EVALUATION OF PRINTED INTERDIGITAL ELECTRODES ARRAY FOR CHEMO-RESISTIVE GAS SENSOR APPLICATION

[1]Vojtěch POVOLNÝ, [1]Alexandr LAPOSA, [1]Jiří KROUTIL, [1]Jan VOVES, [2]Marina DAVYDOVA,
[1]Pavel HAZDRA

[1]Faculty of Electrical Engineering, Czech Technical University in Prague, Prague, Czech Republic, EU,
povolvoj@fel.cvut.cz

[2]Institute of Physics of the Czech Academy of Sciences, Prague, Czech Republic, EU

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Abstract

In this article, we present the design and preparation of a platform for a gas sensor application based on the ink print of silver nanoparticles on flexible substrates. The Inkjet printing allows localized deposition of ink at low temperatures on a large area and can be used for the deposition of both conductive and dielectric materials. The printed platform consists of four interdigital electrodes (IDE), and connector pads printed with silver ink and sintered with an intense pulse light (IPL). The dielectric ink printed around the IDE structures protects the connection pathways and defines the sensing area. The multilayer structure was printed on polyethylene terephthalate (PET) and polyimide (PI) substrates. Before printing, the plasma treatment of the surface of the polyimide is necessary. Three combinations of electrodes with various widths were designed and the structures were printed with three various resolutions to verify the printability on the used combination of substrate and ink. Bending tests, that show the flexibility of printed structures, were performed as well.

Keywords: Inkjet, gas sensor array, PET, Polyimide, IPL

1. INTRODUCTION

Printing technology has been increasingly popular in recent years. Due to its low manufacturing costs, printing technologies such as screen printing [1,2], micro-contact printing [3,4], and inkjet printing [5] have become increasingly popular. Inkjet printing has sparked the most interest among the techniques mentioned because the components produced using this technology have undeniable advantages over conventional ones in terms of the ease of production, on a wide range of substrates, the contactless deposition of the resulting motive without the use of a mask, low-temperature processing without the use of vacuum, and low cost. All-polymer transistors [5,6] polymer light emitting diode (PLED) [7,8], nanoparticle Micro Electro Mechanical Systems [9], and polymer capacitors [10] have all been made with inkjet printing.

Inkjet printing technology has grown in popularity as a technique for producing electronic components in a variety of academic and industrial fields during the last decade. The use of a drop-on-demand or on-demand material deposition technology is a key advantage for inkjet printing and allows the fully additive processing of electronic materials. Inkjet eliminates the requirement for a cleanroom, toxic chemical waste, and expensive photolithographic masks when compared to traditional electronic production processes [11].

In this work, we report the preparation of an inkjet printed sensor array on flexible substrates. We discuss the results obtained for printing with various resolutions, the conductivity of the printed paths according to the annealing method, and the results of a cyclic bend test to analyze the reliability of the printed structures.

2. MATERIALS AND METHODS

2.1. Sensor array design

The prepared platform was designed for the purpose of multiple sensor testing. It was printed with material inkjet printer Fujifilm Dimatix 2831 and cartridge DMC-11610 (16 nozzles, 10 pl drop volume) and the design of the platform was prepared with the CleWin5 layout editor (Wieweb software, Netherland). The dimensions of the array are 13.6 mm x 12 mm and the platform consists of two basic parts – conductive and insulating layers. The conductive layer is divided into two parts – conductive pathways including connector pads and four interdigital electrodes (IDE). The connector part is designed to be simply connected with a flexible tip or to be mounted into an Amphenol Clincher flex connector. The IDE structures have dimensions of approximately 2 mm x 2 mm with finger widths of 20 μm, 40 μm, and 60 μm and were printed with a combination of three various resolutions and three various widths of fingers. The printed structure and the layout of the platform with dimensions can be seen in **Figure 1**.

