3D-inkjet Printing of Flexible and Stretchable Electronics

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Abstract

Inkjet printing of conductive tracks on flexible and stretchable materials have gained considerable interest in recent years. Conductive inks including inks with silver nano-particles, carbon based inks, inks containing poly (3,4-ethylenedioxythiophene) (PEDOT) doped with polystyrene sulfonic acid (PSS) are being researched widely to obtain a printed electronic patterns. In this study, we present drop-on-demand inkjet printing of conductive silver and PEDOT:PSS on a flexible and stretchable substrate. Process conditions for the inkjet printing of silver nano-particles and PEDOT:PSS were optimised and simple geometrical patterns (straight line and sinewave tracks) were printed. Surface profile, surface morphology and electrical resistance of the printed patterns were examined. The printed silver patterns were observed to be highly conductive; however when stretched, the patterns did not conduct due to the origination of cracks. The measured conductivity for the PEDOT:PSS patterns was significantly lower than the silver patterns; however, they remained conductive when stretched for up to 3 mm. When flexed, PEDOT:PSS remained conductive for a lower radius of curvature (10 mm) than the silver. Among the printed patterns, the sinewave pattern was observed to be superior for flexible electronics application.

1. Introduction

Fabrication of flexible electronics is gaining significant interest and the market is rapidly growing. Printed electronics has the potential to eliminate the lengthy, material-intensive and expensive processes used traditionally [1]. Printing technologies including screen printing, soft lithography, flexography, gravure and inkjet are used for the fabrication of flexible electronics. Currently, flexible electronics are fabricated either by assembling a completed circuit on a flexible substrate or by fabricating the circuit directly on a flexible substrate [2]. Assembling electrical components to a substrate being a multi-step process, fabrication of flexible electronics all in a single-step would be beneficial. Inkjet printing of conductive materials over a substrate offers significant advantages including reduced wastage and ease of functionality [3].

Inorganic metal based inks containing gold, copper, aluminium and silver are used for the fabrication of electronic devices including the RFID tags, transistor electrode and other passive components [4], [5]. However, due to the low fracture strain of these metals, fabrication of bendable and stretchable electronics from these inorganic materials is still a challenge. Conductive polymers including poly(3,4-ethylenedioxythiophene): polystyrene sulfonate (PEDOT:PSS) and functional polymers including polyaniline are used for the realisation of inkjet printing of flexible electronics [6].

Conductive tracks with sinusoidal wave patterns were previously reported to offer fully reversible stretchable/compressibility without substantial strains [2]. In this study, silver and poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) tracks were printed on an inkjet printed elastomeric substrates. The stretchability and flexibility of the straight line pattern and the sinewave pattern were explored and compared for both the silver and PEDOT:PSS inks.

2. Materials and Methods

2.1.Materials and Equipment

Objet TangoBlack® FLX973 was used to fabricate flexible substrates in a Stratasys Connex 260 digital materials printer. The nano-silver ink (28 – 33 weight % of silver) used in this study to print conductive tracks was purchased from Advanced Nano Products (ANP). A conductive polymer, poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) was purchased from Heraeus, Germany. Fujifilm Dimatix Materials Printer (DMP-2831) was used to print conductive tracks on the inkjet-printed specimens. Convection oven used in this study was supplied by Lenton Furnaces & Ovens. Nikon optical microscope was used to characterise the printed patterns. A TENMA (72-7725) digital multimeter was used to determine the electrical resistance of the printed conductive tracks. A mechanical stylus profilometer was used to characterise the thickness of the printed conductive tracks.

2.2.Methods

Tensile bars were designed using Magics 14.1 computer aided design (CAD) software according to type 1 of the British Standard BS ISO 37:2011. The dimensions of the bars are given below in Table 1. For flexibility studies, cuboidal samples of 20 mm x 50 mm x 2 mm were designed in Magics. These CAD design was replicated to fabricate the required number of samples in a Stratasys Connex 260 digital materials printer. The patterns were printed using TangoBlack® with glossy finish. On fabrication, the tensile bars were removed from the build platform and cleaned using deionised water.

Table 1 Dimensions for the tensile bars (BS ISO 37:2011)

Description	**Dimensions in mm**
Overall length	115
Width of ends	25
Length of narrow portion	33
Width of narrow portion	6.2
Transition radius outside	14
Transition radius inside	25

Two strain gauge patterns (straight line and sinewave pattern) of track width 2 mm were designed using GIMP open source software. The images were designed with the resolution of 846.666 dpi. The file was exported as bitmap file (.bmp) and converted to .ptn file using the DMP800 software. A tensile bar was aligned in the build platform and the print origin was selected so that the conductive track is in the centre of the test length.

2.2.1. Printing of silver

Since the silver ink used in this study was highly stable, all nozzles worked and hence, all 16 nozzles were used to print the conductive tracks. The print head angle was set to 6.8° to achieve a drop spacing of 30 µm. Print height was 1 mm and firing voltage was 27 V. The cartridge temperature was set to 30°C. After printing one layer of silver, the ink was dried using a heat gun (blows hot air) from approximately 15 cm above the substrate. This was performed in order to dry the excess solvents, so that the next layer can be printed. It should be noted that this procedure was not performed for the sintering of the silver nano-particles. After printing 10 Layers, the tensile bars containing the printed conductive tracks were transferred to an over operating at 130° C. After 2 hours, the tensile bars were transferred from the oven to room temperature (~ 20° C) and allowed to cool before further characterisation.

