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APPLICATION OF THE IMAGE ANALYSIS METHODS FOR THE STUDY OF Al_2O_3 SURAFCE COATINGS

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Abstract: Anodic oxidation of aluminum alloy in a ternary solution of SFS (sulfuric, phthalic and oxalic acids) with 30g of tungsten disulfide (WS_2) powder was carried out in order to obtain the aluminum oxide layer named in article as Al_2O_3/WS_2 coatings. The microstructures of surface of obtained coatings were investigated. The subject of the analysis was a threadlike hill structure visible on the surface of coatings as bright ridges. The computer image analysis was performed in order to study the influence of electrolysis time and temperature of acids bath on the volume fraction V_V of the area of the surface with filamentous hill's structure.
Keywords: computer image analysis, aluminum oxide layer,

1. INTRODUCTION

Aluminium alloys belong to a group of materials which are widely used in motorization, aircraft or food industry, where light weight or corrosion resistance is required. However, the low hardness and abrasion resistance limited the application of aluminum alloys. Until now various surface treatments, such as ion implantation, anodizing and other physical/chemical vapor deposition methods have been used to improve their tribological and mechanical properties. The effect of surface microstructures of coatings formed on aluminum alloy on the tribological properties usually is studied based on micropores and grooves occurring on the surface (Lu at al., 2019). As the authors notices that during the dry sliding process, the soft PTFE particle located into the micropores and grooves as lubricants reduced the friction coefficient evidently and the hard “ridges” of PEO coating increased the anti-wear ability of the whole coating. Wang et al. described (Wang at al., 2010) the surface of ceramic coating fabricated by MAO process, which was made of island-like structures which had the features of porosity and roughness. As they showed the self-lubricating Al_2O_3/PTFE composite coating possess superior tribological properties. As demonstrated tribological studies, the addition of tungsten disulfide (Korzekwa at al., 2009), molybdenum (Kmita and

Skoneczny, 2008) or graphite (Skoneczny and Bara, 2008) to the Al_2O_3 layers causes the reduction of negative friction effects.

The presented work has been devoted to modify the Al_2O_3 layer by WS_2. These results represent the characteristics of SEM studies of Al_2O_3/WS_2 coatings concerning “threadlike hills” observed on top surface layer. The authors assume that quantitative description of threadlike form could be valuable for quality production improvement of Al_2O_3/WS_2 coatings.

2. EXPERIMENTAL

2.1. SAMPLE PREPARATION

The starting material for the process was EN-AW-5251 aluminum alloy. Samples 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 sulfuric, phthalic and oxalic acids with 30g of tungsten disulfide WS_2 powder (Aldrich – Sigma, grain size < 2 μm) per 1 liter of electrolyte. In order to ensure homogeneity of the suspension and to prevent settling of the WS_2 powder, mechanical stirring was applied during the electrolysis process. The hard anodizing process was performed at constant electric charge density of 240 A·min/dm^2. The details concerning the serial number of samples, time and temperature of anodizing process are presented in **Table 1**.

Table 1

The conditions of coatings production

Designation of samples	Temperature of bath [°C]	Time of anodizing process [min]
3A	20	80
3B	25	80
4A	20	60
4B	25	60

2.2. METHODOLOGICAL BASES

In order to analyze the surface of Al2O3/WS_2 coatings research was conveyed with Philips X130 scanning electron microscope. The samples were sprayed with gold. Images were saved in the BMP. The size of the images: 2576x1936 pixels, where 1 pixel was equal to 0,0048 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 the preprocessing of the images had to be done. The analysis was performed according to the order: shade effect correction, contrast enhancement, detection of the threadlike hills and the analysis of the threadlike hill’s skeletons of the initial images [Gadek-Moszczak et al., 2019].

3. RESULTS AND DISCUSSION

The SEM image of the microstructure of the 3A surface layer with marked threadlike hills was shown in **Figure 1**. The examples SEM images of the microstructure of the 3A

(Fig. 2a) and 4A (Fig. 2b) surface layer which were taken into consideration were shown.

