

Characterization of flexo and letterpress printing plate's surface roughness by indirect SEM image-based profilometry

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In this paper indirect, image-based approach to the characterization of surface structure and roughness of flexographic and letterpress printing plates was investigated. Our previous research have shown that direct stylus profilometric method, when used in flexographic and letterpress printing plates surface roughness characterization, resulted in certain usage difficulties. Being a polymeric structure, surface of plates was scratched by stylus diamond tip and the measuring device has shown alt and slowdown in performance. The solution of such a problem lies in indirect and non-contact profilometry approach. According to literature findings SEM micrographs can be used for non-contact surface topography characterization, since they are excellent tool for visualization and qualitative description of surface topography. The indirect approach was based on use of Gwyddion software functions for analysis of SEM images and calculation of standard profilometric parameters. The results of the study have shown that it is possible to obtain profilometric parameters from analysis of SEM micrographs.

The study also involved analysis of influence of different micrograph magnification on final surface roughness results. It is shown that, with appropriately calibrated grayscale intensity distributions, optimal agreement with expected R_a value was achieved using indirect profilometric method. The statistical analysis showed that magnification level had no significant influence on obtained results of R_a parameter (based on p value of 0.05). Its influence was more expressed if the point of interest was shifted toward more specific roughness characteristics like peaks and valleys. Overall, the results indicated that proposed indirect image-based profilometry is a useful tool in the characterization of surface's topographies of flexo and letterpress printing plates.

Key words: surface roughness, direct and indirect profilometric method, flexo printing plates, letterpress printing plates

INTRODUCTION

Surface roughness parameters of the materials are frequently used in many engineering industries as the parameters which clearly depict their surface structure and surface characteristics [1-3]. The surface properties of particular material are defined by its chemical composition and morphology, as well. But, since surface roughness and surface topography often greatly define its functional properties, such as mechanical function, wear, lubrication and appearance, they should not be underestimated and should be precisely determined and characterized [2,3]. In flexography and letterpress printing, printing plates are one of the most influencing on overall printing process. Surface topography of the printing plate highly influence ink transfer during printing process, hence final imprint quality [1,4-9]. Previous research has shown that surface roughness of the printing plate is even more significant than the surface energy when considering print quality [1,10].

There are many methods successfully proposed for the analysis and description of surface roughness and surface topography, where the obtained surface roughness parameters mostly depend upon characteristics of necessary instrument, its settings and data post processing [1,7,9,11]. Well known and widely used surface roughness analyzing methods are imaging (i.e. SEM - scanning electron microscopy or AFM - atomic force microscopy) and profilometric methods (i.e. MSP - mechanical stylus profilometry or non-contact laser profilometry) [1,2,7-9,12,13].

If the primary research goal is visualization of surface topography, then it is recommended to use one of the imaging methods. If the quantitative topographical information is needed, in terms of different surface roughness parameters, than it is more suitable to consider one of the various profilometric methods, contact or non-contact ones [2,14]. The advantage of non-contact ones is in avoiding the potential damages on the specimen's surface associated with the contacting stylus [14]. Namely, in the contact profilometry, the measuring

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unit is equipped with sharp diamond tip mounted on a console which moves along a line on the specimen, measuring directly the surface irregularities, videlicet peaks and valleys. In order to achieve high precision in surface roughness characterization, it is advisable to record a several test lines [1,7,9,12,15]. The average uncertainty in the direct profilometric surface roughness parameters evaluation is found to be up to 6.5% [2].

Even if there is a wide range of parameters which can be used in surface roughness characterization, still the most commonly used ones are amplitude ISO roughness parameters (ISO 4287:1997 and ISO 12218:1997): R_a (average surface roughness), R_q (R_{ms} , root-mean-square deviation), R_{zDIN} (mean value of the single roughness depths Z_i), R_p (leveling depth) and R_v (maximum depth of profile valley) [1,7,9,13,14,16-18]. In more specific applications, such as characterization of surfaces with asymmetric roughness, these parameters cannot successfully describe surface irregularity or complexity. Combined with the metrological limitation of used method, such is in stylus profilometry the limitation to reproduce smaller details [2], it is inevitable to resort to another approach in surface roughness characterization, like the concept of fractals. Estimation of surface roughness via fractals is based on SEM or AFM micrographs, and according to findings, it is well correlated to the profilometric parameters. More detailed description and advantages of this particular method are given in [2,13,15].