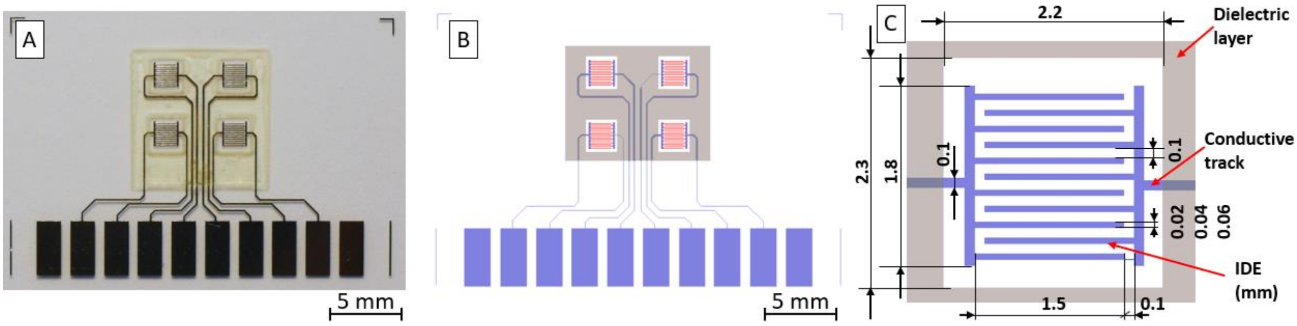

Figure 1 Platform of printed interdigital sensor array: A) Structure printed with silver ink and with the dielectric protection layer, B) Layout of the complete platform, C) Detail of the IDE with dimensions

2.2. Substrates

Two types of flexible materials were used as substrates for printing. The adopted polyethylene terephthalate (PET) substrate Mitsubishi Novele^{TM} IJ-220 by Novacentrix (thickness 140 μm) is a printed electronics mesoporous substrate appropriate for low-cost and low-temperature applications where the flexible and transparent substrate is desired and specifically engineered for inkjet-compatible inks [12]. The second substrate used for printing is polyimide (PI) foil DuPont^{TM} Kapton HN (thickness 75 μm). The parameters of the PET and polyimide materials are listed in **Table 1**. Polyimide can withstand higher temperatures than PET and can be used for the preparation of components in processes with temperatures of several hundred degrees. However, before printing, it is necessary to carry out the plasma treatment that will prevent bleeding of the ink by slightly etching the surface. In this research, we used SF_6 plasma treatment with a power of 200 W of ICP (Inductively Coupled Plasma) source and 30 W of RF (Radio Frequency) generator, respectively with Sentech ICP Etch System SI 500 (SENTECH Instruments GmbH, Germany).

2.3. Inks

The conductive layers were fabricated with the silver nanoparticle ink ANP Silverjet DGP-40LT-15C (solid content 35 %, resistivity 11 μΩ·cm, particle size <50 nm). During the printing, the platen temperature was set to 40 °C and the structures were printed with a resolution ranging from 635 dpi to 1693 dpi at 2 kHz jetting frequency and the print head was preheated to 35 °C. Printed structures were dried at room temperature for 24 h and then sintered with the high-intensity pulse light (IPL) system Xenon X-1100 by Xenon Corporation, USA (up to 9 J·cm^{-2} radiant energy per pulse, broad-spectrum light) by two flashes with the settings of 300 μs and 200 J.

Table 1 Typical properties of selected flexible substrates

Property	Polyethylene terephthalate (PET)	Polyester (PES)	Polyimide (PI)
Tensile strength (MPa)	60 – 85 *	10 – 123 *	72 – 231 *
Flexibility	Excellent	Excellent	Excellent
Dimensional stability	Good	Good	Excellent
Dielectric strength (kV/mm)	20 – 50 *	11 – 30 *	177 – 201 *
Max operating temperature (°C)	100 – 140 *	71 – 200 *	220 – 400 *
Chemical resistance	Very good	Excellent	Excellent
Moisture absorption (%)	0.10	0.20	0.30
Cost	Low	Low	High

* – According to type

The isolation protective layer was printed with dielectric ink Dycotec DM-INI-7003 by Dycotec Materials Ltd, UK (volume resistivity $1.4 \cdot 10^{14}$ Ω·cm, UV curing wavelength 380 nm, UV curing energy 500 $mJ \cdot cm^{-2}$ – 1000 $mJ \cdot cm^{-2}$). The dielectric ink was printed with 15 nozzles at the resolution of 1016 dpi at 2 kHz, the print head was preheated to 32 °C and the substrate was heated to 50 °C. Immediately after printing, the layer was cured by UV radiation with a wavelength of 380 nm and energy of 1.7 $W \cdot cm^{-2}$ using a high-intensity UV LED.

