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SOME PROPERTIES OF PAD PRINTED ELECTRO-CONDUCTIVE ELEMENTS

Sandra DEDIJER[1], Dragoljub NOVAKOVIĆ, Miloš GUŽVICA,
Živko PAVLOVIĆ and Magdolna APRO
Faculty of Technical Sciences,
The University of Novi Sad, RS–21000 Novi Sad

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In this paper pad printed conductive elements using carbon based conductive ink on paper substrate have been analyzed. The investigation was primary directed to checking the possibility to print different elements, using pad printing technique, as narrow as possible but still conductive. The evaluation includes the shape sustainability, thickness and width of printed elements and inevitable resistance, in dependence on the number of wet to wet overprinted layers under predefined curing time. For the research we have printed various lines, straight, curved, right and cut angled in open and closed form, with the width of 50-500 μm. Those elements were single, double and triple printed (wet to wet). The evaluation of printed elements (width and thickness) was carried out using micrographs obtained with optical microscope (magnification of 140×) and Scanning Electron Microscope using image processing software ImageJ. Also, EDS (Energy Dispersive Spectroscopy) analysis was done in order to give a deeper characterisation of used ink. Resistance measurements were carried out using HP

[1] To whom correspondence should be addressed.

4194A Impedance/Gain-Phase Analyzer. Throughout the experiment we have determined the narrowest conductive line that can be printed under specified printing conditions. The results showed the influence of multiple layer printing on resistance and on the changes in the shape of the elements in terms that multiple layer printing decreases resistance but increases line width.

Introduction

During last two decades, significant improvements and rapid growth in the field of the electronic industry has been distinguished. One of the confirmation is certainly the use of different printing techniques in electronic manufacturing. Synthesis of printing process with conductive inks resulted in production of electrically functional devices (printed electronic). Screen printing, flexography, offset, gravure printing, pad printing and ink-jet printing are used for production of conductors, radio frequency identification (RFID) tags, passive radio frequency (RF) circuits, smart labels, photovoltaic devices, organic light emitting diode (OLED) displays and electronic memory and logic [1-6].

Each process has its benefits and drawbacks, depending on the chosen electrically functional device. The screen printing is a common method for the fabrication of interconnections, PEMS devices or RFID tag antenna. Beside screen printing method, RFID tags can also be gravure or flexo printed [1]. Interesting printing methods for printed electronic, mainly RFID tags, are also offset printing and ink-jet printing which is mainly used for manufacturing components for RFID tags [1,7], wafer — scale and board — level electronic devices and colour flat panels [8]. Gravure printing has the benefit when printing with very high throughput, but it has its limitations as regards ink film thickness [1]. Screen printing technique has its drawback in printed line width being limited with screen printing resolution (150-75 μm), and it provides printing of conductors not narrower than 100 μm [3,6]. It also has limitations in printing on curved surfaces where the pad printing is the most suitable technique [1,7]. Ink-jet printing, is a more expensive method and it also lacks in both speed and printed layer thickness [3,7]. On the other hand, ink- jet printing process is an attractive alternative to photolithography for direct patterning conductive lines owing to low-waste and more simple process [9]. The selection of printing technique is also based on the productivity range and ability to create structures with high resolution and homogeneity. With offset and flexo printing a channel length from 50 μm to 100 μm and 50 μm to 200 μm, respectively, was obtained to realize source/drain electrodes [10]. Gravure printing enables printing of source/drain electrodes with a channel length of 100 μm [10,11]. Ink-jet printing technique confirmed justified liability to produce continuous and smooth electric-conductive lines 10 μm in width [12] and 1, 2 and 4 μm thick (multiple layer priniting) [13]. A promising method for cost effective printing electronics is certainly pad printing, since it has significant advantages in speed and pattern thickness and narrowness [3,6]. Conductors with 25 μm width have already been printed using this technique [6]. In fabrication of the electro-luminescence (EL) display device on a curved surface using the pad printing method the minimal pattern was found to be 35 μm wide and 2.4 μm thick [7]. In some other researches the thickness of printed conductive lines was found to be 7-8.5 μm for 150 and 300 μm wide lines [5] as well as 17 μm for 300 μm wide lines, produced from a single print [3]. It must be emphasised that printed line width and thickness are highly dependable on the ink characteristics, pad characteristics, printing form (cliché) characteristics, used printing system and finding of the optimum process parameters.

When characterizing electrical properties of printed conductive elements, characterization is mainly given over electrical resistance, conductivity, resistivity or sheet resistance. Nevertheless, it is undeniable that electrical properties are mainly dependent on ink formulation and postprocessing of printed elements (curing time, curing temperature).

