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Edition 1.0 2021-09

TECHNICAL REPORT

Printed electronics –
Part 402-4: Printability – Measurement of qualities – Classification and measurement methods for morphology

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colour inside

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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FOREWORD

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IEC TR 62899-402-4 has been prepared by IEC technical committee 119: Printed Electronics. It is a Technical Report.

The text of this Technical Report is based on the following documents:

Draft	Report on voting
119/300/DTR	119/357/RVDTR

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

A list of all parts in the IEC 62899 series, published under the general title *Printed electronics*, can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

INTRODUCTION

The IEC 62899-402 series specifies basic measurement methods for printed patterns prepared using printed electronics technology. An overview of the documents in the IEC 62899-402 series is given in IEC 62899-401.

Since the surface morphologies of printed patterns strongly affect the electrical properties of patterns as well as the printing process such as overlay printing onto the patterns, IEC TC 119 plans to prepare other documents to measure the vertical variance of printed patterns, such as the future IEC 62899-402-5 which deals with "'surface roughness", the future IEC 62899-402-6 which deals with "thickness" and the future IEC 62899-402-7 which deals with "surface profile"'. These future documents were designed based on assumptions from classical technologies such as photolithography. However, the cross section of patterns by photolithography has usually a trapezoidal or rectangular shape, and the surface of the patterns is always flat and smooth. In contrast, the actual surface of the printed pattern has various morphologies, because the characteristic surface morphologies are formed by the various printing technologies used in printed electronics, and factors that cannot be controlled perfectly are included in the process of forming the surface. Reflecting those features, the range of variance to be measured in the area of printed electronics becomes very broad, and various measurement methods are used in those measurements. In order to prepare the subsequent documents, the current measurement methods should be reviewed in a technical report. This review will clarify the relation between the morphologies and the appropriate measurement methods.

According to the complicated surface morphologies, it is not easy to specify the measuring point on the surface. This problem will also be reviewed in this document by organizing the definitions of morphologies.

PRINTED ELECTRONICS –

Part 402-4: Printability – Measurement of qualities – Classification and measurement methods for morphology

1 Scope

This part of IEC 62899-402, which is a Technical Report, is a preparatory work for the documents dealing with the measurement method of the vertical direction (surface forms) of printed patterns made by printed electronics technology.

The printed pattern of interest in this document is limited to straight lines on substrates with a flat surface. This document focuses on the classification and measurement methods for surface forms from the nanometer scale to the micrometer scale, and suggests the strategy for the subsequent documents.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
pattern profile
characteristics of the cross sectional form in the width direction

Note 1 to entry: Three shapes, ie trapezoid, arch and side horn are proposed as typical shapes (see Figure 1).

Figure 1 – Typical pattern profile

Note 2 to entry: The side horn shape is called coffee stain in the academic area. However side horn is used in this document.

3.2
micro surface roughness of the printed pattern
amplitude and short-wavelength (less than 10 µm) component of the surface

3.3
thickness of the printed pattern
improved thickness
maximum distance between the top surface and bottom side in the width direction of the printed pattern

3.4
pattern roughness in the line direction
variation in thickness in the line direction

4 Classification of surface forms for future standardization work

Surface forms can be classified as shown in Figure 2. "Pattern micro roughness" expresses the finest unevenness (less than 5 µm) on the surface of the printed lines. "Pattern profile" includes a larger unevenness (from 5 µm to 200 µm). The area of "Pattern roughness" (or waviness) overlaps "Pattern profile", but this shows the waviness with a long range frequency and can be separated from the fine unevenness. "Pattern thickness" can be based on a different concept than the above three parameters, but it is also added to Figure 2 since it has a deep relation with these parameters.

Standards on the measurements for the vertical direction can be prepared according to this classification.

