Energy Policy Studies

1(2) / 2018

IGNACY LUKASIEWICZ
ENERGY
POLICY
INSTITUTE

Cover design: Aku Studio

Typesetting: Aleksandra Szewczyk

Proofreading: Robin Gill

Publisher: Ignacy Lukasiewicz Energy Policy Institute

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http://www.instytutpe.pl/eps/

http://www.instytutpe.pl/en/eps-en/

e-ISSN: 2545-0859

The electronic version of the journal is the original version.

Rzeszów 2018

Ministry of Science and Higher Education
Republic of Poland

"Energy Policy Studies – English language e-edition as a tool for disseminating science" and "Energy Policy Studies – transfer of the best foreign pracities and echange of experience" – the tasks financed as part of contract 687/P-DUN/2018 with the resources of the Minister of Science and Higher Education for science promotion activity.

EVALUATION OF THE QUALITY OF TITANIUM OXIDE AND COPPER OXIDE LAYERS BY MEANS OF OPTICAL MICROSCOPY

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Abstract

The aim of this paper is to analyse high-resolution optical images of the surface layers of titanium oxide and copper oxide. The materials were produced using the PREVAC Modular Platform for layer deposition located at the University of Rzeszow. Images with a magnification of 50x were obtained using an optical microscope. On the basis of the results obtained, the quality of the layers was evaluated based on the image analysis. The quantitative method of analysis was applied, as a result of which the conditions of the manufacturing process for which the obtained layers had the highest homogeneity were indicated. The minimum range of grey shade counting was from 65 to 150 and the maximum range from 135 to 220. The standard deviation was from 8 to 40%.

Keywords: Titanium oxide, Copper oxide, Optical microscopy, Histograms

Introduction

The paper presents the results of research on titanium oxide and copper oxide as potential material for the construction of new types of solar cells based on TiO_2/CuO and TiO_2/Cu_2O connectors. The paper presents a qualitative analysis of optical images of the oxide layers produced. To determine the quality of the structures, images were obtained using the Nikon Eclipse MA 200 light microscope with the NIS-Elements software. This allows the surface of opaque materials in reflected light to be observed. In an optical microscope, an enlarged image of the examined object is generated with the use of light passing through a special optical system usually consisting of a set of several to a dozen or so optical lenses. Fig. 1 shows the photo and structure of the microscope Eclipse MA 200 microscope. The basic parts of the optical microscope are: eyepiece (1), micrometer screw, „revolver" (2), lens (3), macroscrew (4), and workpiece table (5). It is the most advanced metallurgical microscope in the reverse system, optimised for sharpness, aberration correction and ergonomics.

Histograms were created to evaluate the quality of the tested TiO_2 and Cu_2O layers using the online „Image Histogram Generator" software. (www.dcode.fr 2018). The Gauss greyscale curves for each image and the standard deviation were determined on the basis of these curves.

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Figure 1. Nikon Eclipse MA 200 microscope with NIS-Elements software

Preparation of samples

For the purpose of the experiment 4 structures of copper oxide and titanium oxide were created. The layers investigated were produced by reactive magnetron atomisation of titanium and copper disks with the use of a modular PVD platform. N Si (100) type tiles and glass with dimensions of 5x4 and 6x5 mm, respectively, were used as substrates. The samples were produced in an atmosphere of argon Ar and oxygen O_2 in different process parameters. The atomisation time was 10, 20, 30 and 40 minutes. The process parameters are shown in Table 1.

Table 1. Process parameters for creating Cu2O and TiO2 layers

Parameters	P1	P2	P3	P4
Type of substrate	Silicon/glass			
Distance between source and ground [mm]	53	38	38	53
Pressure [mbar]	$1{,}58 \cdot 10^{-2}$	$2{,}41 \cdot 10^{-2}$	$1{,}9 \cdot 10^{-2}$	$2{,}78 \cdot 10^{-2}$
Power [W]	120	110	80	70
Time [min]	30	20	40	10
Oxygen flow [cm^3/s].	5.0	1.5	2.0	8.0
Argon flow [cm^3/s].	1.5	4.0	3.0	4.0
T [°C]	20	150	200	20

Results and discussion

The Image Histogram Generator software (Sekatskii et al. 2001) was used for numerical analysis of the images obtained. This allows numerical greyscale data to be collected for each image based on its bitmap. On this basis histograms, Gauss curves, and the standard deviations for each sample were calculated, as shown in Table 1. The brightness scale is in the range from 0 to 255. In the literature many scientists have analysed the histogram (Scuderi et al. 2017; Rokhmat et al. 2017; Boissenin et al. 2007; Pires et al. 2013; Delbem et al. 2015; Kottler et al. 1997; Sekatskii et al. 2001; Wieclawek 2018).

