1. Introduction

Surface treatment, like anodising of aluminium is a very popular technique, using for obtaining of porous structures [1, 2]. The usability of aluminium after anodising depends mainly on the properties of oxide layers occurred during this process. Such a great interest in surface layer technologies occurs by the fact that every year the world economy losses milliards of dollars, attributable to the product damage due to surface layer degradation [3, 4].

This method changes the texture of the surface structure as well as changes the crystal structure of the metal near the surface [5]. The obtained coatings are normally thick and porous, so a sealing process is often needed to enhance the corrosion resistance [6]. The anodized surface is harder than aluminium but have only moderate wear resistance, which can be improved by applying of suitable sealing substances. Anodic films are normally much stronger and more adherent compared to types of paint and metal plating, they are unfortunately also more brittle, what makes them less likely to crack and less susceptible for aging and wear, but easier to cracking when applied thermal stress [7].

Anodic oxidation processes of aluminium are currently being developed mainly in order to increase competitiveness in the automobile, electronics, and other industries [7]. A strong competition for aluminium anodic oxidation process are the painting technologies (mainly powder spraying). However, one of still unsolved problems is the occurrence of filiform corrosion on the coated aluminium surface [8]. Moreover, taking into account the recycling ability of aluminium products with oxide coating, it can be concluded that oxidation/anodization remains the leading method of surface finishing of aluminium [9].

Colouring of oxide coatings is performed during the anodizing process (one step process) or in a subsequent operation after a clear coating with appropriate porosity is obtained (two-stage process) [10, 11].

In the industry, both civilian and military there are used light metal alloys, differing in mechanical properties and corrosion resistance. Each of technical aluminium alloys require modifying of the surface treatment methods and the optimization of chemical processes. Based on the morphology analysis of the surface there can be found, that it is possible to obtain layers with more or less developed porosity. It depends mainly on the electrolytic process parameters [12]:

- current density (process rate),
- temperature of the electrolyte (granularity),
- process duration.

Despite the assumptions and model presentation of the layer with regular pores, it was found that the columns of the aluminium oxide layer, or even their groups are bent and directed at different angles. This phenomenon may be due to the presence of impurities in the metal substrate.
During the anodizing process, additions are coming from the oxide film, causing a deviation from the columnar structure [13]. In the case of the investigated material the phenomenon described above is possible to occur. The aim of this work is mainly to describe the influence of anodising condition, its influence on the obtained layer thickness, uniformity texture as well as wear resistance.

2. Material for investigations

Investigations were performed on the AlSi9Cu3 alloy which was adopted to the casting process and anodic treatment. Alloy AlSi9Cu3 was cast using high pressure casting and sand casting. The chemical composition of the alloy is presented in Table 1.

| Chemical composition of the AlSi9Cu3(Fe) alloy, wt. % |
|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| Si                | Mg                | Cu                | Mn                | Fe                | Zn                | Al                |
| 9.5               | 1.5               | 3.0               | 0.5               | 0.9               | 0.5               | rest              |

Metallographic investigations were carried out on a light microscope equipped with a digital camera. Recording and processing of images was made on a PC using the dedicated software analySIS.

The aluminium alloy samples were subjected to the anodizing process in a sulphuric acid electrolyte containing H₂SO₄. The conditions and parameters of the electrolytic process are presented in Table 2, the anodization process was carried out in a glass vessel where the anode was connected to the sample and cathode was applied in form of titanium sheet.

| Parameter                                      | Value                                      |
|-----------------------------------------------|--------------------------------------------|
| Electrolyte                                   | H₂SO₄ with a concentration of 315 g/l       |
| Temperature                                   | – 4 ± 2 °C                                  |
| Pulsating current                            | 2 A/dm² during 0.25 s. 1 A/dm² during 0.1 s |
| Concentration of the aluminium ions           | 6 ÷ 9 g/l                                   |

Analysis of surface geometry was based on data obtained from the measurement of selected areas of the castings, performed on a laser profilometer. Measurements were carried out on four materials divided into two groups. The first is the initial material, in as cast state immediately after casting without any surface treatment. The second group was the material after the production of the oxide layer using the galvanic method. The AlSi9Cu3 alloy was cast by two methods: pressure cast and sand cast.