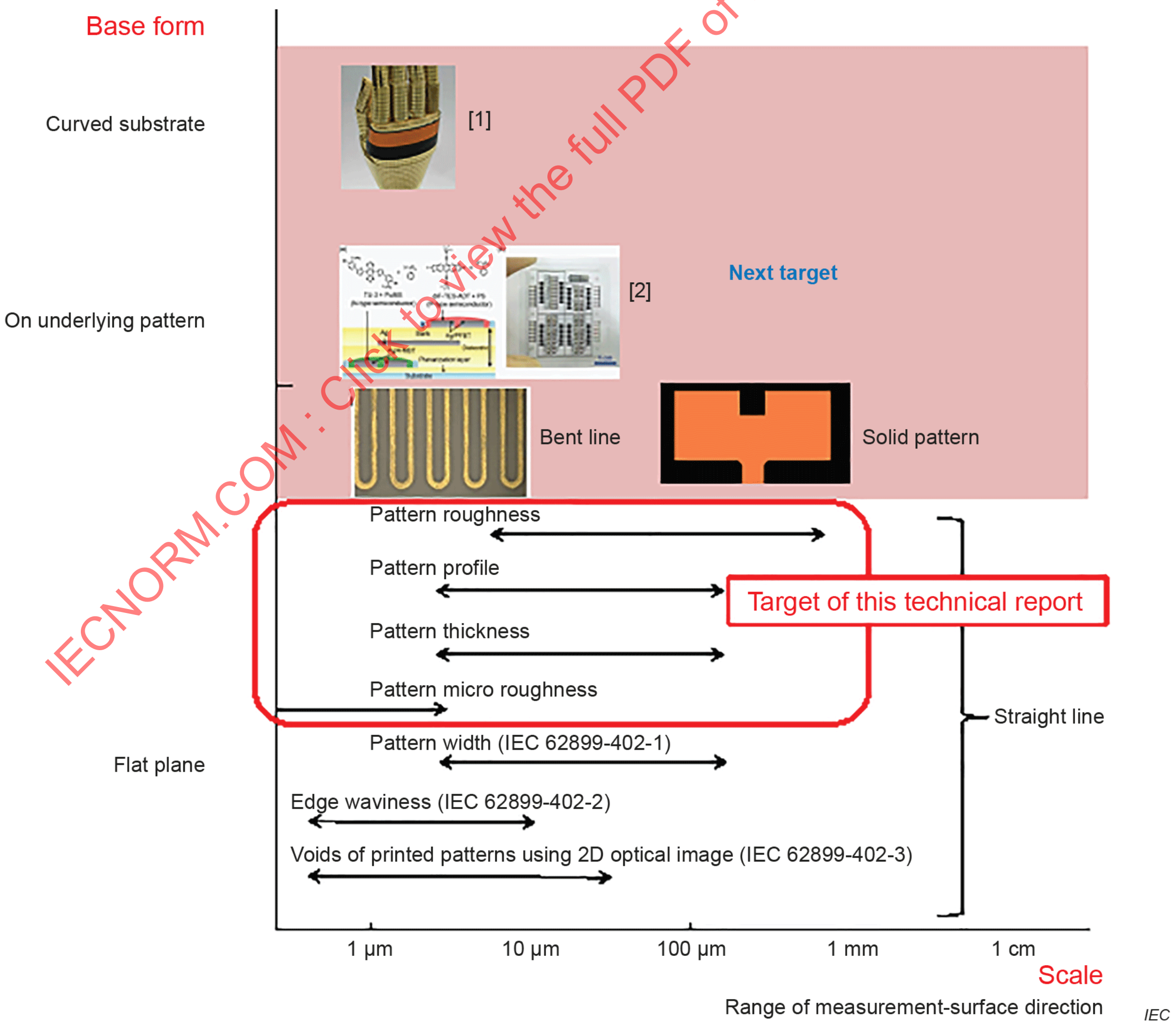


Figure 2 – Classification of parameters and future standardization work in TC 119

5 ISO documents concerning surface roughness

ISO/TC 213 (Dimensional and geometrical product specifications and verification) is a TC that handles product dimensions and tolerances. Since the surface roughness is one of the factors that affects them, ISO/TC 213 has prepared many standards related to the surface roughness such as data processing, definitions of terms (see Figure 3). However, it is not possible to find a document for the measurement method that can be directly used in the printed electronics industry. The summary of the ISO standards is listed in Table 1.

