APPLICATION OF THE IMAGE ANALYSIS METHODS FOR QUANTITATIVE DESCRIPTION OF THE Al_2O_3/WS_2 OXIDE LAYERS WEAR MECHANISM

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Abstract

In the article, the authors attempted to quantify description of the Al_2O_3/WS_2 oxide layers. The description were carried out using image analysis and stereological methods. Objective and precise analysis of the surface topology aim to characterize the Al_2O_3/WS_2 oxide layers obtained by hard anodizing process on an aluminum alloy in differ temperature and different current density. The addition of WS_2 to acid bath reduces the friction coefficient of Al_2O_3/WS_2 - PEEK/BG friction pair. The characteristics of matrix of Al_2O_3 nanofibers with dispered WS_2 phase were performed using scaning electron microscopy and X-ray structural studies. Quantitative description of the surface with set of parameters describing topology of the surface after friction test had aimed to analyse the wear process and assess the parameters which effects on it.

Keywords: Image analysis, stereology**,** alumina coating films, tungsten disulfide, wear mechanism

1. INTRODUCTION

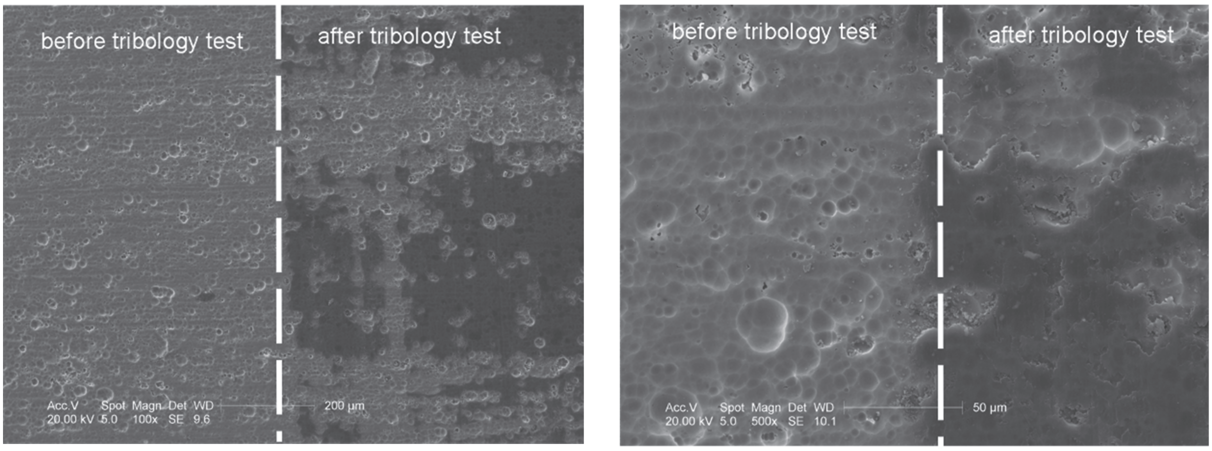
Computer aided material design and analysis is not a novel. Number of variety software assist the scientists in every stage of material research projects, increasing precision, and decreasing time for data analysis, allow to simulate experiments, and predict its properties [1-5]. Computer image analysis is one of tools of computer aided material design systems. Stereology working out methods for analysis and interpretation three dimensional objects on basis of its two-dimensional cross sections [6]. Combination the stereology methods with digital image processing give an opportunity for fast, objective and repeatable analysis of even sophisticated microstructures [7]. Still many kinds of structure are difficult to describe quantitatively due its characteristic difficult to define image like images of Al_2O_3/WS_2 oxide layers. Reilable quantitative and qualitative analysis of the objects strongly depend on the quality of its image, due to influence the occurrence of distortions in the image, such as for instance, noise, the contrast between the subject and the background. Some distortion of the images may be easily reduced, and some, is its saturation is to high may even induce completely erroneous results. When on the image is impossible to carry out the detection of single objects due to its specific view, conventional approach to analysis of the object, by its detection and measures must be replaced by global analysis of conglomerate of objects. For this kind of analysis, texture analysis algorithms are useful, because the analyze the topology of image, and describe its irregularity by set of parameters. Analysis of the surface by texture algorithm, allow to comparing the set of the images presented materials with different technology parameters, and differentiate them. An attempt to prove the relationship between surface characteristics using texture operators and friction mechanisms was undertaken by the authors and presented in this article.