2.2.2. Printing of PEDOT:PSS

The TangoBlack® substrate for printing the conductive track was placed on top of a heated substrate. The surface temperature of the TangoBlack® substrate was maintained to ~ 50°C. The print head angle was set to 6.8° similar to silver to attain a drop spacing of 30 µm. Working nozzles were selected and print origin was selected. Print height was set to 1 mm and the firing voltage range was 19 - 23 V. No heating was applied to the cartridge and the head temperature was maintained at 20°C. After printing 10 layers, the substrate was removed and allowed to cool before further characterisation. It should be noted that no further heating/blowing of hot air was performed during or after printing.

2.3.Surface Characterisation

Nikon optical microscope was used to characterise the morphology of the inkjet-printed TangoBlack® (tensile bar) substrates, silver and PEDOT:PSS patterns. A mechanical stylus profilometer was employed to estimate the thickness of the inkjet printed silver and PEDOT:PSS layer.

2.4. Surface wettability measurements

Surface wettability of the silver and PEDOT:PSS inks on TangoBlack surface was obtained using KRUSS Drop Size Analyser (DSA) 100. The drop volume was 2 µL. The droplet formed on the substrate was allowed to settle for ~ 5 seconds before measuring the contact angle. Measurements were performed on three different TangoBlack® substrates and the obtained contact angle values were averaged.

2.5. Stretchability and Flexibility

An Instron tensile machine was used to stretch the tensile bars. The tensile bars printed with conductive tracks were stretched at a rate of 1 mm per minute. Resistance was measured after every 1 mm elongation until the track stopped conducting. For flexibility measurements, cylinders offering various radius of curvature ranging from 160 mm to 7.5 mm were chosen. Samples were placed flat on the surface of the cylinder and the resistance offered by the conductive tracks was measured.

2.6.Electrical Resistance

Electrical resistance was measured using a TENMA (72-7725) digital multimeter. The probes were gently placed on the contact pads at both ends of the track. The ranges used were 2 Ω to 2 MΩ and the resistance values given by the multimeter was noted.

2.7.Statistical Analysis

A total of three samples were used for each experiment and the results are the average of these three samples. The reported error bars and ± value corresponds to the standard error of the mean all through this study.

3. Results

3.1.Surface morphology of TangoBlack®

Figure 1 shows the surface morphology of the inkjet printed TangoBlack® surface with glossy finish. A wavy pattern can be observed and is likely due to jetting and curing of the ink in swathes. The clean glossy finish of the TangoBlack® substrate was observed to be highly sticky to the substrates including glass slide and the Dimatix build platform (platen).

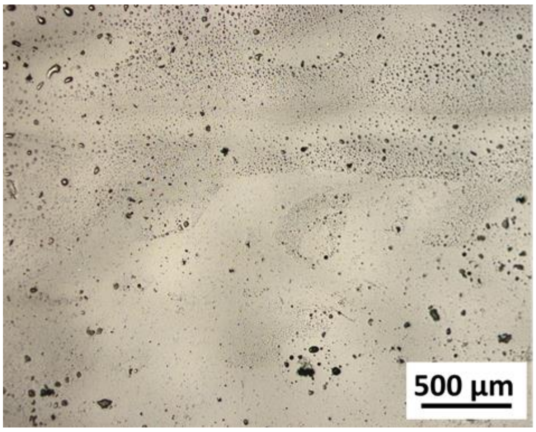

Figure 1 Surface morphology of the inkjet printed TangoBlack® substrate obtained using an optical microscope

3.2.Silver on TangoBlack®

3.2.1. Droplets Characterisation

Figure 2 shows the ANP silver ink droplets ejected from the nozzles and the droplets formed on the inkjet printed TangoBlack® substrate. The measured diameter of the droplets using the inbuilt Dimatix software was approximately 47 ± 3.5 µm. The droplets were mostly observed to be uniform; however, in certain areas a satellite drop was observed next to the main droplet. This is possibly due to the fact that due to the wavy surface profile, the time of flight of the droplet might have been reduced. As a result, coalescence of the satellite with the main droplet might have been affected and thus leading to satellite formation next to the droplet.

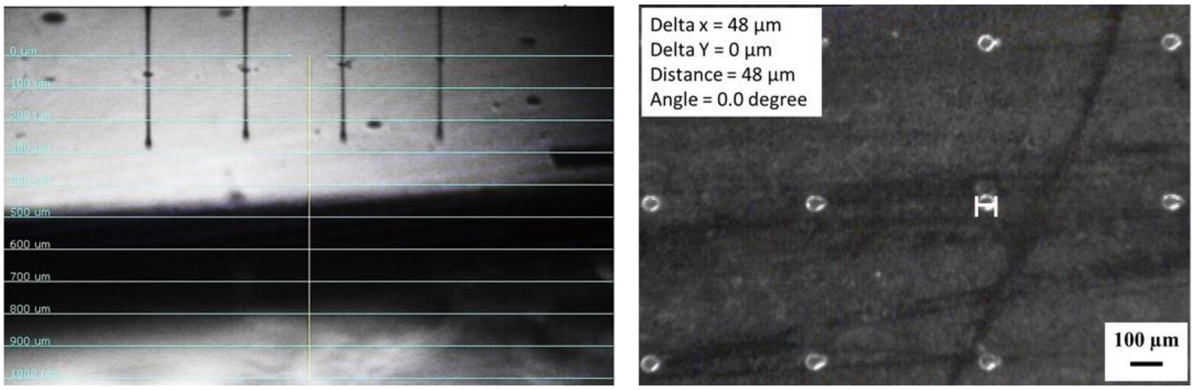

Figure 2 Silver droplet ejection from a Dimatix printer cartridge (left) and subsequent drop formation on the TangoBlack® substrate (right)