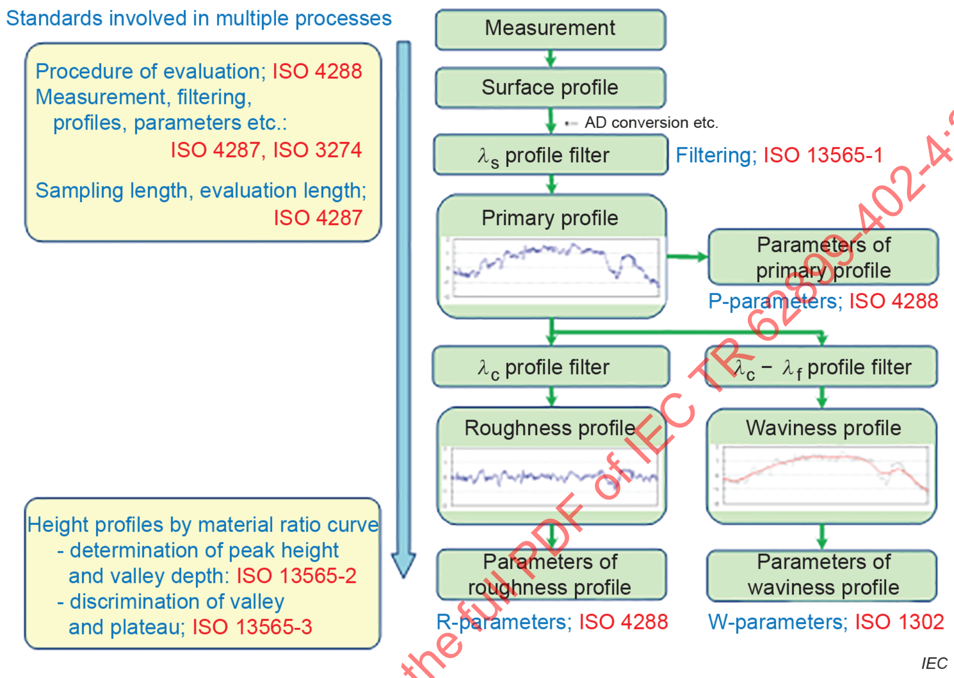


Figure 3 – Flow of data processing and related standards

Table 1 – The ISO standards related to the surface roughness

Document No	Title	Brief summary
ISO 1302 [8]	Geometrical Product Specifications (GPS) – Indication of surface texture in technical product documentation	This document specifies the rules for the indication of surface texture in technical product documentation. The parameters of waviness profile are described.
ISO 3274 [9]	Geometrical Product Specifications (GPS) – Surface texture: Profile method – Nominal characteristics of contact (stylus) instruments	Describes profiles and the general structure of contact (stylus) instruments for measuring surface roughness and waviness.
ISO 4287 [10]	Geometrical Product Specifications (GPS) – Surface texture: Profile method – Terms, definitions and surface texture parameters	Surface profile parameters such as peak, valley, curves and related parameters are defined.
ISO 4288 [11]	Geometrical Product Specifications (GPS) – Surface texture: Profile method – Rules and procedures for the assessment of surface texture	The rules of basic procedure on evaluation are described, and this also gives rules for measuring roughness profile parameters by using stylus instruments according to ISO 3274.
ISO 13565-1 [12]	Geometrical Product Specifications (GPS) – Surface texture: Profile method; Surfaces having stratified functional properties – Part 1: Filtering and general measurement conditions	Describes a filtering method. This filtering method suppresses the valley influence on the reference line such that a more satisfactory line is generated. The method will be effective when the surfaces have a relatively small amount of waviness.

