

A. KOZIK[#], M. NOWAK^{*}, K. GĘDLEK^{*}, D. LEŚNIAK^{**}, J. ZASADZIŃSKI^{**}, H. JURCZAK^{***}**ANODIC OXIDE COATINGS ON ALUMINIUM RESISTANT TO STRONG ALKALI ENVIRONMENT**

The aim of this paper was to test currently available on the market products for sealing anodic oxide coatings as well as to test the use of other alternative substances improving the sealing process. The ability to seal in 10 different solutions and the quality of the seal has been tested. The influence of the applied preparations on corrosion resistance and resistance to strongly alkaline environment was also investigated.

Based on the results obtained, satisfactory results were archived for the sample sealed in a IMN-OML (Institute of Non-Ferrous Metals in Gliwice, Light Metals Division) solution sealant and in solution of nickel acetate in a medium-temperature process. Sealing by means of nickel acetate solutions is economically justified, and its use allows the process temperature to be lowered. When it comes to resistance to alkalis, samples sealed in IMN-OML sealant are the best. Commercial solutions have also achieved positive results in all tests.

Keywords: anodic coatings, sealing of anodic coatings, resistant to strong alkali environment, sealing quality, aluminium alloys

1. Introduction

Anodising is a surface treatment process for aluminium and its alloys to improve properties such as corrosion resistance or mechanical properties (abrasion resistance or hardness). To improve the corrosion resistance of anodised oxide coatings, they are sealed after the anodising process. Traditional sealing method consists in immersion of the element with an anodic oxide coating in demineralised water with pH in the range of 5.5-6.0 and maximum conductivity up to 10 $\mu\text{S}/\text{cm}$, with a temperature of 98°C, for 2 minutes per micrometre of coating thickness. As a result of this treatment, commonly referred to as hot sealing, the porous oxide coating is sealed. After this treatment, the coating obtains a smooth, glossy surface, and thus perfectly protects the metal substrate against corrosion. However, from the economical point of view, the traditional sealing method, i.e. sealing in hot demineralised water, is a very expensive stage, due to the high temperature, the need for continuous water replenishment due to its intensive evaporation and relatively long process time.

It is therefore necessary to look for other alternative, faster and cheaper methods of sealing anodic oxide coatings. In addition to the economic point of view, anodic oxide coatings must meet a variety of requirements depending on the application of the product. Selection of the appropriate anodising technology, including sealing process parameters, is a particular challenge when the technology is to be applied to products resistant to alkaline environments.

Currently, research is also being carried out on sealing in compounds such as nickel fluoride [1,2], water glass, sodium acetate [3-5], molybdenum, cerium or yttrium compounds [6], and even using PTFE [7] and sealing with the sol-gel method [8]. All these methods give positive results in terms of corrosion resistance to acidic and seawater environments, but do not work in a strongly alkaline environment (pH 13-13.5). Resistance to alkalis is required mainly by the automotive industry, which is currently one of the largest recipients of anodised aluminium.

Sealing in nickel or cobalt compounds is usually a medium-temperature process. This method can be used for both decorative – protective as well as dyed in different colours coatings. An important role is played by the reaction of hydroxide precipitation by hydrolysis, which prevents leaching of the dye from the pores. However nickel salts are allergenic so this method has limited application [10]. Low-temperature sealing is carried out in nickel (II) fluoride solutions. Various types of alcohol are used as organic additives. Mechanism of this process has not been fully understood yet, however, it is very likely to hydragilit formation in the pores [11]. It is well known that silicates are an effective inhibitor in the process of sealing anodic oxide coatings. In this process, instead of boehmite, aluminum silicate is formed in the pores. Sealing in silicate solutions should not cause deterioration of abrasion resistance, as they are relatively hard compared to boehmite (6 on the Mohs scale) [9].

The aim of the paper was to compare and test currently available on the market for sealing anodic oxide coatings sub-

* INSTITUTE OF NON-FERROUS METALS IN GLIWICE, LIGHT METALS DIVISION IN SKAWINA (IMN OML) 19 PIŁSUDSKIEGO STR., 32-050 SKAWINA, POLAND,

** AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, AL. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

*** ALBATROS ALUMINIUM SP. Z O.O. ZAKŁAD PRODUKCYJNY 36 POŁUDNIOWA STREET, 78-600 WALCZ, POLAND

Corresponding author: akozik@imn.skawina.pl

stances (both decorative and protective as well as coloured coatings), as well as an attempt to use other alternative substances improving the sealing process. It was then examined the impact of the treatment on the corrosion and strong alkali environment of the sealed anodic oxidation coatings.

2. Materials and methods

1xxx aluminium series (EN-AW 1050 aluminium alloy) was used. The chemical composition of alloy was determined by optical emission spectrometry on the ARL 4460 spectrometer. The results are presented in Table 1.

