14th World Congress in Computational Mechanics (WCCM)
ECCOMAS Congress 2020
Virtual Congress: 11 – 15 January 2021
F. Chinesta, R. Abgrall, O. Allix and M. Kaliske (Eds)

SAMPLE PREPARATION METHODOLOGY OF THE AL_2O_3 SURFACE LAYERS FOR SELF-LUBRICATING SLIDING PAIR

JOANNA KORZEKWA[1], MAREK BARA[1] AND DARIUSZ KARPISZ[2]

[1] Faculty of Science and Technology, Institute of Materials Engineering,
University of Silesia in Katowice
75 Pułku Piechoty 1a, 41–500 Chorzów, Poland
joanna.korzekwa@us.edu.pl (J.K), marek.bara@us.edu.pl (M.B)

[2] Cracow University of Technology, Faculty of Mechanical Engineering
Al. Jana Pawla II 37, 31-864 Krakow, Poland, EU
dariusz.karpisz@pk.edu.pl

Key words: structure of aluminum alloy, anodizing, etching, sample preparation

Abstract. The article discusses the process of preparing samples with an anodic Al_2O_3 layer for tribological tests in the combination of sliding, reciprocating on the T17 tester. The method of cutting, grinding, drilling, threading, gluing, etching, and anodizing samples used in laboratory conditions was presented. As shown in the article, the number of factors influencing the surface quality of the oxide layer produced in terms of tribological associations is significant; therefore, the appropriate sample preparation methodology is so important.

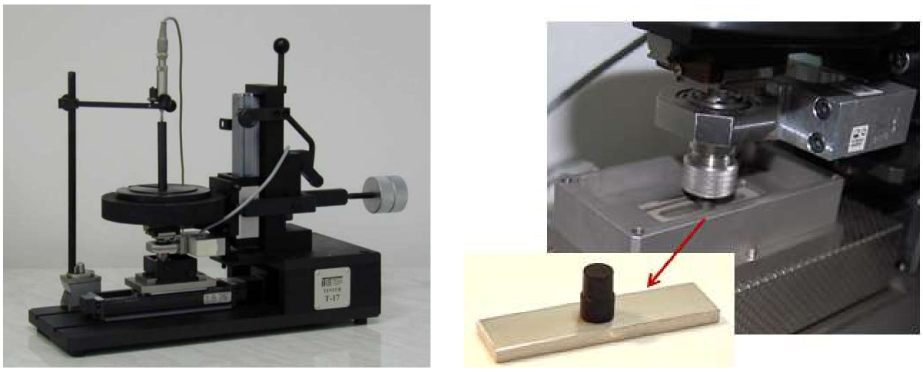
1 INTRODUCTION

Increasing the mechanical properties of aluminum is made by fusing metal with alloying elements to form alloy aluminum, which is even several times more durable while maintaining good plastic properties. The most commonly used alloy additives in aluminum alloys are Cu, Fe, Si, Mg, Mn, Zn. These elements affect in various ways the strength properties of pure aluminum. Some of the aluminum alloys have favorable constructional parameters. The ratio of strength to their specific weight is much greater than, e.g., for steel. This fact largely determines the wide use of alloys aluminum in the industry. Improvement of aluminum's surface properties, e.g., increasing the corrosion or abrasion resistance and hardness, can be obtained by producing aluminum oxide layers in an electrolysis process called anodizing. Anodizing of aluminum alloys leads to the formation of an oxide layer on their surface, due to which the aluminum gains resistance to abrasion. The coatings obtained by this method are widely used in sliding connections of kinematic machine parts. Examples of elements made of anodized aluminum alloys are internal surfaces of cylinders of pneumatic actuators, shock absorbers and compressors, surfaces of pistons of combustion engines, and profiles of sliding door guides. The selection of materials for cooperating tribological elements should be based on preliminary tests carried out in conditions that are closest to real ones. Conducting trials in laboratory conditions often requires special preparation of the test material that allows for testing on a specific stand. Before anodizing, it is necessary to pre-treat or clean the surface of aluminum appropriately. The typical types of surface treatment for aluminum alloys before anodizing are degreasing [1], [2], electroplating [3], [4], mechanical treatment [5], chemical brightening [6]. The article presents the method of preparing samples with an oxide layer intended for tribological tests on the T17 stand, manufactured at the Institute of Materials Science, University of Silesia.