Figure 3 shows the contact angles of the ANP silver ink droplets formed on the TangoBlack® obtained for three different samples. This reveals that the ANP silver ink is hydrophilic and thus wets the surface. The wettability of silver ink on previously printed silver tracks revealed a highly wettable nature with contact angle > 20°.

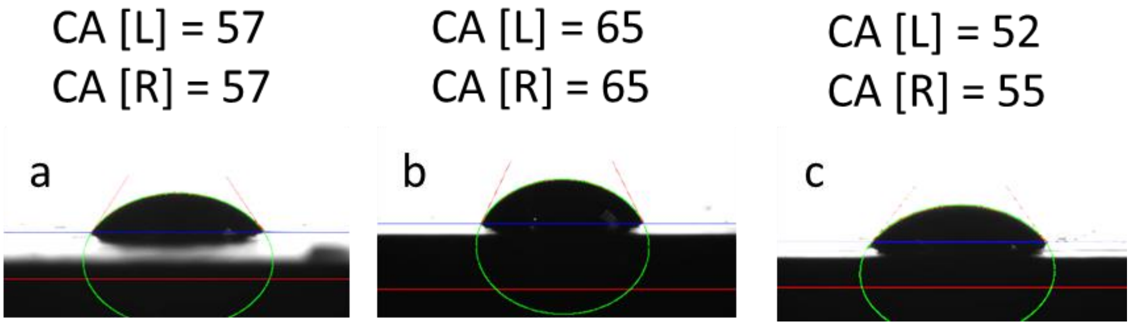

Figure 3 Surface wettability of the silver nanoparticle ink on the inkjet printed TangoBlack® substrates

3.2.2. Characterisation of Silver Tracks

Surface Characterisation

Optical microscopic images of the silver tracks printed on the TangoBlack® surface (before and after sintering) has been shown in Figure 4. It can be observed from the figure that there were very few cracks for the printed silver tracks before sintering. Whereas after sintering, cracks emerge after sintering and this is possibly due to varied thermal expansion behaviour of the TangoBlack® and Silver. The presence of these cracks on the printed silver track may have a strong influence on the resistance offered by these tracks. The thickness of the printed silver tracks was estimated to be approximately 600 nm.

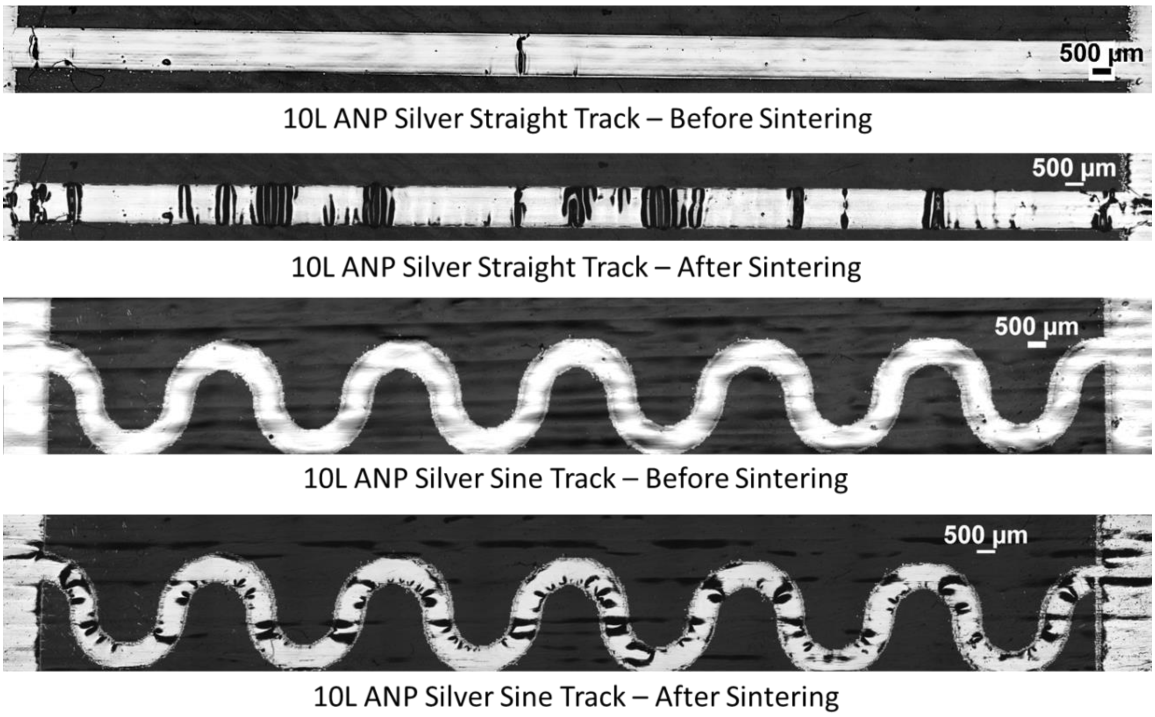

Figure 4 Inkjet printed silver tracks on the TangoBlack® substrates (straight line and sinewave patterns) before and after oven sintering at 130°C