2. RESEARCH MATERIAL

Set of 8 samples of AL_2O_3/WS_2 were prepared due to attempt the quantitative discribtion the surface after tribology test (**Table 1**). The parameters of anodix oxidation were differentiated in order to work out optimal parameters of the process for parameters of the surface layer and its tribology properties. Hard anodizing process was performed on an aluminum alloy EN - AW - 5251 made by PN-87/H-92741/01-03. Anodic oxidation was carried out with GPR-25H30D feeder cable. Aluminium rectangular plates of the field of 10^{-2} dm^2, underwent etching in 5 % KOH and 10 % HNO_3, in order to remove soil on the surface prepared for anodizing. A lead plate of the same work surface was the cathode of the system. Oxidation was carried out in a water electrolyte consisting of sulfuric 18 % (33 ml / l), oxalic (30 g / l) and phthalic acids (76 g / l). The Al_2O_3/WS_2 layers were formed through adding 0 and 30 g of 2H-WS_2 powder (Aldrich - Sigma, grain size <2 μm) per liter of pure electrolyte. The anodizing process was realized in 293, 298 or 303 K temperatures, with current density of 4 A / dm^2 with different electrolysis time.

Table 1 Parameters of tested oxide layers formation

Samples	WS_2 amount (%)	Oxidation temperature (°C)	Oxidation time (min)
2A4C	0	30	60
2D4A	30	20	60
2D3A	30	20	80
2D3B	30	25	80
2D3C	30	30	80
2D2C	30	30	120
2D4B	30	25	60
2D4C	30	30	60



(a) (b)

Figure 1 SEM images of the Al_2O_3/WS_2 presented tested structure (a) 100x, (b) 500x

3. IMAGE ANALYSIS METHOD

In general the purpose of computer image analysis of the microstructure is to deliver quantitative description of chosen elements on the images. In this study the task of selecting the objects for analysis was difficult in order to specific image of the surface layer, where there are not any clearly defined structure elements. Thus, first it was needed to find some distinguishable features on the image of analyzed surface layer, which would help to track the changes in the surface introduced by different preparation parameters, like oxidation time or oxidation temperature. As can be observed on **Figure 1**, the surface layer view presents an image of pores. Unfortunately the contrast between the image of individual pore and the background is low. Therefore the edges of the majority of pores are unclosed, discontinuous. Standard stereological analysis of porosity involves detecting pores and assessing their morphological characteristics such as size, shape, number, spatial arrangement. Standard approach disqualified and rejected tested images due to their low quality for analysis.

Therefore classic stereology's methods was discarded and attempted to carry out global analysis of the images considering images of surface layer as texture image. In order to quantify the images two approaches of texture analysis methods have been applied. There is no strict texture definition, but this feature of the images is naturally perceived and recognized by humans. Texture description may be carrying out using structural, statistical, model-based and transforms methods. Structural approach use well defines primitives to represent texture [8, 9]. Statistical approaches represent texture indirectly by the non-deterministic properties that describe distribution and relationship between the gray levels of an image. An example of method based on the second-order statistics (i.e. statistic given by pairs of pixels), is method indicating texture descriptors from the co-occurrence matrix [8]. This method found its application in classification of medical images. Transform methods based on processing and analysis images in the frequency domain, for instance Fourier, Gabor, wavelet transforms [9-11].

First approach of texture analysis carried out for tested images was structural approach. Since pore analysis cannot be performed, the perforations of the visible peaks, named by authors as “pore ridges,” were detected (**Figures 2** and **4a**). On the friction area detection of the darkness part (sliding film filling pores, named for analysis purpose “sliding film”) was performed (**Figures 3** and **4b**). Due to carry out detection, some image processing was carrying out. Median filtering reduced noise and contrast enhancement was obtained by histogram equalization [12]. Histogram equalization caused a significant increase of contrast, thus exposure previously invisible details on images. In order to “pore ridges” detection, automatic binarization according to Otsu was preceded. Due to reduce the artefacts caused by noise or uneven background, morphological correction was done [13, 14]. Morphological correction was carried out by opening, closing and hole filling operations. Volume fraction (Vv) of “pore ridges” area and on the images of surface layer after friction the area of visible “sliding film” was indicated.

