APPLICATION OF THE IMAGE ANALYSIS METHODS FOR QUANTITATIVE DESCRIPTION OF THE AL_2O_3 OXIDE LAYERS

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Abstract

The article presents a method of description the Al_2O_3 oxide layers structure observed by scanning microscopy. Unusual geometrical features of the structure enforce the use of non-standard techniques for their description. The authors propose the use the descriptors based on skeleton analysis and compare their effectiveness with morphological methods used in standard stereological descriptions of material structures.

The analysis was carried out on a series of photographs presenting the top layers received at 303K and curry density 2A/dm^2 and 4A/dm^2.

Keywords: Image processing, oxide layers, skeletonization, texture

1. INTRODUCTION

A quantitative description of the materials structures is a standard in the material science and material engineering field. Automatic procedures for assessing the typical stereological parameters is not a challenge but everyday routine in laboratories practice [1]. For typical structures, the set of parameters assessing in the quantitative manner their size, spatial organization and shape allow detecting the influence the parameters of the manufacturing process on the material structure and its physio-mechanical properties. The authors considered the problem of the non-typical type of structure, where the object of the analysis is not a cross-section of the compact geometric solid, or compact discontinuity of the material like it is in case of materials with pores. Regarding the possibility of a description of the structure which is exhibited as a partially connected net, the mathematical morphology methods were used in order to simplify the observed structure and enable its description in a quantitative manner. As an example of net-like structure two SEM images of the surface of Al_2O_3 oxide coating were analyzed. The subject of the analysis was a filamentous hill structure visible on the images as a light ridges on the surface of the coating. It is reported that the filamentous hill's structures exhibit the un-uniformity of the bedding material and reveal it in the shape of the filamentous hill's shape.

2. MATERIALS AND SAMPLE PREPARATION

The starting material for the process was EN-AW-5251 aluminum alloy. Samples of a rectangular area of 0.1 dm^2 were etched sequentially with 5 % KOH solution for 45 minutes, and 10 % HNO_3 solution for 10 minutes, at room temperature. After each step of etching, the sample was placed in distilled water to remove residual acid. The electro-oxidation of the aluminum alloy was carried out in a ternary solution of SFS (18 % sulfuric (33 ml/l), oxalic (30 g/l) and phthalic acids (76 g/l)) mixed with 2H-WS_2 powder (Aldrich - Sigma, grain size <2 μm). The hard anodizing process was performed at 2 and 4 A/dm^2 current density in 303K temperatures of electrolyte. In order to ensure homogeneity of the suspension and to prevent settling of the 2H-WS_2 powder, mechanical stirring was applied during the electrolysis process.

3. METHODOLOGICAL BASES

In order to analyse the surface of Al_2O_3 oxide coatings research was conveyed with Philips XL30 scanning electron microscope with EDS attachment (**Figure 1**). The samples were sputter-coated with gold for SEM imaging. Images were saved in the BMP. The size of the images: 2576x1936 pixels, where 1 pixel was equal to 0.0019 µm. Image processing and analyses of the images were performed on the Aphelion software by ADCIS.

Due to observed high noise, shade effect and relatively low contrast between the objects that were the objects of the analysis, the preprocessing of the images had to be done. Noise reduction was performed by Nagao filtering what results in effective noise reduction while maintaining the sharpness of the edges of the objects. Nagao algorithm searching for the most homogeneous neighbourhood area around each analysed pixel of the image, and then set the value of the average grey level of the selected neighborhood area. It removes noise without blurring sharp edges, and preserve the details of the boundary of a region. Nagao smoothing also improves the sharpness of blurred edges [2, 3].

Shade effect is observed when the background of the images has unequal intensity, what unables the detection of the filamentous hills. In the literature, there is many shade effect correction proposed [4, 5]. Authors applied so-called "proportional correction". This method allows to align the lightness of the background proportionally, and with minimal effects on the objects. It consists of a sequence of transformations. First and the most important is to obtain the image of the shade, or background without the objects. It can be performed by averaging filtering with a big mask, or on the way of morphological closing with the size of the structure element as significant as it is needed to obtain the image without objects. Typical procedures for shade reduction on this stage generate the negative of the background and add the negative to the initial image. Obtained image exhibit the even background but the value of the objects might be changed, what will effect on the detection and measurement results. Thus the authors applied the method which is more sophisticated and requires more transformation but gives better results. The proportionality is obtained by dividing the initial image by the image of the shade, and then the resulting image is multiplied by negative of the background. The final image is added to the initial. A detailed description of the method may be found in the literature [4, 5] The last step of the preprocessing stage was to equalize the histogram, which results in good contrast of the objects and background. Detection of the filamentous hills was performed by application of the thresholding transformation. The binary images were the final material for quantitative image description (**Figure 2**).

