investigated the influence of the laser fluence on the printed droplets morphology (reproducibility, spatial resolution) and analysed the resistivity and microstructural evolution of Ag NPs features as a function of annealing temperature.

2. Experimental

2.1. The LIFT process

The liquid phase LIFT process used in this work has been analytically described previously [23].

The laser used for our experiments is a pulsed Nd:YAG laser operating at 266 nm with a pulse duration of 10 ns. A high power mask projection system composed of a variable circular mask and 15× objective lens was used to achieve an almost top-hat beam profile at the imaging plane. The distance between the donor and the receiving substrate was about 300 μm; the laser spot size was 50 μm for the basic parametric study (i.e., droplet diameter dependence on fluence for both donor configurations), while a 20 μm laser beam spot size was used for printing Ag NP lines. Both the donor and the receiver substrate were translated by means of a computer-controlled *x*–*y* motion system (*X*–*Y* stages driven by a LabVIEW program). A CCD camera enabled accurate alignment of the target and substrate materials. The laser fluence was adjusted via a motorized controlled attenuator plate.

2.2. Preparation of donor substrates

Two different kinds of donor substrates were prepared in order to find the best conditions for the transfer and deposition of the material. The donors were prepared by spin-coating (2900 rpm/30 s) a silver NP ink (purchased from SunChemicals, 20 wt.% silver content, solvent: mixture of ethylene glycol, glycerol and ethanol, viscosity: 12 cP, NP size: 80 nm) on a 1 mm thick circular quartz substrate (25 mm in diameter) purchased from UQG Optics (Type 1 donor) and on a similar quartz substrate pre-coated with 40 nm thick titanium sacrificial layer (Type 2 donor). Spin coating ensured homogeneous deposition and film thickness (~5 μm) uniformity of the Ag NP ink on the donor substrate.

The donor substrate was then inverted and placed above the receiver substrate with a small gap in ambient air environment. The receiver substrates used in this study were either wet silicon dioxide (1 μm thick) on silicon substrates or polyimide films (Good-Fellow). No additional treatment was applied at the donors, which were used for a maximum period of 30 min after their preparation to avoid silver ink film drying. Measurement of the donor mass over time revealed an insignificant (4.5%) reduction of the mass of the spin coated silver NP ink layer within a 30 min period. Therefore, we have considered the silver NP ink film as wet and uniform within the time period that each donor was used.

The reflectivity of the titanium/Ag NP ink/quartz donors at 266 nm was experimentally defined to be around 25%, while in the case of the Ag NP/quartz donors the reflectivity was negligible.

2.3. Characterization

Film thickness measurements were performed on a profilometer (Veeco Dektak 150, tip radius 12.5 μm), with a stylus force of 10.00 mg. Micrographs were taken with an optical microscope (Leica) coupled with a digital camera system (Canon PowerShot S50). The size and shape of the sintered Ag NPs, and the microstructures of the printed droplets were observed by scanning electron (FESEM Nova NanoSEM 230) and atomic force microscopy (Veeco diInnova). Typical I–V measurements were performed with an HP4140B pico-amperometer.

3. Results and discussion

3.1. Influence of the donor substrate on printed droplets

Initially, we studied the ink transfer without any sacrificial layer (Type 1 donor). Fig. 2a shows an optical microscopy image of a laser printed microarray of the silver NP ink. Each droplet was transferred onto silicon dioxide on silicon substrates (SiO_2/Si) for a specific range of laser fluences (varying from 40 to 100 mJ/cm^2). The observed threshold laser fluence for the transfer of a droplet with a 50 μm diameter was 50 mJ/cm^2. As the laser fluence increased, satellite droplets and debris appeared around the deposits. It was found that the printed droplets expanded to a 70 μm diameter and lost their shape uniformity at a laser fluence even as low as 100 mJ/cm^2. All in all, the optimum transfer conditions were between 60 and 80 mJ/cm^2 which is a very narrow laser fluence processing window.

