

Research on Printed Conductive Silver Layer based on Inkjet Photo Paper

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Abstract. Printed electronics is an emerging technology that applies traditional printing or coating processes to the manufacture of electronic devices and products. In order to find a low-cost, high-performance, environmentally-friendly flexible substrate suitable for electronic devices, the printability between four kinds of inkjet photo papers and nano-silver ink was investigated. First, different surface morphologies of the inkjet photo papers were measured by a confocal laser scanning microscopy. Then, a pen and a gravure printer were used to test the printability between photo papers and nano-silver ink. It was found that the conductive track and pattern was influenced by the surface morphology of the photo papers. Furthermore, a four-probe test showed that the conductivity of the ink layers on the four photo papers was almost at the same level. Furthermore, a tearing test with 3 M tapes showed that the silk photo paper had the best tearing resistance. In general, silk photo paper has the best overall performance. This research could be beneficial for the development of flexible electronic devices which are low-cost, mass manufacture suitable and environment friendly.
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[DOI: 10.2352/J.ImagingSci.Technol.2022.66.2.020407]

1. INTRODUCTION

Printed electronics involves printing methods used for fabricating electronic devices on various substrates [1–3]. A notable feature of printed electronics is the use of flexible substrates, which reduces production costs and allows fabrication of mechanically flexible devices [4]. Although the current printed electronic devices do not reach the level of traditional silicon-based microelectronic devices in terms

of resolution, integration, and information capacity, printed electronic has the advantages of resource conservation, environmental protection, light weight, low cost, flexibility and large-scale production [5, 6]. While electronic manufacturing technology typically uses rigid substrates like plastic board and silicon, printed electronics can utilize flexible plastic substrate and paper [7–9]. There are mainly two aspects of the research on the conductive ink printed substrate. On the one hand, the corresponding pre-treatment of the printed substrate is made to match with the conductive ink to promote the printed conductive ink to achieve good results. On the other hand, according to the selected base material, suitable conductive inks and sintering printing methods are designed so as to achieve a good conductive effect without exceeding the tolerance of the printed substrate [10].

Paper is a sheet made of plant fibers, which is used for writing, printing and packaging, and it is generally divided into offset paper, coated paper, wood-free paper, letterpress paper, corrugated paper, etc. Compared with plastic substrate, paper substrate has many advantages, such as low cost, mass production, good processing performance, excellent printing performance, light weight and recyclable [11–14]. However, paper substrate also has disadvantages. For example, the ambient temperature and humidity have a great influence on the strength of paper and paperboard. The change of air temperature and humidity will cause the paper and paperboard to change the moisture balance. Finally, the mechanical properties will deteriorate. However, flexible electronic devices need to be used in a variety of complex environments, such as wet or dry outdoor environment. In addition, the manufacturing process also has certain

Received June 18, 2021; accepted for publication Sept. 26, 2021; published online Nov. 3, 2021. Associate Editor: Min Xu.

1062-3701/2022/66(2)/020407/10/\$25.00

requirements to the substrates. Ordinary paper materials cannot meet these requirements. Inkjet photo paper is inkjet paper crafted specifically for printing photograph [15–17]. It is bright white due to bleaching or due to pigments such as titanium dioxide, and has been coated with a highly absorbent material that limits diffusion of the ink. Highly refined clay is also a common coating layer to prevent ink spreading. Inkjet photo paper has several unique advantages, such as waterproof, sun protection, long-term preservation, versatile and suitable for pigment and dye ink printing in a variety of printer types [18–20]. Therefore, inkjet photo paper has better performance and can meet the requirements of flexible electronic device manufacturing and application.

In this research, four kinds of inkjet photo papers sold in the market were investigated with laboratory-made nano-silver ink. In order to study the printability between the conductive ink and the inkjet photo papers, a pen and a gravure printer were used to apply the conductive ink on the inkjet photo papers. Firstly, using a confocal laser scanning microscopy (CLSM), the surface morphology and three-dimensional topography of the inkjet photo papers were analyzed. It was found that different inkjet photo papers have different surface features. Then, the surface morphology, three-dimensional morphology and cross-section of the inkjet photo papers after the application of the nano-silver ink were analyzed. Finally, combined with the four-probe test and the 3 M tape tearing test, the printed conductive layer on the inkjet photo papers was comprehensively analyzed.

2. MATERIAL AND METHODS

The inkjet photo paper purchased from IMACOLOR is microporous resin-coated. The base of the inkjet photo paper is the same as traditional photographic paper, which is coated with polyethylene layer on one side of the base paper. The surface layer of the inkjet photo paper was coated with nano-scale silica material (the particle diameter below 150 nm) to form absorbing layer. Four types of the inkjet photo paper are glossy photo paper, silk photo paper, pearl photo paper and satin photo paper. The weight of the glossy, silk, pearl and satin are 230 g/m^2 , 260 g/m^2 , 240 g/m^2 and 260 g/m^2 , respectively.

The ink used in this research was nano-silver based conductive ink prepared in our laboratory. The silver content of the ink is 60 wt% and the solvent is isopropanol and ethylene glycol. The ink uses poly-vinylpyrrolidone (PVP) as the dispersant for stabilization. The average diameter of the silver nanoparticles is about 100 nm. The viscosity is about 600 cp. After applying on substrates, the ink needs to be dried at 80°C for 10 min.