The anodic oxide coating was produced in a conventional bath, based on H_2SO_4 with a concentration of 180 g/l. Process parameters: temperature 20-21°C, voltage 16 V, time 60 min. The power source was a laboratory power supply unit with regulated and stabilised DC voltage and regulated current consumption. Lead electrodes were used as cathodes. The solution was constantly stirred with compressed air. Before anodising process, the aluminium substrate was degreased in acetone, chemically etched in alkaline solution (based on NaOH 100 g/l) and brightened in HNO_3 200 g/l solution.

After the anodising process, the oxide coatings were sealed. These processes were either high, medium or low temperature. The reference method was the standard hydrothermal method (sealing in boiling demineralised water). For sealing, preparations available on the market were used, as well as IMN OML (Institute of Non-Ferrous Metals in Gliwice, Light Metals Division) baths. The parameters of the sealing process are shown in Table 2.

Quimal Seal 102 and Quimal Seal Cold 501 samples are preparations purchased from the Galvanic Technologies company, used on an industrial scale. Quimal Seal 102 is a preparation designed for hot sealing, while Quimal Seal Cold 501 is designed for sealing at temperatures up to 35°C, its main components are nickel fluoride and cobalt sulphate.

The U30-U31 samples were sealed in two stages, first in a solution of nickel fluoride, in a low-temperature process (20-25°C), and then in demineralised water at 60°C or nickel acetate solution at 70°C, respectively. Samples U16, U19 and U20 were sealed in single-step and low-temperature process (20-25°C) in solutions based on nickel fluoride.

Other samples were sealed in single-step process in temperatures between 70-100°C.

After the sealing process, samples were subjected to tests such as coating thickness, surface roughness, chemical

TABLE 1

Chemical composition of 1050 aluminium alloy

Element	Al	Mg	Si	V	Cr	Mn	Fe	Ni	Cu	Zn	Ga	Ti
Content [% _{weight}]	99.518	0.0027	0.083	0.025	0.002	0.007	0.309	0.004	0.001	0.009	0.012	0.028

TABLE 2

Parameters of the sealing process

No.	Substance	Time [min/μm]	Sample symbol	Comments
1	—	—	U1	—
2	Demineralised water	2	U2	Traditional sealing
3	QUIMAL SEAL 102	3	U3	Commercial preparations
4	QUIMAL SEAL COLD 501	0.6	U17	
5	IMN OML1 sealant	0.7	U8	Single-stage sealing
		1.4	U13	
		2.8	U26	
6	IMN OML2 sealant	0.7	U10	
		1.4	U11	
7	Nickel acetate	1.4	U15	
		2.8	U28	
8	Nickel fluoride	1	U16	
		2	U19	
9	Nickel fluoride Isoamyl alcohol	0.7	U20	
10	Nickel acetate Triethanolamine	1.4	U22	
		2.8	U27	
11	Nickel fluoride	1	U30	Two-stage sealing
	Demineralised water	1.4		
12	Nickel fluoride	1	U31	
	Nickel acetate	1		

composition of coatings, microstructure observations and sealing quality tests. Corrosion tests of anodic oxide coatings obtained were also performed.

Thickness tests of anodic oxide coatings were carried out using the DUALSCOPE MP20E-S device with the ETA3.3H probe, by eddy current method.

The roughness was measured using a Hommel-Etamic W10 roughness tester, by contact method. The tests were carried out at a distance of 4.8 mm, with a measurement speed of 0.5 mm/s, 3 measurements were made on each sample.

The microstructure and chemical composition of anodic oxide coatings were examined by scanning electron microscope (SEM) Inspect F50. The observations were carried out on the cross-section of samples.

Sealing quality tests were carried out using four methods. Admittance was measured according to PN-EN 2931:2010 [12]. The test was carried out after 1h after sealing and cooling to room temperature. The electrolyte used was aqueous solution of potassium sulphate 35 g/l. A dye spot test was performed according to PN-EN ISO 2143:2010 [13]. The method involves applying a spot of 25 ml/l H_2SO_4 plus 10 g/l KF solution to the clean and dry anodic oxide coating for exactly 1min. The acid spot is then washed off and the surface dried, afterwards a spot of dye solution (5 g/l Aluminium Blue 2LW) is applied also for exactly 1 min. The depth of colour (if any) remaining

of the surface of the coating is then compared with a printed colour chart giving a range of colour densities between 0 and 5 where 0 is complete freedom from retained colour. Measurement of weight loss after immersion in H_3PO_4/CrO_3 solution was performed according to PN-EN ISO 3210:2010 [14] standard. The principle of this method is that an unsealed anodic oxidation coating on aluminium is dissolved rapidly by acid media, whereas a well-sealed coating will withstand long immersion without appreciable attack. Before the test sample is degrease and drying, after it sample is weight. Next step is to immerse the test specimen in test solution (35 ml/l H_3PO_4) for 13 min at $38 \pm 1^\circ C$, then specimen is rinse, then dried and weight again.

