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# Improvement of the evaluation of inkjet printed silver based layers' adhesion

Peter Lukacs, Alena Pietrikova and Ondrej Kovac

Department of Technologies in Electronics, Technical University of Kosice, Kosice, Slovakia

**ABSTRACT**

This paper presents the improved method of analysis and evaluation of the silver layers' adhesion printed by inkjet printing technology. The generally known method for adhesion measurement of layers and coatings according to the standards ASTM D3359 and ISO 2409 has been improved by development of the automatic evaluation software of achieved results from cross cut tests. The developed evaluation tool offers the possibilities of calculation the detached area, rotation of the image, setting the threshold and the automatic classification according to the mentioned standards. The inaccuracy caused by samples' assessment by humans is mainly eliminated by using of this tool. The developed evaluation tool has been applied to experimental works oriented to analysis of the adhesion of two commercially available silver based nano-inks on two polymeric based substrates (PI and PET). For this purpose, different sintering conditions have been applied to analysed samples. The achieved results show the differences between the investigated nano-inks' adhesion on two types of polymeric substrates. The adhesion of silver layers can be optimized by controlling the sintering conditions. By developing the software tool the simple and inaccurate method of adhesion analysis has been upgraded to the appropriate technique to the silver layers' adhesion testing. The improved method for adhesion measurements can serve as a tool for fast and relatively simple technique for technologist.

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InkJet printing; adhesion; silver layer; measurement; cross cut

## 1. Introduction

The special deposition technology of nano-inks onto polymeric substrates, known as inkjet printing technology, extends the application capabilities of printing technologies to unexplored areas. Low thickness, high printing accuracy and electrical conductivity of applied materials are only the small part of benefits of described technology. The inkjet printing technology is more and more often used not only for prototyping but also in production of electrical structures. There are also shortages which limit the applicability of inkjet printing technology. The deposition process of nano-inks brings a lot of challenges. Trajectory of drops flight, optimal control signal

CONTACT Peter Lukacs peter.lukacs@tuke.sk

of the print head, surface properties of polymeric substrates and the interaction of the nano-ink and substrate presents the main issues in this field. Each improperly selected technological step can be a cause of the irreversible changes in the final quality of printed structure [1–11].

Low sintering temperature and high reliability of inkjet printer layers move this technology to the forefront in the field of precise printing of electronic structures. Nano-particles contained in the nano-ink are coated by what they are not electrically conductive. To achieving the electrical conductivity of these particles the sintering process is necessary. The particles are welded each to other at temperature below their melting point during sintering. After the sintering process electrically conductive necks are created. The adhesion of silver layers deposited by inkjet printing technology on polymeric substrates is mainly influenced by the surface properties of polymeric substrates, wetting, spreading, penetration and by complexity of applied nano-ink. The solvents evaporation rate, drying, the sintering temperature and time have significant impact on the adhesion as well. The uncontrolled sintering process of silver layers or incorrect post processes can cause the excessive tension in the layer resulting in cracks or in separation of layers [12–17].

The adhesion of layers especially in the cases of electrically conductive thin layers has to fulfil a strict conditions. Bending of the silver layers is critical factor for initializing of the disruption of its adhesion mechanism. By optimizing of the silver layers' adhesion the multilayer structures can be manufactured for the reason of achieving the high signal integrity and miniaturization of electronic circuits. For achieving the homogenous electrically conductive layer the deposition of multilayer structure is necessary. Parameters of the first layer are decisive for formation of the adhesion between the silver layer and the substrate. Creation of bonds by diffusion sintering applied in the inkjet printed silver layers presents the possible technique to implementation of the inkjet printing technology to the field of precise electronics [18–20]. Size reduction of particles to the nano area causes the reduction of the sintering temperature and the sintering time as well. The initial stage of the adhesion formation begins during the deposition of the first layer because the substrate is heated up. At the temperature higher than room temperature, the evaporation of the mixture of solvents from the nano-inks occurs [17,21–24].