Stretchability

Figure 5 shows the conductive tracks (both straight line and the sinewave pattern) printed on test specimens. These conductive tracks printed on the TangoBlack® substrates were stretched using an Instron tensile machine at the rate of 1 mm/min with 1mm steps. The resistance offered before stretching by straight line tracks were 1.1 ± 0.1 Ω and the sinewave tracks were 2.4 ± 0.2. When stretched for 1 mm, no conductivity was observed on both the straight line and sinewave track patterns; whereas after relaxing the tensile bars, the tracks were observed to conduct with slightly a higher resistance. This shows that the cracks were formed due to stretching of the specimens.

Figure 5 A photographic image showing the inkjet printed silver tracks (straight line and sinewave track patterns) on the tensile bar specimens

On examining the surface morphology of the elongated/stretched tracks through optical microscope, it was confirmed that the loss of conductivity after stretching was primarily due to the origination of cracks due to stretching (Figure 6).

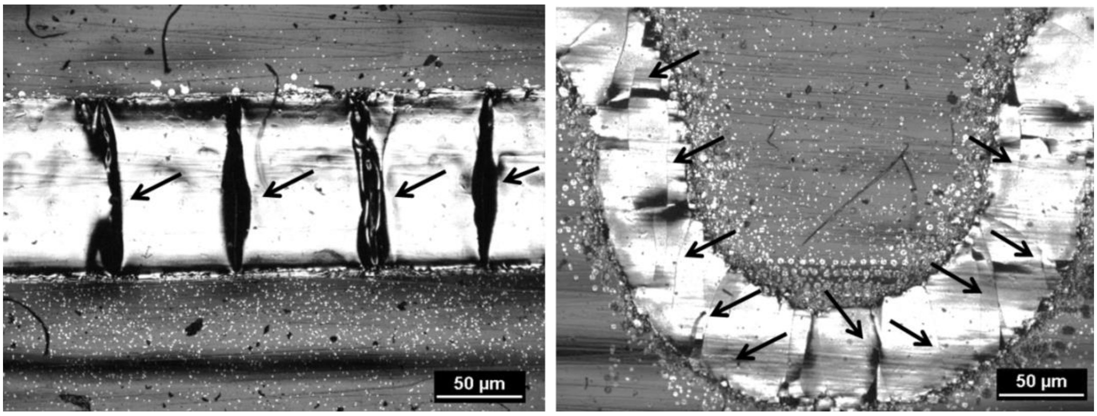

Figure 6 Cracks formed on the silver tracks after stretching. (a) Straight line pattern and (b) Sinewave pattern

Flexibility

The substrates printed with silver were flexed to different radius of curvatures ranging from 160 – 7.5 mm. Table 2 shows the resistance obtained for the printed silver tracks on the TangoBlack® substrates. It can be observed from the table that the sinewave pattern was capable of conducting at a lower radius of curvature than the straight line pattern.

Table 2 Resistance measured for straight line pattern and sinewave pattern at various radius of curvatures

Sample	Radius of Curvature (mm)							
	No Flex	***160***	***60***	***55***	***45***	***35***	***30***	***15***
Resistance (Ω) for Straight	1.6 ± 0.1	2.1 ± 0.1	2.5 ± 0.3	2.5 ± 0.3	-	-	-	-
Resistance (Ω) for Sinewave	2.4 ± 0.2	2.5 ± 0.2	2.8 ± 0.2	3 ± 0.2	3.7 ± 0.5	4 ± 0.3	6.6 ± 0.3	0

Figure 7 and 8 depicts the cracking behaviour of the straight line and the sinewave pattern of the printed silver tracks on the TangoBlack® substrate respectively. It can be observed from the figures that the straight line pattern was more prone to cracks than the sinewave pattern. This further confirms the ability of the sinewave pattern to withstrand for low radius of curvature compared to the straight line pattern.