A pen and a gravure printer (AYDJ, Shanghai Modern Environmental Engineering Technology Co., Ltd.) is applied to transfer the conductive ink to the inkjet photo papers. The gravure printer has a rectangular pattern with $35 \text{ mm} \times 45 \text{ mm}$. The cells on the gravure plate are inverted pyramid shape. The ink was injected into the pen. Then the track was drawn on the inkjet photo papers. The printing steps

are as follows: first, adjust the knob on the gravure printer, so that the scraper and cylinder can be adjusted to the right position. Then, cutting four kinds of inkjet photo paper into $24 \text{ mm} \times 12 \text{ mm}$ rectangular shape, wrap the paper on the surface of the roller and stick it with transparent adhesive. Furthermore, 1 ml nano-silver conductive ink is evenly spread on the gravure cylinder with syringe. Finally, the pattern was printed on the photo paper when the cylinder was rotated. The samples with ink track or pattern were immediately sent to an oven. The sintering treatment in the oven is conducted at 80°C for 10 min. After thermal post-treatment, the conductive track and layer on the inkjet photo paper were obtained. The conductive track and layer were characterized by a confocal laser scanning microscopy (VK-X200 series, KEYENCE). In this research, the CLSM was a major experimental apparatus. The CLSM provided optical images and three-dimensional images. In addition, it was capable of measuring the size of the target object in the image and the height of the surface undulation. The four-probe tester (RTS-9, Guangzhou Four Probe Technology Co., Ltd.) was used to test the resistance of the conductive layer on the four photo papers. 3 M tape was used to test the adhesion between the conductive layer and the inkjet photo papers.

3. RESULTS AND DISCUSSION

3.1 The Morphological Analysis of the Inkjet Photo Papers

According to the information provided by the manufacturer and visual observation, the feature of the glossy photo paper is very smooth and shiny. Notable feature of the silk photo paper is its strong texture, with small concaves or pits while that of the pearl photo paper is clear texture, crystalline and sparkling. The feature of the satin photo paper is soft and delicate.

Through the observation of the confocal laser scanning microscope (Figures 1 and 2), it can be found that the surface of the glossy photo paper is very flat, and only a few cracks exist. There are evenly arranged elliptical concaves on the surface of the silk photo paper, and the area outside the concave is relatively flat. The surface of the pearl photo paper is very rough and has large undulations. The surface morphology of the satin photo paper is similar to the pearl photo paper, but its undulations are relatively smaller. The 3D images of the inkjet photo paper generated by software based on optical images also show these morphological features. Through CLSM software analysis, it can be found that the average surface height difference of the glossy photo paper is only $1.75 \mu\text{m}$ which is the lowest. Due to the presence of the concave, the average surface height difference of the silk photo paper is $42.55 \mu\text{m}$. The average surface height difference of the pearl photo paper and the satin photo paper is $23.15 \mu\text{m}$ and $17.45 \mu\text{m}$, respectively. The results are shown in Table I. The greater the surface height difference of the photo papers, the greater the surface roughness is. In a printing process, photo paper with a rough surface absorbs more ink easily and dries faster. In addition, through the calculation of the laser confocal microscope software, the surface roughness of the photo paper can be obtained.

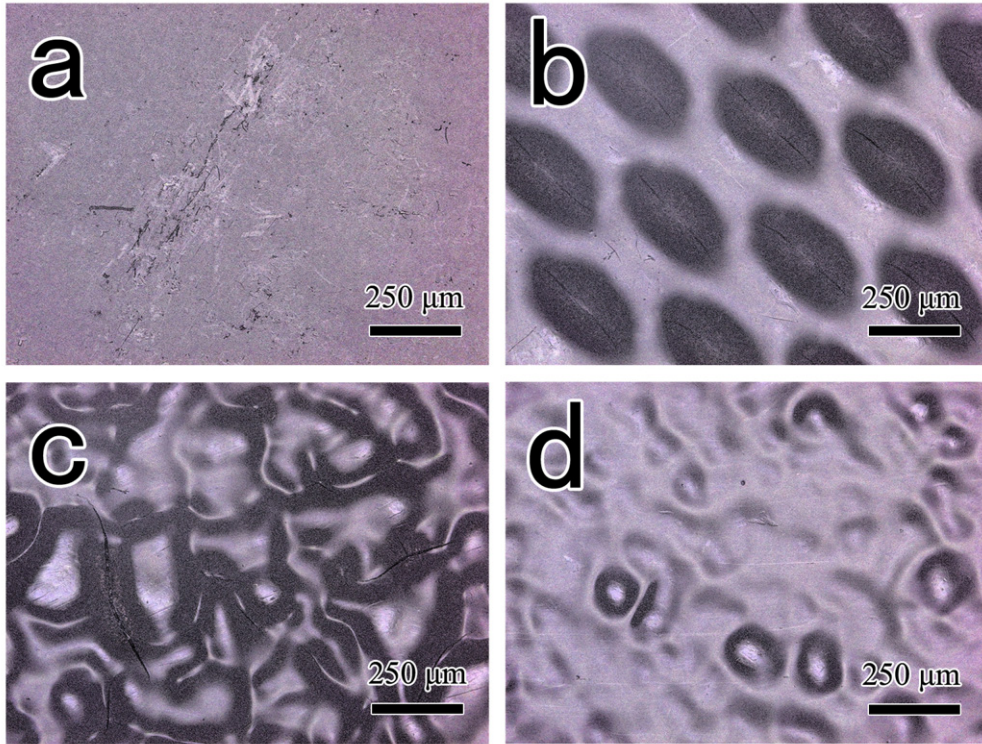


Figure 1. The morphology of the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