The pull-off tests often applied for analysis of the silver layers' adhesion deposited by inkjet or screen printing technology require a special additive technological layers [25–27]. These tests lay on the addition of epoxy adhesive between the stud and the investigated layers as well as between the flexible substrate and the rigid board. Next method for the adhesion's analysis is a shear test, where the die is glued to the investigated silver layer and the shear tool acts a force horizontally with the substrate [28]. The disadvantage of these methods is a necessity of adding the special adhesive to glue a stud or a die. Next shortcoming of described test methods lays in samples creation where a special technological steps have to be applied. Also the chemical consistence of the glue can influence the psychical changes in the silver layer especially when the thickness of investigated layer is too low. The plastic deformation of the soft substrate during the test can also distort the results as well. These test methods are not primarily useful for thin layers deposited by inkjet printing technology. The

Table 1. Applied sintering conditions [33].

| Substrates/Ink | Nano-ink No. 1 | Nano-ink No. 2 |
|---|---|---|
| PI DuPont™ Kapton® HN 200 | 160 °C/30 min | 200 °C/120 min |
| | 160 °C/60 min | 200 °C/150 min |
| | 180 °C/30 min | 230 °C/60 min |
| | 180 °C/60 min | 230 °C/90 min |
| | 200 °C/30 min | 230 °C/120 min |
| | 200 °C/60 min | 230 °C/150 min |
| | 220 °C/30 min | 250 °C/60 min |
| | 220 °C/60 min | 250 °C/90 min |
| | 250 °C/30 min | 250 °C/120 min |
| | | 250 °C/150 min |
| PET Mylar® A | 160 °C/30 min | Not applied |
| | 160 °C/60 min | |
| | 180 °C/30 min | |
| | 180 °C/60 min | |
| | 200 °C/30 min | |
| | 200 °C/60 min | |

solution for adhesion analysis of thin silver layers created by inkjet printing technology is admittedly the cross cut test [29,30]. This method lays in the mechanical disruption of the investigated layer and in the following observations of the cut area. The results are evaluated and classified according to the standards ISO 2409 and ASTM D3359 [31,32]. By means of the developed software the main disadvantage of this method has been eliminated which lays in the inaccurate, subjective evaluation of results by humans.

This paper deals with the improvement of the cross cut test method by development of the evaluation software, which is implemented in the evaluation of results from the experimental works oriented to the investigation of silver based layers' adhesion. In addition, the application of the developed software to the silver layers' adhesion is presented.

## 2. Samples preparation and selected test method

Investigation in the field of adhesion mechanism between inkjet printed silver based layers and polymeric substrates were realized by application of cross cut test method. For this reason the samples in rectangular shape with dimensions 50 × 50 mm were created by using the inkjet printer MicroFab Jetlab 4 × l-A with orifice diameter 70 µm. After the detailed evaluation of preliminary results the 2 layers samples were used for cross cut tests. Silver layers were sintered immediately after the deposition process according to the Table 1. For achieving the more detailed results two kinds of silver based nano-inks were applied and investigated. Marked as No. 1 in the Table 2 is a silver based nano-ink produced by UTDots – UTDAgIJ, the second analysed nano-ink marked as No. 2 is AX JP-6n by Amepox LTD. The printed silver layers were heat sintered in conventional oven (Binder FP 53) under standard conditions without vacuum or accelerates. The applied sintering conditions were selected in regard to recommended sintering conditions by manufacturers as well as by physical specifications of the nanoparticles contained in the investigated nano-inks.

As it is generally known, the adhesion mechanism can be totally different in dependence on the surface properties of polymeric substrates. For this reason the

Table 2. Selected specifications of investigated silver based nano-inks [33].

| Silver basednano-inks | Nano-ink No. 1 | Nano-ink No. 2 |
|---|---|---|
| Percentage of silver [%] | 25–60 | 40–60 |
| Viscosity [mPas] | 1–30 | 7.5–10.5 |
| Particle size in diameter [nm] | 10 | 3–7 |
| Recommended sintering conditions | 120–300 °C | 220–230 °C<br>60 min |

polyimide substrate Kapton HN and PET substrate Mylar A with thickness of 125 μm were selected for our experiments. The roughness of selected substrates are comparable, Ra = 0.044 μm for PI Kapton HN and Ra = 0.046 μm for PET Mylar A. The coefficient of thermal expansion for PI substrate Kapton HN is 20 ppm and for PET substrate Mylar is 0.17 ppm [34,35]. For every sintering condition 15 samples were created for both of investigated nano-ink and substrate. The used polymeric flexible substrates were prepared for printing by applying the cleaning bath in isopropyl alcohol for 1 min, which has the proved negligible impact onto the surfaces' properties [1]. After the cleaning bath the surfaces were rinsed by methanol which flashes the particles of dust or other impurities. Next the substrates has been heated up to 60 °C for the reason of evaporation of the rest of applied chemical agents.