Another promising approach in surface roughness characterization is software-based extraction of profilometric parameters from SEM or AFM recordings of specimen surface. It seems to be a promising tool, gaining more attention with development of free and open source software for Scanning Probe Microscopy (SPM). This method is based on spatial grayscale intensity distributions

analysis of SEM/AFM micrographs and direct calculation of amplitude ISO roughness parameters. This approach was found to be utilized in surface roughness characterization of wide range of substrates: silicate glasses [19], graphene [20], ZnO thin films [21,22], CL optic surface [23], membranes [24], siliconized cellulosic substrates [25] as well as offset printing plates [2].

In this context, we aimed through this study to present a studious comparison of two basically different approaches to the surface roughness characterization of printing area on polymeric flexo and letterpress printing plates. The research encompassed detailed analysis of surface roughness parameters obtained from indirect SEM image-based profilometry against those obtained with the standard, direct stylus profilometric method. Through the evaluation of the performances and usefulness of indirect, image-based method in the characterization of surface topography of flexographic and letterpress printing plates, we have tried to set a stepping stone towards standardizing the image analysis technique in printing plate's surface roughness characterization.

EXPERIMENTAL

Materials

In this paper we have used three type of specimens: two conventional, solvent-washable photopolymer flexographic printing plates (Nyloflex FAH 2.84 mm and Nyloflex FAR 2.84 mm) and one water-washable photopolymer letterpress printing plate (Nyloprint WF-F 0.88). All specimens were prepared by UVA radiation exposure, in order to develop proper hardness of printing elements, followed by conventional developing process, solvent or water based, helped by brushing. [26,27]. The processing parameters used were in compliance with manufacturer's recommendation [28-30] and are given in Table 1.

Table 1. Plate processing parameters

| Printing plates | Back exposure [s] | Main exposure [min] | Washout speed [mm/min] | Washout time [min] and temperature [°C] | Drying time [min] | Drying temperature [°C] | Post exposure UV-A [min] | Light finishing UV-C [min] |
|------------------|-------------------|---------------------|------------------------|---|-------------------|-------------------------|--------------------------|----------------------------|
| Plate I (FAH) | 120 | 24 | 160 | / | 180 | 60 | 10 | 15 |
| Plate II (FAR) | 80 | 24 | 160 | / | 180 | 60 | 10 | 15 |
| Plate III (WF-F) | / | 4 | / | 4 (28) | 15 | 60 | 3 | / |

Surface roughness analysis

As we have mentioned before, the main scope of this paper was in-depth analysis of possibility to use the indirect SEM image-based profilometry in surface roughness characterization of flexographic and letterpress printing plate.

Albeit, there are many roughness parameters which can be used for the surface characterization, in this investigation the four most common amplitude ISO roughness parameters have been used, namely R_a , R_q (R_{ms}), R_p and R_v . These parameters are compliant to the geometric product specification standards (ISO4287:1997 and ISO 12218:1997) and they are the most suitable for the aims of this study [7,12,16]. The R_a and R_q parameters are two amplitude-averaging quantities, which are the most widely used for the industrial applications, while R_p and R_v are two peak-valley parameters, optimal to quantify the importance of extreme peaks and valleys on the surface [14,16].

Thus, surface roughness parameters [1,7, 2,14,16-18] were as follows:

a) R_a , average surface roughness, defined as the average deviation of the surface profile from the mean line, geometrically represented as a total ruled area divided by the evaluation (sampling) length l , and analytically given with the expression:

$$R_a = \frac{1}{l} \int_0^l |y(x)| dx \quad (1)$$

b) R_q (R_{ms}), root-mean-square deviation or the rms roughness, represents the square root of the arithmetic mean of the squares of profile deviation from mean within sampling length, mathematically described as:

$$R_q = \sqrt{\frac{1}{l} \int_0^l y^2(x) dx} \quad (2)$$

c) R_p , leveling depth, is the distance between the highest peak and the reference line (Fig. 1),

d) R_v , maximum depth of profile valley, measured below the reference line (Fig.1).