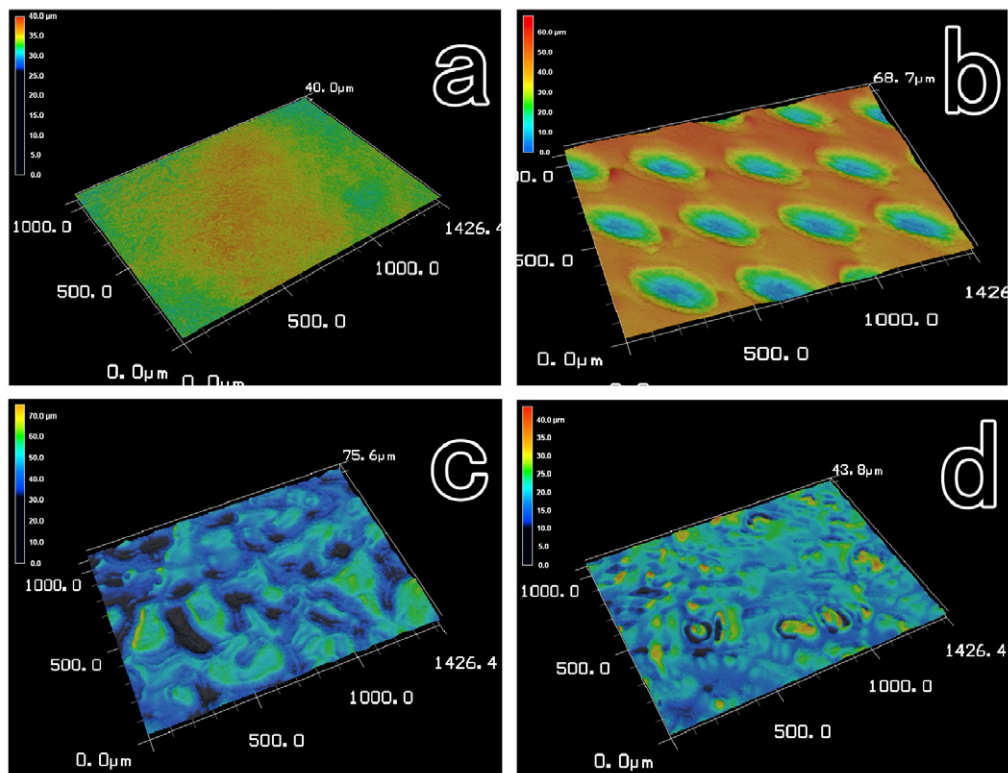


Figure 2. The 3D images of the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

The surface roughness of the glossy, silk, pearl and satin are $1.27 \mu\text{m}$, $10.28 \mu\text{m}$, $12.87 \mu\text{m}$ and $7.5 \mu\text{m}$, respectively. This

result is consistent with the observation of 3D topography pictures in Fig. 2.

Table 1. The average height difference of the inkjet photo papers.

| Inkjet photo papers | Glossy | Silk | Pearl | Satin |
|--|--------|-------|-------|-------|
| Average height difference/ μm | 1.75 | 42.55 | 23.15 | 17.45 |

3.2 The Analysis of the Inkjet Photo Papers with the Ink Track

Since the nano-silver ink from a pen can be easily written on the substrate, it can be used to initially characterize the printability of the photo papers. Through the observation of CLSM (Figures 3 and 4), the ink track by pen-writing were shown. It can be seen that the nano-silver ink track on the glossy photo paper is uniformly distributed. The edge of the ink track is flat and the roughness of the ink track is low. The distribution of the nano-silver ink track on the silk photo paper is influenced by the concave. Part of the ink track fell into the concave area and the edge of the ink track is relatively straight. It can be seen that the color of the silver layer on the silk-faced photo paper is brighter. This is due to its larger surface roughness, which absorbs more solvents and additives in the ink, which makes the silver layer denser. The edge of the nano-silver ink track on the pearl photo paper is clear. The ink track is also affected by the surface undulation of the pearl photo paper. Moreover, the distribution of the nano-silver ink is also not uniform. The nano-silver ink track on the satin photo paper is relatively straight and uniform. The ink track can be clearly detected in the 3D image, because the glossy

photo paper and the area outside the concave of the silk photo paper are relatively flat. The surface roughness of the pearl photo paper and the satin photo paper is high so that the ink track is submerged in the fluctuation of the surface of the inkjet photo paper, which is not easy to find. However, there are still some signs that there is almost no deep valley in the area of ink track due to the filling of the nano-silver ink.

Combined with the analysis of the cross-sectional view (shown in Figure 5), the distribution state of the ink layer on the photo papers can be further analyzed. The bottom layer of the inkjet photo papers is resin-coated film. The middle layer is paper base. The top layer is the nano-scale absorbing layer. The top layer will prevent nano-silver particles of the ink from permeating to the middle layer. The nano-silver particles of the ink exist only in shallow area at the top layer. It indicates that the sandwich structure of the inkjet photo papers successfully controls the deposition of the nano-silver particles. Therefore, a uniform conductive layer can be formed on the surface of the photo papers.

3.3 The Analysis of the Inkjet Photo Papers with the Ink Layer

It can be seen from Figure 6(a), the coverage of the printed nano-silver ink on the glossy photo paper is very high, almost 100%. It is related to high smoothness of the glossy photo paper. Moreover, because the surface of the glossy photo paper is very smooth, the ink cells on the surface of the gravure cylinder are apparently influenced the printed conductive layer on the glossy photo paper. The ripple