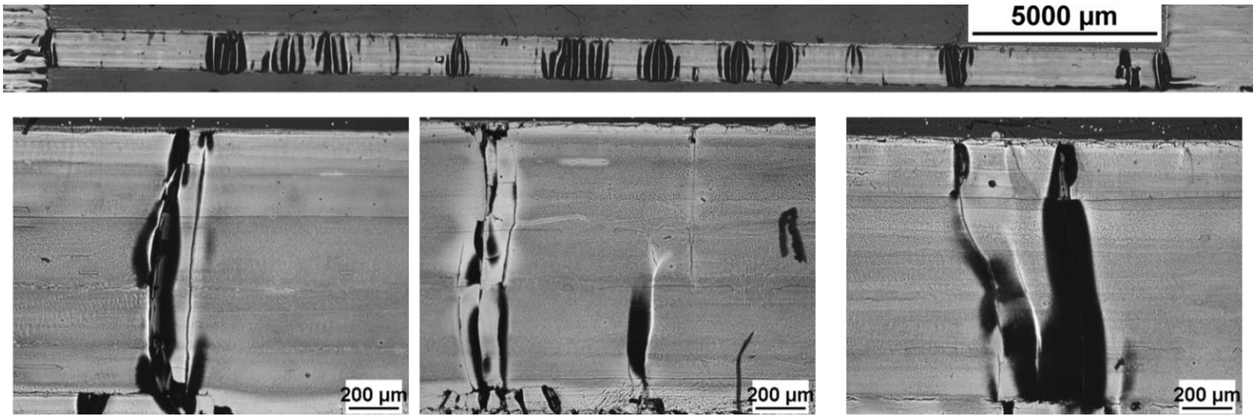

Figure 7 Cracks formed on a straight line silver track when flexed

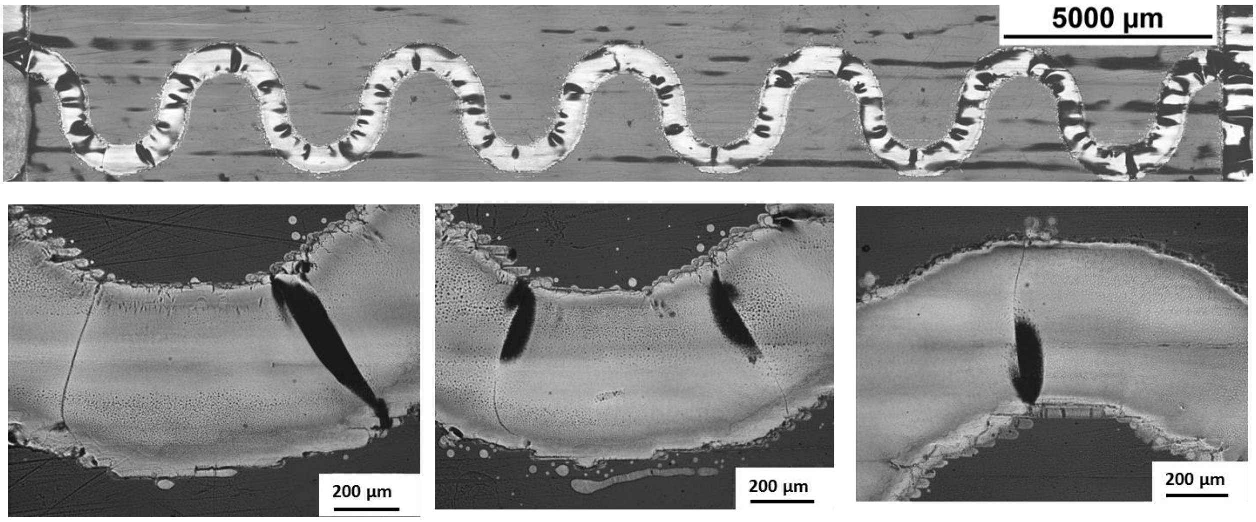

Figure 8 Cracks formed on a sinewave silver track when flexed

3.3. PEDOT:PSS on TangoBlack®

3.3.1. Droplets Characterisation

Droplets formed by the PEDOT:PSS ink on the TangoBlack® substrates were almost in-visible due to its transparency. Hence it was hard to characterise the droplets for size in Dimatix. Figure 9 shows the contact angle formed by the PEDOT:PSS on TangoBlack® substrates. Wettability studies showed that PEDOT:PSS ink was wettable on the TangoBlack® substrates yielding a contact angle of 47° ± 1°. PEDOT:PSS droplets also exhibited a wettable nature (~ 35°) on PEDOT:PSS substrate.

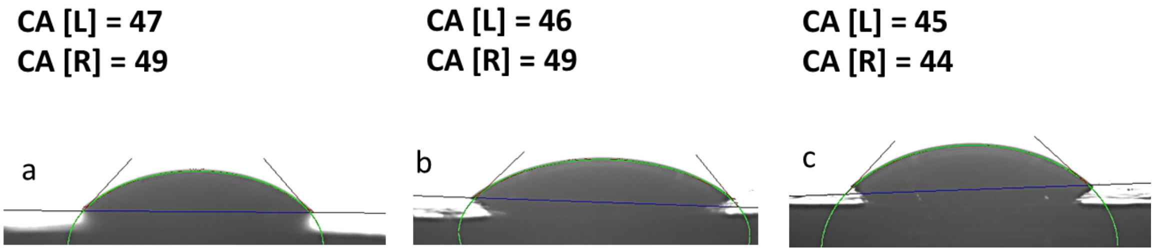

Figure 9 Wettability of the PEDOT:PSS ink on the TangoBlack® substrates

3.3.2. Characterisation of PEDOT:PSS Tracks

Surface characterisation

Figure 10 shows the surface morphology of the thin film of PEDOT:PSS printed on the TangoBlack® surface. A wavy profile can be observed and this is likely to be due to the waviness of the substrate itself. However, the printed swathes of PEDOT:PSS can also add to this waviness. The layer thickness of the film was measured to be approximately 2 µm.