A statistical approach of analysis was also performed, using a method dedicated to texture description, Haralick's method. Analysis of texture features on tested images was performed using Haralick's descriptors, like for example contrast, entropy, energy, moments, which inform us about image contrast or homogeneity of the texture [15]. Texture analysis was carried out on the images without quality enhancement, but after median filtering.

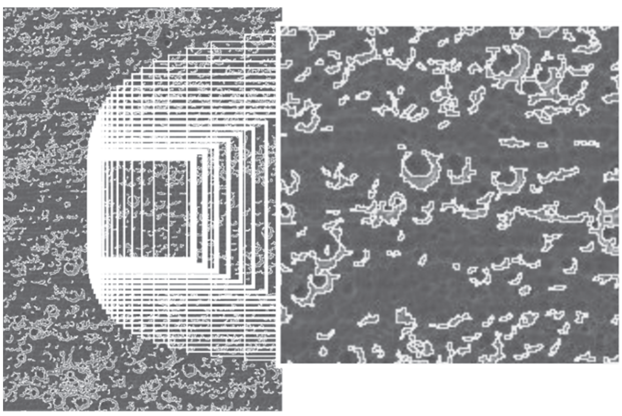


Figure 2 Visual example of detection of pores of ridges before tribology test on sample 2A4C

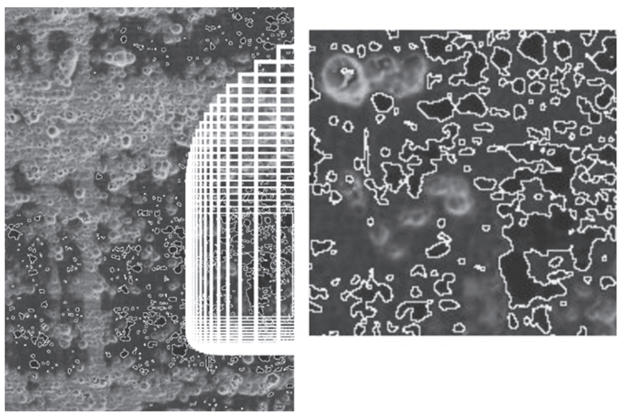


Figure 3 Visual example of detection of sliding film on surface layer after tribology test on sample 2A4C

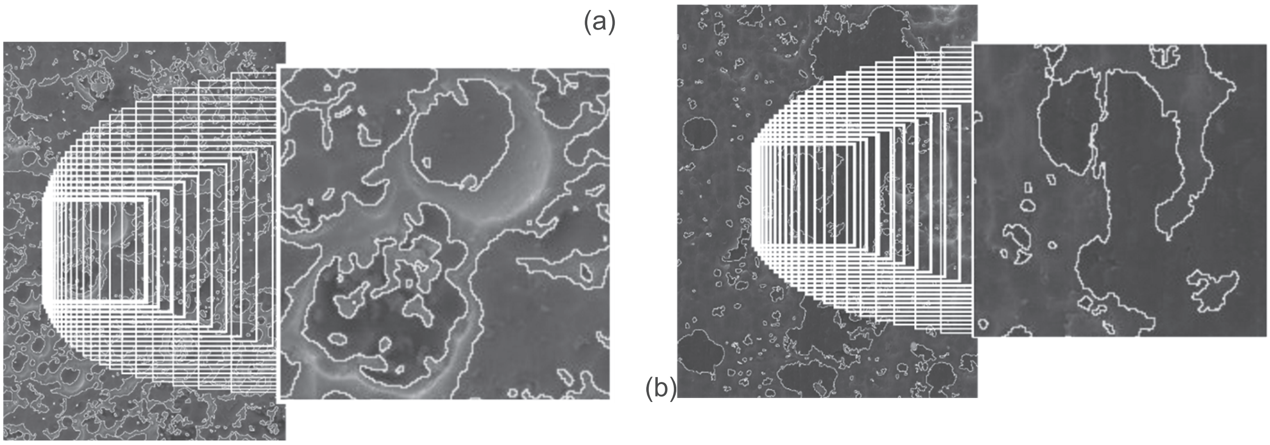


Figure 4 Graphic presentation of detection result of specific area of surface layer (a) before tribology test ("ridge"), (b) after tribology test ("sliding film") on sample 2D3A