Due to the increasing use of aluminium in the automotive industry, the test of resistance to alkalis according to General Motors standard GMW 14665 [15] was also carried out. This standard divided anodic oxide coatings into 3 classes of alkali resistance (A, B, C). Class A – resistance to extremely alkaline environment (pH = 13.5), class B – resistant to high alkaline environment (pH 13.0) and class C – resistant to alkaline environment (pH 12.0 and 11.5). During the test samples are dip at $20^\circ C$ to $23^\circ C$ in the test solution for 10 minutes then rinse with water and air dry. There shall be no surface appearance changes (dull areas, lost colour, etc.).

Corrosion resistance tests were carried out in neutral salt spray (NSS) in the DURA HKT 1000 salt chamber.

Electrochemical tests were carried out using the AUTO-LAB PGSTAT 302 electrochemical test set and software for recording results and analysis GPES data ver. 4.9. The samples were tested using a potentiodynamic method with a scan rate of 0.001V/s. The polarization started after 10 minutes of establishing the stationary potential in the conditions of the open circuit potential (OCP). The electrodes were samples with an anodic oxide coating sealed with various methods, the counter

electrode was a platinum electrode and the reference electrode was an Ag/AgCl 3M KCl electrode. Measurements were carried out in 1M NaCl solution at $25^\circ C$. The corrosion current density (I_{corr}) was deduced by extrapolating the cathodic branch of the polarization curves to the OCP and corrosion rate (V_{corr}) were calculated using following equation:

$$V_{corr}(mm/year) = \frac{K \times I_{corr} \times EW}{\rho}$$

Where, the constant, $K = 0.00327$, defines the unit of corrosion rate (mm/year), I_{corr} is the corrosion current density in $\mu A/cm^2$, ρ is the density of corroding material, EW is the equivalent weight of the alloy calculated using equation shown below:

$$EW = \frac{1}{\sum \left[\frac{n_i \times f_i}{w_i} \right]}$$

Where f_i is the weight fraction of the i th element in the alloy, w_i is the atomic weight of the i th element of the alloy and n_i is the valence of the i th element of the alloy.

3. Results and discussion

The thickness of coatings on all produced samples ranged from 22 to 25 μm .

The parameter that best reflects the value of roughness is R_a , because it eliminates the influence of single, irregular peak and valley. It is also the most commonly used in technique. The results of roughness measurements are presented on the Figure 1. The value of R_a parameter for unsealed anodic coating is 0.362 μm , for a coating sealed in demineralised water it is more or less at the same level of 0.378 μm . The samples (U30