Figure 2a. Photo and histogram of the structure P1 deposited on silicon

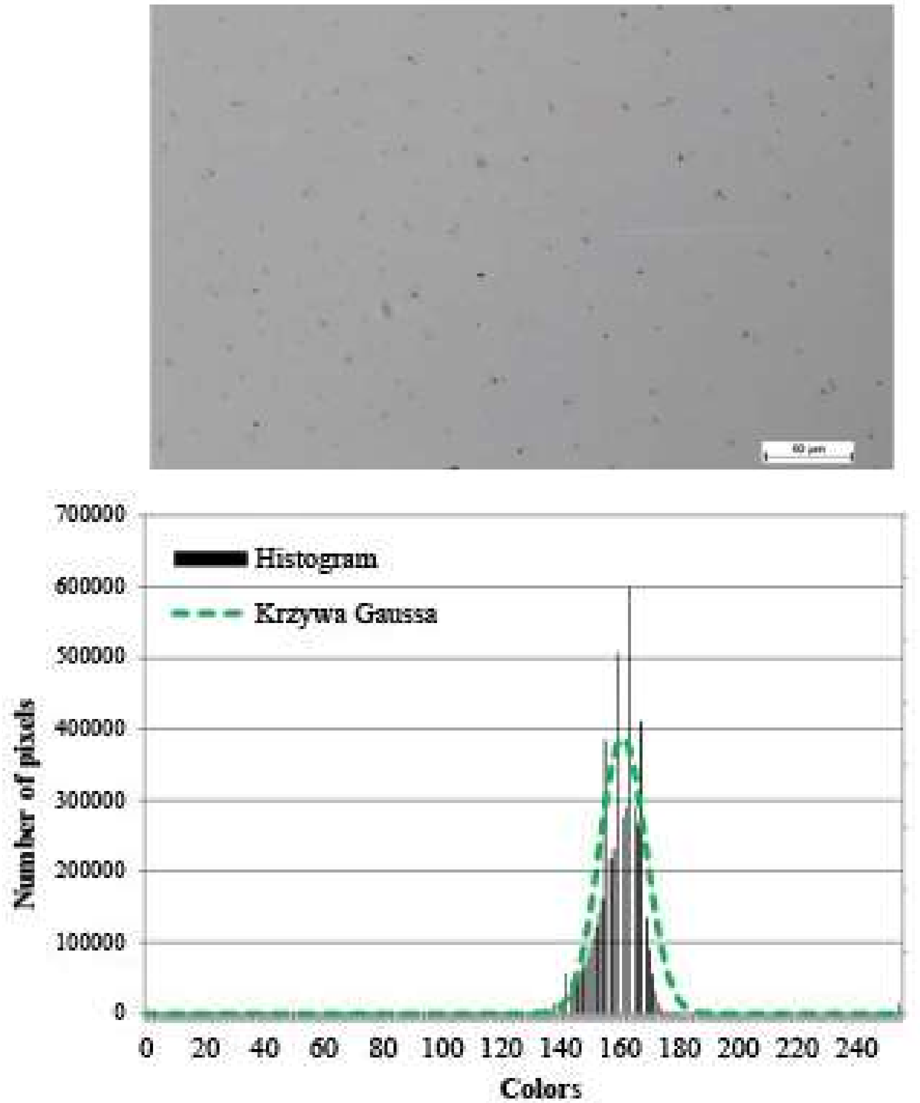
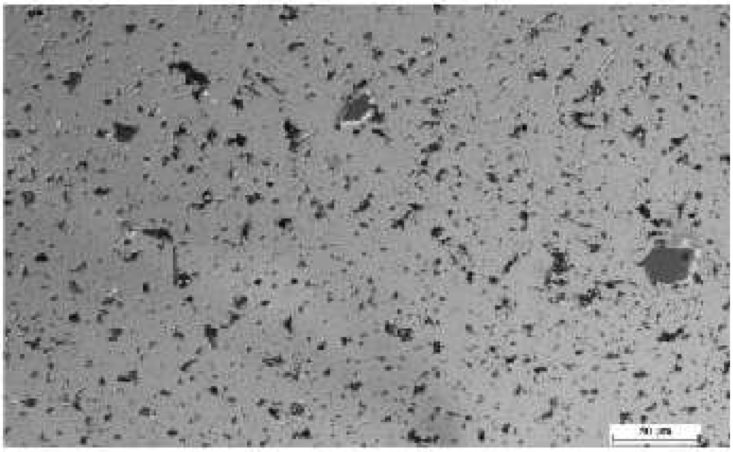