As it was mentioned earlier, the cross-cut test method has been selected in according to the standards ASTM D3359 and ISO 2409. For this reason, the measuring equipment TQC CC 3000 which creates six straight cuts into the analyzed silver layer, has been used. The depth of cuts can be controlled by retractable blades. In the Figure 1 the basic principle of cross cut test is shown. Hence, the silver layer is damaged by equipment's blade on X and Y axis. The critical places are in the cross sectional areas, where the silver layers are mainly damaged. The main principle of cross cut tests lays in the making of two cuts perpendicularly to each other and in the brushing of the pattern lightly with the brush several times back and forth along each of the diagonal lines of the lattice pattern. After these steps, the examination of the cut area and classification of the test by mark according to percentage of the affected or destroyed area especially along the edges and intersections are realized.

The number of blades and spacing is specified by standard ISO 2409 [31]. For the soft substrates with the thickness of layer up to 60 μm, the number of blades is 6 with 2 mm spacing. In our experiments, the thickness of layers is <5 μm. In the Figure 2, the example of cut silver layer on polyimide substrate after the cross cut test is shown.

As the previous experiments presented in [36] show, the homogenous structure is achieved when the temperature of the substrate during the deposition process not exceed 60 °C. In the cases of silver based nano-inks the temperature above 60 °C causes the coffee ring effect when the evaporated volume is smaller in the middle than the volume in the edge area. The evaporation rate is mainly depended on mixture of solvents, surface properties of substrates but mainly on the temperature of substrate. For the reason of achieving the homogenous silver layer during the deposition process the substrates were heated up to 60 °C, which ensures the homogeneity of the deposited structure in the whole volume and not allows the creation of the coffee ring effect. The cross cut tests were realized at room temperature.

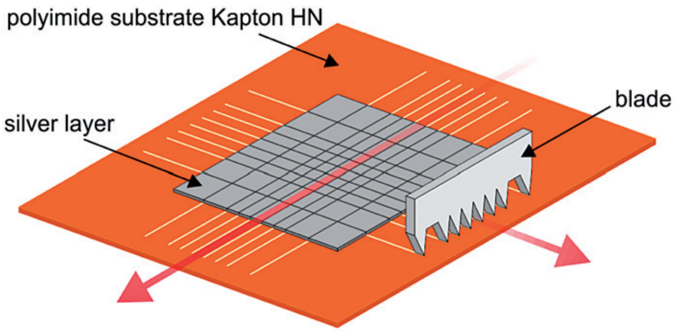


Figure 1. The principle of cross cut test method.

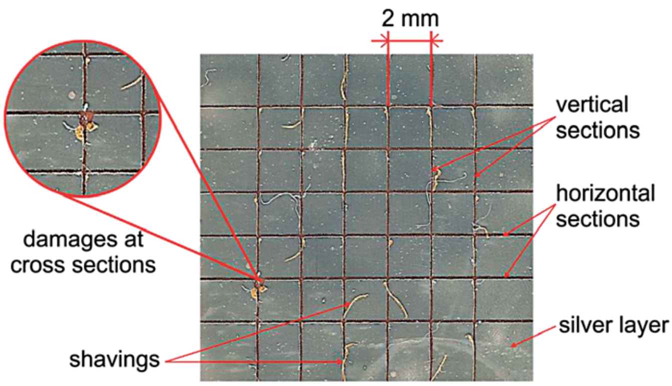


Figure 2. Example of the result of cross-cut test on silver layer.