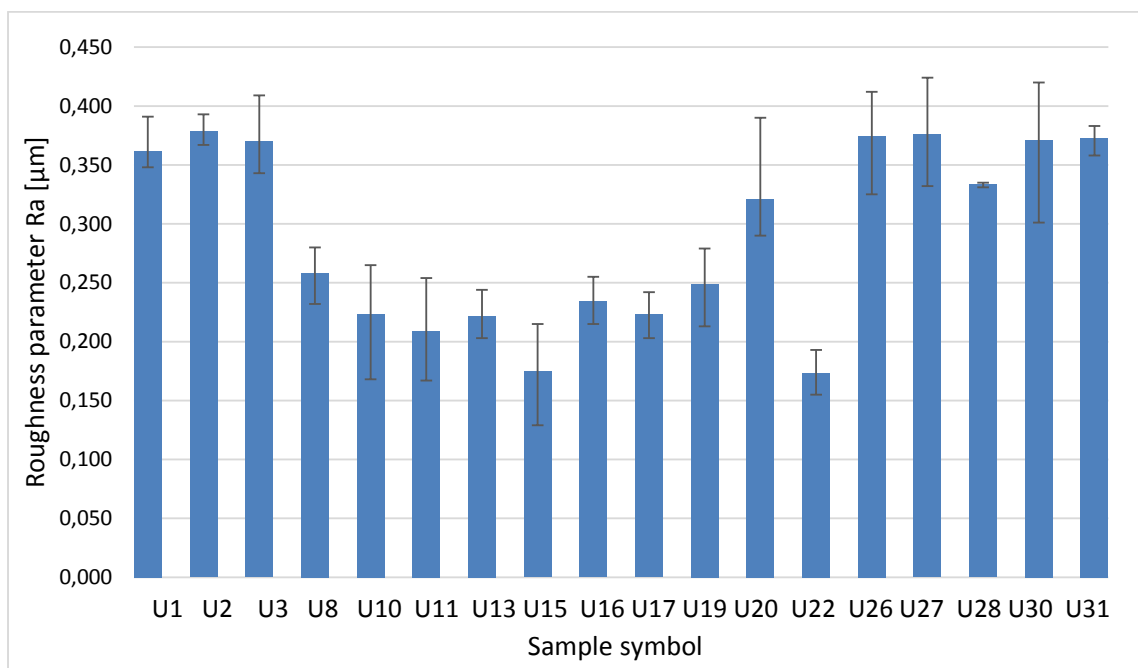


Fig. 1. Roughness parameter R_a for coatings with different sealing methods

and U31) sealed in two-stage process as well as sample sealed in QUIMAL SEAL 102 solution (U3) and during $2.8 \mu\text{m}/\text{min}$, regardless of the solution used, were characterized by a similar roughness value to the unsealed sample. The remaining samples were characterized by a decrease in surface roughness to the level of about $0.2 \mu\text{m}$.

Example images of cross-section and surfaces of coatings are shown on Figure 2. All observed coatings were continuous

and well-adhered to the substrate. There are significant differences in the pore size between the samples sealed in nickel compounds and IMN OML sealants. Sample U31 (Fig. 2c) sealed in two stages process and U28 (Fig. 2e) sealed in nickel acetate are characterized by significantly larger pores than samples U8 (Fig. 2d) and U11 (Fig. 2f) sealed in IMN OML sealants.

The results of chemical composition in micro-regions analysis are presented in Table 3.

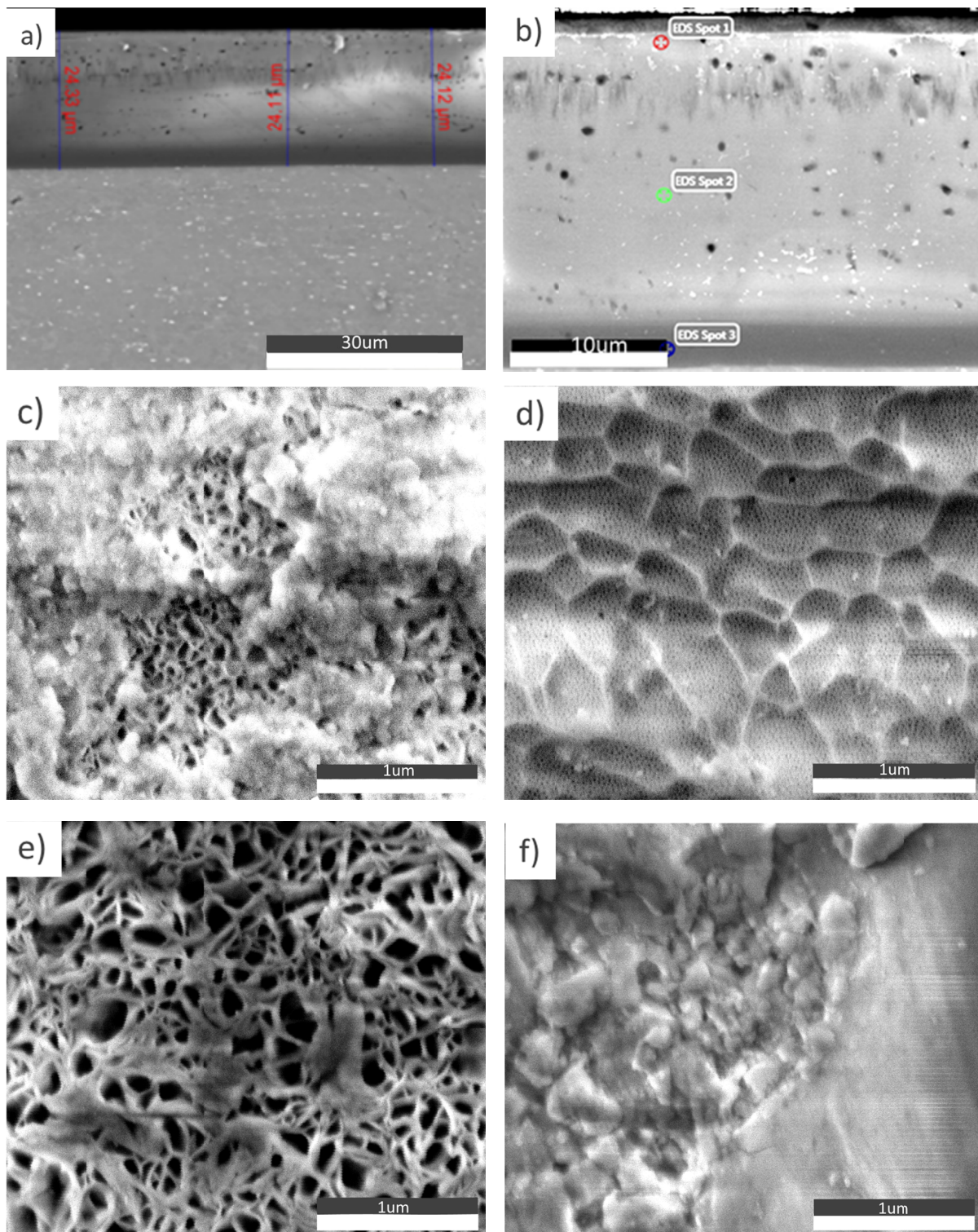


Fig. 2. SEM pictures of anodic coating a) example of cross-section of coating; b) example of analysis in micro-regions; c) surface of U31 sample; d) surface of U8 sample; e) surface of U28 sample; f) surface of U11 sample