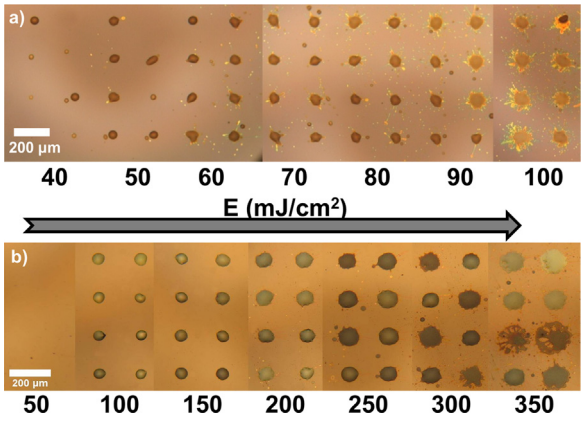

Fig. 2. Optical microscope image of laser-printed droplets of silver nanoparticles ink for different energies from (a) Ag NPs ink/quartz donor substrate (b) Ag NPs ink/Ti/quartz donor substrate.

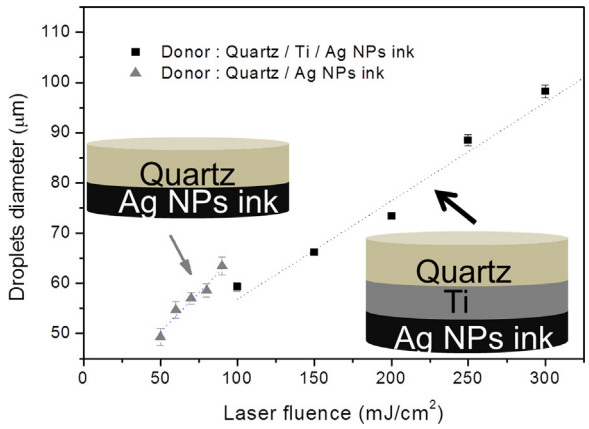

Fig. 3. Plot of the laser printed Ag NPs droplets diameter vs. laser fluence energy: gray triangles are without the sacrificial layer, black rectangles are with 40 nm Ti sacrificial layer.

As previously mentioned, the use of a sacrificial layer prevents the direct exposure of the material to be transferred from to the incident beam. In this context, the ink transfer from donor substrates pre-coated with 40 nm Ti layer (Type 2 donor) was investigated. The minimum laser fluence required to print reproducible circular shaped droplets was 100 mJ/cm^2. At this low fluence, droplets with a 60 μm diameter were printed with no debris around them as shown in Fig. 2b and Fig. 4. In addition, it was observed that for laser fluences between 100 and 250 mJ/cm^2 the printed droplets kept their circular shape and the debris were limited and only appeared above 200 mJ/cm^2. This effective laser fluence processing window is wider than the one observed for printing without using a sacrificial layer. Above 300 mJ/cm^2, many debris appeared around the printed droplets which expanded to a 100 μm diameter.

These results are in accordance to our recent work on the dynamics of the silver NP ink ejection during the LIFT process [18], where considerably different ejection velocities were observed for the two different types of donors. The explanation of the different dynamics was attributed to the main mechanisms involved in the laser pulse energy to impulse pressure conversion for each LIFT configuration [18]. The latter is governed by either heat diffusion (LIFT with sacrificial layer) or field enhancement and cavitation around the NPs (LIFT without sacrificial layer). The reflection of the different ejection mechanisms on the deposition quality can be clearly seen in Fig. 2a and b. When a sacrificial layer is used, the low velocity and directional ejection results to well define droplets free of satellite drops for a wide fluence range due to smooth impingement and accumulation of the Ag ink material onto the substrate. These printing results are of great importance to a potential commercialization of the process, were high process stability is essential (i.e., high tolerance to processing instabilities, wide laser fluence processing window, control of droplet size/morphology).

The dependence of the droplet diameter on the laser fluence is depicted in Fig. 3 for both donor configurations. In the case of Ti covered donors, the droplets diameter expands linearly with laser fluence, varying from 60 μm to 100 μm for laser fluence ranging from 100 mJ/cm^2 to 300 mJ/cm^2. On the other hand, in the case of donors without Ti, the droplets diameter varies from 50 μm to 65 μm for laser fluence ranging from 50 mJ/cm^2 to 90 mJ/cm^2. Rapp et al. [8] observed similar dependence of the droplet diameter on the laser fluence for the effective laser fluence range (i.e., printing of circular droplets). However, they reported an effective laser fluence range shifted to significantly higher fluences.