2 SAMPLE PREPARATION METHODOLOGY

2.1 Test stand

Tribological tests carried out on stands whose friction node reflects the real association most accurately constitute the basis for inferring about cooperating materials' tribological properties under given operating conditions. For example, materials for the pneumatic actuator's sliding elements should be tested on the T17 tribotester (produced by Łukasiewicz Institute for Sustainable Technology in Radom), where the tester's reciprocating sliding movement is consistent with the type of motion of the actuator piston rod – Fig.1a. The T-17 tester enables testing according to the American Society for Testing and Materials F 732 standard requirements. Before the test run, the frequency and amplitude of the cycles and the friction junction load are set on the T-17 device. The friction node consists of a stationary pin pressed against the plate, making a reciprocating movement. The pin is fixed in a holder, which is connected to a force sensor –Fig.1 b.



(a) **(b)**

Figure 1: *An example of the correct selection of the T 17 tribological tester for testing the sliding connection of pneumatic actuator elements* ***(a)****. Friction node of T-17 tester (b).*

2.2 Sample preparation

The material for tribological tests is plates cut from a 4 mm thick rolled sheet from various aluminum alloys (e.g., EN AW 5251). Plates with 62.5 x 16 mm dimensions are cut on a milling plotter or water jet cutting machines – Fig.2.[7] Plates are cut in external companies. Plates for tribological tests must maintain surface parallelism. Failure to keep this parallelism causes erroneous loading of samples and cooperation of elements on the surface.

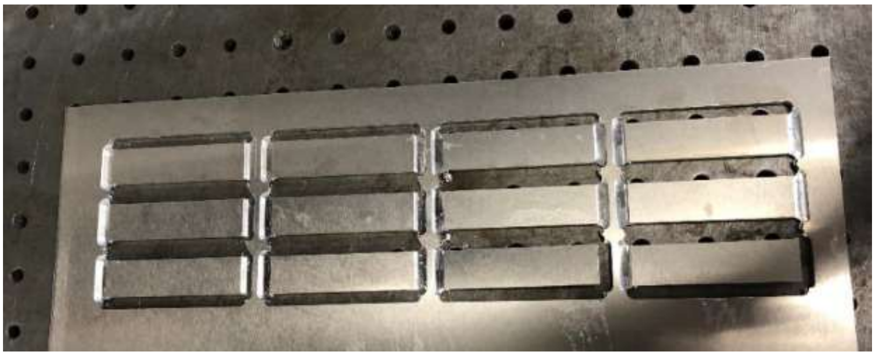


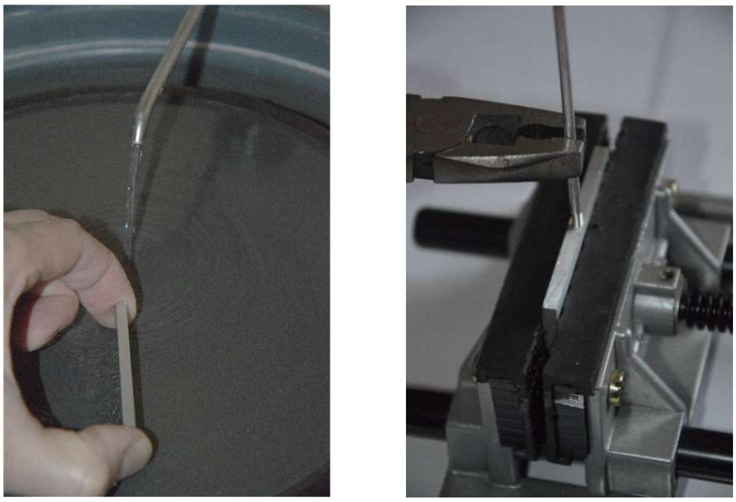
Figure 2: *Plates cut out from aluminum alloy* [7]

Machine-cut plates are drilled in the central part of the long side of the sample with a drill of ⌀2.4 mm in diameter to a depth of 10 mm –Fig.3a, and then the drilled hole is threaded with M3 mm taps – Fig.3b. The M3 die is also used to thread an aluminum rod made of the same aluminum alloy grade as the sample – Fig.3c. The threading process is carried out using fluids that reduce the tool's effect sticking to the inserted material.