Document No	Title	Brief summary
ISO 13565-2 [13]	Geometrical Product Specifications (GPS) – Surface texture: Profile method; Surfaces having stratified functional properties – Part 2: Height characterization using the linear material ratio curve	This document contains the determination of peak height and valley depth and the evaluation process.
ISO 13565-3 [14]	Geometrical Product Specifications (GPS) – Surface texture: Profile method; Surfaces having stratified functional properties – Part 3: Height characterization using the material probability curve	This part of ISO 13565 provides a numerical characterization of surfaces consisting of two vertical random components, namely, a relatively coarse "valley" texture and a finer "plateau" texture.

6 Examples of measurement

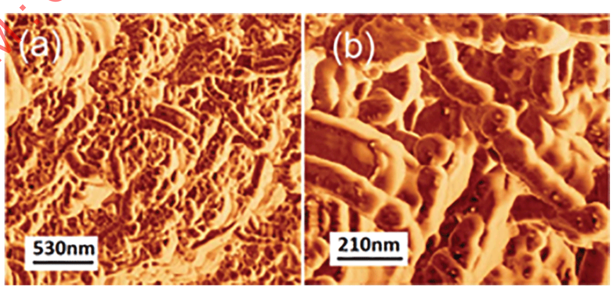
6.1 Micro surface roughness of the printed pattern

6.1.1 AFM approach

AFM (atomic force microscope) is one of the tools that are able to quantitatively measure the surface roughness. This measurement can be applied to the even surface of the printed pattern, and preferably applied to measure the small unevenness caused by particles since it has a resolution of 5 nm to 10 nm laterally and sub-nanometer vertically. The measurement area of AFM is generally 1 μm^2 to 10 μm^2.

Examples of the AFM measurement can easily be found, but there are not so many cases applied to the surface of printed layers.

AFM analysis on the printed pattern is reported by Menon et al [3][1]. The pattern was prepared by the screen printing and carbon nanotube (CNT) ink (multiwalled carbon nanotubes (MWCNTs) are used as CNT). Intertwine between MWCNTs is quite important since it greatly affects the conductivity and other properties. The resolution of AFM measurement is appropriate for the detection of the diameter and length of CNT and is suitable for evaluating intertwine. (see Figure 4). The RMS of surface roughness is obtained in this document. However, the detail of the relationship between the surface roughness and conductivity is not mentioned.



SOURCE: See [3].

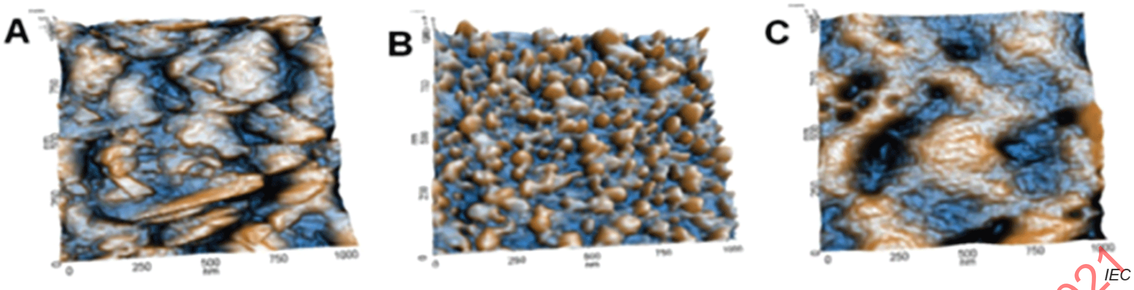
Figure 4 – AFM image of MWCNT printed pattern

The R-parameters on the printed carbon electrode are reported by Wongkaew et al. [4]

Immobilization of a bio-recognition element on the surface of a functional working electrode (screen-printed carbon electrode) is fundamental for effective biosensor development. The surface morphology of the working electrode is remarkably changed by enzyme immobilization.

[1] Numbers in square brackets refer to the Bibliography.

This change can be detected quantitatively by the AFM measurement (see Figure 5 and Figure 6). The R-parameters reported in this document are listed in Table 3.