With gravure printing process, when producing indium tin oxide coatings (ITO coatings), mainly used for production of transparent front diode in organic light emitting diode (OLED) devices, gained electrical sheet resistance is of the order of 1 to 2 kΩ square^{-1} for thicknesses of approx. 600 nm, and also can be decreased to below 1 kΩ/square by an additional treatment under inert or reducing conditions at elevated temperatures [14]. Also, with gravure-printed method the printing of transparent PEDOT:PSS (poly(3,4 ethylenedioxythiophene):poly-(styrene sulfonate)) electrodes was realized on a flexible PET substrate with a sheet resistance of 359 Ω square^{-1} indicating the possibility of using gravure printing method for production of flexible organic solar cells [15].

In the study reported by Chiolerio *et al.* [16], the realization of low resistance silver lines by ink-jet printing of a commercial silver nanoparticles (Ag NPs) based ink on a flexible kapton was reported. Electrical resistivity values of 4.9 μΩ cm (corresponding to the 32.65 % of pure bulk silver conductivity) were calculated for 6 and 8 layer thick films, which indicated clear possibility for producing low resistivity inductors and coupled LC systems (such as those for chipless radio frequency identification tag, RFID) with ink-jet printing technique [16]. Similar resistivity values were reported in study given by Lee *et al*. Namely, ink-jet printed lines with Ag nanoparticle colloidal ink (containing 57.3 wt.% Ag) of two polyimides with different thicknesses and one slide glass, showed resistivity values of 4.7 μΩ cm and 8.0 μΩ cm, respectively [17]. Kim *et al.* in their experimental study gained resulting resistivity of about 5 μΩ cm for electrodes printed with copper nano-ink on a flexible bismaleimide triazine (BT) core substrate with a drop-on-demand (DOD) piezoelectric ink-jet printing method [13]. Park *et al*. reported that conductive lines using Cu conductive ink ink-jet printed reached resistivity to 17.2 μΩ cm [18].

Cu pattern ink-jet printed using copper nanoparticles (Cu NPs) ink on flexible PI substrate and thermally treated in a furnace under reducing atmosphere showed the resistance of 0.88 ± 0.08 Ω on average [19]. On the other hand, Loffredo *et al*. reported resistance of 90 kΩ and 20 kΩ for devices with polymeric carbon films deposited with ink-jet technique on ink-jetted silver transducer and for devices with polymeric films ink-jetted on photolitographed gold transducer, respectively [20].

With screen printing technique using conductive copper ink that includes an organic metal complex of copper (II) neodecanoate and a copper hydroxide as metal precursors (used in printing RFID antenna) resistivity of 12.5, 5.4 and 4.4 μΩ cm (when heated at the temperature of 250 °C and 320 °C, respectively) was obtained for printed elements [17,21].

In the research which was presented by Deganello *et al*., a sheet resistance of 1.26 Ω square^{-1} for conductive flexo printed tracks on ITO coated flexible polyethylene terephthalate (PET) film with nano-particle silver ink was achieved, which indicates the feasibility and value of industrial flexographic process for the patterning of fine conductive structures [22].

In this paper we have aimed our investigation on checking the possibility to print conductive line elements in different shapes, as narrow as possible but still conductive, using pad printing technique. The shape sustainability, thickness and width of printed elements as well as resistance of conductive elements in dependence on the number of wet to wet overprinted layers were evaluated. In addition, energy dispersive spectroscopy (EDS) analysis was done in order to provide deeper insight in composition of used conductive ink. EDS (or EDAX — Energy Dispersive X-ray Spectroscopy) is a valuable tool in analytical testing where X-ray energies are used to identify and quantify the elements of any sample placed inside SEM. During analysis the sample is being bombarded with electrons, the various elemental constituents are generating their own characteristic X-rays which are collected and displayed in a spectrum. Namely, backscattered electron images in the SEM display compositional contrast that results from different atomic number elements and their distribution where through EDS are identified those particular elements and their relative proportions [23]. Here, EDS was used in order to define used conductive ink via the indentification of elemental constituents of a printed sample.

Materials and Methods

The printing process was carried out with ELEKCTRA$^{\Omega}$D'OR ED 5020 carbon based polymer ink (5 % of reducer was added) on a coated paperboard (300 g m^{-2}) on a one colour pad printing machine "TSH print swiss 1-1010" (used pad TP63 "TAMPON PRINT"). According to specifications given by the ink manufacturer, curing time by IR radiation was 50 minutes at the temperature of 150 ºC. Single, double and triple layer prints (wet to wet) were made.