Table 3. Classification of test results.

| Classification | | |
|---|---|---|
| ISO 2409 | ASTM D3359 | Type size of detached area [%] |
| 0 | 5B | The edges of the cuts are completely smooth; none of the square of the lattice is detached. |
| 1 | 4B | <5 |
| 2 | 3B | 5–15 |
| 3 | 2B | 15–35 |
| 4 | 1B | 35–65 |
| 5 | 0B | >65 |

In the Table 3, the decision levels of detached area according to standards ASTM D3359 and ISO 2409 are shown [31,32]. These levels are implemented into the developed software tool.

## 3. Results analysis

For the reason of elimination of the human influence on the classification of cross cut tests results, the special software (CCT_Analyzer) tool has been developed. For this purpose, programming environment of Matlab has been used. The developed software detects and calculates the detached area from the analysed substrate and

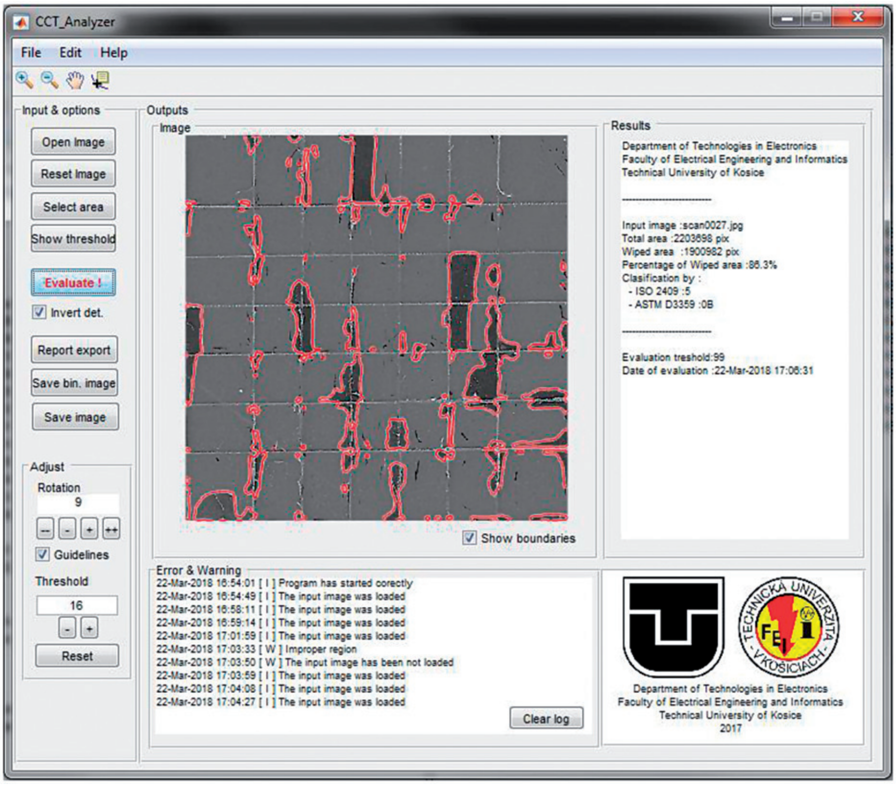


Figure 3. GUI of CCT_Analyzer.

automatically ranks the adhesion into the classification according to mentioned standards. The GUI of the described software tool is shown in the Figure 3.

The developed software tool allows the working with the image regardless of the image format. An obvious part of the software is rotation of the image or selection the analysed area, using the guidelines or reset the image. With changing the threshold, the technologist can manually adjust the selection of detached area. If the selected area and the value of the threshold is appropriate, a report with required data can be exported. The exported report contains a basic info about the evaluated image, the percentage of detached area and the classification according to mentioned standards as well. The image with areas marked by software can be also saved for next processing.