(A) bare screen printed carbon spot area of a screen printed carbon electrode (SPCE);

(B) chitosan modified screen printed carbon spot;

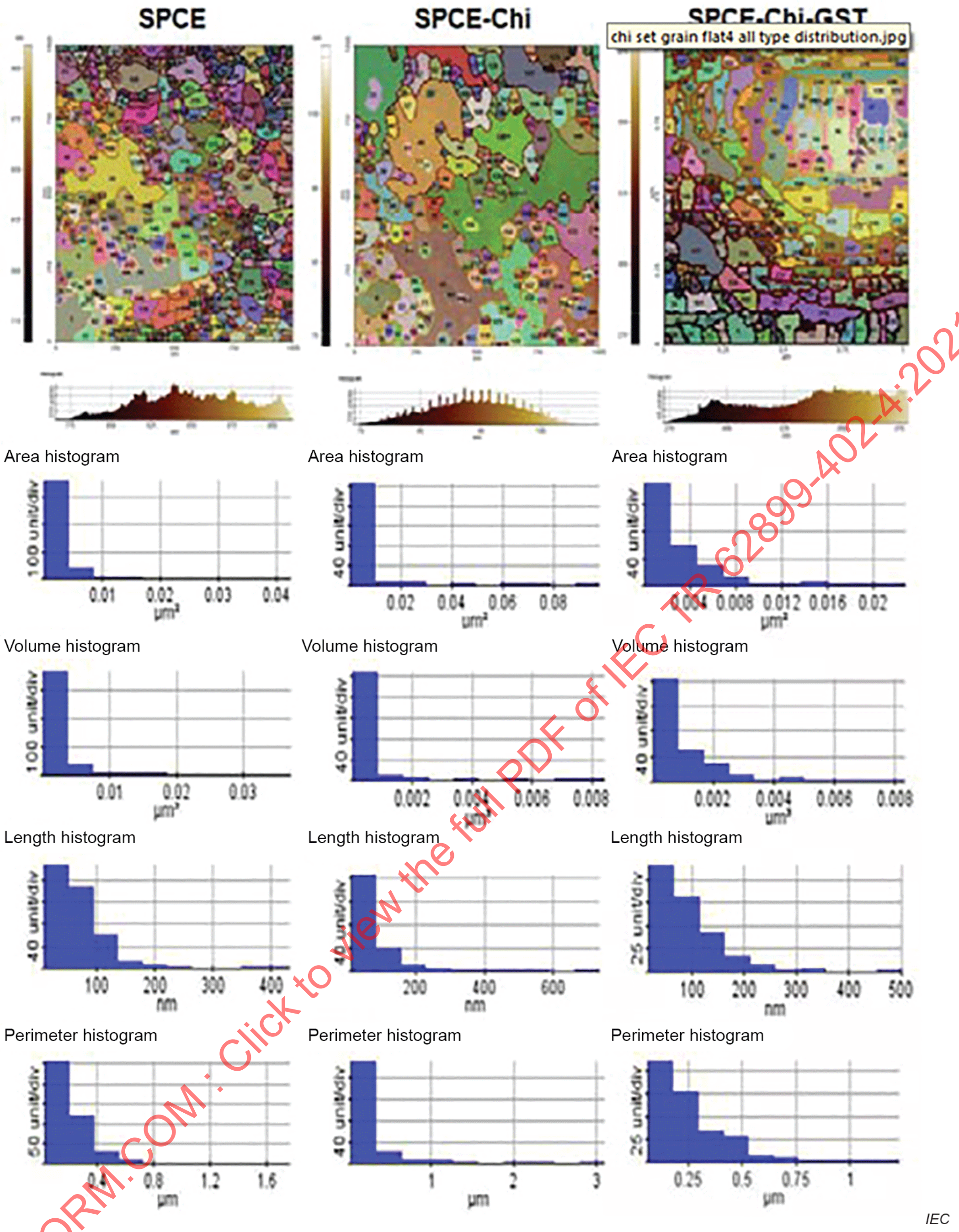
(C) chitosan modified screen printed carbon spot after immobilization with glutathione-s-transferase.