Fig. 2a. shows that the structure is homogeneous with a small number of point defects, which reflect the characteristic lines of the histogram. Its small blurring indicates the high homogeneity of the layer. The largest number of counts in this case is in the range 140-170.

Figure 2b. Photo and histogram of structure P1 deposited on glass



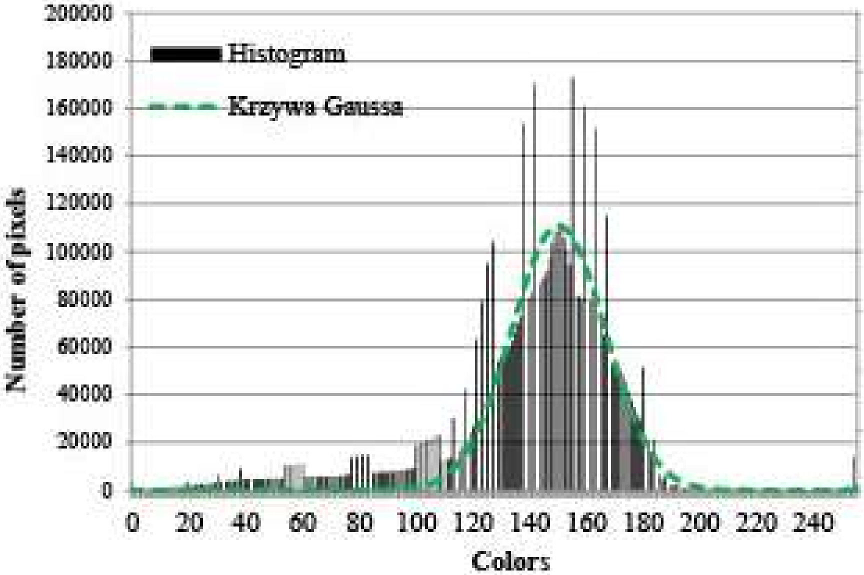
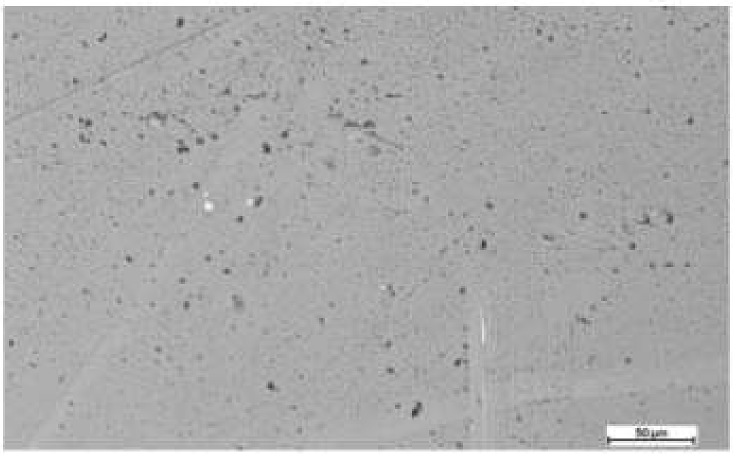
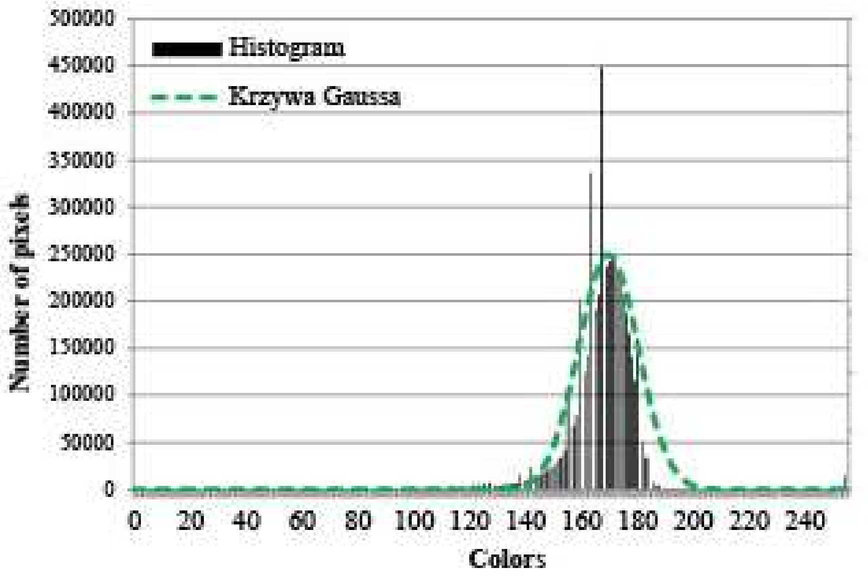