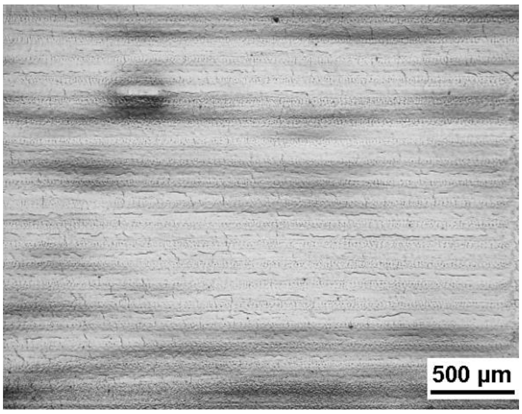

Figure 10 Surface morphology of a thin film of PEDOT:PSS printed on a TangoBlack® substrate

Stretchability

Similar to silver on TangoBlack® substrates, printed tracks of PEDOT:PSS on TangoBlack® substrates were stretched in Instron tensile machine. Resistance measured at various extensions were graphically shown in Figure 11. As it can be observed, both PEDOT:PSS patterns withstood to about three millimetres of extension before cracking. However, the tracks conducted back once relaxed, but with a higher resistance (10 – 15 KΩ) compared to the resistance measured before elongation (1 – 2 KΩ). Optical microscopy images of the tracks before and after extension were presented in Figure 12.

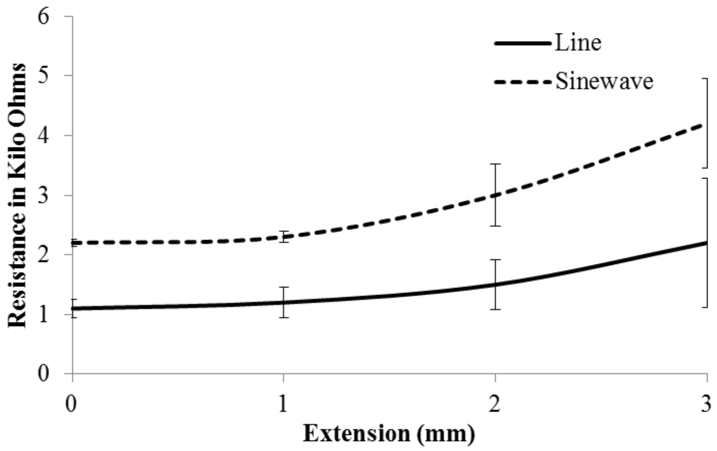

Figure 11 Resistance measured using an Ohmmeter for the straight line and sinewave tracks at different extensions

Few cracks on the straight track patten can be wittnessed before elongation. These cracks prossibly originated due to the sticky nature of the TangoBlack® while transfering the substrates with the printed tracks from the Dimatix printer to microscope for characterisation. However, after elongation to 4 mm, a series of cracks all through the straight line pattern can be observed. When this is compared with the sinewave pattern, cracks originated exactly perpendicular to the direction of the applied force. No cracks were visible in the other areas of the printed tracks.

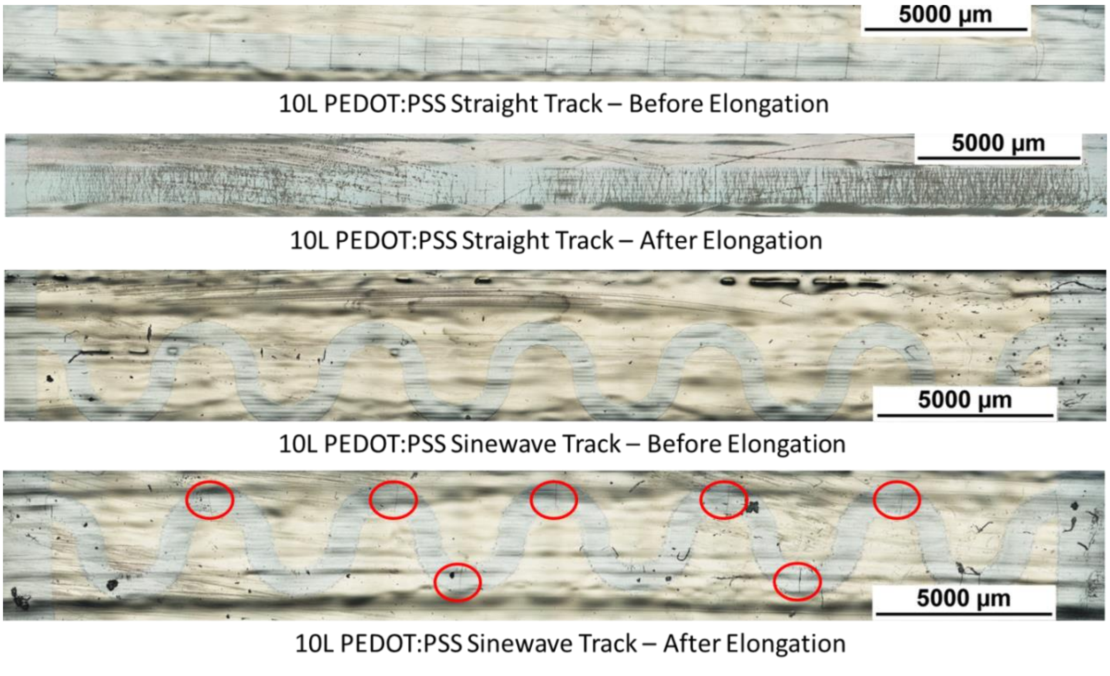

Figure 12 Optical microscopy images showing the straight line and sinewave pattern printed using PEDOT:PSS before and after elongation. Red circles points the cracks originated in the printed sinewave track.