2.4. Mechanical test

A bending endurance test was carried out for the verification of the flexibility and stability of the inkjet printed layers. For this purpose, an in-house made bend test apparatus was used, see **Figure 2**. This machine performs cyclic bending of a flexible substrate with a printed test structure, which puts mechanical stress on the entire structure. The bending radius (6 mm) and the distance (25 mm) are precisely specified according to the standard for mechanical bending tests IPC-TM-650 2.4.3.

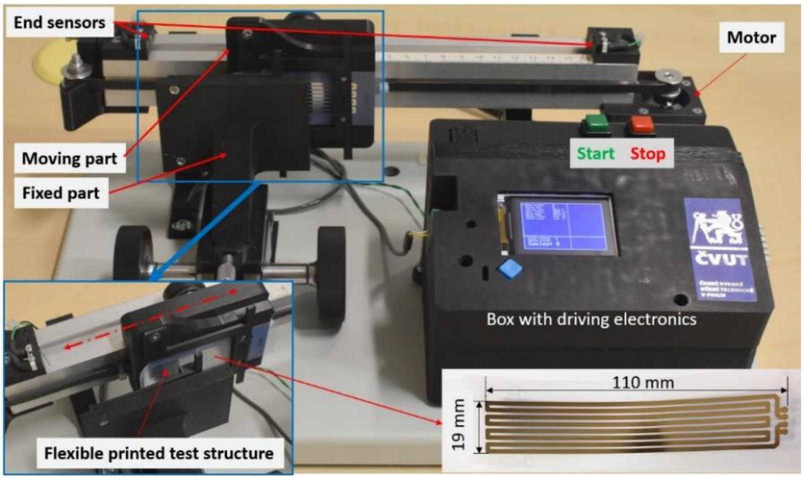

Figure 2 Setup for mechanical test of flexible printed circuit board

3. RESULTS AND DISCUSSIONS

3.1. PET

To ascertain the behavior of the silver ink on the PET substrate surface, a single nozzle line was prepared and printed using built-in control software. Optical characterization and measurement of printed structures were performed on a digital microscope Olympus BX 60. **Figure 3** shows silver lines printed at resolutions of 635 dpi, 725 dpi, 847 dpi, 1016 dpi, 1270 dpi, and 1693 dpi and also with measured widths. It can be seen that the lines printed with 635 dpi and 725 dpi are formed from separated drops and the lines are not continuous. For the line printed with 1693 dpi, we observed that the line is not uniform due to the deposition of a big amount of the material. The resolutions of 847 dpi, 1016 dpi, and 1270 dpi showed the most suitable shape of the lines and were chosen for further printing. The measured widths were varying from 30 μm to 33 μm. For the thickness evaluation of silver ink, a 200 µm wide and 2 mm long line was printed with a various number of layers at the resolution of 1016 dpi. The thickness of printed structures was measured with Stylus Type 3D Surface Profilometer (Taylor Hobson, UK). Mean heights with maximal deviation are listed in **Table 2**.