Surface grain structure estimation on a working platform of bare SPCE, SPCE-Chi (chitosan modified screen printed carbon electrode), and SPCE-Chi-GST (GST = glutathione-s-transferase), at 1 µm x 1 µm AFM scan size is described in Table 2.

Figure 5 – Three-dimensional AFM nanoscale surface images and parameters

Table 2 – Surface grain structure estimation on a working platform

Parameters		Bare SPCE	SPCE-Chi	SPCE-Chi-GST
Area (nm^2)	Mean	2,215	4,185	3,165
	Std.	3,385	1,029	3,792
Volume (nm^3)	Mean	1,882	0,037	1,063
	Std.	2,943	0,089	1,307
Length (nm)	Mean	67,544	83,854	89,109
	Std.	43,259	82,999	64,534
Perimeter (nm)	Mean	205	273	252
	Std.	155	335	179
Rpv (nm)	Mean	10,212	6,942	5,683
	Std.	5,995	3,164	3,777
NOTE Rpv = peak-to-valley, Std = root mean square or standard deviation				



SOURCE: See [4]

Figure 6 – Surface image of printed electrode and sensor and grain size distribution histogram on working surface of SPCE, SPCE-Chi, and SPCE-Chi-GST at 1 µm x 1 µm AFM scan size

Table 3 – R-parameters exploration from AFM micrographs at 1 µm x1 µm scan size

Analysis parameters	SPCE	SPCE-Chi	SPCE-Chi-GST
Ra (nm)	18,69	21,74	26,72
Rq (nm)	24,49	25,15	33,17
Rpv (nm)	157,23	111,62	143,38
Rz (nm)	155,24	108,88	142,81
Rsk	-0,19	-0,18	0,59
Rku	3,32	1,96	2,67
Rsm (µm)	0,39	0,11	0,09
Fractal (triangular)	2,14	2,27	2,02

NOTE *Ra* = roughness average, *Rq* = root mean square roughness or standard deviation of the height value, *Rpv* = peak-to-valley, *Rz* = ten point height, *Rsk* = skewness, *Rku* = kurtosis, *Rsm* = mean spacing average.

6.1.2 Shape of the droplet on substrates using inkjet printing

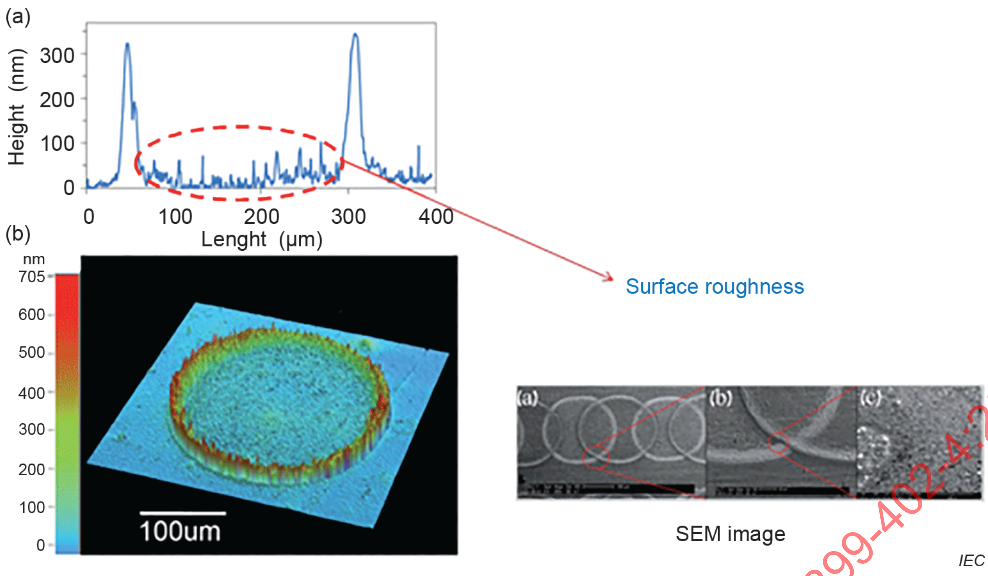
Everyone knows that when coffee falls and dries it makes circular marks. That mark is known as "coffee stains" or "coffee rings." Even in inkjet printing used in PE technology, the ink may dry to the similar shape depending on the conditions. In the transparent electrode, the conductive layer may be intentionally formed in this shape for the purpose of improving transparency, etc. From the electrical perspective, the contact of rings is important. The current may flow along the edges of the rings. In other words, it is recommended to always be aware of the cross-sectional appearance, and the ring shape should be called the "side horns".