Nyloprint WS (water washable) 0.73 mm thick steel based printing plate was used as a printing plate. It was processed on BASF Nyloprint CW 22×30 combined machine with first exposure time of 50 sec (exposure through positive film with solid printing elements), second exposure time of 90 sec (exposure through positive 90 % 200 lines per inch film), washout time of 2.5 min at the temperature of 28 ºC and drying time of 12 min at the temperature of 80 ºC, which was determined according to specifications given by printing plate manufacturer.

We have chosen to print line elements in width range from 50 up to 500 μm (gradation ratio of 50 μm) in a form of straight line, curved line, right angled line and cut angled line single, double and triple printed (wet to wet) (Fig. 1). In further text they will be referred as line 1 (50 μm), line 2 (100 μm), line 3 (150 μm), line 4 (200 μm) up to line 10 (500 μm).

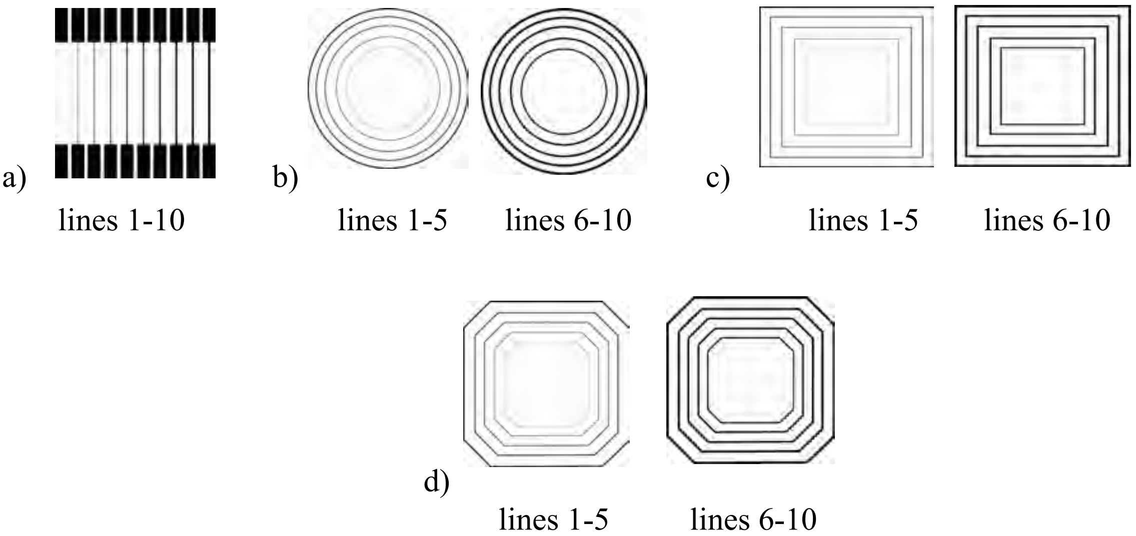


Fig. 1 Printed elements: a) straight lines, b) curved lines (circuits), c) right angled lines and d) cut angled lines

It must be emphasized, that due to multiple layer printing, the attention was paid to obtain precise print register. During printing process, the printing substrate was fixed with clamps on carrier plate on printing machine. Since overprinting was done with the same colour, it was not possible to track the changes in print register via register marks in it genuine sense. Therefore, each overprinted sample was firstly observed with digital microscope SibressPit in order to detect and dismiss prints with poor register. The width of printed lines was evaluated using image processing software ImageJ on micrographs made by digital microscope SibressPit with magnification of 140×. For each printed line and each shape, nine micrographs were made (on three different places on three printed samples). For each micrograph nine measurements were done. The results presented in this paper are average values of made measurements. The ImageJ software was also used for thickness determination of printed straight lines according to micrographs obtained by scanning electron microscope (SEM) 6460 JSM (JEOL, Japan) at magnification of 500×. The measurements were made on micrographs of cut sections for lines 8, 9 and 10, single, double and triple printed.

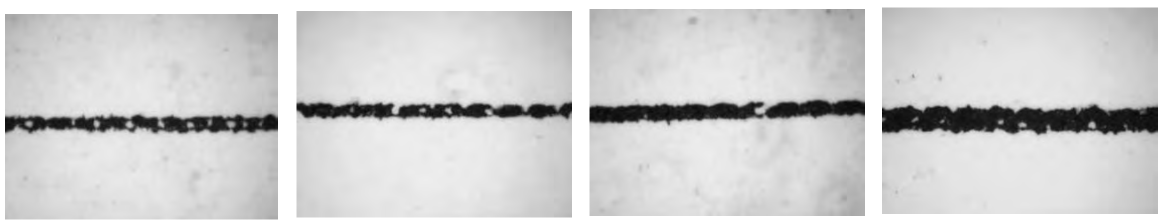
Resistance measurements for triple printed straight conductive lines were carried out using HP 4194A Impedance/Gain-Phase Analyzer for a frequency range from 1 to 30 MHz. Preliminary measurements were done with commercial unimeter, and the results led to the decision to measure resistance exclusively for triple printed lines, since the results gained for the other two groups of samples (single and double printed) were rather high.