Results of the chemical composition in micro-regions – distribution on the cross-section of the coating

Substance	Sample symbol	Results of the quantitative analysis, [%wag.]						
		O	Al	S	Na	Si	F	Ni
—	U1	45,72	48,96	5,32	—	—	—	—
Demineralised water	U2	48,66	46,29	5,05	—	—	—	—
QUIMAL SEAL 102	U3	50,07	45,29	4,65	—	—	—	—
QUIMAL SEAL COLD 501	U14	48,67	43,27	4,66	—	—	2,70	0,70
IMN-OML 1 sealant	U8	47,55	44,46	5,08	1,13	1,77	—	—
IMN-OML 2 sealant	U11	48,46	44,30	5,00	—	2,24	—	—
Nickel acetate	U28	51,87	43,47	4,66	—	—	—	0,29
Nickel acetate Triethanolamine	U27	51,91	43,07	4,47	—	—	—	0,55
Nickel fluoride	U19	47,68	44,90	4,60	—	—	2,24	0,59
Nickel fluoride Isoamyl alcohol	U20	46,64	45,34	4,73	—	—	2,43	0,86
Nickel fluoride Demineralised water	U30	48,78	43,48	4,70	—	—	2,50	0,53
Nickel fluoride Nickel acetate	U31	49,70	43,31	4,56	—	—	2,17	0,27

Analysis of coatings chemical composition in the micro-regions showed mainly the presence of O, Al and S. In some cases, Si, Ni, Na and F and C were also analysed. The elements analysed in the coatings occurred primarily at the surface of the coating. The closer to the substrate, the content of elements decreased until complete extinction.

Admittance measurement was performed in accordance with [9], however, it is a standard dedicated mainly to the coating sealed with the traditional method. In the literature studies were carried out on the possibility of applying admittance measurement method for coatings sealed in solutions of nickel salts [5]. The authors of this paper noted that the samples sealed in solutions containing nickel compounds show completely different values of apparent conductivity when the measurement is carried

out directly after the sealing process, and different when the measurement is carried out after a few days or more. The value of admittance decreased over time. Therefore, the admittance measurement was performed twice, for the first time directly after the sealing process (blue bars in the graph) and after 14 days (orange bars in the graphs).

It is assumed that the coating is well sealed when the apparent conductivity value is $\leq 20 \mu\text{S}$, but this is not a normalised value but only good engineering practice. The graphs show a value of $300 \mu\text{S}$ – maximum measurement value of the device for samples for which the apparent conductivity value was outside the measurement range of the device.

The results of admittance measurements are presented on Figure 3.

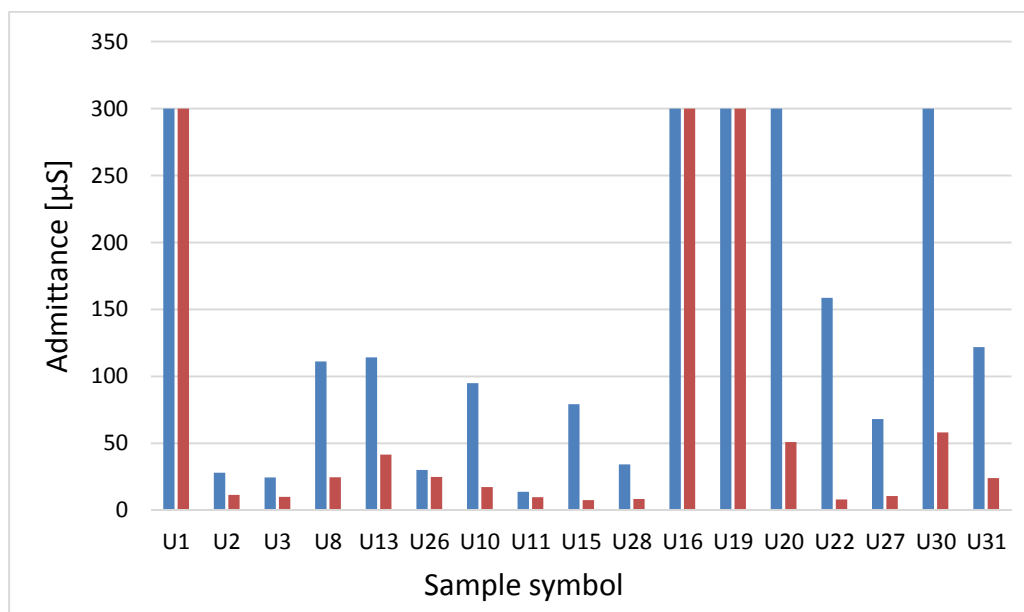


Fig. 3. Results of measurement of apparent conductivity (blue – measurement within 24 hours from the formation of the coating, orange – measurement after 14 days from the formation of the coating)