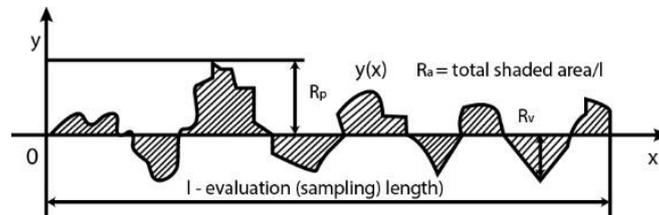


Figure 1. Surface roughness parameters

Direct profilometric surface roughness measurements. For the purpose of direct profilometric measurements, the portable surface roughness tester TR 200 (Micro Photonics, Inc.) was used. It is provided with a 2 mm radius diamond tip. The unit is compatible with ISO 4287, DIN 4768, ANSI B 46.1 and JIS B601 standards [31]. The device measurement parameters were as

presented in Table 2. Measurements of the roughness parameters were carried out on the solid printing area of each specimen by 18 different measuring lines, 9 per in two principal directions, printing and cross printing direction, in order to avoid possible variations caused by the measuring direction.

Table 2. Device measuring parameters

| Filtering method | Measuring range | Resolution | Standard | Traversing speed | Cut-off |
|------------------|-----------------|------------|-------------------|------------------|---------|
| Gauss filtering | ± 20 μm | 0.01 μm | ISO 4287 standard | 0.135mm/s | 0.80 mm |

Indirect surface roughness measurements

The indirect determination of the roughness parameters was done based on the analysis of the SEM micrographs in Gwyddion v.2.38 software. It is free and open source software for SPM data visualization and analysis, supported by the Czech Metrology Institute [32-35]. It is rather versatile software, which is not only limited to surface topography characterization. Gwyddion can be successfully used for particle size measurements [36-38], particle automatic counting and covering

area calculation [39,40] as well as line profiling and 3D imaging [32,34,35]. The SEM micrographs were captured on JEOL JSM 6460 LV Scanning Electron Microscope. Before imaging, the samples were gold-coated (15.0 nm thick and 19.32 g/cm³ dense layer of gold) in order to enable uniform electrical properties of the surface. For the experiment purposes, the specimens were imaged at three different tilt angles and four different magnification values. The SEM recording parameters are given in Table 3.

Table 3. SEM recording parameters

| Working distance | Voltage | Tilt angle | Magnification |
|------------------|---------|------------|--------------------------|
| 15 mm | 20 kV | 0°, +/- 5° | 600x; 800x; 1000x; 1500x |

Previous research [2, 24] has shown that the precision and certainty of image-based profilometry is highly dependable on several parameters including pixel size, number of pixels, working distance, rotational and tilt angles, but far the most is the intensity or height calibration [2]. The image-based profilometry relies on the analysis of the spatial distributions of intensities in grayscale SEM image whereas the bright areas on the image represent the elevated (peaks) while dark areas represent the depressed areas (valleys) of the imaged surface. Definition of surface roughness follows from the assumption that the intensity in a particular pixel is proportional to its elevation [2]. Thus, the scaling and calibration of SEM images with respect to their intensities are essential for the lowest result uncertainty.

For the scaling procedure in Gwyddion software, three user defined input values are needed: length (x), width (y) and depth (z) of an SEM micrograph, expressed in micrometers. Appropriate scaling of the x and y dimensions of an image is ensured according to the scaling mark on the image (usually a white line), of a known length in micrometers as well as in pixels. The determination of z-value (depth), the maximum roughness value which Gwyddion will calculate [24] is accomplished according to the procedure established for quantitative analysis of SEM images given in [2,41]. We have selected this particular methodology, since the alternative one proposed in [24] based on the effective penetration depth of

secondary electron beam needed to produce the SEM images, directly influence the large discrepancies between the surface roughness values measured experimentally using the AFM profilometer and those quantified using Gwyddion. Conversion of the grayscale intensity (0–255) to the corresponding height scale (z) was accomplished by using the images recorded at three different tilt angles (0°, ±5°), defining the corresponding coordinates of black and white points on the images through the image matrix, calculating the distances between those points and defining z factor as the median value of determined distances [2,41]. Since the black level in the images was not 0 but higher, we have used corrected calibration factor given by:

$$z_{cor} = z * 255 / (255 - x) \quad (3)$$

where x is the median value of black levels of points used in calculation.

On the appropriately scaled SEM micrographs, further analysis of surface roughness was conducted using software build-in function ISO tool. This tool provides a roughness analysis along the straight-line arbitrarily drawn by the means of cursor over the imported SEM micrograph. Roughness profile (Fig. 2), derived as a one-dimensional texture profile along the cursor line, compounds of high frequency/short wavelength component – roughness and low frequency/long wavelength component – waviness, videlicet form of the profile [2,35,42].