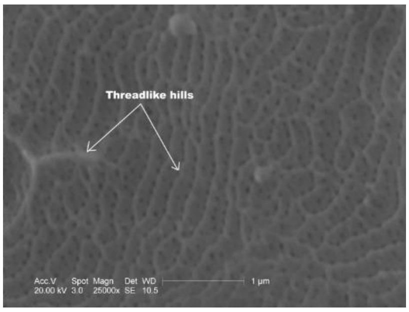

Fig. 1. SEM image of the microstructure of 3A surface layers

Detection of the threadlike hills was presented in Fig. 2c for 3A sample and in Fig. 2d for 4A sample. This kind of detection was used to calculate area fraction A_A parameter which characterized the relative surface of threadlike hills as a part of the entire analyzed area. Area fraction (A_A) delivers general information about the per cent of the area which represents analyzed structure. The highest A_A parameter has the samples 3A which was obtained at the lowest bath temperature equal 20 ^oC. Reducing of anodizing time caused the slightly reduction of A_A parameter. It could be also notice that increasing the bath temperature had also influence on reduction of A_A parameter. Result of the skeletonization is presented in Fig. 2e for 3A sample and in Fig. 2d for 4A sample. Skeleton consists of intersection points and lines which are called branches. The compilation of those parameters allows demonstrating the quantitative differences of the examined structures (Table 2).

Table 2
The results of computer image analysis

Designation of samples	**A_A parameter [%]**	**Number of intersection points/area [1/ µm^2]**	**Number of branches/area [1/ µm^2]**
3A	44	96.17	270,36
3B	38	164.18	327.17
4A	34	73.37	132.69
4B	31	84.69	114.29

The results of the image analysis are presented in Fig. 3a and Fig. 3b. The more number of intersection points and the more branches are in the analyzed object, the more complex shape it has. Analyzing those parameters we can state that there is a difference in the number of threadlike hills in the samples. The rising of time with the same anodizing temperature affected decreasing the number of branches and intersection points per area. Comparing the number of interaction points per area for analyzed samples it could be observed that its numbers increasing for the samples obtained in higher temperature with the same time of anodizing. Similar behavior is observed for the number of branches per area but just for samples 3A and 3B. Trying to link the obtained results of computer image analysis with tribological research, the authors reached for research (Korzekwa J., 2012) where friction coefficient was analyzed. In the cited literature the sample 3B and 4B are characterized by friction coefficient equal μ=0,16 and μ=0,19 respectively. Combine the results of computer image analysis with referenced results one can observed that slightly reduction of A_A parameter and decreasing the number of branches and intersection points per area had influence on value of friction of coefficient. For farther evaluation more examples should be done. Nevertheless the authors assume that quantitative description of threadlike form could be valuable for quality production improvement of Al_2O_3/WS_2 coatings.