The admittance values for a U2 sample sealed with the traditional method in boiling demineralised water were 28.14 μS – immediately after sealing and 11.59 μS after several days, however, according to the standard, the measurement for such samples should be carried out within 48 hours since sealing. Among the other samples sealed with high-temperature methods, the lowest apparent conductivity value is the U11 sample, sealed with IMN OML 2 sealant. For this sample, the apparent conductivity value immediately after the sealing process was 13.86 μS , and after 14 days it did not differ much and amounted to 9.89 μS . Both of these values meet the condition of a „good“ seal ≤ 20 μS . For U22 and U27 samples sealed in high-temperature processes, significant differences were observed between the apparent conductivity measurements immediately after the sealing process and after 14 days.

U16 and U19 samples were sealed with NiF_2 solution in a low-temperature process, for the samples of these values of both the first and second measurement were outside the measuring range of the device, which indicates a very poor quality of the seal.

Table 4 presents the interpretation of colour spot test results according to [13].

TABLE 4

Interpretation of dye-spot test according to [13]

Sample symbol	Intensity	Loss of absorption capacity
U1	5	none
U2	1	strong
U3	2	medium
U8	5	none
U10	4	very weak
U11	5	none
U13	5	none
U15	1	strong
U16	5	none
U17	3	weak
U19	4	very weak
U20	3	weak
U22	1	strong
U26	2	medium
U27	1	strong
U28	2	medium
U30	3	weak
U31	2	medium

The reference sample, i.e. the sample sealed in boiling demineralised water, showed an absorption capacity of 1, similarly as the samples U15, sealed in nickel acetate, U22 and U27, sealed in nickel acetate with the addition of triethanolamine. The samples U3 (sealed in Quimal Seal 102 preparation), U26 (sealed in IMN OML 1 sealant), U28 (sealed in nickel acetate solution) and U31 (sealed in two stages with $\text{NiF}_2 \rightarrow \text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$) were characterised by acceptable sealing quality (intensity at level 2). The sealing quality of the other samples was very poor.

Table 5 presents the results of measurements of weight loss of samples after immersion in $\text{H}_3\text{PO}_4/\text{CrO}_3$ solution

according to [14]. The loss of weight of the U1 sealed sample is 402.96 mg/dm^2 , while the U2 sealed sample is 13.94 mg/dm^2 . For all other samples the results were significantly lower than the value of the unsealed sample. The values of weight loss of about 30 mg/dm^2 , indicating good quality of sealing, were obtained for U3 samples (sealing in QUIMAL SEAL 102) – 15.49 mg/dm^2 , U8 (OML 1 sealant) – 14.37 mg/dm^2 , U27 (sealing in nickel acetate with addition of triethanolamine) – 33.52 mg/dm^2 and U28 (sealing in nickel acetate) – 29.01 mg/dm^2 .

TABLE 5

Results of weight loss measurements after immersion in $\text{H}_3\text{PO}_4/\text{CrO}_3$ solution according to [14]

Sample symbol	Starting mass [mg]	Mass after immersion in acid [mg]	Weight loss [mg]	Weight loss [mg/dm^2]
U1	16858.9	16572.8	286.1	402.96
U2	17533.0	17523.1	9.9	13.94
U3	17310.9	17299.9	11	15.49
U8	16856.4	16846.2	10.2	14.37
U10	16839.5	16748	91.5	128.87
U11	16567.8	16436.7	131.1	184.65
U13	17146.3	17053.7	92.6	130.42
U15	17785.1	17747.9	37.2	52.39
U16	17149.5	16835.5	314	442.25
U17	17054.8	17008.5	46.3	65.21
U19	15052.0	14959.5	92.5	140.15
U20	17123.9	17046.5	77.4	109.01
U22	16903.5	16867.2	36.3	51.13
U26	17209.2	17077.7	131.5	185.21
U27	16457.2	16433.4	23.8	33.52
U28	17281.5	17260.9	20.6	29.01
U30	15415.3	15333	82.3	115.92
U31	15140.3	15095.4	44.9	63.24

Measurement of weight loss after dissolution of the coating in solution $\text{H}_3\text{PO}_4/\text{Cr}_2\text{O}_3$ was also tested by Y. Shang et al. [16]. For any of the coatings sealed by different methods, they did not obtain a result below 30 mg/dm^2 . The best results were obtained for the sample sealed in stearic acid solution, for which the weight loss was 46 mg/dm^2 .