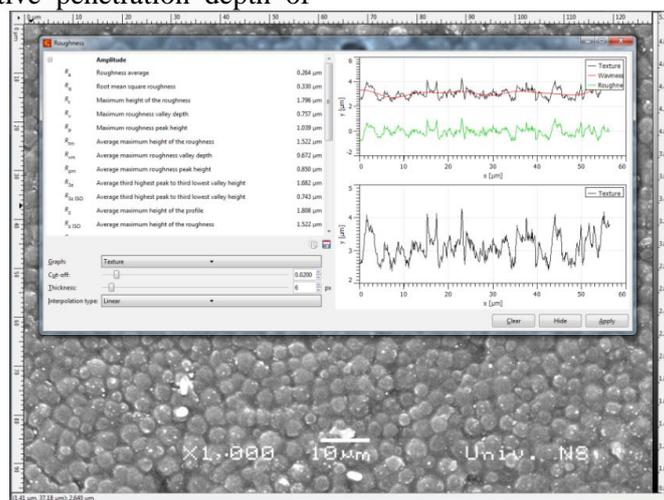


Figure 2. Roughness profile of a particular surface presented in Gwyddion.

Cut-off frequency, thickness value and interpolation type are supposed to be preselected. They are influencing factors on final surface roughness parameters values as well.

The cut-off frequency is specified in the units of the Nyquist frequency and is to be set according to ISO 4287 standard, at 1/5 scan length [2].

Thickness value is in direct proportion to the quantity of data used in the evaluation of one profile point – the higher the value the more neighboring data perpendicular to the profile direction is used in the evaluation [2,43].

Concerning interpolation type, linear interpolation was found as optimal [2,24,35] where

the interpolated value of the point was calculated from the three vertices of the Delaunay triangulation containing that point.

Input x and y values were 213.33 μm and 160 μm , 160 μm and 120 μm , 128 μm and 96 μm , 84.21 μm and 63.16 μm for micrographs imaged at magnification level of 600x, 800x, 1000x and 1500x, respectively. Input z value was 0.23 μm , 0.12 μm and 0.02 μm for micrographs of Plate I, Plate II and Plate III, respectively.

We have used thickness value of 6 px and linear interpolation. Cut-off frequency was set to 0.04 for images magnified 600x and 800x, and 0.02 for images of 1000x and 1500x magnification value.

The profilometric parameters were determined from SEM images recorded at zero tilt, along eight straight horizontal and eight straight vertical lines, each 60 μm long, avoiding apparent fallacies on image, resulting from residual dust or other impurities on specimen surface.

The values of each profilometric parameter used for the graph generation in Results and discussion section represent the average value obtained from the measurements with corresponding standard deviations.

RESULTS AND DISCUSSION

SEM micrographs presenting surface topographies of used printing plates taken with magnification of 1000x and corresponding 3D

surface plot, generated in Gwyddion via built-in function for converting 2D captured images into 3D maps of the surface, are given in Fig. 3 and Fig. 4, respectively.

As it was expected, plates' surface topography of flexography Plate I and Plate II is visually pretty similar and manifested as medium rough surface with clearly visible surface cracks. The white areas, as well as easily spotted larger bulges are residual impurities left behind after plates airbrush treatment. The surface of flexographic printing plate can be found to be still partially tacky even after UV-C light post treatment [26, 27] and consequently micro-impurities can easily remain on its surface. The letterpress printing plate (Plate III) has visually somewhat different surface topography with distinguished pores and grainy structure, therefore it was expected to have difference in surface roughness parameters values in comparison with the other two plates.

These imperfections or characteristic areas on the observed printing plates were allowing precise SEM imaging and later on a proper image calibration in Gwyddion. However they should be avoided during the image-based measuring process, since they can lead to misinterpretation of roughness parameters, due to false decrease or increase of the height parameters (shadow area of the impurity particle, the particle itself).