Finally, EDS was done providing the indentification of elemental constituents in used electro conductive ink. For the analysis and generation of X-ray spectrum from the scan area of the SEM software INCA X sight (Oxford Instruments) was used. The magnification was 1000×, depth of 2 μm and accelerating voltage of 20 kV. The results were also presented in weight % and atomic %.

Results and Discussion

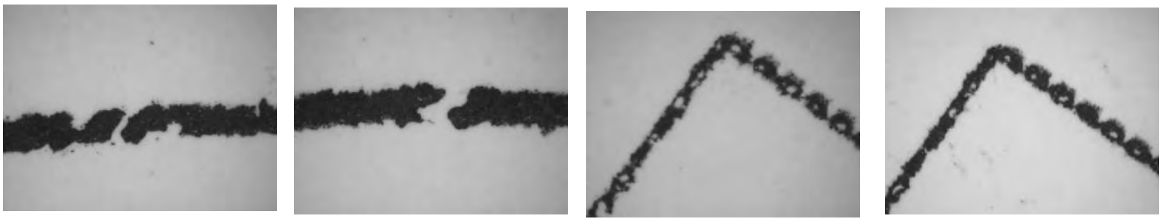
Line Printability

From the micrographs presented in Fig. 2 it is obvious that lines 1 and 2, single and double printed are not continuous, regardless of form, and consequently not conductive which was later confirmed with resistance measurements. This statement is also true for triple printed line 1 (Fig. 2).

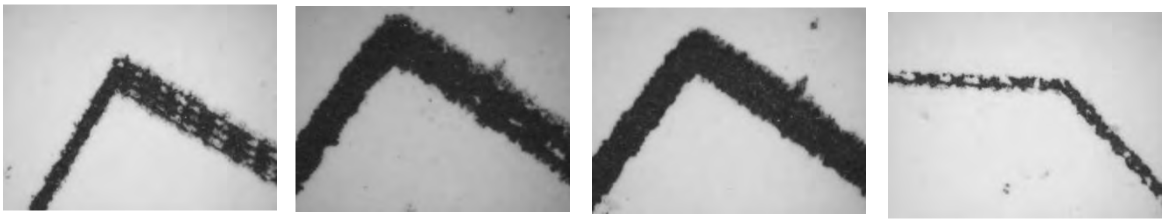


a) line 1 single printed b) line 1 double printed c) line 1 triple printed d) line 2 single printed

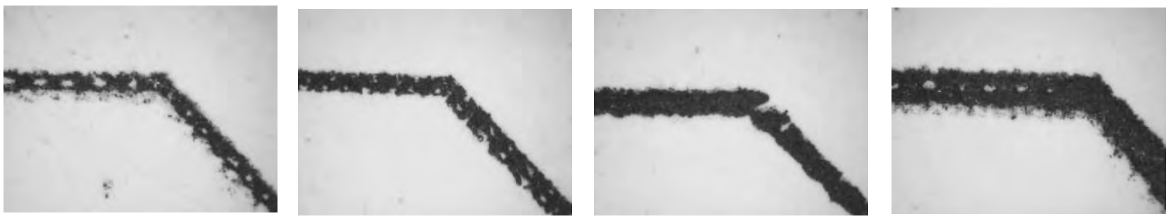
e) line 2 double printed f) curved line 1 single printed g) curved line 1 double printed h) curved line 1 triple printed



i) curved line 2 single printed — j) curved line 2 double printed — k) right angled line 1 single printed — l) right angled line 1 double printed



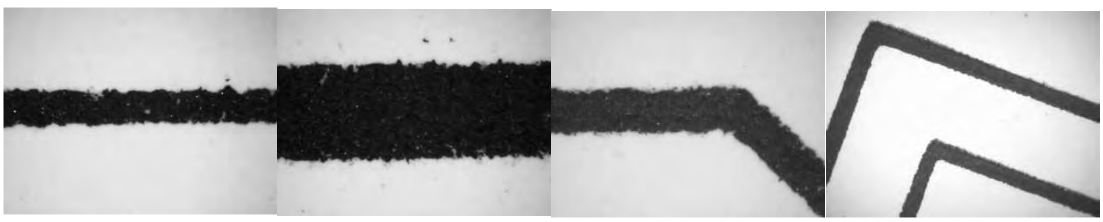
m) right angled line 1 triple printed — n) right angled line 2 single printed — o) right angled line 2 double printed — p) cut angled line 1 single printed



q) cut angled line 1 double printed — r) cut angled line 1 triple printed — s) cut angled line 2 single printed — t) cut angled line 2 double printed