4. RESULTS

Set of 8 images representing different samples with Al_2O_3/WS_2 produced in different parameters (**Table 1**). Surface layer was observed on SEM with 500x magnification. Images processing and analysis was carried out using Aphelion 4.2 software. Obtained results of images quantitative analysis were presented on the charts below (**Figures 5 - 10**). Results for analysis SEM images with 100 magnifications due to high dispersion of results were not considered.

In the **Figures 5 - 8** the 2A4C sample is shown as reference sample [16] for the rest of presented samples in this paper. Wear mechanism of presented samples was shown by authors in [17]. The goal of comparision of texture, presented in this article, is an attempt to discribe new tool which can help to show the difference in samples obtained in variety technology conditions.

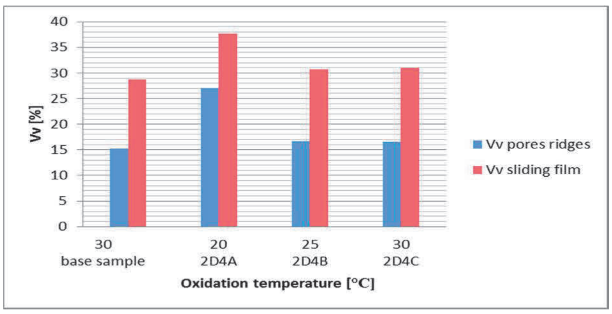


Figure 5 Comparison the surface layer before ("pores ridges") and after ("sliding film") tribology test

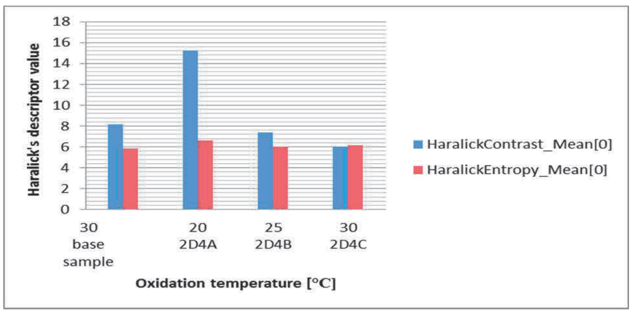


Figure 6 Description the surface layer structure by chosen Haralick's texture descriptors

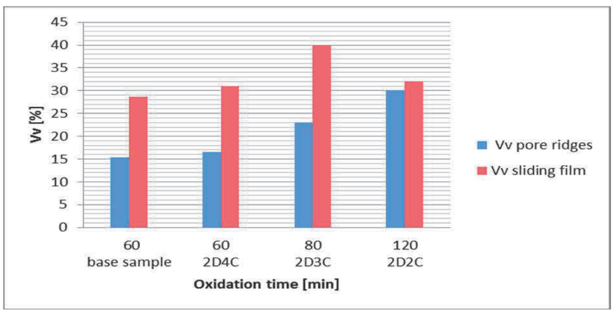


Figure 7 Comparison the surface layer before (pores ridges) and after (sliding film) tribology test

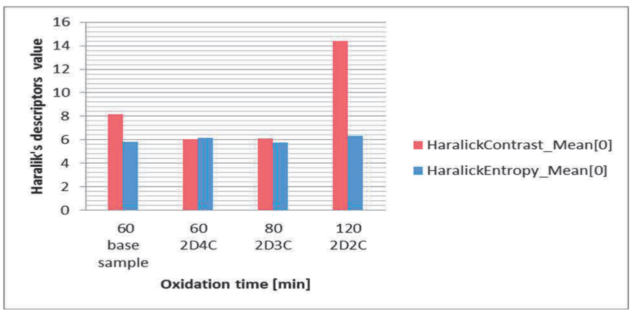


Figure 8 Description the surface layer structure by chosen Haralick's texture descriptors