(a) (b) (c)

Figure 3: *Drilling of sample (a), threading of sample (b), threading of rod (c)*

All surfaces previously milled or cut with a water jet are sanded on a polisher with water-based sandpaper, 400 grit, to obtain a smooth surface – Fig.4a. After pre-washing and degreasing the plates' surface and the rod with ethyl acetate, the plate is connected to the rod by twisting the threaded elements until a slight resistance is obtained –Fig.4b.



(a) **(b)**

Figure 4: *Grinding of the surface plate (a), connecting of plate and rod (b)*

To reduce the amount of heat generated on the aluminum plate during the electrochemical process, all plate surfaces that do not take part in the slip test and the rod attached to the plate are coated with a distal classic two-component epoxy adhesive - Fig. 5a. The taped tiles are left to dry for a minimum of 48 hours. After this time, the adhesive is resistant to acids, which are components of electrolytes. When in contact with the atmosphere, the surface of the aluminum and its alloys is spontaneously covered by the passivating layer of aluminum oxide. This thin oxide layer (0.001-0.1 μm) isolates the metal from contact with the environment. Therefore, before the anodizing process, this layer must be removed using various acids or bases [8]. In our research, the etching is carried out in 5% potassium hydroxide solution for 40 minutes and then neutralized in 10% nitric acid solution to reverse the etching reaction – Fig.5b. Etching and neutralization treatments end with rinsing in distilled water.

(a) **(b)**

Figure 5: *The plate surfaces covered with classic distal glue (a), the etching and neutralization of plates (b)*

Fig. 6 shows the roughness values of the aluminum alloy substrate surface for five samples. Measurements were made in the horizontal and vertical positions of the samples. For most samples, the roughness measured in the horizontal direction has higher values than that measured vertically. This difference is caused by the surface of aluminum related to heat-plastic treatment (sheet rolling). Higher roughness values are obtained for a direction that is perpendicular to the rolling direction. The exception is sample No. 4, the value of the average roughness of which could have been affected by sheet scratches. Fig. 7 shows the roughness values for the etched aluminum alloy substrate surface for five samples. The surface roughness values after etching are higher due to the action of chemicals on the alloy surface.