Fig. 2 Micrographs of line 1 and 2 (single, double and triple printed), 140× magnification

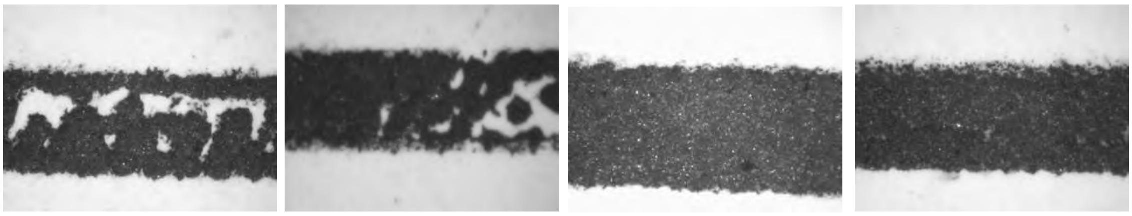
In case of all other, wider lines, regardless of shape (straight, curved, cut angled, right angled) continuous and shape permanent printing was obtained. Some examples of these lines are shown in Fig. 3.



a) straight line 3 single printed — b) straight line 10 triple printed — c) cut angled line 4 double printed — d) right angled line 2, 3 single printed

Fig. 3 Some examples of printed lines, 140× magnification

It must be emphasized that, during the experiment it was noticed that, considering pad printing technique, printing elements should never be parallel with the doctor blade trajectory, since ink transfer onto printing plate and consequently printing substrate will not be adequate. Figure 4 shows the difference in single printed lines when lines are parallel with doctor blade trajectory and when they are angled for approximately 5 degrees.



a) b) c) d)

Fig. 4 Single printed lines 10: parallel with doctor blade trajectory (a, b), angled for approximately 5 degrees ©, d) at 140× magnification

Line Width and Thickness

The calculated average values of line width measurements are presented in Table I.

Table I Average values of line width for all printed objects

Line	Straight line, mm			Right angled line, mm		
	Single printed	Double printed	Triple printed	Single printed	Double printed	Triple printed
1	0.07	0.08	0.09	0.10	0.10	0.13
2	0.13	0.15	0.16	0.22	0.19	0.24
3	0.19	0.21	0.22	0.30	0.27	0.31
4	0.22	0.24	0.25	0.36	0.34	0.37
5	0.27	0.29	0.31	0.45	0.40	0.43
6	0.32	0.34	0.35	0.44	0.47	0.45
7	0.37	0.38	0.40	0.49	0.55	0.50
8	0.43	0.46	0.48	0.56	0.61	0.56
9	0.47	0.51	0.53	0.59	0.64	0.59
10	0.50	0.55	0.58	0.66	0.69	0.65

Table I – Continued

Line	Cut angled line, mm			Circled line, mm		
	Single printed	Double printed	Triple printed	Single printed	Double printed	Triple printed
1	0.09	0.12	0.12	0.10	0.11	0.12
2	0.14	0.20	0.20	0.18	0.20	0.19
3	0.19	0.28	0.27	0.25	0.26	0.25
4	0.24	0.32	0.32	0.32	0.31	0.31
5	0.28	0.36	0.37	0.37	0.33	0.34
6	0.37	0.44	0.44	0.38	0.45	0.46
7	0.42	0.50	0.48	0.41	0.50	0.53
8	0.47	0.54	0.54	0.46	0.53	0.58
9	0.49	0.57	0.57	0.50	0.58	0.59
10	0.55	0.62	0.61	0.51	0.59	0.52