Table 6 shows the results of alkali resistance tests carried out according to [15].

Resistant to highly alkaline environments (pH 13.5) were samples sealed with IMN OML sealant solutions. Samples U8, U13 and U26 were sealed in IMN OML 1 sealant, U10 and U11 – sealed in IMN OML 2 sealant. The only sample sealed in another solution and resistant to pH 13.0 was the U28 sample, sealed in nickel acetate solution.

Similar tests according to GMW 14665 were carried out by B.A. Manavbasi and others [17]. They conclude that all coatings sealed in boiling demineralised water, nickel acetate and even in chromium acid are not resistant to the strongly alkaline environment. They invented a new sealant called MLT (it does not contain heavy metals), which gives positive results in the test of resistance to alkalis.

TABLE 6

Test results for resistance to alkalis according to [15]

Sample symbol	pH 12.0	pH 13.0	pH 13.5
U1	matting	matting	matting
U2	unchanged	matting	matting
U3	matting	matting	matting
U8	unchanged	unchanged	unchanged
U10	unchanged	unchanged	unchanged
U11	unchanged	unchanged	unchanged
U13	unchanged	unchanged	unchanged
U15	matting	matting	matting
U16	unchanged	matting visible at an angle	matting
U17	unchanged	matting	matting
U19	unchanged	matting	matting
U20	matting	matting	matting
U22	matting	matting	matting
U26	unchanged	unchanged	unchanged
U27	unchanged	matting	matting
U28	unchanged	unchanged	matting visible at an angle
U30	unchanged	matting visible at the angle	matting
U31	unchanged	matting	matting

The results of electrochemical tests are presented in Table 7 and on Figure 4. In typical polarization curve, a lower I_{corr} and a higher E_{corr} corresponds to a lower corrosion rate and a better corrosion resistance. All samples with an anodic oxide coating sealed with different methods were characterised by better corrosion resistance parameters in comparison to an unsealed sample (U1). Compared to the conventionally sealed sample (U2), only

U16 and U19 samples were shifted in a more anodic direction and had a higher corrosion current (I_{corr}). The other samples had a more cathodic or lower corrosion current in relation to the sample sealed in boiling demineralized water. The smallest corrosion rate was found for samples U3, U13 and U17.

TABLE 7

Results of electrochemical tests

Sample No.	I_{corr} [A/cm ²]	E_{corr} [mV]	V_{corr} [mm/year]
U1	6,5E-6	-1154	1,50E-3
U2	3,3E-9	-693	8,03E-7
U3	3,4E-9	-822	8,28E-7
U8	7,1E-8	-782	1,73E-5
U10	1,3E-8	-714	3,16E-6
U11	2,4E-8	-723	5,84E-6
U13	2,9E-9	-947	7,06E-7
U15	5,2E-8	-800	1,27E-5
U16	1,1E-5	-805	2,68E-3
U17	3,7E-9	-674	9,01E-7
U19	3,4E-6	-1256	8,28E-4
U20	4,9E-9	-1045	1,19E-6
U22	6,5E-9	-1096	1,58E-6
U26	5,3E-9	-1015	1,29E-6
U27	4,3E-9	-1067	1,05E-6
U28	1,8E-8	-778	4,38E-6
U30	1,3E-8	-693	3,16E-6

After 720 h of exposure to neutral salt spray, the first signs of corrosion were visible on the surface of the U1 unsealed sample. The surface of the residual samples after 1000 h of exposure to neutral salt spray remained intact.