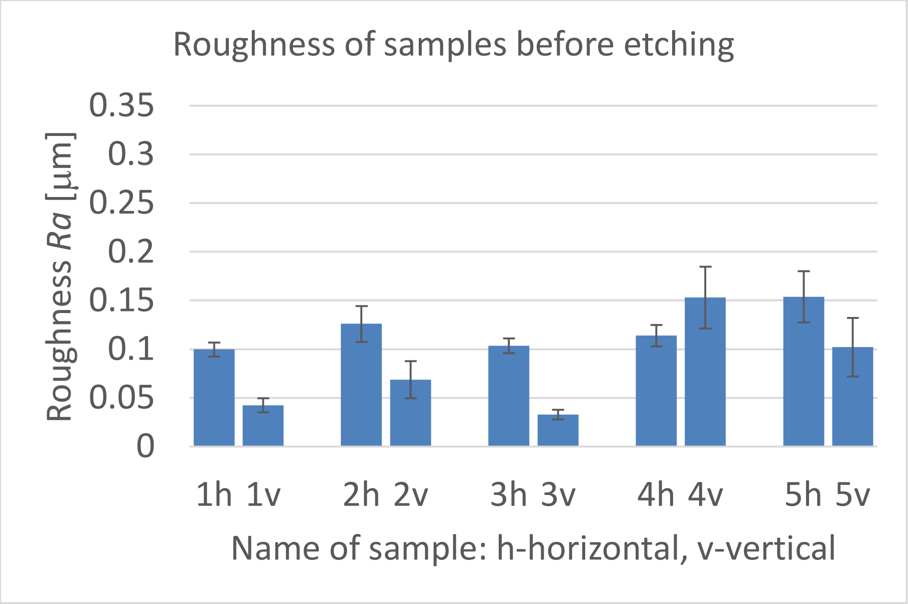


Figure 6: *Roughness of samples before etching.*

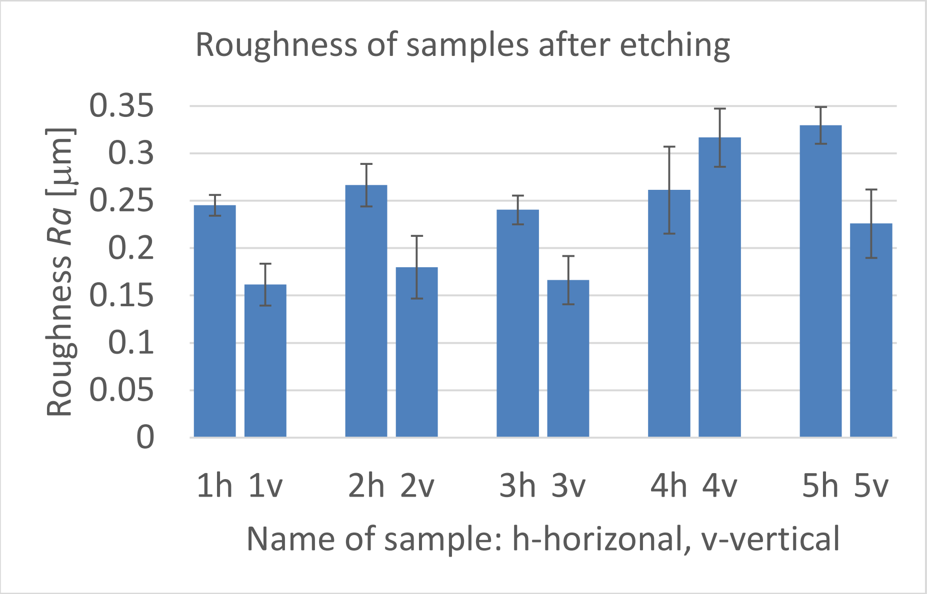
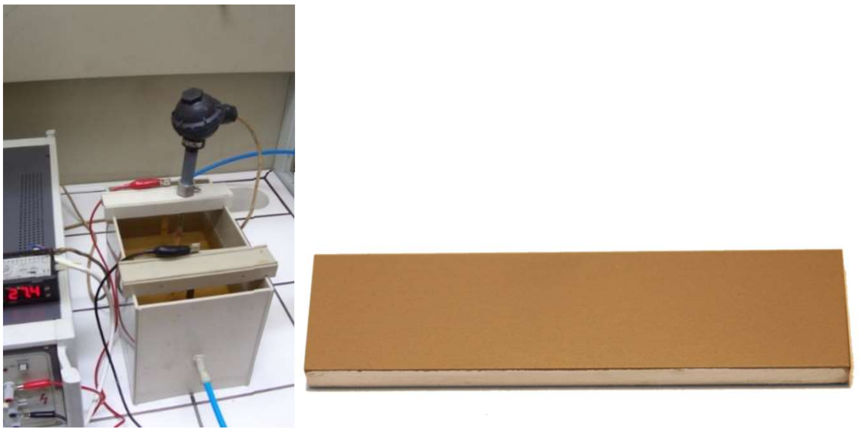


Figure 7: *Roughness of samples after etching.*

An essential element in preparing samples is the appropriate selection of the material in terms of texture after rolling and roughness after etching. The influence of these processes on the substrate's properties is inherited in the anodizing process of aluminum alloy [9], which in its consistency may contribute to disturbances in tribological tests.

2.3 Anodizing of samples

Anodizing is performed by the direct current [10] or the impulse method [11] using the laboratory power supply – Fig. 8a. Anodizing carried out in oxalic or sulfuric acid requires intensive cooling of the electrolyte, which increases the cost of the process. In order to avoid intensive cooling, electrolytes with other acids are used in the composition [12]. Hard anodizing requires cooling the electrolyte, which is passed through an automatic temperature control system. The heat release in the layer growth area results from aluminum's exothermic reaction with acid and the so-called Joule's warmth. Too high electrolyte temperature in the oxide layer growth area and too shallow immersion of the sample in the electrolyte increases the acid's ability to redissolve the oxide and reduces its surface hardness. Such samples are not suitable for further research. For this reason, a rod (Fig. 5a) is attached to the test plates, allowing the sample to be placed at the appropriate depth in the electrolyte. After the anodizing process, the plate with the oxide layer formed is rinsed in distilled water to rinse the electrolyte from its porous structure. Thanks to the characteristic structure of the layer and variable properties, the hard, porous aluminum oxide layer (Fig. 8b) can be used in many technology fields.