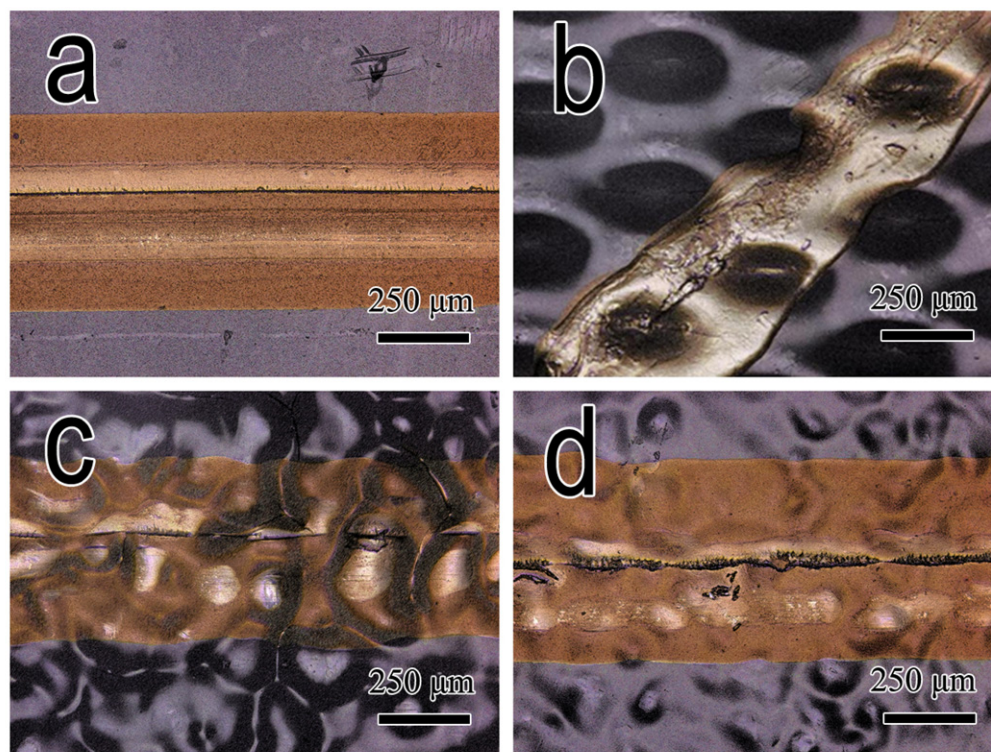


Figure 3. The morphology of the conductive track on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

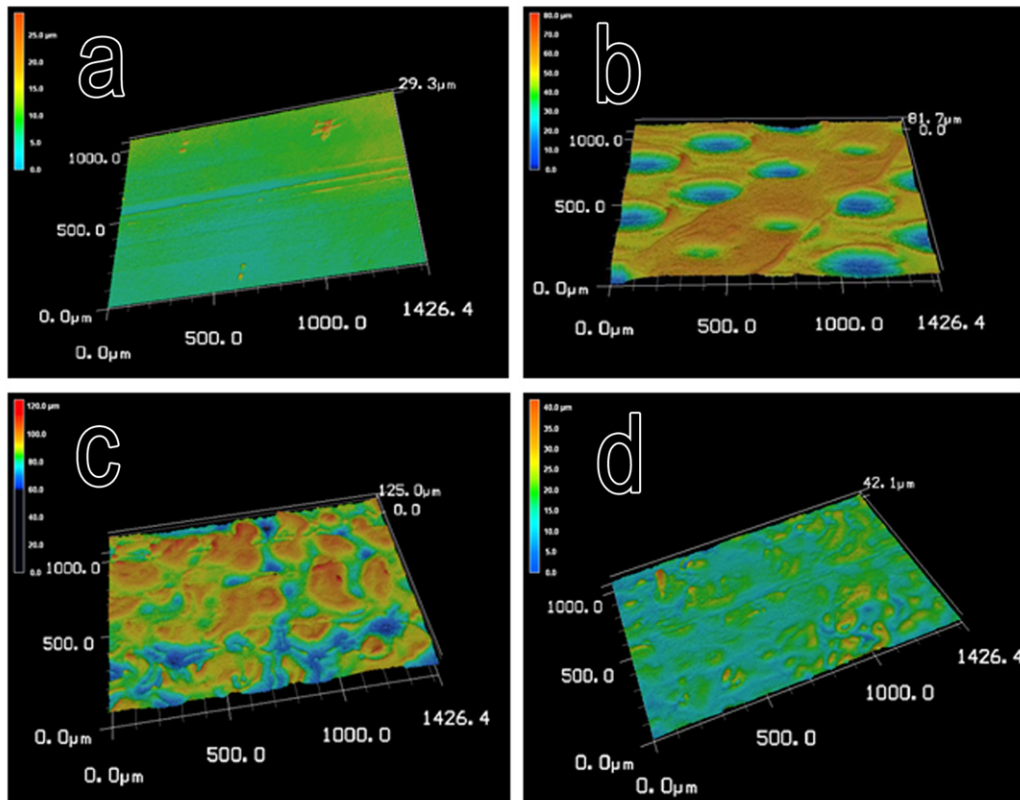


Figure 4. The 3D images of the conductive track on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