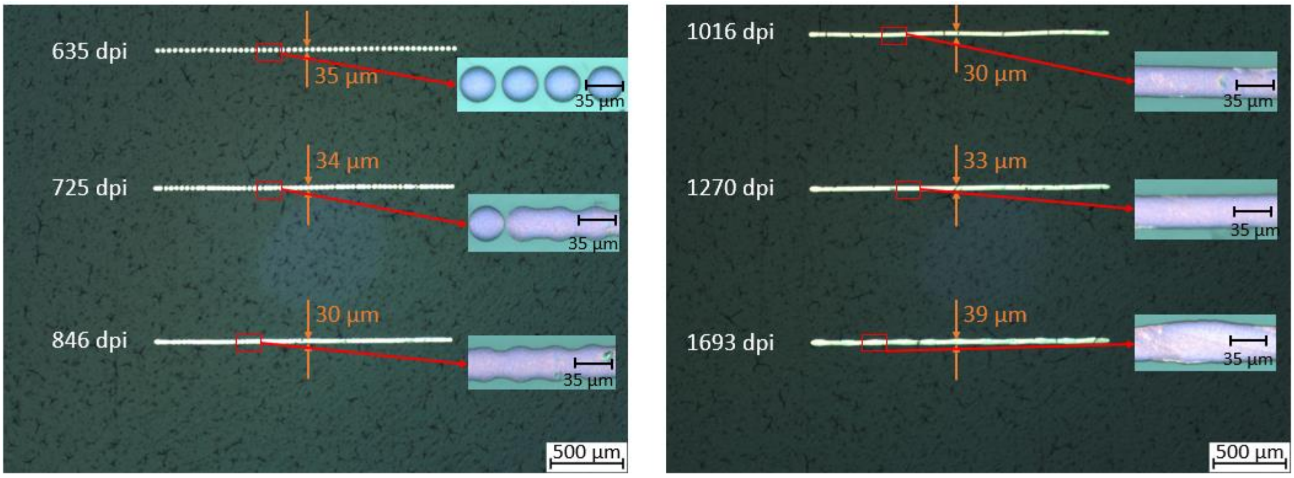

Figure 3 Resolution test of silver ink printed on PET substrate

Table 2 Thickness and maximal deviation for the various number of silver layers printed on PET

Number of layers	Thickness (µm)	Max deviation (µm)
1	0.45	0.19
2	0.88	0.23
3	1.37	0.25
4	1.73	0.26
5	2.13	0.31
10	3.93	0.34

Line width and shape

The interdigital electrodes were printed with two layers and resolutions of 847 dpi, 1016 dpi, and 1270. From the structures printed with the resolution of 847 dpi, we observed that the lines are not homogenous because of the greater drop spacing. Even if the structure was printed with two layers an incomplete connection may occur. This can make the platform unusable. For the structures printed with the resolution of 1016 dpi, the lines are homogenous and all patterns are completely connected. However, the edges of the IDE fingers are still not absolutely smooth. The best results of the printed structures with high smoothness and uniformity were achieved at the resolution of 1270 dpi. However, printing with higher resolution is more time-consuming. The printing time of conductive tracks and connection pads was 6 min, 7 min, and 9 min for the resolutions of 847 dpi, 1016 dpi, and 1270 dpi, respectively. For printing larger areas, it is important to take these parameters into account.

Sintering

Figure 4A shows the dependency of resistance on a various number of layers for different materials or sintering processes, respectively. From the plot can be seen that for structures printed with silver nanoparticle ink the values of the resistance are approximately 4 times higher compared to the calculated values of resistance for the specific resistivity of the bulk silver. This means that if we want to achieve these values with silver ink the thickness of the printed structure would be 4 times greater. **Figure 4A** also shows that we achieved better results than one can expect considering the values of resistance for the manufacturer´s specific resistivity. The reason for this phenomenon is caused by different sintering methods. The producer declares the specific resistivity of 11 µΩ·cm for structures sintered at 250 °C for 3 hours even though the recommended sintering temperature is 100 °C – 150 °C. The temperature of 250 °C is too high for a majority of substrates and can cause their damage. Therefore, it is unusable for printed electronics.

In our case, we use an intense pulse light system to sinter printed structures. The calculated resistivity of the structures is approximately 8.3 µΩ·cm, see the small plot in **Figure 4A**. To compare, **Figure 4B** shows the calculated resistance of silver ink sintered with a laboratory oven from the previous measurements. From this comparison, it can be seen that the resistance of the structures printed with five layers and sintered with the oven is 3 times higher than that of the IPL sintered structures.

When using IPL to sinter printed structures, the multiple flashes have the effect of decreasing the resistance, see **Figure 4B**. With each flash, the silver nanoparticles melt and gradually form a uniform layer, leading to an improvement in the conductivity of the layer.