The block diagram of the developed software tool is shown in the Figure 4. The evaluation works on the base of thresholding of input image, which leads to creation of a binary image. For minimization of user interference the automatic threshold detection has been implemented into the software tool. The optimal threshold is defined as a local minimum between two most occurring places in the image achieved from the histogram. The edges of the input image (result of the cross cut test) are enhanced by summation of the input image and the Laplacian image [37]. Next the enhanced image is subsequently filtered by median filter which removes the noise and small particles of dust or shavings of substrates caused by the blade. The

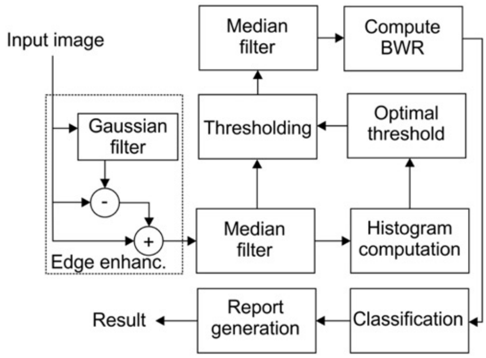


Figure 4. Block diagram of the evaluation tool.

Gaussian filter implemented in the algorithm is realized by computation of convolution of the input image $x$ and the impulse response $h$ of Gaussian filter based on Equation 1.

$$y(n_1, n_2) = x(n_1, n_2) * h(n_1, n_2) = \sum_{i=-\infty}^{\infty} \sum_{j=-\infty}^{\infty} x(i,j).h(i-n_1, j-n_2) \quad (1)$$

Examples of the evaluation of results from cross cut tests are presented in the Figure 5. As it can be seen, the software detects rather the layer or the substrate in dependence on the settings. The technologist can easily choose what should be evaluated. In the Figure 5 the percentage of the detached area and the classification according to the standards ISO 2409/ASTM D3359 respectively is shown. For example in the first image in the Figure 5 the silver layer on polyimide substrate Kapton HN with 86% of detached area is shown. Cross cut tests have been realized immediately after the sintering process. After the cross cut tests layers have been brushed for removing shavings or dust resulting from the tests according to the mentioned standards. Next the samples have been scanned in high resolution (more than $3000 \times 3000$ px) and evaluated in the developed software tool.

The developed software has been implemented into the evaluation of silver layers' adhesion. The adhesion of two kinds of silver based nano-inks used for inkjet printing technology on polymeric substrates has been investigated at different sintering conditions. In the case of nano-ink No. 1, the significant improvement of the adhesion has been achieved by raising the temperature during the sintering process. As it can be seen in the Figure 5, sintering of the nano-ink No. 1 on PI Kapton at the temperature 220 °C causes that the detached area is $<$11.5% (2/3B). Longer duration of the sintering process at 220 °C or increasing the temperature reduces the detached area below 5%.

In the Figure 6, the adhesion between the nano-ink No. 1 and PET substrate Mylar is excellent already at the temperature 180 °C, can be seen. For the reason of low thermal resistance, PET substrate Mylar has not been applied for higher temperatures than 200 °C and for nano-ink No. 2.

The homogenous silver based layer is achieved above the temperature 180 °C for the time 60 min in the case of the nano-ink No. 1. Below this temperature, the silver

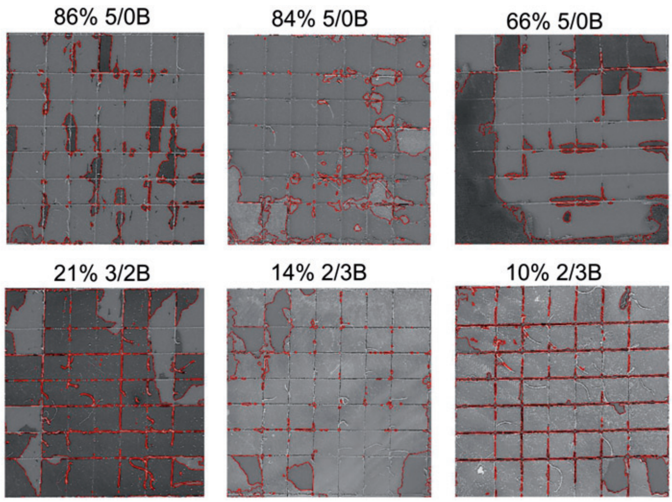


Figure 5. Examples of evaluated results from cross cut tests based on ISO 2409/ASTM D3359.