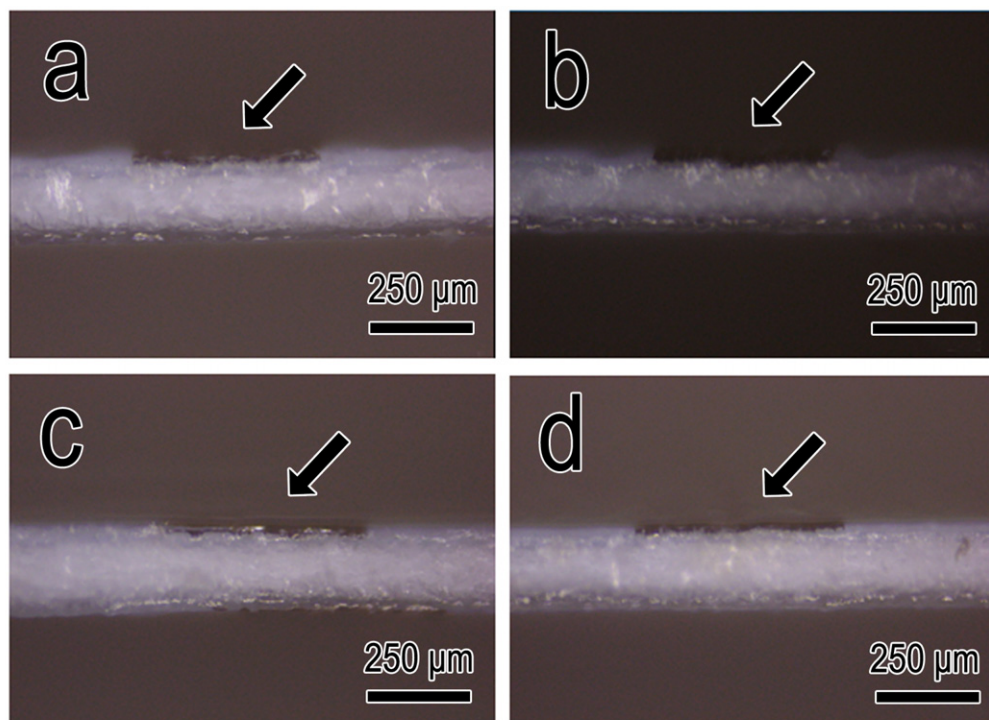


Figure 5. The cross-section picture of the conductive track (indicated by the arrow) on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

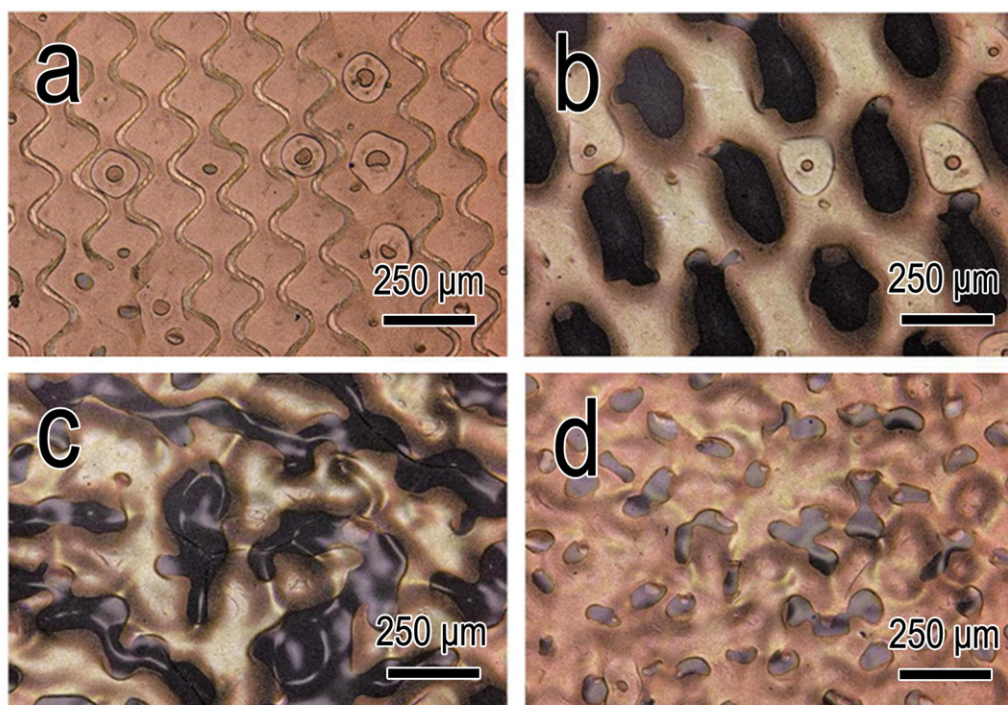


Figure 6. The morphology of the printed conductive layer on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

periodically appeared on the nano-silver ink layer. Although, the structure and materials of commercial inkjet photo paper are trade secrets. Typically, the ink-absorbing layer of the photo paper uses nano-scale titanium oxide or zinc oxide. Therefore, its surface wettability is very good, and it easily blends with water-based, alcohol-based or solvent-based inks. In Fig. 6(b), it can be seen that there is no nano-silver ink in the concave area, since the gravure cylinder can't contact the bottom of the concave when printing. Therefore, the nano-silver ink can't be transferred into the concave due to the large depth of the concave of the silk photo paper. The area outside the concave was printed with nano-silver ink. The ink coverage is very high. As for the pearl photo paper, the lower area was also unable to contact with the cylinder in the process of printing, since the surface roughness is high. Only the shallow area was printed, thus there is also a considerable area without nano-silver ink covering, as shown in Fig. 6(c). In Fig. 6(d), only a few areas are not covered by the nano-silver ink, because of the surface roughness of the satin photo paper is relatively small. It shows that the printed ink coverage is mainly determined by the surface roughness of the inkjet photo papers. The 3D images of the inkjet photo papers with printed ink layer can be seen in Figure 7. The printed pattern is apparent on the glossy photo paper, due to the ink cells of the gravure cylinder. The rectangular-like pattern is slightly different from the previous ripple pattern, but it is more realistic in revealing the thickness of the printed ink layer on the glossy photo paper. The 3D image shows the depth of the concave of the silk photo paper is still persists after printing. Moreover, deep and shallow valley areas of the pearl and satin photo paper are shown, due to lack of contact

with the cylinder. Calculated by the software of the confocal microscope, the average thickness of the dried silver layer is $1 \pm 0.2 \mu\text{m}$. Since the gravure ink is not heavy, the thickness of the dried silver layer is relatively thin after the solvent absorbing and thermal post-treatment.