Flexibility

The printed PEDOT:PSS tracks were flexed to different radius of curvatures ranging from 160 mm and 7.5 mm (until the tracks stopped conducting). Table 3 shows the resistance obtained for the tracks flexed at various radius of curvatures. It can be noted from the table that both the straight line and sinewave patterns had same resistance within their sets when flexed until a radius of curvature of 15 mm. When flexed beyond this point, the resistance offered by the tracks increased slightly and subsequently cracked at the radius of curvature of 7.5 mm for both patterns. Figure 13 shows the optical microscopic image of the previously flexed straight line PEDOT:PSS tracks. Cracks can be noted all through the track. Some of these cracks completely cuts the track and some are intermediate. If when flexed more, these intermediate crack may propogate further to completely cut the track.

Table 3 Resistance offered by the straight line and sinewave patterns printed using PEDOT:PSS when flexed to various radius of curvatures

Radius of Curvature (mm)	Resistance (KΩ)	
	Straight Line	*Sinewave*
160	1.1	2.1
60	1.1	2.1
55	1.1	2.1
45	1.1	2.1
35	1.1	2.1
30	1.1	2.1
15	1.1	2.1
12.5	1.3	2.2
10	2.1	2.2
7.5	Dead	Dead

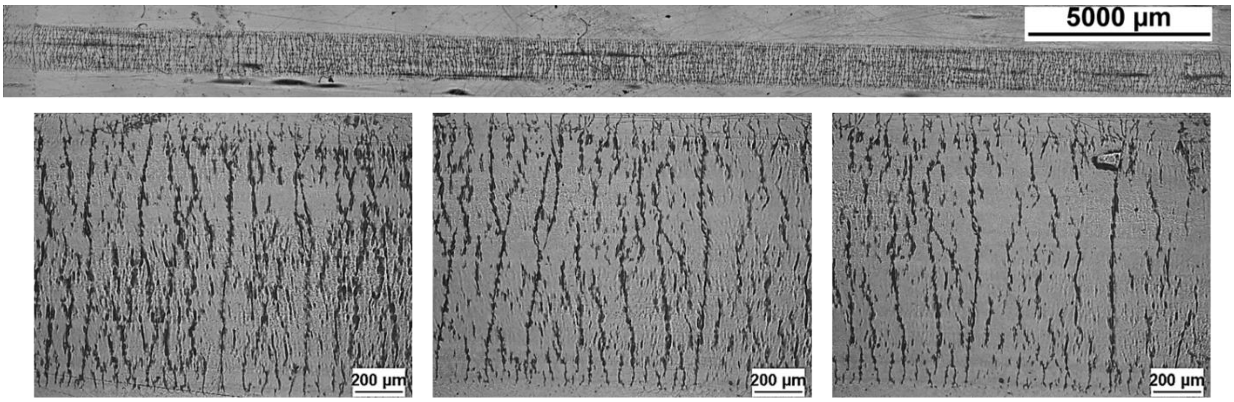

Figure 13 Optical microscopy image showing the cracks that originated in the printed PEDOT:PSS straight line tracks when flexed

Figure 14 shows the cracks propagated for the sinewave pattern after flexing. Similar to the stretched sample in the tensile machine, cracks originate at the points perpendicular to the applied force. The zoomed-in image (below) shows both complete and intermediate cracks in the pattern.

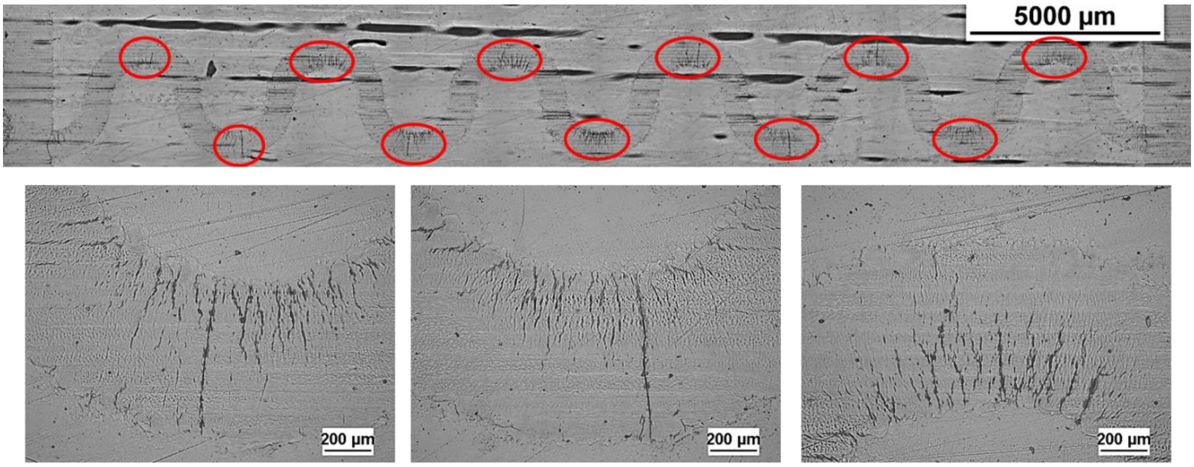

Figure 14 Optical microscopy image showing the cracks that originated in the printed PEDOT:PSS sinewave tracks when flexed. Red circles points the cracks originated in the printed sinewave track as a result of flexing.