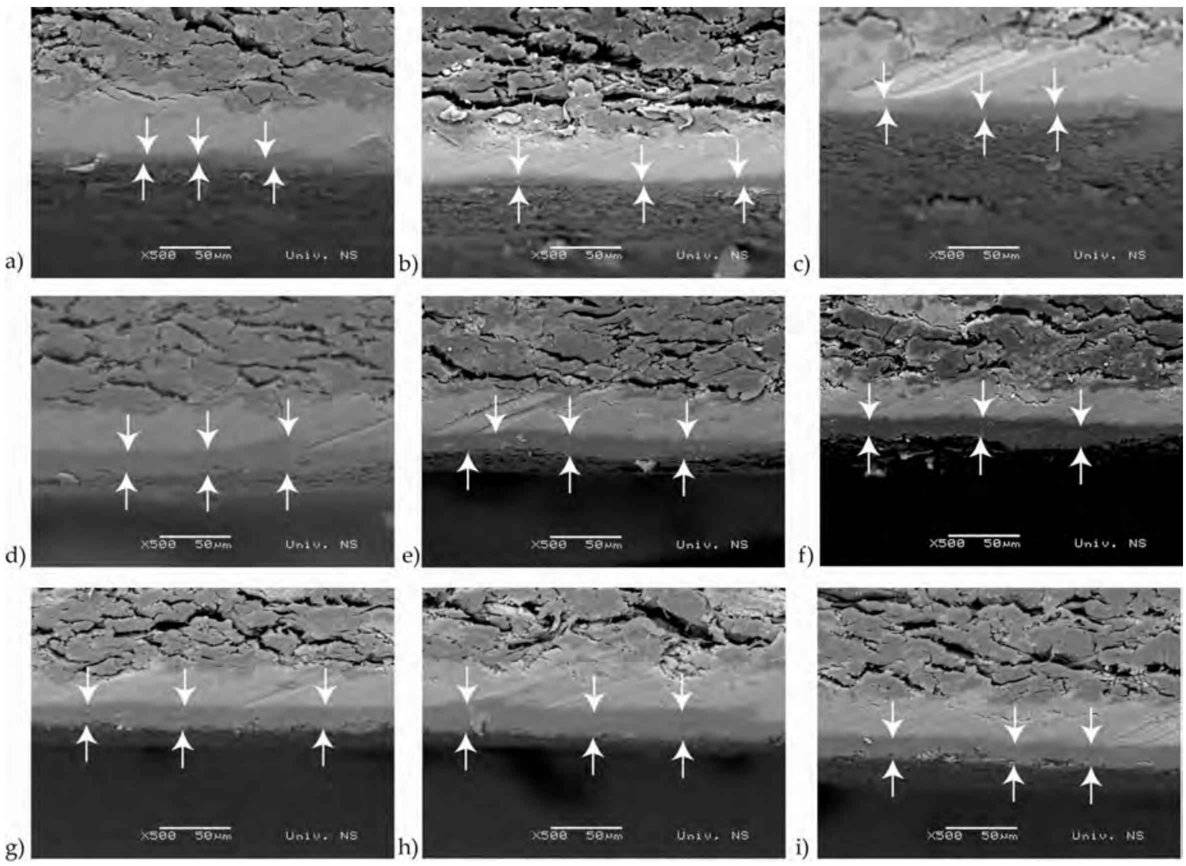


Fig. 5 Micrographs of cut sections of printed conductive lines: single printed line 8 (a), line 9 (b), line 10 (c); double printed line 8 (d), line 9 (e), line 10 (f) and triple printed line 8 (g), line 9 (h) and line 10 (i)

Knowing the target width of each line, it is obvious that printed lines are wider regardless of number of printed layers, where straight lines showed the lowest deviation. The values given in Table I also indicate that line width depends on the number of printed layers. Greater difference in line width is noticed between single and double rather than double and triple printed lines. It can be assumed that distortion was influenced by printing pressure, the hardness of the used pad as well as friction [8]. Partly, the changes in line width might be a consequence of changes in print register.

Figure 5 presents SEM micrographs of cut sections of straight lines used for line thickness determination. White arrows point out the conductive ink layer. The obtained results of line thickness are presented on Fig. 6.

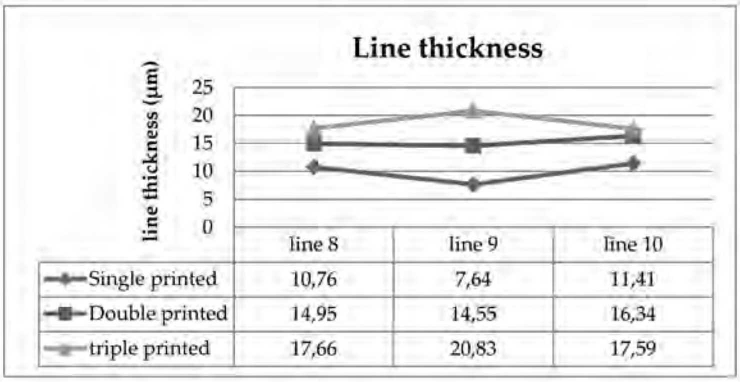


Fig. 6 Line thickness (average values) of single, double and triple printed lines 8, 9 and 10

The graph presented in Fig. 6 shows that line thickness increases with the number of printed layers. The highest thickness of 20.83 μm was detected for triple printed line 9. The difference in line thickness is greater between single and double printed layer than double and triple printed layer which might be influenced by ink drying, printing pressure, pad hardness and certainly surface tension and surface roughness of interacted materials.