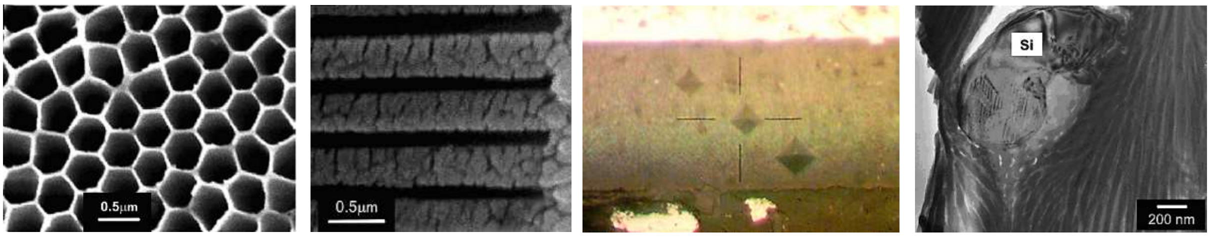


(a) **(b)**

Figure 8: *The Al_2O_3 layer production process (a), the Al_2O_3 surface layers (b)*

2.4 Layer test proposals

The basic properties of the oxide layers produced on the aluminum alloy substrate, such as porosity – Fig.9a [13], structure – Fig. 9b [13], thickness and microhardness – Fig.9c, can vary widely and depend mainly on the current density, electrolyte temperature, anodizing time, and the composition and ph of the electrolyte. An important issue is the selection of an appropriate aluminum alloy for anodizing. In particular, alloys containing 2÷ 5% Mg and 0.1÷ 0.4% Mn, deserve attention (e.g., EN-AW-5251). They are characterized by good corrosion resistance, and they are plastic, weldable, resistant to fatigue, and easy to work smoothly. Additionally, an advantage of these alloys is the low content of intermetallic phases, which increases susceptibility to anodic oxidation, carried out to obtain thick and hard wear-resistant surface layers. Some aluminum alloys are subjected to precipitation hardening, i.e., the process of supersaturation and aging, after which the strength properties are comparable to those of many steel grades. Application for some alloys (e.g., EN-AW-6060), plastic deformation after a period of aging leads to an increased strength by up to 30% compared to precipitation hardening without deformation. The disadvantage of casting alloys used to produce an oxide layer on their substrate is a coarse-grained structure with primary silicon crystals – Fig.9d [14], which leads to lower mechanical properties of the alloy and silicon transition inclusions to the oxide coating structure.