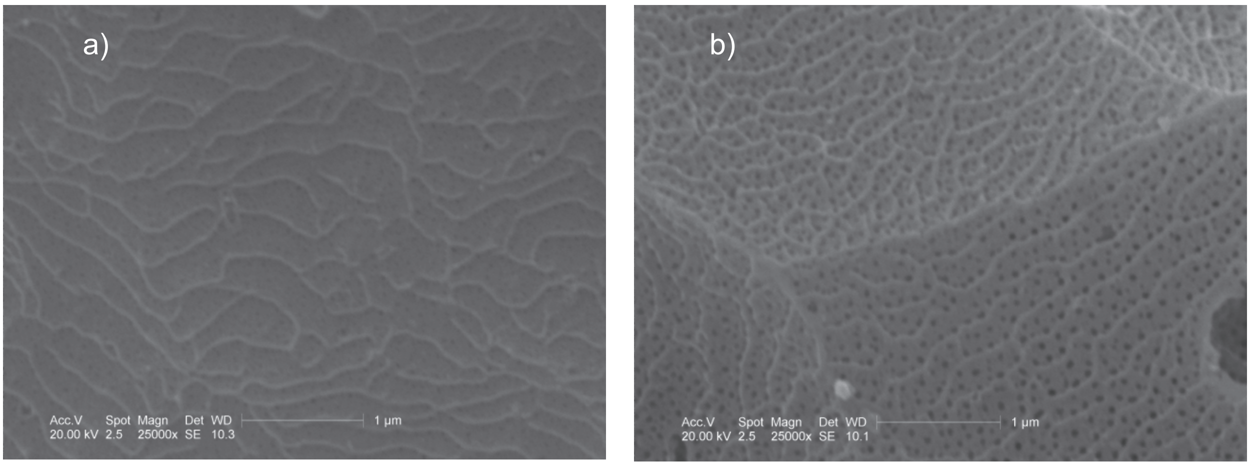

Figure 1 Comparative micrographs (25000x) show the change in microstructure of Al_2O_3/WS_2 surface layers obtaining at a temperature of 303K and current density: a) 2 A/dm^2, b) 4 A/dm^2

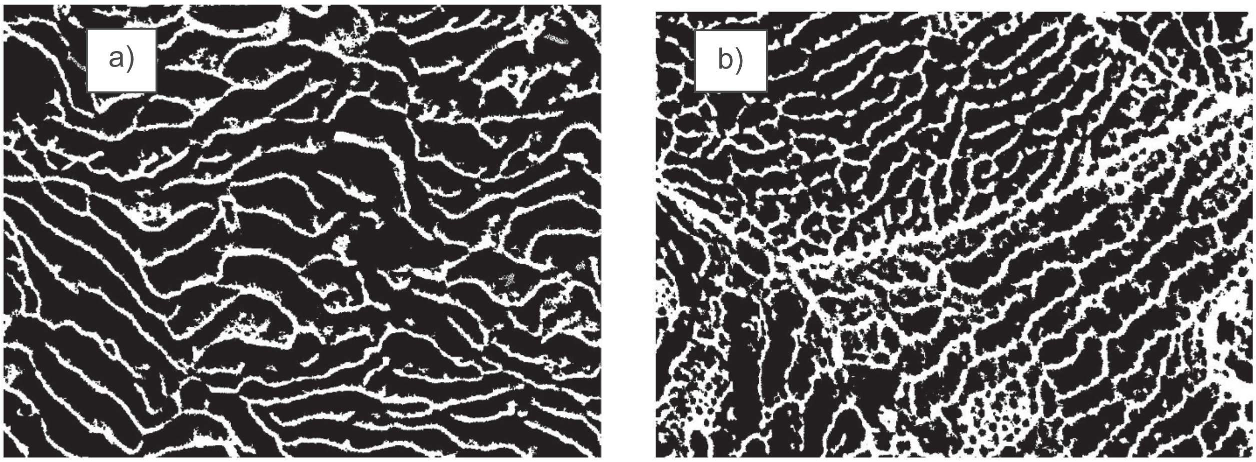

Figure 2 Result of detection the filamentous hills on the images presented the sample of Al_2O_3/WS_2 surface layers obtaining at a temperature of 303K and current density: a) 2 A/dm^2, b) 4 A/dm^2