Resistance (Rs)

Preliminary measurements of printed lines resistance were done using unimeter (Table II). Line printability analysis showed that lines 1 and 2 single and double printed as well as line 1 triple printed are not continuous, thus it was expectable that those lines are not conductive.

Table II Resistance values obtained with unimeter

Line	Resistance, kΩ		
	Single printed	Double printed	Triple printed
1	-	-	-
2	-	-	40.59
3	142.3	38.35	14.18
4	126.2	18.93	17.51
5	67.8	13.93	10.81
6	46.48	10.45	7.11
7	41.03	10.12	8.11
8	34.51	10.60	3.62
9	29.32	9.96	3.45
10	23.62	8.25	3.05

As it can be seen from Table II, single and double printed lines showed high resistance values, thus precise resistant measurements with HP 4194A Impedance/Gain-Phase Analyzer were done only for triple printed conductive lines. The resistance values for a frequency range from 1 to 30 MHz are presented in Fig. 7.

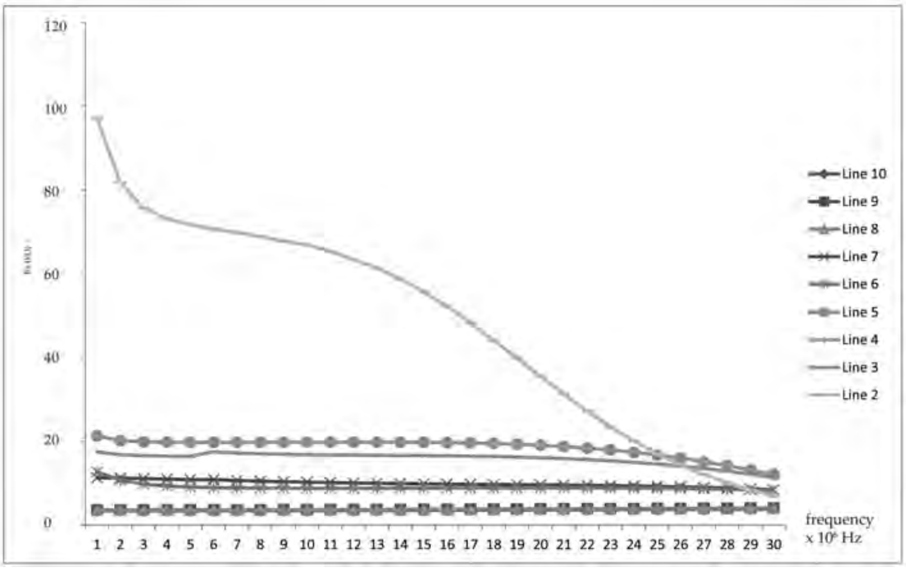


Fig. 7 Resistance (Rs) for triple printed straight lines

The highest resistance values, thus the lowest conductivity, through the whole frequency range are given for the narrowest line, line 2. For other printed lines, resistance values are not higher than 22 kΩ. The lowest resistance values, thus the highest conductivity, observed in lines 9, 8 and 10 (in the range of 3.35-4.05 kΩ), than lines 6 and 7 (in range of 8.5-12.68 kΩ) and then lines 3, 4 and 5 (in the range of 10.52-21.34 kΩ).

EDS – Energy Dispersive Spectroscopy Analysis of Used Materials

The morphology of used materials, substrate and ink, was studied with EDS based on the printed sample placed in SEM (micrograph presented in Fig. 8).

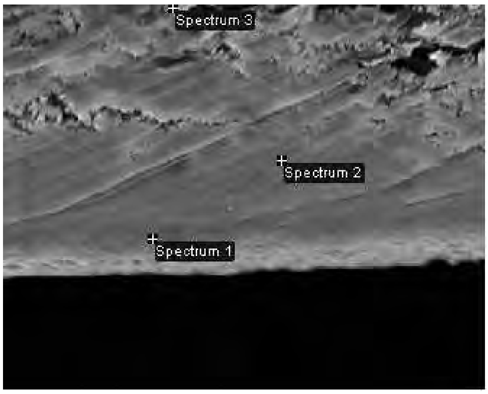


Fig. 8 SEM micrograph used for EDS

The generated X-ray spectrums of scanned areas are presented in Fig. 9. Spectrum 1 refers to electro conductive ink, Spectrum 2 refers to paper coating and Spectrum 3 refers to basic paper material.