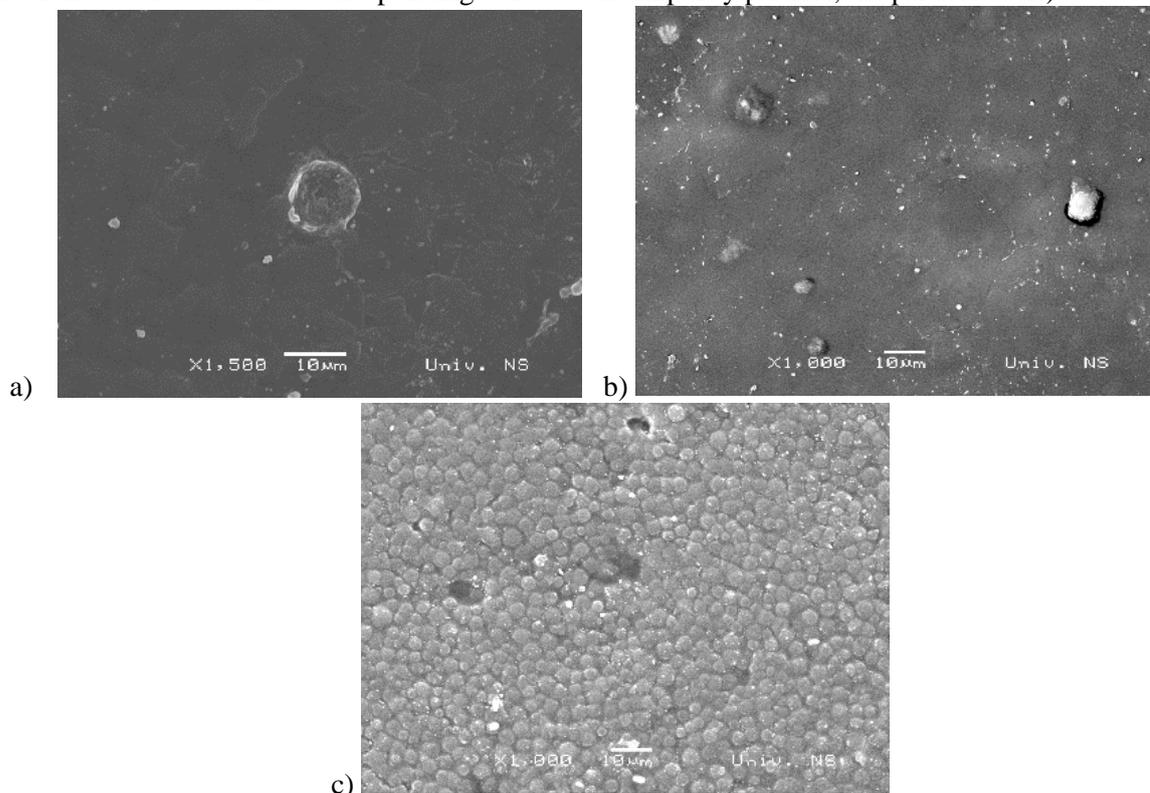


Figure 3. SEM images of printing plate' surface topography, magnification 1000x a) Plate I, b) Plate II, c) Plate III.

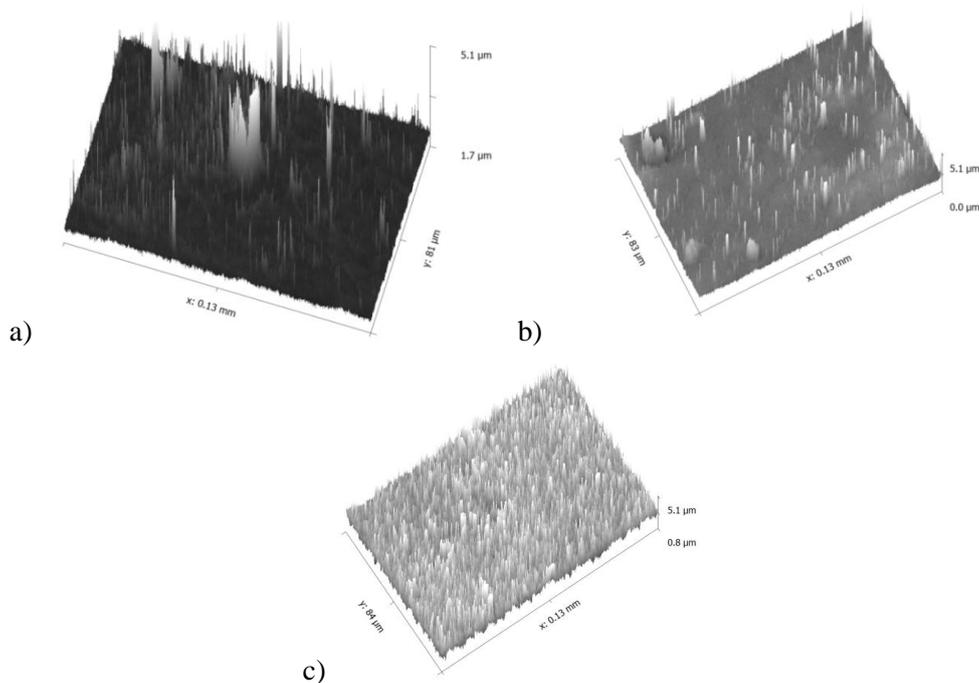


Figure 4. 3D surface plot of analyzed printing plates a) Plate I, b) Plate II, c) Plate III.