3.2. Morphological characterization of the printed droplets

Printing of Ag NPs inks by means of ink-jet usually results in a non-uniform droplet morphology, which is attributed to the "coffee ring" effect [24]. A ring-like deposit along the perimeter of a drying droplet is obtained due to migration of particles to the edge of the drop [25]. By using optimized processing conditions (i.e., use of a titanium sacrificial layer, laser spot size 20 μm), a microarray of droplets was obtained on SiO_2/Si substrate for a laser fluence of 100 mJ/cm^2. The deposited droplets were then cured at a temperature of 100 °C for 60 min to evaporate the solvent. SEM visualization of the printed microarray after the curing is illustrated in Fig. 4a.The observed droplets had a uniform circular shape with a diameter as small as ~25 μm and an average thickness of 150 nm. The AFM image of a single cured droplet revealed a uniform surface morphology with no coffee ring effect, as shown in Fig. 4b. The elimination of the coffee ring effect may be attributed to the different evaporation rates between the solvents of the Ag ink: ethanol (boiling temperature T_b = 78.4 °C), glycerol (T_b = 290 °C) and ethylene glycol (T_b = 197.3 °C). It is assumed that the differences in the evaporation rates of the solvents cause an additional Marangoni-type flow during the drying phase and thus obtaining morphologically uniform deposits [24].

3.3. Influence of the curing temperature

3.3.1. On Ag NP sintering

The curing temperature is defined as the temperature where the organic solvent of the Ag ink evaporates and the particles start showing conductance by direct physical contact. Sintering takes place at higher temperature when all the organic solvent has been totally evaporated and necks begin to form between particles. The melting temperature of bulk silver is 960 °C, but Ag NPs start to melt at lower temperatures (~150 °C) [26].

In order to study the curing treatment, Ag NPs ink droplets deposited on SiO_2/Si and polyimide substrates under optimum processing conditions (i.e., use of a sacrificial layer, 100 mJ/cm^2) where dried and sintered in an oven at temperatures varying from 70 to 150 °C isothermally held for 60 min. The samples were then gently cooled down to room temperature.

Fig. 5a–c illustrate the resulting SEM images of the silver lines on SiO_2/Si samples. As expected, we see an increase in grain size with increasing sintering temperature. More specifically, at 70 °C, each silver nanoparticle could be easily distinguished as it is shown in Fig. 5a. After heating at 100 °C, the sintering effect became obvious

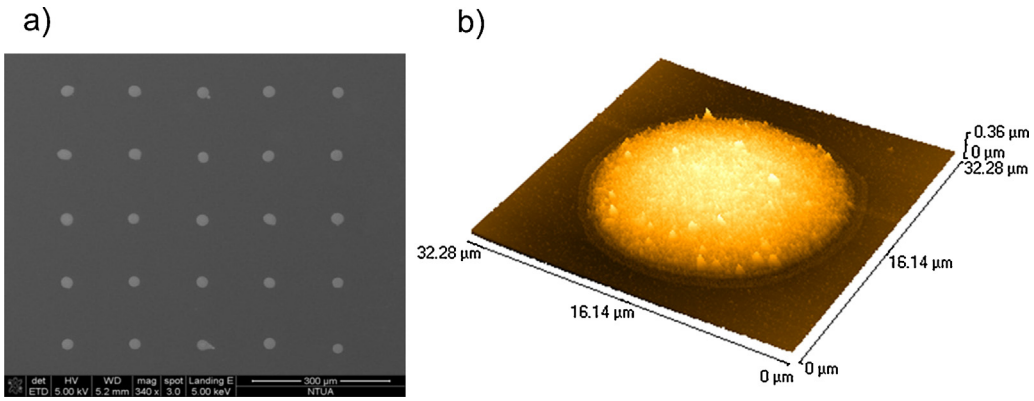

Fig. 4. Laser printed Ag NPs droplets from Ag NPs ink/Ti/quartz donor substrate: (a) SEM image of laser printed droplets at 100 mJ/cm2 and cured at 100 °C for 60 min, (b) AFM 3D image of the printed droplet: uniform surface morphology (without coffee ring effect).

and the size of particles increased (Fig. 5b). Above 150 °C, densification of the silver particles occurred as shown in Fig. 5c.