4. Discussion

Several studies reported inkjet printing of both conductive silver and PEDOT:PSS on various flexible substrates including polyethylene terepthalate (PET), polyethylene napthalene (PEN), parylene and polyimide for various sensing applications [7]–[10]. TangoBlack® is a flexible and stretchable (to a certain extent) elastomer that could be printed in Objet machine. Hence in this study, conductive inks were printed on a 3D inkjet printed TangoBlack® substrates with a view of printing flexible and stretchable electronics using one process ie., ink jetting.

TangoBlack® substrates fabricated in the Objet® machine showcased a wavy surface and obtaining a microscopically smooth surface without waviness is almost imposible in this jetting machine. However, the impact of this wavy profile on the printed surface can be reduced by choosing appropriate part orientation. In the present study, the requied substrates (both tensile bars and cuboidal samples) were printed parallel to the printing direction to minimise the wavy profile. Printing parts perpendicular to the printing direction will increase the the number of swathe per print layer, potentially leading to a highly wavy profile. Printing conductive tracks on a highly wavy surface may lead to uneven printing and inturn affect the conductivity.

Previous literature suggested oven sintering of silver at 130°C - 200°C for 2 hours to achieve highly conductive tracks [11]. Although 130°C was used as the sintering temperature for silver nanoparticles in this study, oven sintering of the printed silver tracks was observed to induce cracks in the printed tracks. This is most likely due to different thermal behaviour of the silver and the TangoBlack®. As it would be expected, both the straight line and sinewave patterns of printed silver tracks did not survive on elongation; however when flexed, they remained conductive for upto a certain radius of curvature. Survival of the sinewave pattern for a lower radius of curvature compared to the straight line pattern reveals that, in addition to the material properties, track geometry also plays a part in yielding a flexible conductive tracks. Thus, optimisation of the track geometry can offer a highly conductive and flexible circuitry. Though silver tracks printed on TangoBlack® substrates may not be useful for stretchable electronic devices, it has the potential to be used in flexible electronics. However, the flexibility of these printed silver tracks are limited.

PEDOT:PSS, the conductive polymer on the other hand was seen promising due to its transparency, thermal stability and good electrical conductivity. The PEDOT:PSS ink used in this study was a water based colloidal dispersion. Several studies, reported inkjet printing of PEDOT:PSS as an efficient and clean choice of fabricating components [12]–[14]. In reality, for inkjet printing, there are issues with this colloidal dispersion of PEDOT:PSS in water. The PEDOT:PSS ink (Clevios®) supplied by Heraeus was not stable in the Dimatix print head. Although most of the nozzles (more than 12 out of 16) worked when the ink was filled, after printing few layers, the nozzles died one by one leaving with less than 4 working nozzles. If the printing was stopped for an hour or two, these nozzles were also observed to die. Ultimately, leaving the Dimatix catridge with PEDOT:PSS ink overnight led to clogging up of all nozzles. Repeated puring and wiping of the nozzles, and cleaning of nozzles with deionised water did not help to get all nozzles to work. This is an alarming issue and a major set-back for the inkjet printing of this water based PEDOT:PSS ink, especially in the Dimatix.

Admists of these issues, the tracks printed with PEDOT:PSS showcased resistance in kilo ohms, an order of magnitude of three compared to silver ink (usually in ohms). Due to the increased track length, the sine wave pattern was more resistive (~ 2.2 KΩ) and nearly double that of the straight line pattern (~ 1 KΩ). Both the printed PEDOT:PSS patterns remained conductive upto an elongation of 3 mm and to the radius of curvature of 10 mm when flexed. PEDOT:PSS is certainly a promising material for the flexible electronics; however, the stability issues are yet to be solved. Be it a printed silver or PEDOT:PSS track, cracks were observed to originate perpendicular to the direction of the applied force and propogate further. Designing smart patterns such as the sinewave pattern shown in this study, origination of cracks can be controlled and limitted thereby the performance of the printed patterns can be enhanced. In contrast to straight line tracks, modification of the conductive tracks in micron scale can add more value to the printed electronics.

5. Conclusion

The use of TangoBlack® as a substrate for printing silver nanoparticle ink was not promising for stretchable electronics, but has some potential for the flexible electronics. TangoBlack® can be used as substrates to print PEDOT:PSS for both stretchable and flexible electronics. However, poor stability of the PEDOT:PSS ink for inkjet printing should always be reminded. Sinewave pattern was witnessed to be a better design compared to the straight line pattern. This reveals that in addition to the material property, track geometry also plays a part in determining the flexibility of the ink jet printed circuits.

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Future Work

- To optimise the geometry of the conductive track for better performance
- To perform cyclic tests on the printed conductive tracks for repeatability

- To enhance the stability of the PEDOT:PSS ink for inkjet printing
- To compare the obtained results with other jettable elastomers.

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