Fig. 2b clearly shows that in the case of this structure we can observe a much greater blurring of the histogram. This is due to the large number of point defects. The highest number of counts in this case is in the range of 120-180, which indicates the heterogeneity of the coating.

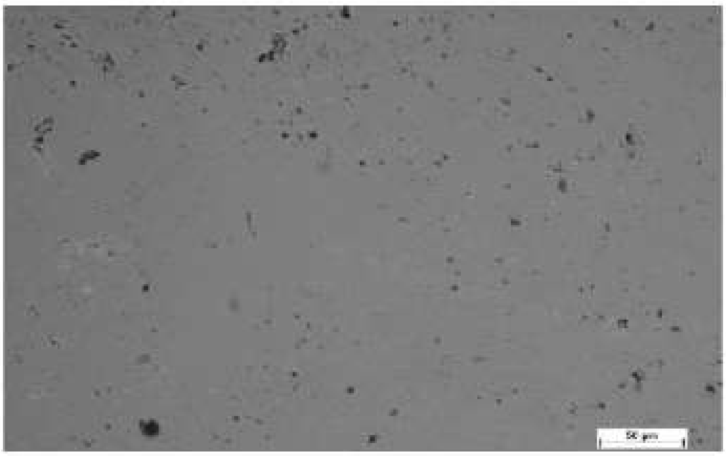
Figure 3a. Photo and histogram of the structure P2 deposited on silicon

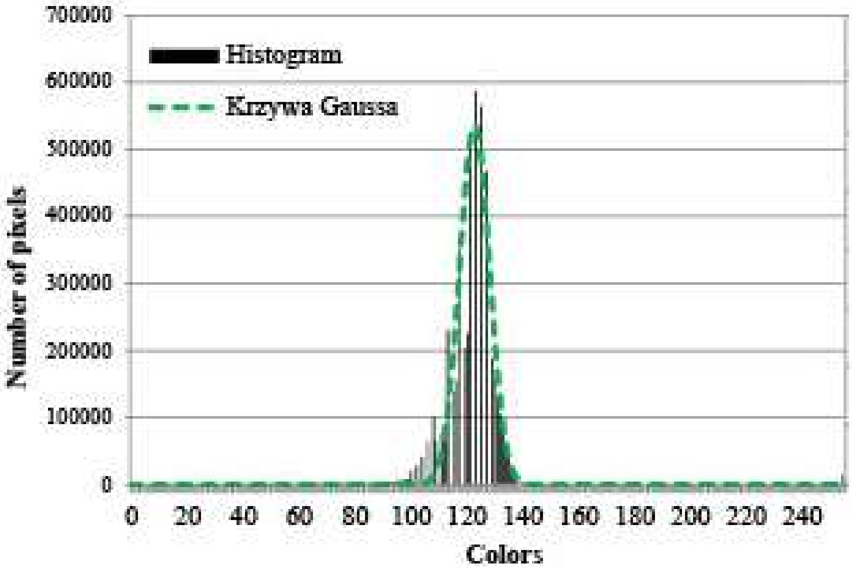




The structure is homogeneous with a small number of point defects of several μm in size. On the surface of the structure there are also surface defects in the form of longitudinal streaks, which probably result from errors in the substrate preparation. It should be emphasised that the streaks are not reflected in the histogram, because their greyscale is very similar to the colour of the layer. The two histogram lines to the left of the histogram illustrate several bright points in the image, the occurrence of which is probably the optical effect of the interaction of the microscope radiation with the layer. The largest number of counts in this case is in the range 150-180.