The cross-section of the printed inkjet photo paper can be seen in Figure 8. It can be seen that the distribution of the printed conductive ink layer on the inkjet photo papers is different from the conductive track drawn by the pen. With the pen drawing line, the tip of the pen is in full contact with the surface of the inkjet photo papers. The nano-silver ink penetrates well into the ink absorbing layer. Therefore, the side views of the inkjet photo papers with pen drawing are similar. However, the side views of the printed conductive layer on the inkjet photo papers are different, due to the characteristics of the inkjet photo papers. It can be seen that the continuous conductive film layer only can be clearly visible in the side view of the glossy photo paper. For silk photo paper, the conductive layer is divided into equal length by periodic spaces, since there is no ink in the concave. For pearl photo paper and satin photo paper, the conductive layer has poor consistency, due to the presence of partially irregular ink-free regions. It can also be seen from the cross-sectional pictures that the nano-silver particles penetrated into the ink-absorbing layer of the photo paper, in the relatively thin dried silver layer.

By using the four-probe tester to measure the conductivity of four gravure printed photo papers, the measurement was tested on 20 points and averaged to reduce the experimental error. The result can be seen in Table II. It can be seen that the conductivity of the printed conductive layer

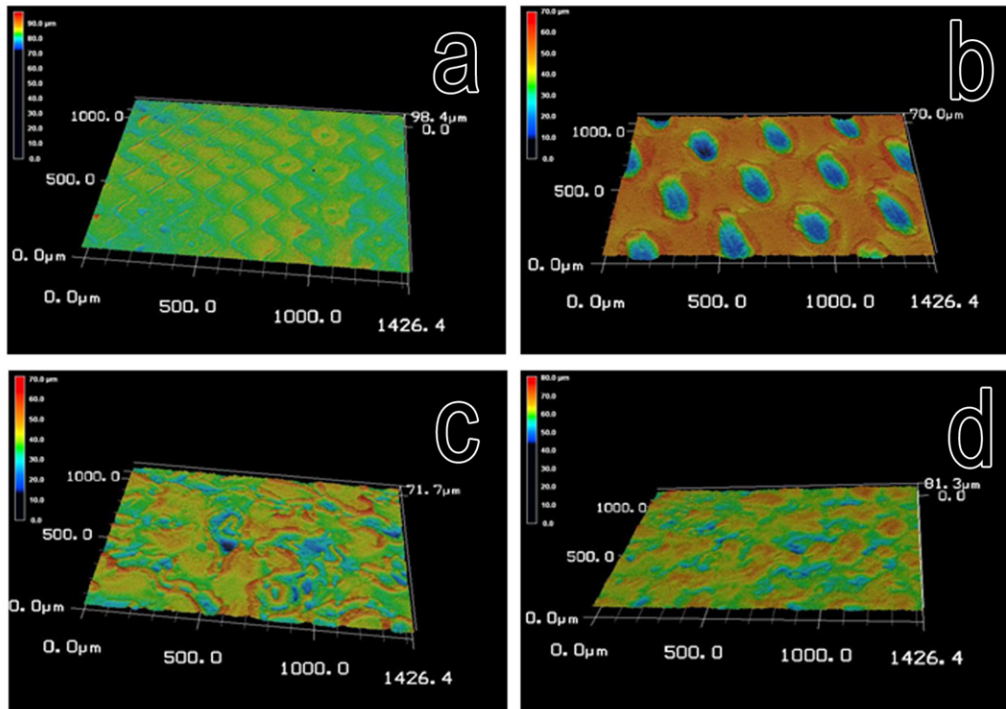


Figure 7. The 3D images of the printed conductive layer on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

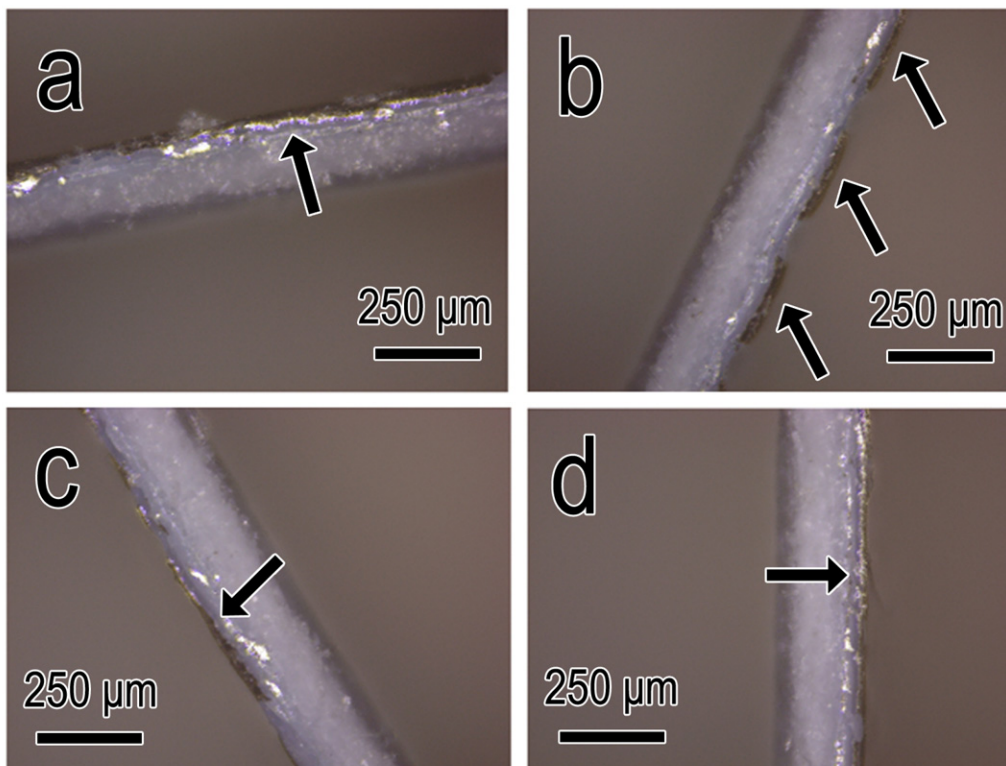


Figure 8. The cross-section picture of the conductive layer (indicated by the arrow) on the inkjet photo papers by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

of the inkjet photo papers is almost at the same level, since a sufficient amount of nano-silver ink is provided during printing. The thickness of the conductive layer is relatively

large. Therefore, the property of the inkjet photo papers is no longer a limiting factor to the conductivity of the printed conductive layer.