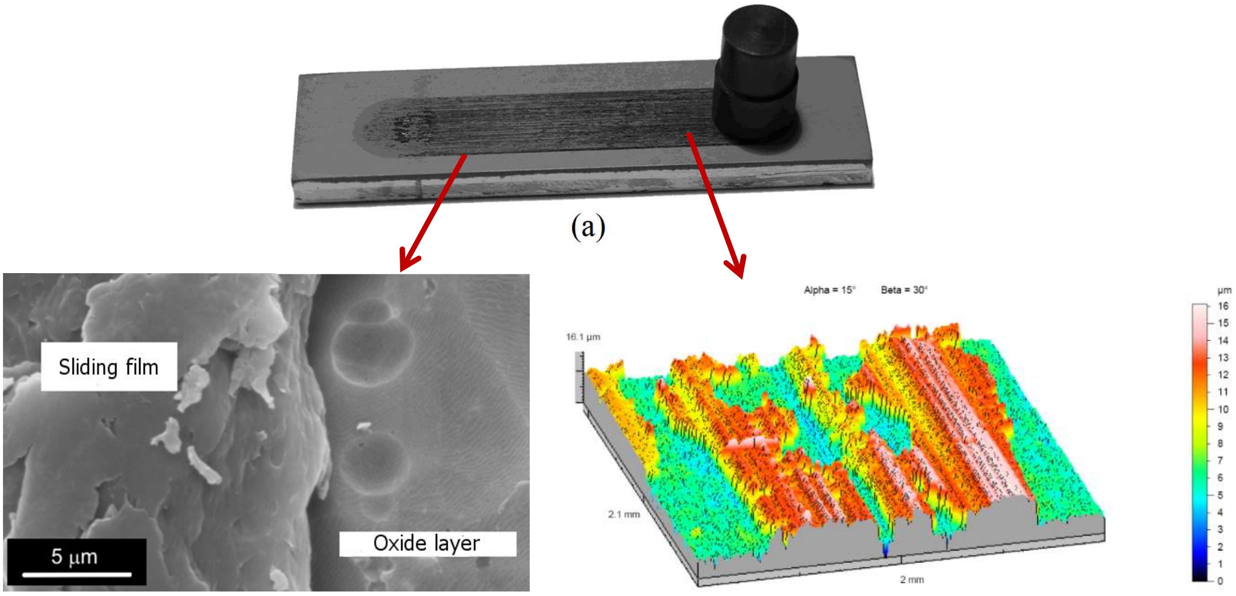


(a) **(b)** **(c)** **(d)**

Figure 9: *The basic properties of the Al_2O_3 oxide layers: porosity (a)*[13], *the parallelism of the fibers (b)*[13], *thickness and microhardness (c), inclusions (d)*[14]

2.5 Tribological test

The sliding test's tribological partner with oxide layers is a pin with a diameter of 9 × 10-3 m, made of different polymers. Polymer pins are made on a CNC lathe from a bar of ø25 mm by machining. As a result of the tribological tests carried out using the above-described material samples, the surfaces with a polymer sliding film are obtained – Fig. 10a. The formation of a polymer sliding film in dry friction sliding associations is closely related to the wear process of the polymeric mandrel and the distribution of the mandrel material on the oxide coatings' surfaces. The sliding film can be analyzed by scanning electron microscopy (Fig. 10b), Energy Dispersive Spectrometer, and profilographometric tests (Fig. 10c). Based on the data measured with the Spider 8 analog-digital converter, which is equipped with the T17 tester, it is possible to plot the friction characteristics, calculate the friction force of the cooperating elements, determine the ambient temperature of the friction junction and the total linear wear of the friction junction elements.



(a)

(b)

(c)

Figure 10: (a) *Al_2O_3 layer with a sliding film, (b) SEM micrograph of Al_2O_3 coating, (c) profilographometric image of the Al_2O_3 layer*

3 CONCLUSIONS

The paper presents sample preparation steps for tribological tests of the sliding connection on the T17 tester. A method of cutting, grinding, drilling, threading, gluing, etching, and anodizing samples used in laboratory conditions was presented. The surfaces of the aluminum alloys on which the oxide layers are formed are usually pre-prepared for the anodizing process by selecting the aluminum alloy, surface texture, type of chemicals, and etching time. An adverse selection of the conditions mentioned above may, to no small extent, have a negative impact on the substrate surface of these layers already before the process of producing the oxide layers. Equally important factors affecting the tribologically tested surface result from the application of appropriate anodizing parameters, i.e., current density, electrolyte temperature, anodizing time, and the composition and ph of the electrolyte. An important parameter during anodizing is the depth of sample immersion in the electrolyte and the electrolyte flow rate, affecting the rate of secondary dissolution of the oxide layer by the acids contained in the electrolyte, and thus its hardness. As shown in the article, the number of factors influencing the surface quality of the oxide layer produced in terms of tribological pairings is significant; therefore, the appropriate sample preparation methodology is so important.

The results presented in this article may be useful in industrial in order to improve the durability of machine parts by strengthening the surface layer [15, 16], modifying its functional properties [17, 18] and wear resistance [19, 20]. It can also be used to improve the techniques enhancing the resistance of parts working in chemically aggressive environments [21, 22], thermomechanically loaded [23] and exposed to biocorrosion [24-26].

For scientific purposes, it provides data sets for the development of analytical methods [27, 28]. The experience gained may be an inspiration in the field of analytical [29-31] and organizational methodologies, providing them with improved information on the durability of parts [32, 33]. It can also be useful when designing the structure of specific industrial databases [34, 35], as well as non-contact shaping techniques [36, 37].

REFERENCES

[1] F. J. Monteiro, M. A. Barbosa, D. R. Gabe, and D. H. Ross, Surface pretreatments of aluminium for electroplating. *Surf. Coatings Technol.* (1988) **35**(3–4): 321-331.