The example of the side horns is shown in Figure 7.

The profile shown in the upper side of Figure 7 is measured by a mechanical profilometer (stylus type thickness instrument) and the image shown in the lower side of Figure 7 is obtained from an optical profilometer (confocal microscope).

In the printed layer having the side horn shape, there are difficult problems such as where to consider the surface of the printed layer and where to determine the thickness of the printed layer. From the appearance of the cross section, the plateau area might be specified to the surface and the thickness of the printing layer to be the thickness of the plateau area. Considering the electrical influence, however, it seems that the parts of horns are largely influenced and the volume of these parts cannot be ignored. In this document, the height and width of the horns are described, and it is mentioned that the connection of these parts functions as an electrode.

When the measurement methods of surface roughness are standardized, it is necessary to decide which area to be measured firstly in this type.



SOURCE: See [5].

Figure 7 – Example of "side horns"

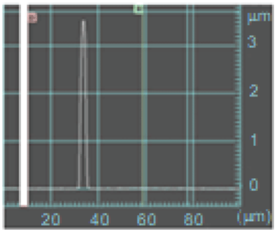
6.2 Thickness of the printed pattern (improved thickness)

6.2.1 Measurement of trapezoid and semi ellipse

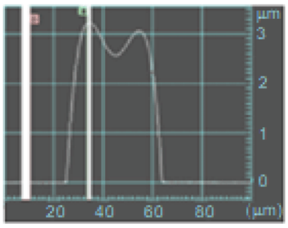
When a printed line has an appearance of cross-section classified to trapezoid or semi ellipse type, the thickness of the line would be easily defined. In the case of the semi elliptical type, it might not be so easy to define the surface, but there would not be various opinions about the thickness of the line.

In many cases, the thickness of the line classified to these types can be measured by stylus type equipment.

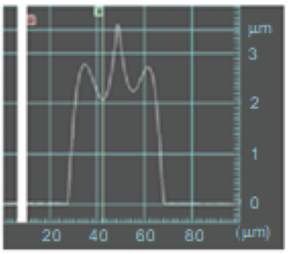
There are many reports on these types, but a typical example is shown in Figure 8 [6]. This is an assistant electrode of transparent film, and made from Ag using gravure offset printing. The thickness of the line (electrode) is measured by the stylus type thickness instrument. The appearance of the line is a typical semi ellipse type, and it would be reasonable to think that the height of the semi-elliptical shape is the thickness of the line.



(a) Assistant Ag electrode
3,54 µm



(b) Insulating paattern
3,2 µm



(c) Assistant Ag electrode + insulating paattern
3,57 µm

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SOURCE: See [6].

Figure 8 – Electrode formed by gravure offset printing

6.2.2 Measurement of side horn

The issue mentioned here is already described in 6.1.2. The thickness of the side horn type can also be measured by the mechanical profilometer or optical profilometer. However, what is more important is where to define the thickness. There may be several options to define the thickness.

For example;

a) the average thickness of the plateau between the horns,

b) the maximum height of the horns themselves, and

c) the average height calculated from the area of the cross section.

In the example described in 6.1.2, since the transparent electrode is the application and the plateau area is made thinner, b) would be a reasonable decision even when considering the electrical aspect. However, c) may be appropriate when the plateau area becomes thicker and the current flowing in that area cannot be ignored. In any case, subsequent documents on vertical variance should firstly determine those definitions.