Table II. The average surface resistance of the conductive layer on the inkjet photo papers.

| Inkjet photo papers | Glossy | Silk | Pearl | Satin |
|---|------------|------------|------------|------------|
| Average surface resistance Ω/\square | 48 ± 8 | 42 ± 9 | 51 ± 5 | 44 ± 8 |

3.4 The Analysis of the Inkjet Photo Papers with the Ink Layer after Tearing

In order to investigate the mechanical property of the conductive layers on the inkjet photo papers, the tearing test to the conductive layers of the inkjet photo papers was conducted. The conductive layer was torn with 3 M tape to obtain the relationship between the number of tearing and the square resistance of the conductive layer. As shown in Table III, it can be seen that only the conductive layer of the silk photo paper still has conductivity after 1 time tearing. The surface resistance can't be measured on the other three inkjet photo papers after 1 time tearing. All the inkjet photo papers with the conductive layer could not measure the surface resistance after two times tearing. Therefore, the anti-tearing performance of the silk photo paper is the best.

After the tearing test with 3 M tape, the edge of the tearing area on the inkjet photo papers was characterized by the CLSM. As can be seen from Figures 9 and 10, the tearing area of the glossy photo paper has little ink residue. The tearing edges are neat, leaving only wavy stripes. The wavy stripes were imprinted by the walls of the gravure cells. The printing pressure at the stripe is large, so the ink

Table III. The average surface resistance of the conductive layer before and after tearing on the inkjet photo papers.

| Inkjet photo papers | Before tearing | Tearing 1 time | Tearing 2 times |
|---------------------|---------------------------|---------------------------|-----------------|
| Glossy | $48 \pm 8 \Omega/\square$ | / | / |
| Silk | $42 \pm 9 \Omega/\square$ | $55 \pm 7 \Omega/\square$ | / |
| Pearl | $51 \pm 5 \Omega/\square$ | / | / |
| Satin | $44 \pm 8 \Omega/\square$ | / | / |

of the stripes does not fall off easily. Thus, it was unable to measure the surface resistance after tearing, since most of the conductive layer was torn. For the silk photo paper, the conductive layer that has been torn by 3 M tape still has a layer of ink film remaining. The residual ink film is relatively neat and flat. Therefore, the silk photo paper is amenable to measure the surface resistance after one tearing. The surface of the pearl photo paper is rough and the conductive layer on the raised area of the inkjet photo papers was easily torn. The edge of the tearing area was irregular. The edge of the tearing area of the satin photo paper was apparent and the tearing edge was serrated. The reason why the conductive layer of the pearl photo paper and the satin photo paper cannot be measured after being torn possibly due to the uneven surface structure. The undulating surface structure makes the conductive network of the conductive layer three-dimensional rather than planar. The conductive layer in the upper region is most likely to be destroyed after

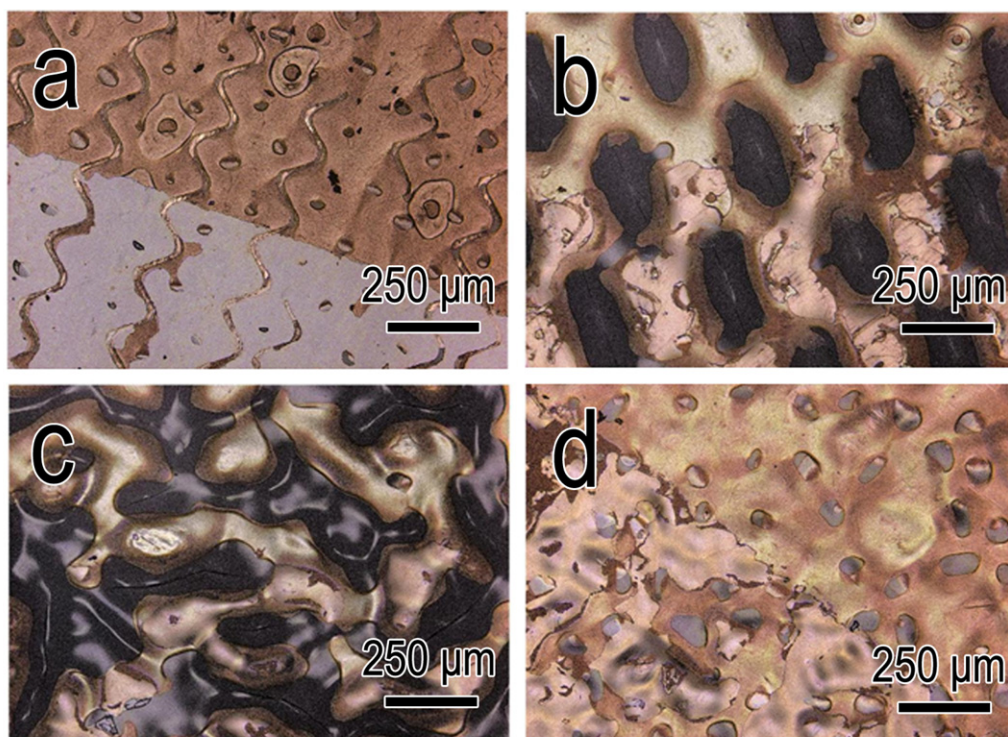


Figure 9. The morphology of the printed conductive layer with/without tearing by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