[2] M. Michalska-Domańska, M. Norek, W. J. Stępniowski, and B. Budner, Fabrication of high quality anodic aluminum oxide (AAO) on low purity aluminum – A comparative study with the AAO produced on high purity aluminum. *Electrochim. Acta* (2013) **105**: 424-432.

[3] A. E. Kozhukhova, S. P. du Preez, and D. G. Bessarabov. Preparation of anodized aluminium oxide at high temperatures using low purity aluminium (Al6082). *Surf. Coatings Technol.* (2019) **378**: art. 124970.

[4] E. Feschet-Chassot, P. Chennell, R. Cueff, B. Mailhot-Jensen, and V. Sautou. Anodic alumina oxide surfaces prepared by dual hard and mild anodization at subzero temperature: Surface microscopic characterization and influence on wettability. *Surfaces and Interfaces* (2020) **19**: art. 100473.

[5] U. Tiringer, J. Kovač, and I. Milošev. Effects of mechanical and chemical pre-treatments on the morphology and composition of surfaces of aluminium alloys 7075-T6 and 2024-T3. *Corros. Sci.* (2017) **119**: 46–59.

[6] B. Chatterjee. Chemical brightening of aluminium. *Mater. Chem. Phys.* (1984) **10**(4): 357-364.

[7] D. Pancerz. *Analysis of the repeatability of tribological tests carried out on a modified holder of the T-17 tester in a roller-plate combination*. University of Silesia in Katowice, 2020.

[8] M. Bara. An estimation of the influence of the substrate preparation on tribological properties of nanoceramic oxide layers. *Tribologia* (2014) **4**(32): 9-20.

[9] M. Bara and M. Kubica. Influence of substrate preparation on the shaping of the topography of the surface of nanoceramic oxide layers. *Appl. Surf. Sci.* (2013) **293**: 306-311.

[10] W. Skoneczny. Effect of the base material condition on the structure and properties of Al2O3 oxide layers. *Indian J. Eng. Mater. Sci.* (2020) **27**(1): 128–132.

[11] T. Kmita and W. Skoneczny. Increase of operational durability of a plastic material-oxide coating couple as a result of the application of a pulsed anodizing process. *Eksploat. i Niezawodn.* (2010) **45**(1): 77-82.

[12] K. H. Rashid. Comparative Study for Anodizing Aluminum Alloy 1060 by Different Types of Electrolytes Solutions. *First Sci. Conf. Mod. Technol. Oil Gas Refin.* (2011):21.

[13] Y. Jia, H. Zhou, P. Luo, S. Luo, J. Chen, and Y. Kuang. Preparation and characteristics of well-aligned macroporous films on aluminum by high voltage anodization in mixed acid. *Surf. Coatings Technol.* (2005) **201**(3-4): 513–518.

[14] Fratila-Apachitei, L.E., Tichelaar, F.D., Thompson, G.E., Terryn, H., Skeldon, P., Duszczyk, J. and Katgerman, L. A transmission electron microscopy study of hard anodic oxide layers on AlSi(Cu) alloys. *Electrochimica Acta* (2004) **49**(19): 3169-3177.

[15] Korzekwa, J., Gądek-Moszczak, A. and Zubko, M. Influence of the Size of Nanoparticles on the Microstructure of Oxide Coatings. *Materials Science* (2018) **53**(5): 709-716.

[16] Bochenek, D., Niemiec, P., Korzekwa, J., Durtka, B. and Stokłosa, Z. Microstructure and properties of the ferroelectric-ferromagnetic PLZT-ferrite composites. *Symmetry* (2018) **10**(3): art. 59.

[17] Dwornicka, R., Radek, N., Krawczyk, M., Osocha, P. and Pobędza, J. The laser textured surfaces of the silicon carbide analyzed with the bootstrapped tribology model. *METAL 2017 – 26th Int. Conf. on Metallurgy and Materials* (2017): 1252-1257.

[18] Radek, N., Szczotok, A., Gądek-Moszczak, A., Dwornicka, R., Brонček, J. and Pietraszek, J. The impact of laser processing parameters on the properties of electro-spark deposited coatings. *Archives of Metallurgy and Materials* (2018) **63**(2): 809-816.