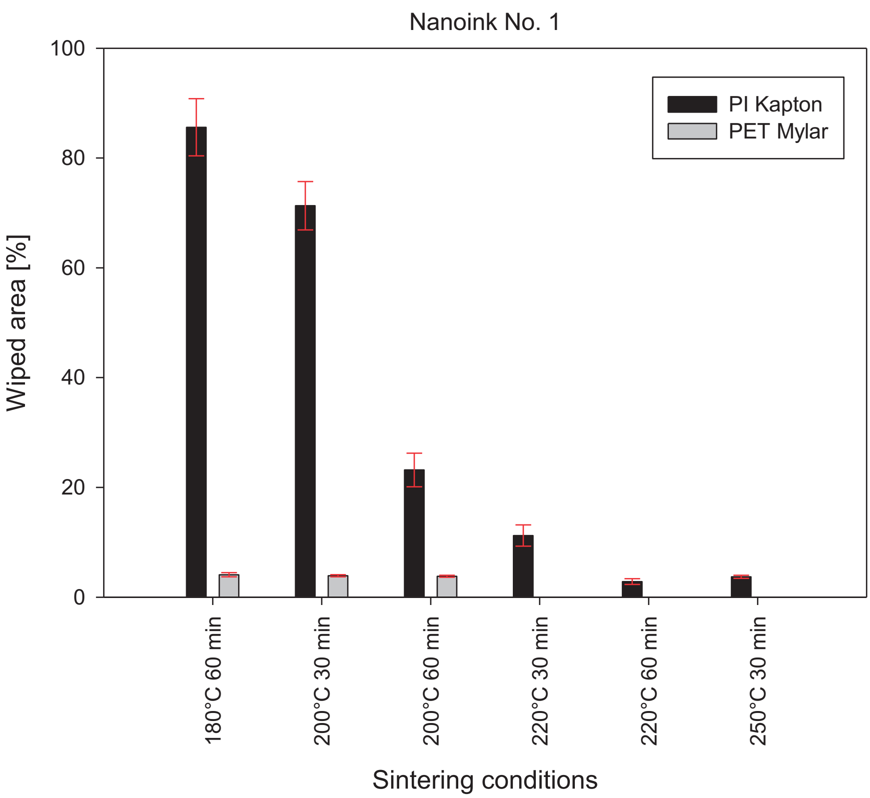


Figure 6. Influence of the sintering conditions on the adhesion for nano-ink No. 1 on polyimide substrate Kapton and PET Mylar.