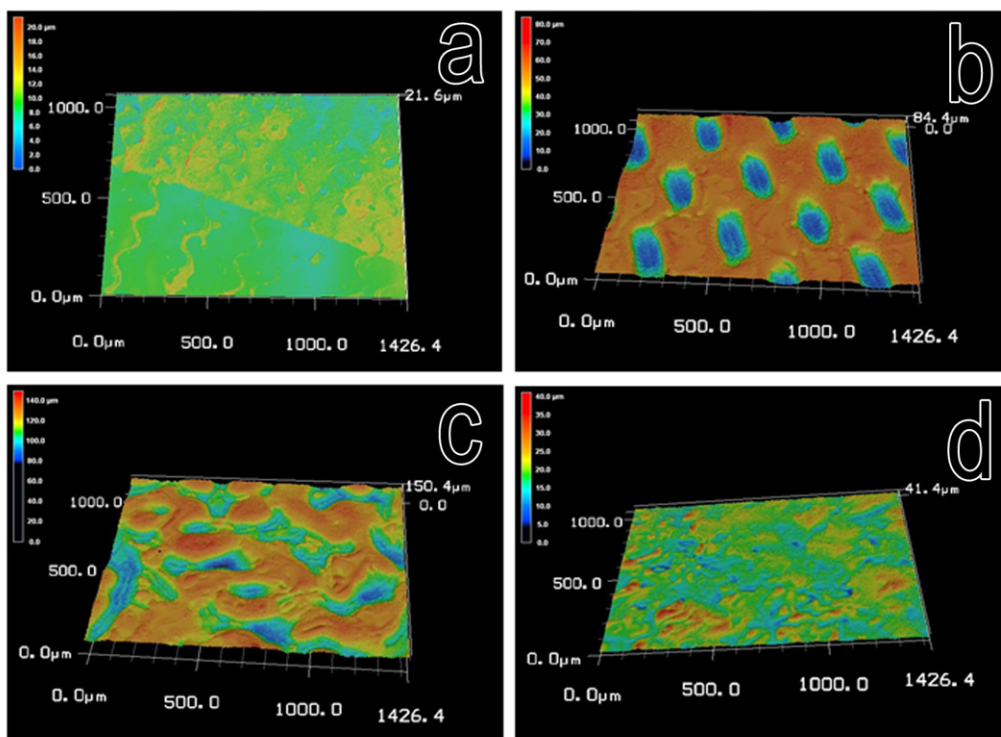


Figure 10. The 3D images of the printed conductive layer with/without tearing by the CLSM ((a) glossy; (b) silk; (c) pearl; (d) satin).

tearing, resulting in the connection being broken and thus unable to conduct electricity. However, silk photo paper is different. Except for the concave structure, the rest area on the surface of the silk photo paper is flat and broad, which is one reason why it can withstand the tearing test.

In addition, the thickness variations of the conductive layers before and after the tearing was tested by using the software of the CLSM. It was found that the thickness of the conductive layer on the glossy photo paper is changed from about $1\ \mu\text{m}$ to $0\ \mu\text{m}$, and the peeling ratio is about 100%. The thickness of the conductive layer on the silk photo paper changed from about $1\ \mu\text{m}$ to $0.8\ \mu\text{m}$, and the peeling ratio is about 20%. The thickness of the conductive layer on the pearl photo paper is changed from about $1\ \mu\text{m}$ to $0.7\ \mu\text{m}$, and the peeling ratio is about 30%. The thickness of the conductive layer of the satin photo paper is changed from about $1\ \mu\text{m}$ to $0.5\ \mu\text{m}$, and the peeling ratio is about 50%. The above data is basically consistent with the results of the surface resistance of the photo papers after the tearing. The main reason for the loss of conductivity of photo papers is that all or part of the conductive layer is peeled off. The conductive layer on the silk photo paper has the best adhesion and the strongest anti-peeling performance.

4. CONCLUSIONS

In this research, four inkjet photo papers and the printability between the photo inkjet papers and the nano-silver ink were investigated. The surface of the glossy photo paper is very flat. The surface of the silk photo paper exist concave, while the area outside the concave is relatively flat. The surface of the

pearl photo paper and the satin photo paper are relatively rough. The cross-section pictures showed that when the nano-silver ink was used by the pen and the gravure printer, it has a different penetrating effect on the ink absorbing layer of the inkjet photo papers. The conductivity of the inkjet photo papers with printed conductive layer is almost at the same level. All the photo papers demonstrate good printability to the nano-silver ink and printer. The adhesion of the conductive layer on the silk photo paper is the best, while the glossy photo paper is the poorest. Overall, silk photo paper has the best overall performance on anti-tearing, surface conductivity and roughness. This work could be beneficial for the development of low-cost, environmentally friendly paper-based electronic devices.

ACKNOWLEDGMENT

This work is financially supported by the Beijing Municipal Education Commission's Science and Technology Program General Project (KM201910015010), Beijing Municipal of Education 2011 Collaborative Innovation Center, Printed Electronics Technology & Engineering Discipline Construction (III) (grant number 21090116001), 2017 Cultivation and development of innovation base (Z171100002217032), "Practical Training Program" Project of Cross-training High-level Talents in Beijing Colleges and Universities, Beijing Education Committee Key Item (KZ202110015019), Beijing Institute of Graphic Communication R & D Plan: (Ef202002), (Eb202102), (Ec202005).

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