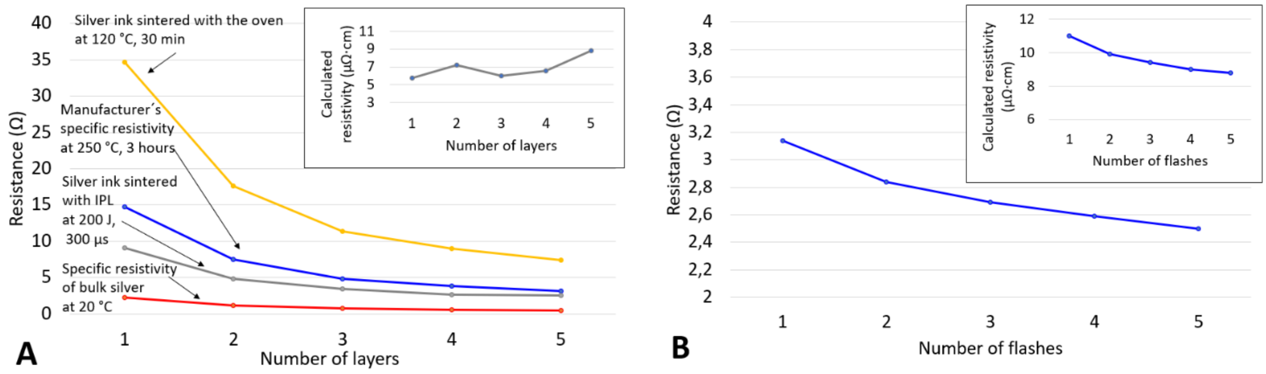

Figure 4 Dependency of the resistance on various number of layers for various value of the resistivity (left), dependency of resistance on various number of flashes (right)

3.2. Polyimide

Printing on polyimide is more demanding due to the necessity of the surface treatment. When no cleaning and surface activation has not been done the printed structure didn't create the desired shape due to the flooding of the ink. Therefore, the SF_6 plasma treatment was selected for the surface treatment of the substrate. The original process time of 30 seconds was gradually reduced to 5 seconds. This value was chosen as the most suitable in terms of ink behavior on the surface. The sensor array platform was printed with the same conditions as it was on the PET substrate. **Figure 5** shows the printed structure on the PI. The measured values of finger widths printed with two layers are summarized in **Table 3**.

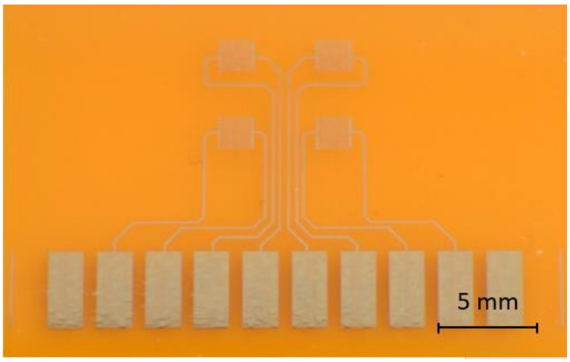

Figure 5 Interdigital sensor array printed on polyimide substrate

3.3. Mechanical test

The meander testing structure was printed on the PET substrate to verify the flexibility and stability of the printed electronics. The structure was tested with 1000 cycles which correspond to approximately 2 hours of test time. Before testing, the resistance of the structure was measured and compared with the value after testing. It was observed that the bending of the structure caused a relative change of the test structure of about 3 %. Furthermore, the printed structure has been shown to withstand the bending test without any destructive effect.

Table 3 Measured widths of printed fingers of IDE structures for each resolution

Designed width (µm)	**Measured width on PET (µm)**			**Measured width on PI (µm)**		
	847 dpi	1016 dpi	1270 dpi	847 dpi	1016 dpi	1270 dpi
20	26.5	25	21	48	25	38.1
40	58.5	37.5	29.5	51	51.6	54.3
60	66	55.5	42	72	65.7	70.6

4. CONCLUSION

In this work, the interdigital sensor array was fabricated on two flexible substrates by inkjet printing with various printing resolutions and various widths of IDE fingers. It was proved that for printing narrow lines (ca 40 µm) is better to use a higher resolution (1270 dpi), which is not so affected by the change in layout size of printed structures. On PET substrate, the thickness of printed layers was measured for a various number of layers. It was proved that the thickness increases linearly with a higher number of layers. Moreover, it was observed that with the use of the IPL sintering system we can obtain better resistivity (8.3 µΩ·cm measured after 2 x 200 J pulse energy and 300 µs each pulse length) than those indicated by the manufacturer (11 µΩ·cm measured after dry oven baking at 250 °C for 3 hours). By testing the flexural endurance of printed circuit boards, it was shown that printed silver structures can withstand at least 1000 cycles without any evidence of damage or delamination.

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