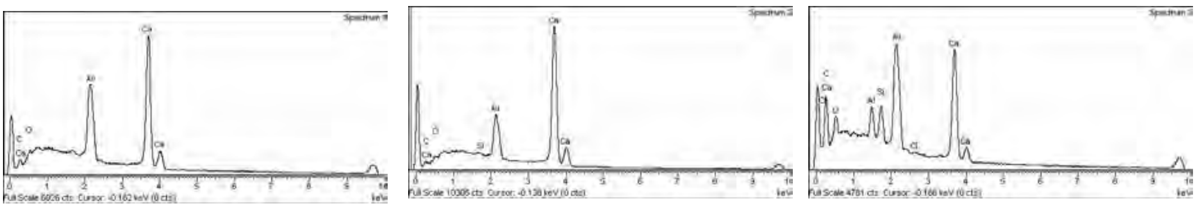


Fig. 9 Generated X-ray spectra: a) Spectrum 1 for electro conductive ink, b) Spectrum 2 for paper coating and c) Spectrum 3 for paper

Table III lists the detected elements (peaks in Fig. 9), their weight% and atomic%. Au was omitted, since the observed specimen was gold steamed in the standard preparation procedure for SEM.

The spectrum of the most interest is Spectrum 1 since it represents a spectrum generated from conductive ink. The results presented in Table III indicate that the conductive ink is composed of C (Carbon), Ca (Calcium) and O (Oxygen). Since this is carbon based ink, through this analysis the weight% in printed ink layer is determined and it amounts to 12.2 ± 0.86 %. The high weight% of Ca might come from calcium carbonate since it is usually found in commercial ink formulations or it might be from the migration from the second layer (Spectrum 2), since Ca is also found there in high percentage.

Table III Weight (*Wt*) and atomic % of detected elements *via EDS*

Element	Spectrum 1		Spectrum 2		Spectrum 3	
	Wt, %	*Atomic*, %	*Wt*, %	*Atomic*, %	*Wt*, %	*Atomic*, %
C	12.2 ± 0.86	24.74	7.19 ± 0.57	16.85	40.07 ± 1.03	55.91
Ca	64.49 ± 2.42	39.43	75.46 ± 2.08	53.00	23.08 ± 0.52	9.65
O	23.39 ± 2.81	35.83	16.86 ± 2.24	29.67	27.53 ± 1.27	28.84
Si	-	-	0.48 ± 0.17	0.48	4.75 ± 0.18	2.84
Al	-	-	-	-	4.08 ± 0.17	2.53
Cl	-	-	-	-	0.5 +0.11	0.23

Conclusion

The experimental work presented in this paper was focused on evaluating the possibility to print different conductive elements, using pad printing technique, with carbon based conductive ink on paper substrate. The shape sustainability, thickness and resistance of conductive elements in dependence on the number of wet to wet overprinted layers were evaluated.

Line printability analysis showed that the narrowest continuous line element in the shape of straight line, curved line, cut angled line and right angled line which can be printed under defined conditions is 150 μm wide when single and double printed and 100 μm wide when triple printed. These analyses also indicated that when printing is performed, engraved line printing elements on printing form should not be parallel to doctor blade trajectory, otherwise ink transfer will be inadequate and will result in discontinuous elements.

From the images taken with digital microscope at magnification of 140× and image processing software ImageJ, the changes in line width due to printed layers were evaluated. It was shown that printed lines, regardless of number of printed layers and shape, are wider than nominal. It is clear that the number of layers printed contributes to line width, and it can be concluded that overprinting of the second layer increases the line width more than the third layer.

Micrographs of cut sections of printed straight lines taken with SEM allowed line thickness analysis in software ImageJ. The performed measurements showed that the thickness of printed lines increases with the number of printed layers. The highest thickness of 20.83 μm was detected for triple printed, line 9, with nominal width value of 450 μm.

The resistance measurements showed that the thicker and wider the line the lower the resistance, thus the higher the conductivity.

Generated X-ray spectrum using EDS showed that the weight% of Carbon in the conductive ink layer is 12.2 ± 0.86 %.

Finally, it must be emphasized that printed line wideness, thickness and consequently resistance are highly dependable on the ink characteristics, pad characteristics, printing form characteristics, used printing system and the process parameters. Also, the curing time and temperature play important role in achieving ink resistance (conductivity). In the presented experiment, curing time and drying temperature were adopted according to manufacturers' specifications, but further investigations could be directed to determine if longer or shorter curing time improves resistance due to different number of printed layers and different width and shape of printed line elements.

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