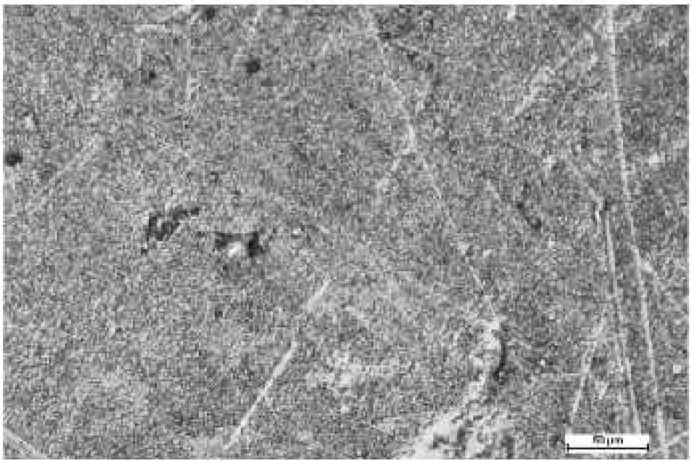
Figure 3b. Photo and histogram of structure P2 deposited on glass

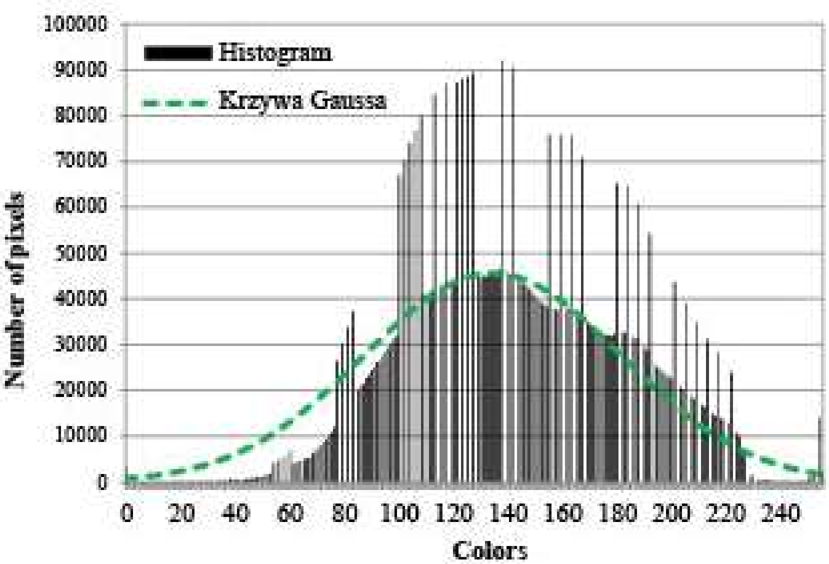




Figures 3a and 3b show that the structure P2 on silicon and glass is very similar. The layer on glass is homogeneous and has fewer point defects than the structure on silicon (Fig. 3a), but they are larger. The highest number of counts in the case of structure P2 deposited on glass is in the range of 110-135; the small range of greyscales indicates a good application of the coating on the substrate and confirms its homogeneity.