To further investigate the curing treatment of the silver ink, samples of printed silver lines on polyimide substrate were also oven heated at 85 and 150 °C for 60 min as depicted in Fig. 5d and e, respectively. It can be seen that when the sample is heated at 85 °C (Fig. 5d) the silver nanoparticles form small agglomerates with many voids in between. When increasing the temperature to 150 °C, as shown in Fig. 5e, the agglomerates have grown and the voids have minimized, forming a denser layer. It is believed that the morphology of sintered Ag layers with curing temperature plays a crucial role in determining the electrical transport properties [27,28]. In order to study the effect of sintering temperature, electric resistance measurements were performed on printed Ag lines.

3.3.2. On resistivity

LIFT printing of Ag NP lines was carried out in order to investigate the influence of the curing temperature on the NP sintering. The lines were printed on both SiO_2/Si and polyimide substrates by using optimum laser printing conditions (i.e., use of a sacrificial layer, printed with a laser fluence of 100 mJ/cm^2) and appropriate adjusting of the separation distance between the droplets. An overlap of about 30% was selected to result in relatively uniform lines with sharp edges.

Fig. 6 shows the variations in the resistivity of laser printed Ag lines as a function of the curing temperature. Note that the resistivity dependence with the curing temperature of the printed pattern on the polyimide substrate is similar to that of the printed pattern on the SiO_2/Si substrate. For both cases, as the curing temperature increased from 70 to 100 °C, the resistivity of the printed line drastically decreased. From 100 to 200 °C the resistivity tends to saturate. The lowest resistivity ($\sim 1.8 \times 10^{-5}\ \Omega$ cm which is about eleven times that of bulk silver) was achieved for a curing temperature of 150 °C for the printed lines on SiO_2/Si substrate. This temperature dependence of the resistivity on the curing temperature may be associated with the surface morphology of the Ag NP ink patterns described in the previous section were with increasing temperature the silver ink particles agglomerate into larger clusters forming a silver network, and thus resulting in lower resistivity