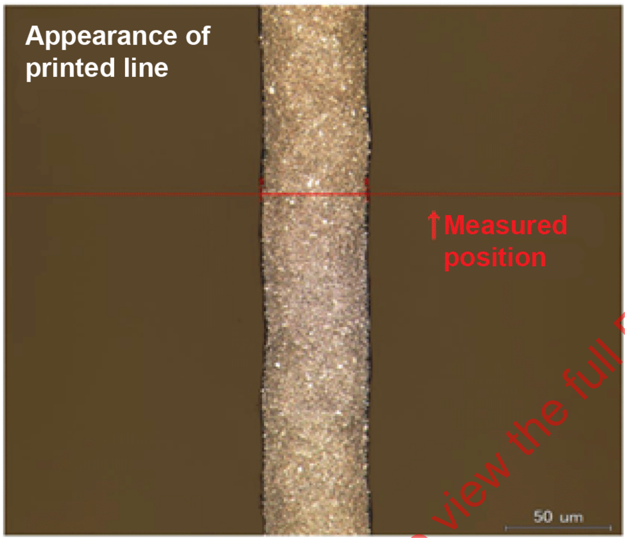
6.3 Roughness for the printed conductive line

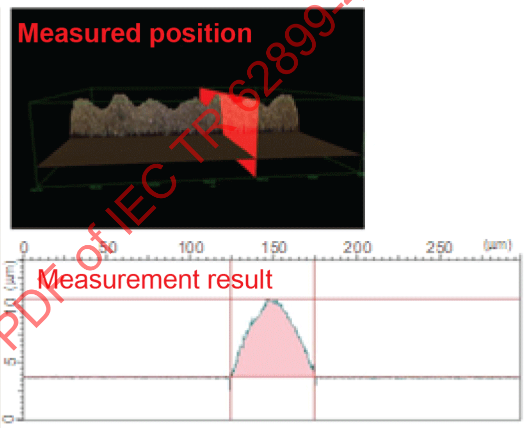
The roughness such as Ra is very important particularly for multilayered patterns. However, when one focuses on the conductivity of the conductive patterns, the cross-sectional area should be paid attention to since the conductivity is dependent on the area. Although, from the point of view of the smoothness of the surface of printed lines, the maximum height and dent in a specified section may be an indicator of conductivity, the conductivity is also affected by the variation of the line width, and therefore, the quality of the printed line related to the conductivity should be evaluated by mean of a cross-sectional area for a specified section of the printed line. Figure 9 and Figure 10 [7] illustrate this problem.

The printed lines shown in Figure 9 and Figure 10 were prepared by using screen printing, and a confocal microscope was used in the analysis. In Figure 9, the target cross section is made on the red line, and the cross-sectional area is obtained at the red plane in the right side picture.

The analysis shows the area of 210,791 µm^2 with the line having a height of 6,662 µm and a width of 50,263 µm. In contrast, the red section for the same printed line is analysed in Figure 10. The mean cross-sectional area of this figure becomes 182,062 µm^2 (the line height is 5,040 µm, the width is 35,124 µm.). Unfortunately, this document does not discuss the relationship between the surface roughness and cross-sectional area in terms of conductivity, despite the fact that periodic unevenness due to the mesh of the screen printing can be found on the printed lines. However, there is a 32 % difference between the height in Figure 9 and in Figure 10, there is only a 15,7 % difference in cross-sectional area in them. This should become a clear difference in the evaluation of conductivity.

It will be possible to specify the measurement methods of surface roughness in the subsequent parts of this series. Those methods would be good enough to evaluate the appearances of printed lines. However, the methods may not be sufficient for measuring the quality of printed lines, which is a case that directly affects conductivity. Further discussion will be necessary from various perspectives other than simply knowing the surface roughness.





Height (µm)	Width (µm)	Area (µm^2)
6,662	50,263	210,791

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Figure 9 – An example of roughness in the cross-section in the width direction of a printed line