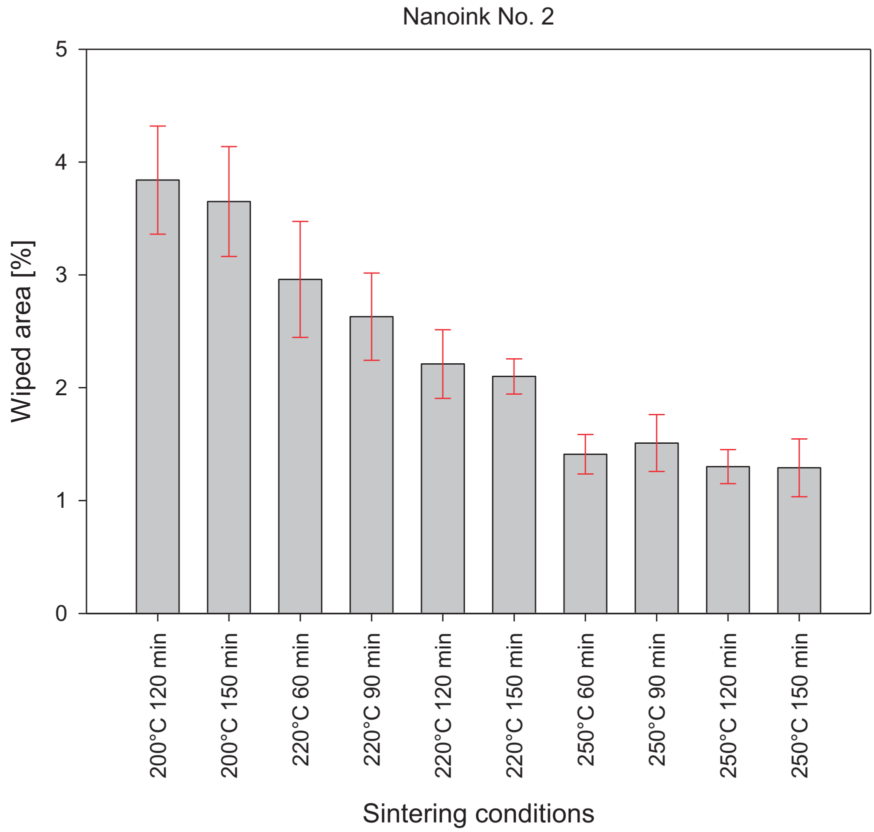


Figure 7. Influence of the sintering conditions on the adhesion for nano-ink No. 2 on polyimide substrate Kapton.

layers have been separated from each other and not from the substrate after brushing the layers according to mentioned standards. The inhomogeneity of the inadequate sintered silver layers has been investigated in previous experiments [33,36]. For this reason, the results from cross cut tests have been not evaluated below this temperature.

In the Figure 7, the influence of sintering conditions on the adhesion of nano-ink No. 2 is presented. For achieving the great adhesion on polyimide substrate Kapton classified as 0/5B and 1/4B, there is the necessity of sintering at the temperature 200 °C for time longer than 120 min or at 220 °C at least 60 min.

In the Table 4, the complete results of adhesion analysis of two kinds of silver based nano-inks on two polymeric substrates are listed. Results are shown by the detached area after the cross cut test and by the classification according to the standards.

The achieved results of adhesion analysis by application of developed software tool has been compared with results by human assessment. For this purpose, two groups of people has been asked for subjective classification of standard according to mentioned ISO and ASTM standards. First group consists of 14 selected technologist in electronics and material engineers from Technical university of Kosice. The second

Table 4. Complete results of adhesion analysis.

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| PI Kapton® HN | | | | | | | | | | |
| Nano-ink No. 1 | | | | | | | | | | |
| Sintering conditions | 160 °C 30 min | 160 °C 60 min | 180 °C 30 min | 180 °C 60 min | 200 °C 30 min | 200 °C 60 min | 220 °C 30 min | 220 °C 60 min | 250 °C 30 min | |
| Detached area in % | not evaluated | | | 85.6% | 71.31% | 23.18% | 11.23% | 2.84% | 3.71% | |
| Standard | **not evaluated** | | | **5/0B** | **5/0B** | **3/2B** | **2/3B** | **1/4B** | **1/4B** | |
| Nano-ink No. 2 | | | | | | | | | | |
| Sintering conditions | 200 °C 120 min | 200 °C 150 min | 230 °C 60 min | 230 °C 90 min | 230 °C 120 min | 230 °C 150 min | 250 °C 60 min | 250 °C 90 min | 250 °C 120 min | 250 °C 150 min |
| Detached area in % | 3.84% | 3.65% | 2.96% | 2.63% | 2.21% | 2.1% | 1.41% | 1.51% | 1.3% | 1.29% |
| Standard | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** | **1/4B** |
| PET Mylar® A | | | | | | | | | | |
| Nano-ink No. 1 | | | | | | | | | | |
| Sintering conditions | 160 °C 30 min | 160 °C 60 min | 180 °C 30 min | 180 °C 60 min | 200 °C 30 min | 200 °C 60 min | 220 °C 30 min | 220 °C 60 min | 250 °C 30 min | |
| Detached area in % | not evaluated | | | 4.09% | 3.9% | 3.81% | not applied | | | |
| Standard | **not evaluated** | | | **1/4B** | **1/4B** | **1/4B** | not applied | | | |
| Nano-ink No. 2 | | | | | | | | | | |
| Sintering conditions | 200 °C 120 min | 200 °C 150 min | 230 °C 60 min | 230 °C 90 min | 230 °C 120 min | 230 °C 150 min | 250 °C 60 min | 250 °C 90 min | 250 °C 120 min | 250 °C 150 min |
| Detached area in % | not applied | | | | | | | | | |
| Standard | not applied | | | | | | | | | |

Bold terms represent the final classification of achieved results due to standards for adhesion testing.

group has been built up by 46 students of the study program Technologies in electronics at Technical University of Kosice. In the Table 5, the comparison of achieved results by proposed method and subjective evaluation is presented.