The 3D images (Fig. 4) represent three-dimensional reconstruction of surface topography providing better understanding of the surface imperfections and also displaying the visual comparison of flexographic and letterpress printing plates. The 3D surface plots, again, illustrate the similarity in a surface topography of two flexo plates, characterized by extremely high, sharp, needle-like pinnacles randomly distributed, which are actually mostly impurities on plate surface. Letterpress printing plate has more uniform structure with deeper valleys, not as sharp, but uniformly distributed peaks, without many extremes.

Direct determination of profilometric parameters

Direct profilometric measurements of three different polymeric printing plates resulted in a range of profilometric parameters' values (Fig. 5), providing a basis for a comparison with those obtained with indirect, image-based surface roughness analyzing method. The measured roughness profiles indicate a remarkable similarity in corresponding surface roughness parameters values for flexography printing plates, Plate I and Plate II. Letterpress printing plate, Plate III, has lower average surface roughness and lower maximum peak height but higher maximum valley depth. Decrease in overall surface roughness might be due to different polymeric composition as well as difference in applied processing parameters.

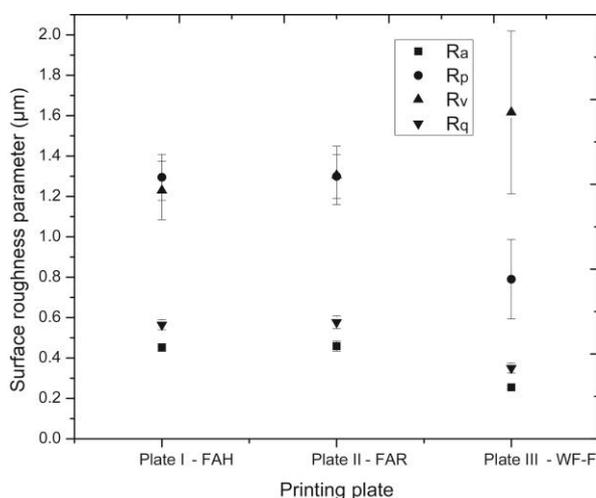


Figure 5. Surface roughness parameters of analyzed printing plates – direct profilometry.

During measuring process, we have found that the physical interaction of diamond tip of measuring device and polymeric surface of a printing plate can lead to scratching of the plate surface, interruption in measuring, consequently disable reading, prolong measuring process and potentially causing irretrievable damages on a plate surface.

Higher values of standard deviation can be observed for R_p and R_v roughness parameters (Fig. 5). But, it cannot be indicative of problems in the data or experiment disadvantages, considering the parameters nature as well as the fact that the coefficient of variation, as a measure of dispersion of data relative to the mean [44], is not greater than 30% [45].