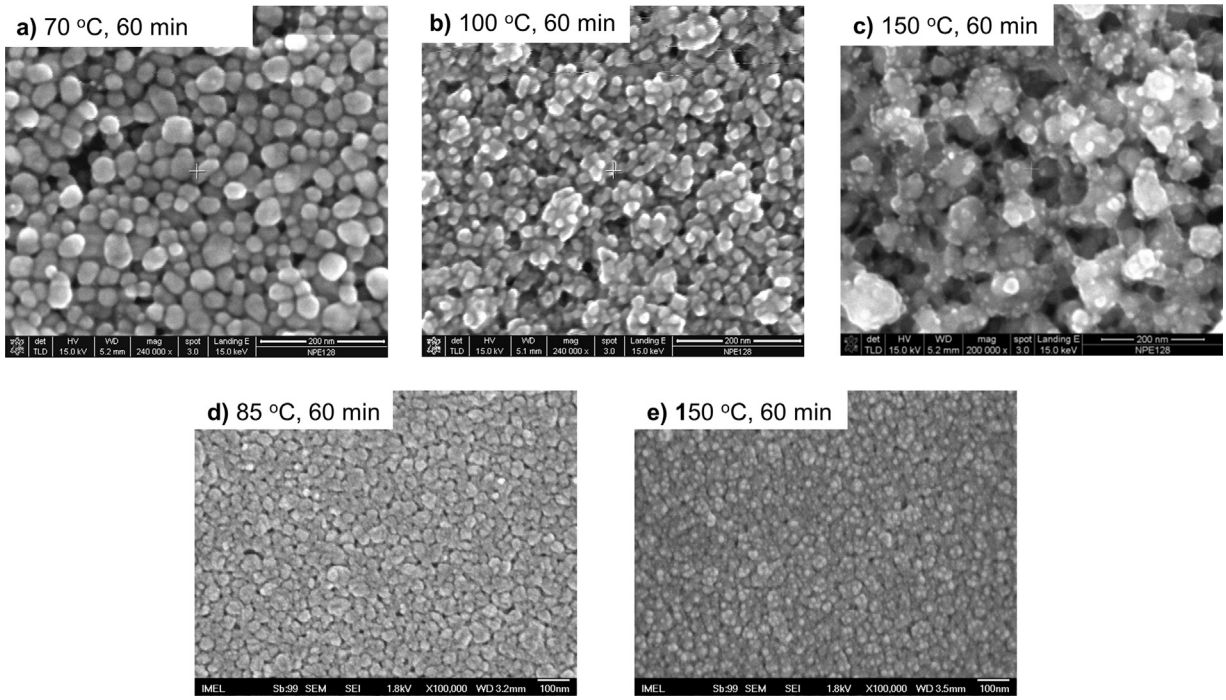

Fig. 5. SEM pictures depicting the surface morphology of LIFT printed Ag NP ink patterns on SiO2/Si (a–c) and polyimide (d and e) substrates for various treatment temperatures.

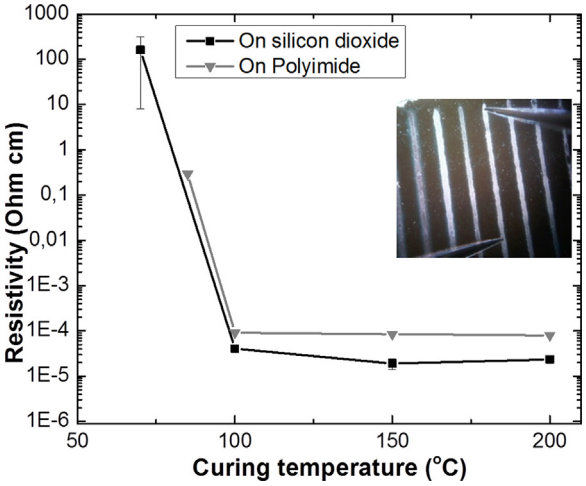

Fig. 6. Resistivity of Ag lines, printed with LIFT on SiO2/Si and polyimide substrates and cured at various temperatures for 60 min.

values. In further analysis, the electrical resistivity of the silver lines on polyimide substrate decreased from $2.9 \times 10^{-1}\ \Omega$ cm at 85 °C to $9.1 \times 10^{-5}\ \Omega$ cm at 100 °C, to $8.4 \times 10^{-5}\ \Omega$ cm at 150 °C. This shows that the electrical resistivity is sufficiently low to behave as an electrical conductor, even though the silver nanoparticles were sintered at relatively low temperatures. The applicability of the sintering process at low temperatures between 100 and 150 °C enables the production of conductive features with reasonably low resistivity on more flexible substrates, such as polyethylene naphthalate (T_g = 120 °C) and polycarbonate T_g = 147 °C).

4. Conclusions

This work has shown that reproducible, circular and uniform Ag droplets could be printed for a wide laser fluence range processing window when using a thin Ti sacrificial layer. In addition, no "coffee ring" effect was observed on the printed droplets from the mixed-solvent-based Ag NPs ink. The ability of LIFT to print both a low and high viscosity ink allows the printing of various ink compositions with high boiling point solvents and thus minimizing or eliminating the "coffee ring" effect. Also, LIFT printed Ag NP lines on SiO_2/Si and polyimide substrates cured at a sufficiently low sintering temperature (150 °C for 60 min) exhibited resistivity of $1.8 \times 10^{-5}\ \Omega$ cm and $8.4 \times 10^{-5}\ \Omega$ cm, respectively. These values are close to the resistivity of the bulk silver ($1.59 \times 10^{-6}\ \Omega$ cm). Further work and optimization on the laser sintering of printed Ag NP lines on temperature-sensitive substrates would open new possibilities for applications in flexible and plastic electronics.

Acknowledgments

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