Hardness measurements were performed on an automatic hardness tester, using a penetrator in the form of sintered carbide ball with a diameter of 1/16 inch.

Microstructure investigations were made using light microscope equipped with an electronic camera configured with a computer as well as using atomic force microscope (AFM) for surface structure analysis working in the NC-AFM topography mode on an area of 20µm x 20µm using a scan rate of 0.8 Hz.

Abrasive wear tests as well as the profile measurement were conducted according to the specification and requirements of the standard ISO 8251. A load of 4.9 N was applied, at a slip velocity of 40 cycles/min. The test surface area was 12 x 30 mm. The grindability tests were performed at temperature of 23°C, humidity of 63%. The test was repeated 5 times.

3. Investigation results

3.1. Wear resistance test results

As a result of the performed wear test, it was found that anodization reduces wear resistance (Fig.1). The best wear resistance was achieved when the thickness of the anode layer is relatively high (approximately 48 µm). Partial removing of the coating was observed for all high pressure casts, where the coating thickness is lower (about 10 µm). Sand cast samples show increased weight loss which is caused by the fact that the test area is close to the electrode mounting (the layer is at this point is probably thinner). Analysing the test results presented in Table 3, there can be found that for the high pressure cast the anodized samples have about half the mass loss compared to the non-anodised sample. For sand cast the measured reduction in weight loss is equal 36%.

| Sample designation | Minimal thickness. µm | Maximal thickness. µm | Average thickness. µm | σ |
|--------------------|-----------------------|-----------------------|------------------------|---|
| Sand cast          | 26.1                  | 108.3                 | 65                     | 32.7 |
| High pressure cast | 65                    | 207.5                 | 136.25                 | 27.95 |
3.2. Light microscope investigation results

Metallographic investigations were performed on the light microscope Leica MEF-4A. There was measured the thickness of the oxide layer in various areas of each of the samples (Fig. 2). The results of measurements, statistical analysis, are presented in Table 3. On the basis of the thickness measurements of the anodized layer, it was found that the thickness of the anodized layer formed is higher in the case of the sand cast material, and lower for high pressure cast material measured under the same anodizing parameters.

3.3. Hardness measurement results

Based on the obtained hardness measurement results it was found, that in case of the sand cast alloy, the average hardness of the non-anodised alloy is 10 HRF and for the anodized is alloy 11 HRF (Table 4). The difference between these values is caused by the measurement error, so it can be state, that for the sand cast alloys the occurrence of anodic layer does not change the hardness. While the average hardness for the non-anodised high pressure cast alloy die-cast is 9 HRF and for the anodized - 14 HRF. Therefore it was found an increase of the hardness after anodizing by 46%. Furthermore, the standard deviation of the hardness measurement for anodized alloy is lower (3.77) than the non-anodised alloy (6.165).

3.4. Stereological microscope surface evaluation

On Figures 3 to 8 are presented results of the Al₂O₃ layer surface investigations, made on the abrasive wear test device.

3.5. Extent investigations

Comparing the images of the surface structure (two- and three-dimensional), and analysing the various schedules of roughness it can be unequivocally stated, that the chemical composition of the tested samples has no impact on the formation of the surface (Fig. 3, Fig. 4, Fig. 6, Fig. 7). This result repeats for both samples coated with Al₂O₃ alumina layers as well as for the material in the initial state.