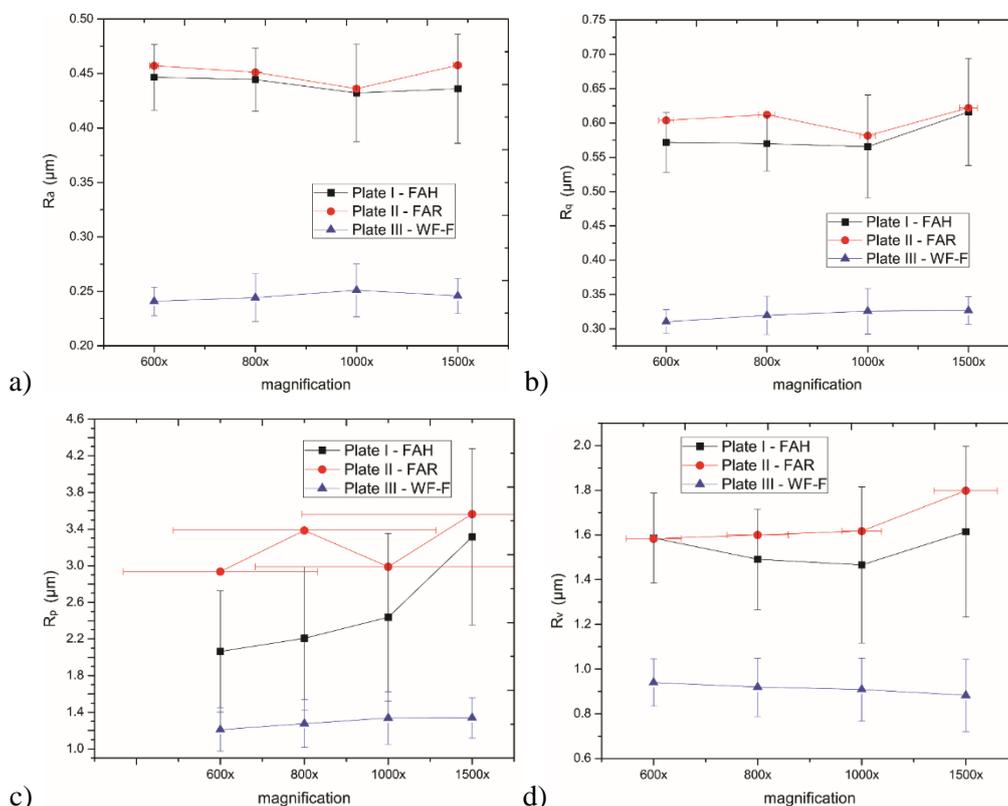


Figure 6. Surface roughness parameters of analyzed printing plates – indirect SEM image-based profilometric method
 a) R_a , b) R_q , c) R_p , d) R_v

Indirect determination of profilometric parameters

Mean values, with corresponding standard deviation, of surface roughness parameters R_a , R_q , R_p and R_v obtained by indirect, SEM image – based methodology are presented in Fig. 6.

The profilometric parameters, derived from SEM images obtained at zero tilt angle, are discussed in terms of magnification level used during imaging. Graphs presented indicate that with higher magnification level overall surface roughness (reflected through R_a and R_q parameter) is slightly changing and exhibit the similar trend for examined specimens. The values of roughness parameters which reflect the deepest valley and highest peak on the surface are much more influenced by the used magnification level during imaging.

This was expected, since the SEM image-based profilometric method is relying on the analysis of

the spatial distribution of gray intensities of an image whereas the gray intensity of each pixel is directly proportional to depressed (valleys: darker pixel, lower grayscale value) or elevated (peaks: lighter pixel, higher grayscale value) areas. The magnification level directly influences the gray intensity value of each pixel which represent the lowest valley (R_v) and highest peak (R_p) value, but the overall surface roughness will be slightly changed.

It is also noticed that standard deviation as well as corresponding coefficient of variation are higher with higher magnification level, which might indicate significant influence of magnification level on the measuring precision and stability as well as result accuracy. This is especially expressed in case of R_p and R_v surface roughness parameter. But again, this can be also partially attributed to methodology itself, as it was already explained, as

well as to the nature of these parameters (representing the highest and deepest point of the profile along measuring line, thus expecting to exhibit a greater variety in comparison to averaged surface roughness).

Statistical analysis of profilometric measurements

Deeper analysis of surface topography using SEM micrographs of the different magnification levels and their comparison with the corresponding parameters obtained from the measurements with TR 200 was done using statistical technique for testing the equality of means: one – way analysis of variance (one-way ANOVA).

Preliminary analyses of normal distribution and homogeneity of variance have shown that assumption of the homogeneity, according to Levene's test for equality of variances, was violated. Since the literature findings suggest that analysis of variance is reasonably robust to violations of this assumption, if the size of groups is reasonably similar [46] (in our case they are the same size of 18), we have believed that conclusions derived from one-way ANOVA tests are legitimate. According to advisable procedure we have used Dunnett's T3 post hoc test and Welsh and Brown-Forsythe test.

A one-way between-groups analysis of variance indicated that there is no statistically significant difference at the $p < 0.05$ level for R_a surface roughness parameter in case of all three printing plates. Practically, this means that mean values of R_a parameter resulting from different SEM micrographs captured at 4 different magnification levels as well as from TR 200 measuring device do not differ significantly.

The same result was found to be in case of R_q parameter and flexographic printing plates but not for the letterpress printing plate where statistically significant difference was found at the $p < 0.05$ level. Posthoc comparisons using the Dunnett's T3 post hoc test indicated that the mean value obtained with TR significantly differ from mean values obtained from SEM micrographs but, importantly, they do not differ from each other, emphasizing once more the insignificant influence of magnification level on overall surface roughness derived using image-based profilometry.