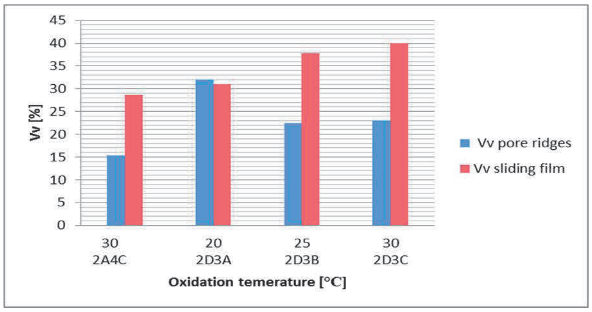


Figure 9 Comparison the surface layer before (pores ridges) and after (sliding film) tribology test

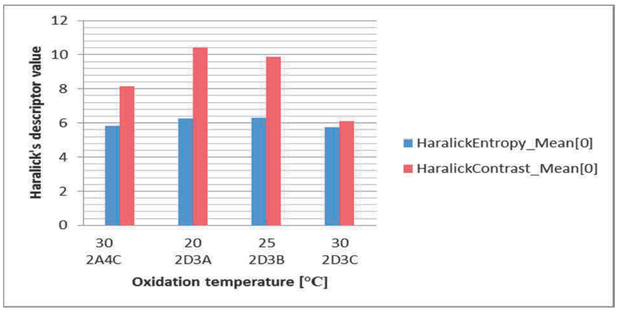


Figure 10 Description the surface layer structure by chosen Haralick's texture descriptors

The authors belived that further texture comparision could be also of help to determine degree of tribological wear of counter body and hence nature of friction process. In **Figures 5** and **7** the comparisons of the surface layer before ("pores ridges") and after ("sliding film") tribology test for samples obtained in different temperature are shown. The main difference in technological process between samples presented in **Figure 5** (2D4A, 2D4B, 2D4C) and in **Figure 7** (2D3A, 2D3B, 2D3C) is time of oxidation. For first group of samples time was 60 minuts for the second one 80 minutes. As is visible for each of samples (exeption 2D3A sample) the same trends of Vv parameters have been seen - the Vv "sliding film" is higher then Vv pores ridges. The same tendency of Vv parameters have been seen for samples in **Figure 9**. This may be due to the fact that in computer image analysis it is easier to determine the volume of craters on the surface after the tribological test than before the test. This notice is also consistent with the observations generated by computer analysis of the areas in **Figures 2** and **3**. From the **Figures 5** and **7** it is also possible to determine volumetric contribution of sliding film, which has been tamped / inserted into the craters of the structure during the friction process. From the practical point of view it could be usful also for indicate the volume share of containers e.g. for solid lubricants on surface [17]. The presented observations for 2D4B, 2D4C, 2D3B and 2D3C samples are according with the results obtained by authors [16]. In the citated research the wear intensity of counter body was higer for 2D4C and 2D3C compare with 2D4B and 2D3B, respectively. That results are accoring with bar charts presented for mentioned samples (**Figures 5** and **7**) in this article - for a higher oxidation temperature the Vv sliding film on surface increased, as well (as was shown in [17]) as incresed the wear intesity of counter body. In **Figures 6, 8** and **10** the descriptions the surface layer structure by chosen Haralick's texture descriptors on images x500 were shown. The values of that parameters indicate the comparability of the presented surface textures.

5. CONCLUSION

Objectification of the analysis the results of scientific experiments, as well production quality control are crucial for verifying hypothesis and seeking the failure in actual procedures. Quantitative analysis of material structure applying image processing techniques and measurements algorithms is essential tool to evaluation as it was presented in this paper. Texture description carried out by two methods - structural, which consider the detected features of both type of surface layer, and statistical, by Haralick's descriptors, confirmed each other, as well as the hypothesis of the authors presented in previous papers [16-18]. Further investigation should include additional image analysis techniques especially 3D [19] possible to acquire from a computer micro-tomography with a specific preparation [20, 21]. Finally, the process of image analysis, even automated, need to include a human factor to select objects and this fact and possible related human errors should be considered during a further analysis [22]. It unavoidable will lead analytical techniques to the fuzzy approach [23-24]. In summary, the proposed approach of surface layer analysis as textures will yield satisfactory results and will be further developed by the authors.

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