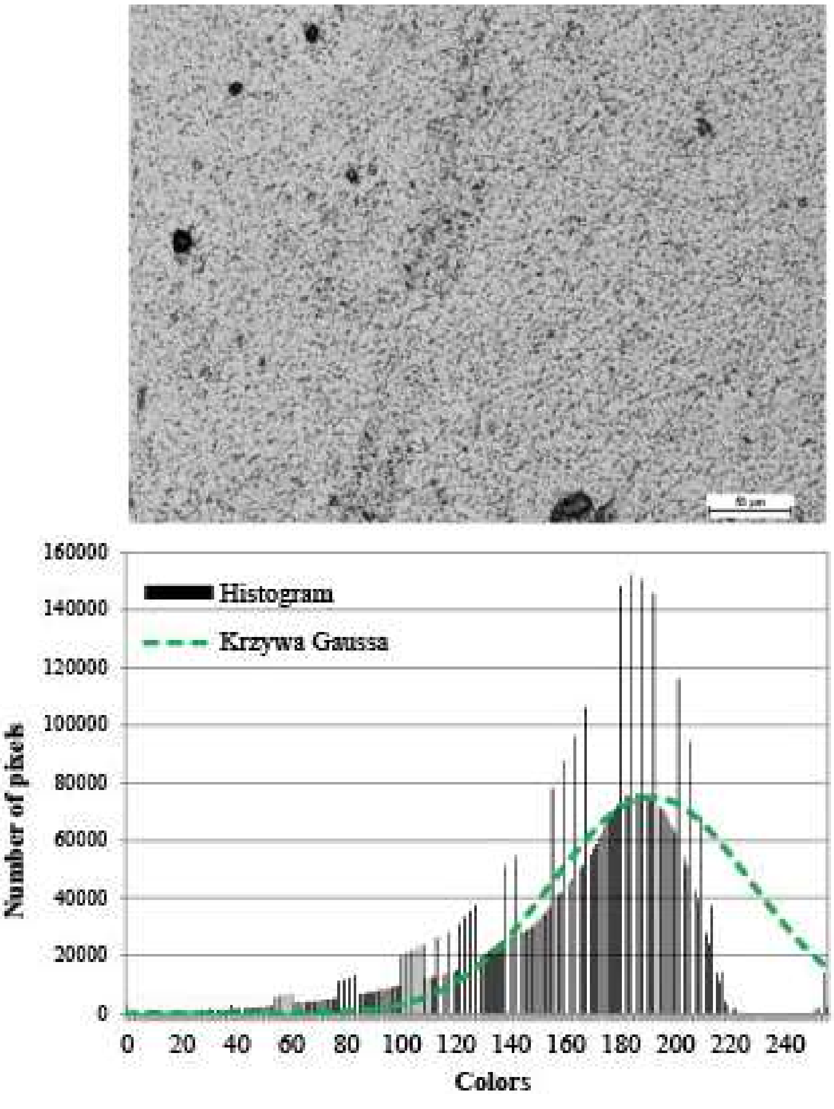
Figure 4a. Photo and histogram of the structure P3 deposited on silicon





The wide distribution of greyscales means that the sample is highly granular and, to some extent, this reflects a defect in the substrate. We conclude that the fine-graininess is due to the optical effect in the form of strong light scattering. The wavelength of the light is comparable to the characteristic grain size. The histogram detected surface defects with a lighter shade (a strong characteristic line on the left, towards the light pixels). The largest number of counts in this case is in the range of 65-220; the wide range of grayscale confirms the heterogeneity of the coating.

Figure 4b. Photo and histogram of structure P3 deposited on glass



Analysing the image obtained during the examination of the coating with the optical microscope, it can be stated that, similar to the structure P3 deposited on silicon, the coating is fine-grained. From the histogram for layer P3 deposited on glass we can see that the layer is characterised by a large variety of surfaces. The greyscale counting range from 80 to 220 shows that there are many phases and surface defects. The largest number of counts in this case is in the range 150-210.