As it is clear from the Table 5, the different classification of detached area according to the standard ISO 2409 by software and human assessment can be observed in the case of samples 3, 4 and 6. These limit cases present the biggest issue in the field of exact evaluation of detached area.

## 4. Discussion

The paper provides the results of adhesion measurements of two silver based nano-inks on two polymeric substrates. It was proved, that PET substrate Mylar achieved the excellent adhesion when the nano-ink No. 1 has been applied. In the case of the PI substrate Kapton at same nano-ink, the adhesion classified as 1/4B has been achieved at the temperature 220 °C for 60 min. It can be probably caused by different reaction between the solvents included in the nano-ink and polymeric substrates surfaces.

The higher coefficient of thermal expansion can causes the cracking of the thin diffusion layers during the initial stages of adhesion formation. For this reason, the preheating of the substrate during the deposition process, the drying of nano-inks and sintering profiles have to be strictly adhered [36]. The roughness of the investigated substrates has no proved impact on the adhesion of analyzed silver layers. In the case of polyimide substrate Kapton, the better adhesion has been achieved at nano-ink No. 2.

The developed software tool for the automatic evaluation of the results from cross cut tests has been tested. The achieved results proved the usability of this tool for

**Table 5.** Comparison of achieved results with human assessment.

| | Proposed method | | Subjective evaluation – students | | | | Subjective evaluation - technologist | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Sample ID | DWR [%] | ISO | Mean | Std. | ISO (based on median) | ISO (based on mean) | Mean | Std. | ISO (based on median) | ISO (based on mean) |
| 1 | 86 | 5 | 4,83 | 0,48 | 5 | 5 | 4,93 | 0,26 | 5 | 5 |
| 2 | 84 | 5 | 4,7 | 0,46 | 5 | 5 | 4,64 | 0,48 | 5 | 5 |
| 3 | 66 | 5 | 4,39 | 0,53 | 4 | 4 | 4,43 | 0,49 | 4 | 4 |
| 4 | 21 | 3 | 2,22 | 0,83 | 2 | 2 | 2,57 | 0,49 | 3 | 3 |
| 5 | 14 | 2 | 1,52 | 0,65 | 2 | 2 | 1,57 | 0,49 | 2 | 2 |
| 6 | 10 | 2 | 0,63 | 0,57 | 1 | 1 | 1,36 | 0,48 | 1 | 1 |

analysis of the results of cross cut tests. The application of this software tool significantly improves the accuracy and the area of applicability of cross cut tests for the adhesion investigation of silver layers. The results presented in this paper offer the improved testing methodology of inkjet printed silver layers.

The presented software tool improves the generally known method for adhesion analysis of layers by automatic methodology of the results assessment. The main benefit of the improved method is a suitability for simple and cheap analysis of the adhesion of layers without the necessity of microscopic analysis. The main value of the improved method has been proved by the comparison of the achieved results with human assessment where the developed software tools offered the more precise results according to mentioned standards for cross cut tests.

## 5. Conclusion

The presented paper aims at the evaluation of the adhesion of silver based layers deposited by inkjet printing technology. For the reason of elimination the inaccuracy caused by human evaluation of the achieved results by cross cut tests, the automatic evaluation software has been designed and developed. The developed software tool significantly increases the usability of the simple and cheap cross cut test method for investigation of inkjet printed layers' adhesion. This software offers the set of tools oriented to detailed analysis of the image from the cut layers.

The achieved results present the significant benefit in the field of evaluation of silver based layers created by inkjet printing technology. Experiments show the dependence of sintering conditions on the adhesion of the silver layers. The paper also offers the comparative study of silver layers' adhesion deposited by inkjet printing technology.

The investigated nano-ink No. 2 offers the excellent adhesion on polyimide substrate Kapton HN already at temperature 200 °C. In the case of the nano-ink No. 1, for achieving the excellent adhesion on polyimide substrate Kapton HN, there is a necessity of sintering at the temperature 220 °C for 60 min. The nano-ink No. 1 is more suitable for the substrate PET Mylar, in which the adhesion classified as 1/4B was achieved at temperature 180 °C.

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