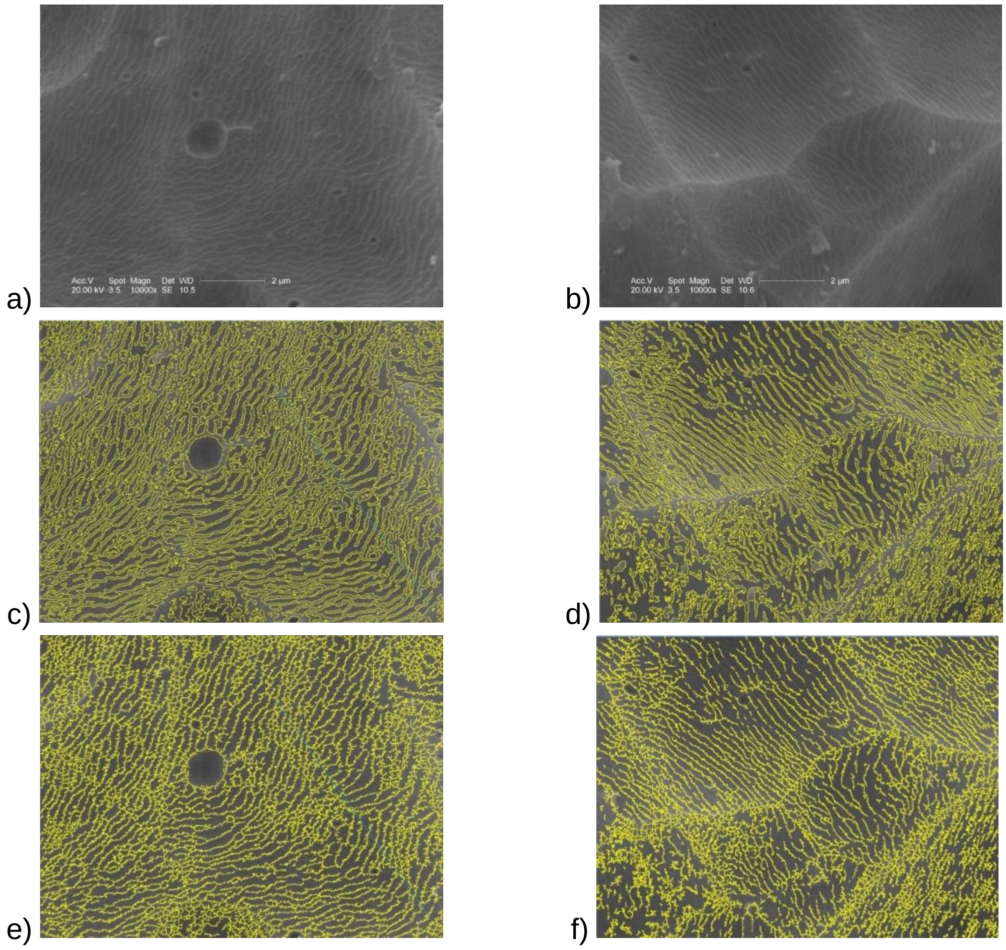

Fig. 2. SEM images of the microstructure of the 3A (Fig. 2a) and 4A (Fig. 2b) surface layer, result of detection the threadlike hills on the images - 3A (Fig. 2c) and 4A (Fig. 2d), result of the skeletonizing detected threadlike hills - 3A (Fig. 2e) and 4A (Fig. 2e)

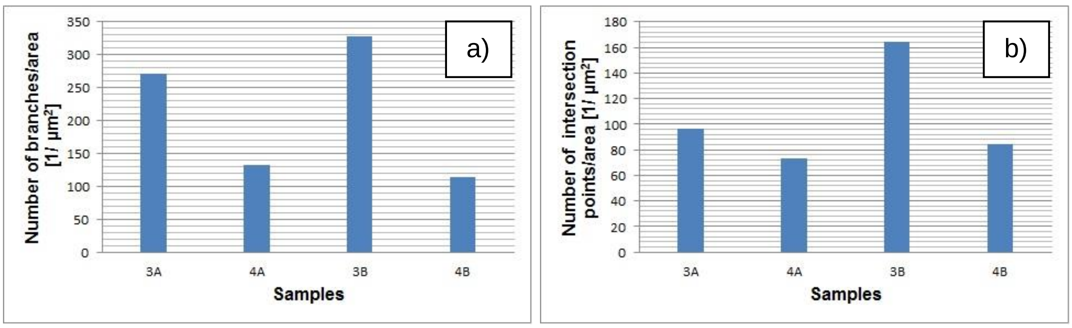

Fig. 3. Number of branches per area (Fig. 3a)
and number of intersection points per area (Fig. 3b) for analyzed samples.

4. CONCLUSION

Computer image analysis included detection of the threadlike hills and the analysis of the threadlike hill's skeletons of the initial images of Al2O3/WS_2 coatings were done. Quantitative analysis of the structure comprised the area fraction A_A parameter and the number of branches and intersection points per area can extend the typical stereological parameters of Al_2O_3/WS_2 coatings. Moreover, combine the results of computer image analysis with the results from tribological measurement could be valuable for quality production improvement of Al_2O_3/WS_2 coatings. Farther evaluation of computer image analysis connected with tribological estimation could be promising method used to characterize tribological properties of Al_2O_3/WS_2 coatings even before tribological cooperation.

Obtained results may be interested for other researchers focused on coatings and surface layer modifications (Skoneczny and Bara, 2007; Bara et al., 2009; Kmita and Bara, 2012; Skoneczny et al., 2018; Bara et al., 2019), even in the combustion technology (Opydo et al., 2016). They may be also inspiring for related areas of materials science (Lipinski, 2015; Lipinski 2018; Szczotok et al., 2018), heavy-duty machines design (Fabis-Domagala et al., 2018) and experimental design methodology (Pietraszek and Goroshko, 2014; Pietraszek et al., 2016), especially with the uncertainty quantification (Pietraszek et al., 2017) and fuzzy logic decision scheme (Filo et al., 2018; Krawczyk and Sobczyk, 2018). The obtained data should be disseminated in scientific databases e.g. (Gawlik et al., 2015).

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