Figure 5a. Photo and histogram of P4 structure deposited on silicon

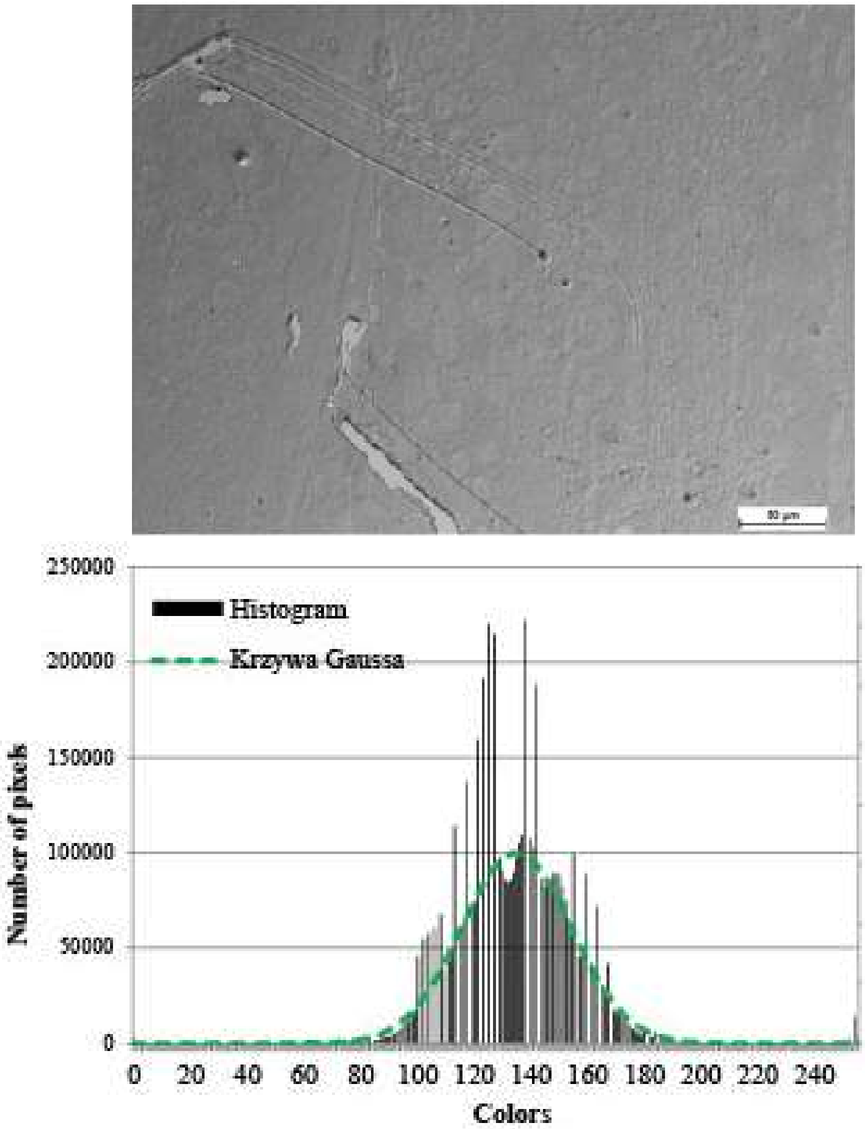
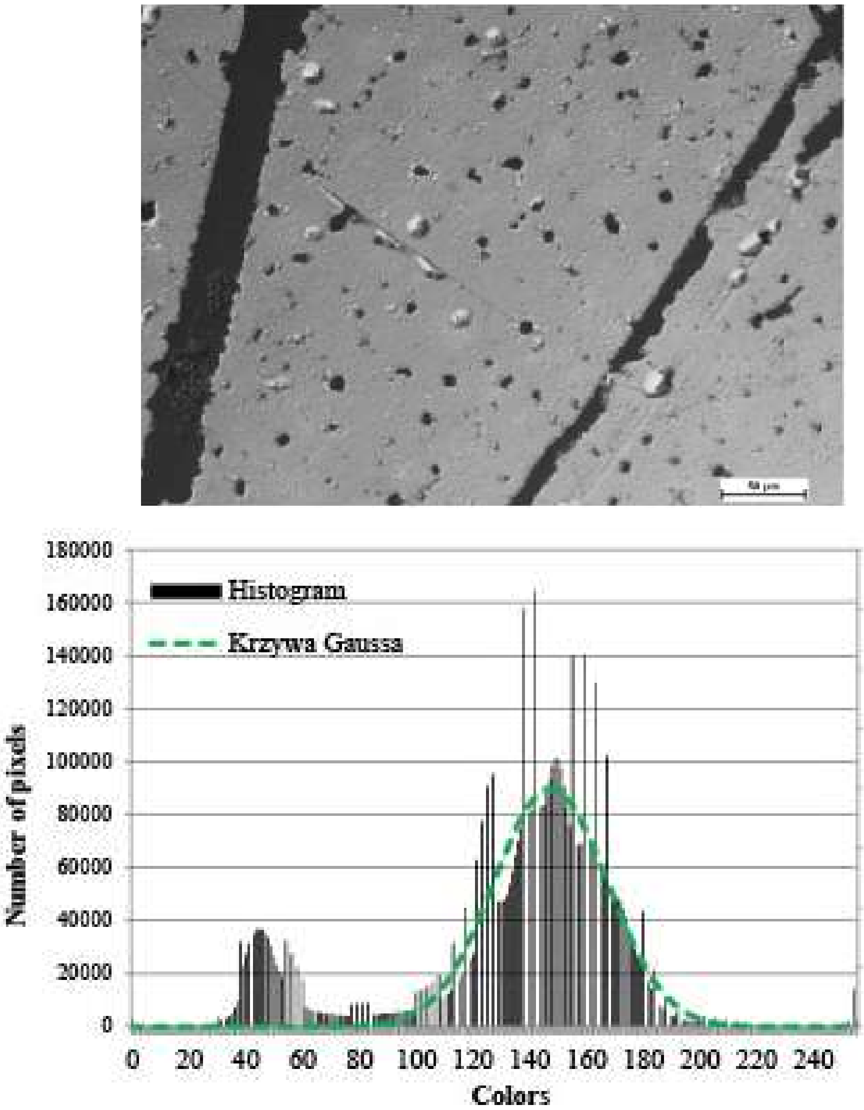