However, the differences between the mean values of R_p parameter, both for flexographic as well as letterpress printing plate significantly differ at the $p < 0.05$ level. Dunnett's T3 post hoc test revealed that significant difference is reported between the mean values derived from direct and indirect profilometry. Statistically significant

difference was also established between R_p values obtained from micrographs captured with magnification of 1500x and other three, lower ones. In the case of two flexo printing plates, the difference between mean values of R_p parameter obtained from micrographs with 1000x and 1500x magnification was also found to be significant.

The difference between mean values of R_v parameter are not found to be statistically different in the case of letterpress plate, but in the case of flexographic plates, however, these differences were only established between mean values of direct and indirect profilometry.

This analysis pointed out the greater influence of magnification level of SEM micrographs used for surface roughness analysis if the point of interest is shifted toward more specific roughness characteristics, like peaks and valleys, rather than overall, average roughness. Also, it is rather important to emphasize that the derived significant differences between the mean values of R_v and R_p parameters from direct and indirect profilometry are directly reflecting the nature of methodologies themselves. Increased values of R_p parameter derived from SEM micrographs in comparison to those obtained with stylus profilometer might be direct consequence of rather small pixel dimension (i.e. 100 μm on image magnified 1000x) thus precise registration of the deepest pores, as well as insufficiently small size of a diamond tip of measuring device and thus disability to reproduce the smallest details. The increased values of R_v parameter might be found in influence of the local tilt angle of the surface structure on brightness level in the SEM micrograph, thus evaluation of the topographic contrast of SEM images [2].

CONCLUSIONS

In this paper, we have analyzed indirect, image-based approach to the characterization of surface roughness of flexographic and letterpress printing plates against standard, profilometric method. In the analysis we have included SEM micrographs captured at four different magnification levels. The conclusions derived from the conducted research are as follows:

- The average surface roughness values obtained by the indirect profilometric method correspond to average surface roughness (R_a) obtained by direct stylus profilometric method. The differences between average values were not found to be statistically different.
- Greater differences were found in case of roughness parameters which describe more specific roughness parameters – peaks and valleys. The

reasons might be found in the methodologies themselves, since determination of these parameters are directly influenced by physical resolution of the measuring device on one side and pixel dimension as well as pixel grayscale level on the other side.

- The higher influence of magnification level of SEM micrographs was established if the point of interest is shifted toward more specific roughness characteristics, rather than overall, average roughness.

- In terms of results consistency, repeatability, accuracy and dissipation, both methodological approaches have exhibited the same trend, where more consistent results were obtained for R_a and R_q parameter over the R_p and R_v parameter.

- Direct profilometric method has exhibited deficiencies in terms of leaving scratches on the plate surface, interruptions in measuring and prolongs measuring process.

Overall, the results have shown that proposed indirect image-based profilometry can serve as a flexible, valuable and useful tool in the characterization of the average surface roughness of flexo and letterpress printing plates.

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ИЗСЛЕДВАНЕ НА ПОВЪРХНОСТНИТЕ ХАРАКТЕРИСТИКИ НА ФЛЕКСО И ЛЕТЪРПРЕС ПЕЧАТНИ ФОРМИ ЧРЕЗ SEM БАЗИРАН АНАЛИЗ

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(Резюме)

В това изследване е използван нов метод базиран на анализ на изображението за оценка на повърхностната структура и неравност (нееднородност) на флексо- и летърпрес печатни форми. Предишни наши изследвания показаха, че директното използване на профилографски метод за анализ на гореспоменатите печатни форми води до редица трудности като отклонения в точността на измерване и забавяне на работата при измерване на полимерни материали. Беше установено, че безконтактните методи като SEM, са особено подходящи за визуализация и количествено описание на повърхностната структура.

Индириктния метод базиран на Gwyddion софтуерни функции за анализ на SEM изображения и изчисляване на стандартни профилографски резултати. Резултатите показаха, че е възможно получаването и използването на данни от SEM анализа на подробни данни за повърхностната структура и профил на изследваните печатни форми.

Извършен е анализ на влиянието на различните увеличения върху анализа на повърхността. Статистическия анализ показва, че различните мащаби и увеличения, не оказват съществено влияние върху получените резултати за Ra.

Като заключение, резултатите показаха, че предложения и изследван индириктен метод за характеризиране на повърхността на печатните форми е особено полезен и достатъчен за точен анализ на интересуващите печатната индустрия параметри.