[19] Pliszka, I. and Radek, N. Corrosion Resistance of WC-Cu Coatings Produced by Electrospark Deposition. *Procedia Engineering* (2017) **192**: 707-712.

[20] Lipiński, T. and Karpisz, D. Corrosion rate of 1.4152 stainless steel in a hot nitrate acid. *METAL 2019 – 28th Int. Conf. on Metallurgy and Materials* (2019): 1086-1091.

[21] Skrzypczak-Pietraszek, E., Reiss, K., Żmudzki, P. and Pietraszek, J. Enhanced accumulation of harpagide and 8-O-acetyl-harpagide in Melittis melissophyllum L. agitated shoot cultures analyzed by UPLC-MS/ MS. *PLoS ONE* (2018) **13**(8): art. e0202556.

[22] Skrzypczak-Pietraszek, E., Piska, K. and Pietraszek, J. Enhanced production of the pharmaceutically important polyphenolic compounds in Vitex agnus castus L. shoot cultures by precursor feeding strategy. *Engineering in Life Sciences* (2018) **18**(5): 287-297.

[23] Trzewiczek, K., Szczotok, A. and Gadek-Moszczak, A. Evaluation of the state for the material of the live steam superheater pipe coils of V degree. *Advanced Materials Research* (2014) **874**: 35-42.

[24] Lipinski, T. and Karpisz, D. Effect of animal slurry on carbon structural S235JR steel at 303 K. *Engineering for Rural Development* (2020) **19**: 1482-1487.

[25] Lipinski, T. and Karpisz, D. Effect of animal slurry on carbon structural S235JR steel at 318 K. *METAL 2020 – 29th Int. Conf. on Metallurgy and Materials* (2020): 643-648.

[26] Wojnar, L., Gadek-Moszczak, A. and Pietraszek, J. On the role of histomorphometric (stereological) microstructure parameters in the prediction of vertebrae compression strength. *Image Analysis and Stereology* (2019) **38**(1): 63-73.

[27] Pietraszek, J. and Goroshko, A. The heuristic approach to the selection of experimental design, model and valid pre-processing transformation of DoE outcome. *Advanced Materials Research* (2014) **874**: 145-149.

[28] Pietraszek, J., Dwornicka, R. and Szczotok, A. The bootstrap approach to the statistical significance of parameters in the fixed effects model. ECCOMAS Congress 2016 – *Proc.*

of the 7th European Congress on Computational Methods in Applied Sciences and Engineering (2016) **3**: 6061-6068.

[29] Gądek, A., Kuciel, S., Wojnar, L. and Dziadur, W. Application of computer-aided analysis of an image for assessment of reinforced polymers structures. *Polimery/Polymers* (2006) **51**(3): 206-211.

[30] Patek, M., Konar, R., Sladek, A. and Radek, N. Non-destructive testing of split sleeve welds by the ultrasonic TOFD method. *Manufacturing Technology* (2014) **14**(3): 403-407.

[31] Gadek-Moszczak, A. and Zmudka, S. Description of 3D microstructure of the composites with polypropylene (pp) matrix and tuf particles fillers. *Solid State Phenomena* (2013) **197**: 186-191.

[32] Maszke, A., Dwornicka, R. and Ulewicz, R. Problems in the implementation of the lean concept at a steel works – Case study. *MATEC Web of Conference* (2018) **183**: art. 01014.

[33] Pacana, A., Czerwińska, K. and Dwornicka, R. Analysis of non-compliance for the cast of the industrial robot basis. *METAL 2019 – 28th Int. Conf. on Metallurgy and Materials* (2019): 644-650.

[34] Karpisz, D. Design of manufacturing databases. *Technical Transactions* (2016) **113**: 73-77.

[35] Pietraszek, J., Sobczyk, A., Skrzypczak-Pietraszek, E. and Kołomycki, M. The fuzzy interpretation of the statistical test for irregular data. *Technical Transactions* (2016) **113**: 119-126.

[36] Radek, N., Kurp, P. and Pietraszek, J. Laser forming of steel tubes. *Technical Transactions* (2019) **116**: 223-229.

[37] Danielewski, H. Laser welding of pipe stubs made from super 304 steel. Numerical simulation and weld properties. *Technical Transactions* (2019) **116**: 167-176.