Measurement of the volume fraction V_V of the area of the surface with filamentous hill's structure was performed. Proposed approach based on the analysis of the filamentous hill's skeletons. Skeleton of the object in the image processing meaning is the one-pixel width line build up with the pixels which are located in equal distance to the boundaries of the objects (**Figure 3**). Skeleton results in the thinning process, which reduced objects to the line. Skeleton analysis is used to distinguish the objects that have a complex structure, and the standard shape analysis methods are not efficient. It is applied in object recognition, writing recognition, OCR etc. Skeleton consists of lines, call branches and intersection points [6, 7]. The compilation of those parameters allows to significant differences in the examined structures. Result of the skeletonization is presented on the **Figure 4.**

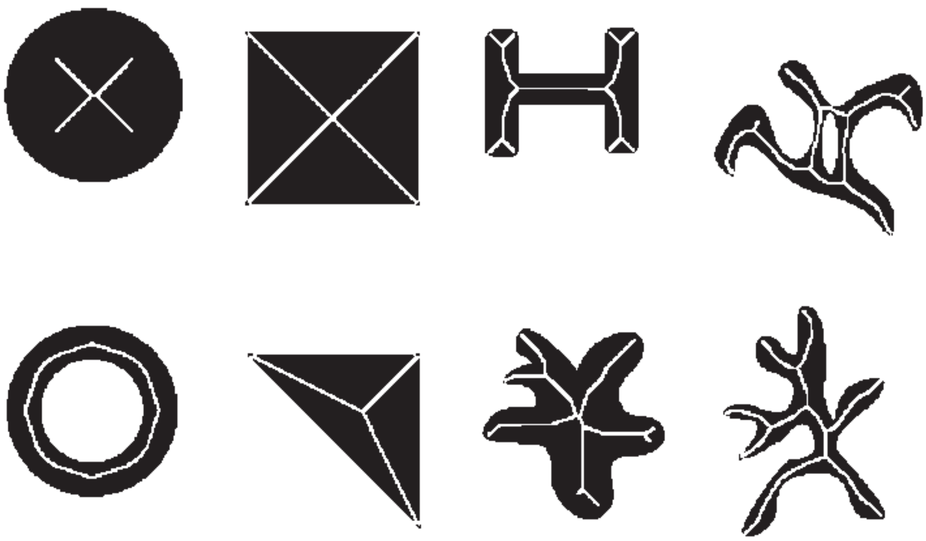

Figure 3 Example of the skeletonization results on the objects with significantly different shapes

The last stage of the measurement process was to analyze the images as the value of the variance can characterize textures. The variance in texture analysis is the parameter which value give the information about the level of variability of pixel values. The values of the variance of each image were indicated.