![Fig. 3. Geometric shape of the test surface, colour intensity map (left), numeric photograph of the sample surface (right), sand cast alloy](image)

![Fig. 4. Selected 2D profiles cut from the test surface: a) in the W-E plane, b) in the N-S plane, sand cast alloy](image)

The differences in the surface geometry of the tested samples significantly depend on the method of casting. For the sand cast material, the surface structure is similar for the uncoated and coated material. It is characterized by a significant roughness. The roughness reaches a maximum value of up to 80 µm. Analysing the 3D image there can be observed “island” of regularly distributed hills on the surface (Fig. 5, Fig. 8).
Comparing the distance between “peaks” should be treated as mapping of surface roughness of the sand mould. Surface shape retains the characteristic features, even after coating. In other words, applying a coating does not change the characteristics of the surface geometry.

The carried out studies of the surface geometry allow the conclusion that the determining factor for the studied group material for the quality of the surface and their geometric features, is the casting technique (Fig. 9, Fig. 10).

3.6. Surface AFM investigations

Comparing the three-dimensional NC-AFM images of the surface structure and analysing the given roughness scale in the range up to 5 µm it was found, that the observed roughness of the tested samples is higher for the non-anodised material reaching even 4.4 µm, whereas for the anodised material it was measured up to 2.3 µm. So the anodized alumina surface reveals better quality of the obtained surface with higher smoothness compared to the naturally grove layer (Fig. 11).

3.7. Layer thickness measurement

The layer thickness measurements results were presented on Fig. 12 for the anodised and non-anodised samples. It can be recognised that the sand cast treated material reveal a thicker surface layer after anodisation reaching 72 µm, compare to 66 µm high pressure cast. Whereas for the non-anodised material the difference is negligible reaching 3 µm for the sand cast alloy.
4. Conclusions

The reported investigation results allows unequivocally to determine, which of the tested surfaces in combination with an appropriate manufacturing method (pressure or sand casting) has the highest abrasion resistance. The best results should give a combination of the sand cast AlSi9Cu3 alloy.

The anodized alloy surface show less weight loss compared to non-anodised material. It can be seen that the casting method affects the abrasion resistance; sand cast alloys exhibit less weight loss during the performed test, so it have a higher abrasion resistance.

Based on the hardness measurement results, it can be concluded, that the samples after the anodization process have higher hardness compared to those that have not been subjected to this process. After anodizing its hardness has increased to 14 HRF.

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REFERENCES

[1] P.G. Sheasby, R.Ch. Pinner, The Surface Treatment and Finishing of Aluminum and its Alloys, Materials Park, Ohio & Stevenage, UK: ASM International & Finishing Publications 2001
[2] H. Adelkhani, H. Forati Rad, Iranian Journal of Surface and Engineering 16, 9-17 (2013)
[3] J.G. Castaño, F. Echeverría, Ingeniería & Desarrollo. Universidad del Norte. 28 1-14 (2010)
[4] T. Araújo, Faculdade de Engenharia da Universidade do Porto 2012
[5] M. Michalska-Domańska, M. Norek, W.J. Stepniowski, B. Budner, Electrochim Acta. 105, 424-432 (2013)
[6] J. Konieczny, L.A. Dobrzanski, K. Labisz, J. Duszczyk, J Mater Process Tech. SPEC. ISS. 718-723 (2004)
[7] Y. Goueffon, L. Arurault, C. Mabru, C. Tonon, P. Guigue, J Mater Process Tech. 209, 5145–5151 (2009)
[8] M.B. Spoelstra, A.J. Bosch, D.H. Van Der Weijde, J.H.W. De Wit, Mater Corros. 51 (3), 155-160 (2000)
[9] J.M. Montero-Moreno, M. Sarret, C. Müller, Surf Coat Tech. 201, 6352–6357 (2007)
[10] L. Kwiatkowski, P. Tomassi, Surface Eng. 3, 39-48 (2006)
[11] P. Tomassi, Steel Construction (in polish), Special ed. 30-31 (1998)
[12] W. Skoneczny, Surface Eng. 2, 21 – 26 (2000), in polish
[13] F. Ishigure, S. Inayoshi, J of the Vacuum Soci of Japan. 58 (12), 437-441 (2015)