Figure 5a shows that the structure is heterogeneous. There are point defects arising from the manufacturing process and large surface defects in the form of scratches probably of a physical nature, which are not the result of the applied deposition process, but of subsequent tests carried out on the sample. The histogram shows that the analysed layer is homogeneous, but the counting observed in the range 100-160 of greyscales indicates slight deviations in the surface parameters of the analysed structure.

Figure 5b. Photo and histogram of P4 structure deposited on glass



From the histogram for layer P4 we can see that, apart from the dominant homogeneous structure giving greyscales from the range 110-180, a large number of counts in the range 40-55 illustrates the lack of continuity of the layer (uncovered substrate). In addition, it should be noted that in this case we are dealing with mechanical damage to the layer resulting from the scratch--test test (two clear scratches shown in Fig. 5b). The picture and histogram (Fig. 5b) show that the material is heterogeneous, there are a significant number of point defects of a large size of 10 µm, in addition, there are two characteristic scratches formed mechanically.

Table 2, with the minimum and maximum value of greyscale counts and the calculated standard deviation for all structures imposed by magnetron atomisation on silicon and glass, is presented below.

Table 2. Greyscale range and standard deviation

Greyscale range	P1		P2		P3		P4	
	Silicon	Glass	Silicon	Glass	Silicon	Glass	Silicon	Glass
Min	140	120	150	110	65	150	100	110
Max	170	180	180	135	220	210	160	180
Standard deviation [%]	10	20	15	8	40	25	20	30

The grayscale ranges and standard deviations listed in Table 2 allow the surface quality of the analysed layer to be assessed. The greatest heterogeneity is observed for the P3 layer deposited on silicon and glass. In this case there is the largest dispersion of values and the largest value of standard deviation. The best homogeneity is observed for layer P1 on silicon and layer P2 on glass. In the case of layer P4 on glass, the interpretation of the results and evaluation of the quality of the layer is difficult due to the clear mechanical scratches on the layer.

Conclusion

The paper presents a method of quantitative analysis of surface defects of samples by applying histograms of greyscales. The analysis technique used has certain advantages. The distributions presented quite effectively reflect particular types of defects, although the disadvantage of the method is the fact that the overlapping of similar greyscales from particular types of defects may lead to misinterpretations. The largest greyscale range was determined for structure P3 deposited on silicon and amounted to 65-220 (sample with fine grain structure with defects), whereas the smallest number of counts was determined for structure P2, deposited on glass and amounting to 110-135 (homogeneous sample with a small number of defects). The smallest standard deviation is shown by the structure P1 deposited on the silicon substrate and P2 applied to the glass substrate, which indicates the highest uniformity of its surface. This is consistent with the optical image made on the microscope and the histogram placed next to it (Fig. 2a, 3b).

Further studies on this analytical method are necessary, as it appears to be effective, but its weaknesses and methods for correcting errors should be learned, as well as clearly linking the nature, number and size of defects and the shape of histograms.

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