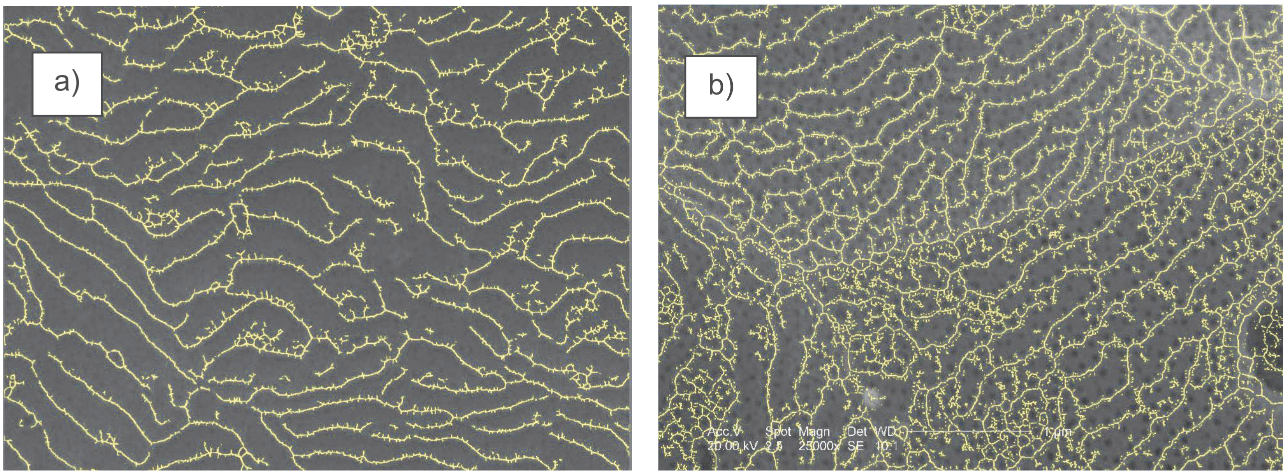

Figure 4 Result of the skeletonizing detected filamentous hills overlapping the initial images

4. RESULTS AND ITS DISCUSSION

Comparative micrographs (25000x) show the change in microstructure of Al_2O_3/WS_2 surface layers obtaining at the same temperature of 303K with different current density were presented in **Figure 1**. The results of the image analysis where presented on the **Figures 2** and **4**. The results of the quantitative analysis were presented in the **Table 1**.

Table 1 Results of the quantitative analysis of filamentous hill structure

Sample	Volume fraction of the filamentous hill's area (%)	Total length of skeleton (µm)	Number of intersections (1)	Number of objects (1)	Number of branches (1)	Variance (1)
A	21	0.2951	797	218	1348	70.49
B	31.24	0.3829	1539	501	3108	247.62

Subjective analysis of the filamentous hill structure based on the visual assessment by experts results in a statement that two observed structures are different. That gives an impulse for authors to work out the method which might allow assessing more precisely the differences between two observed structures not only based on the fuzzy and subjective statements. The proposed approach consists of the combination of the typical stereological parameters and parameters of the skeletons obtained from the detected structure. Volume fraction (VV) delivers general information about the per cent of the area which represents analyzed structure. When the aim of the analysis is a more detailed description of the structure morphology, usually there are used the parameters which assess the shape factors. In presented structures as can be observed in **Figure 1**, there is no distinct difference in the shape of the structural elements. There are elongated, with different spatial orientation. The application of the skeleton analysis (**Figure 3**) shown that this method delivers new and valuable information about analyzed structures.

The number of intersections provides information about the number of points where at least two branches have one common point. The number of intersection and the more branches are in the analyzed object, the more complex the shape it has. As is shown in the **Table 1**. The difference in values of the volume fraction between the sample A and B is close to 50 % of the value for sample A. Analyzing this parameter we can state that there is a difference in the number of filamentous hills in both samples. Comparing the parameters describing the skeletons of filamentous hill's structure on both images, we can observe much higher diversity than the volume fraction. The structure on the image B is much more complicated than on sample A what finds its reflection in the number of intersections, which is higher 100 %, number of objects (separated filamentous hill) 250 % and number of branches over 200 % in comparison to the values of this parameter for sample A.

Variance analysis also showed significant differences in compared images. The value for the image of sample B is 300 % higher than for image of sample A. Analyzing the results of variance it should be noticed that this parameter is addressed to the whole image, not only the filamentous hill structure. Thus it reflects the differences in other elements of analyzed structures.

5. CONCLUSIONS

Quantitative analysis of the structure can extend the typical stereological parameters. The wide range of abilities of image processing and analysis allow proposing new, dedicated to the analysed type of structures parameters, which can more effectively differ and describe it.

The proposed method of the skeleton analysis adapted to the filamentous hill structure exhibited on the Al_2O_3/WS_2 surface layers obtaining at a temperature of 303K and current density: a) 2A/dm^2, b) 4A/dm^2 confirmed their high sensitivity to changes in the structure, what was presented in **Table 1**. Other parameters (volume fraction, variance analysis) are too global and are not sufficiently precise to be used for detection less different structures.

Methods presented in this article may be useful in similar materials science analysis e.g. dental alloys [8], magnetic materials [9], material hardening [10], special coatings [11] or during operational inspection [12-14]. Due to their effectiveness, these methods can be of economic importance in industrial applications [15-19]. These methods should be included into the knowledge database of a decision support systems [20-22].

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