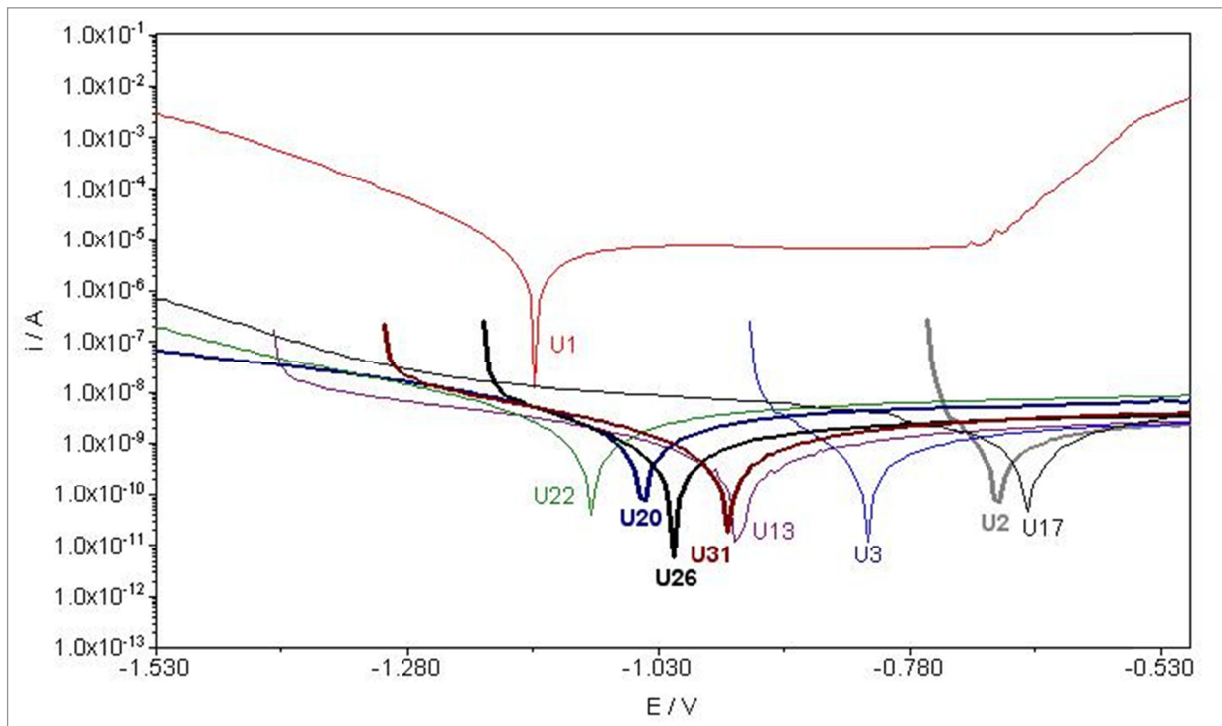


Fig. 4. Polarization curves

4. Conclusions

- On the basis of the results obtained, the best results in all conducted studies were obtained for the U28 sample. The U28 sample was sealed in nickel acetate solution at 90°C and for 2.8 min/μm.
- In the tests carried out, the samples sealed in preparations supplied by the Galvanic Technologies company also performed very well, however, these samples are not resistant to strongly alkaline environment.
- The worst sealing solution was nickel fluoride. The samples sealed by this method not pass any seal quality test. It is a very expensive preparation and, in addition, harmful to the environment, therefore, its use is not economically justified.
- The combination of sealing in NiF₂ solution with sealing at elevated temperatures, nickel acetate solution or demineralised water gives positive results.
- In terms of resistance to the alkaline environment, IMN OML sealants are the best performers.
- From the economical point of view, further research should be directed towards nickel acetate solutions, which is relatively cheap, and in addition, its use reduces the temperature of the process from 100°C to about 65-70°C and a small amount of process time.

REFERENCES

- [1] W. Liu, Y. Zuo, S. Chen, X. Zhao, J. Zhao, The effect of sealing on cracking tendency of anodic films on 2024 aluminium alloy after heating up to 300°C, *Surface & Coating Technology* **203**, 1244-1251(2009).
- [2] J. Lee, U. Jung, W. Kim, W. Chung, Effects of residual water in the pores of aluminium anodic oxide layers prior to sealing on corrosion resistance, *Applied Surface Science* **283**, 941-946 (2013).
- [3] V. Lopez, J.A. Gonzalez, A. Bautista, E. Otero, R. Lizarbe, The response of anodized materials sealed in acetate-containing baths to atmospheric exposure, *Corrosion Science* **40**, 4/5, 693-704 (1998).
- [4] M.R. Kalantary, D.R. Gabe, D.H. Ross, A model for the mechanism of nickel fluoride cold sealing of anodized aluminium, *Journal of Applied Electrochemistry* **22**, 268-276 (1922).
- [5] M. Rezaei-Kalantary, Development of cold sealing processes for anodized aluminium, *Praca doktorska*, 1990.
- [6] F. Mansfeld, C. Chen, C.B. Breslin, D. Dull, Sealing of Anodized Aluminium Alloys with Rare Earth Metal Salt Solutions, *J. Electrochem. Soc.* **145**, 8 (1998).
- [7] P. Kwolek, P. Górecka, A. Oblój, U. Kwolek, K. Dychtoń, M. Drajcewicz, J. Sieniawski, The Teflon impregnation of anodic coating onto aluminium substrate, *Chemik* **70**, 7, 361-368 (2016).
- [8] M. Zemanowa, M. Chovancova, New Approaches for Sealing Anodic Coatings – Aluminium substrates can be sealed with sol-gel method after anodic oxidation in sulfuric acid, *Aluminium anodizing compendium*, www.metalfinishing.com, October 2005.
- [9] J.A. Gonzalez, M. Morcillo, E. Escudero, V. Lopez, E. Otero, Atmospheric corrosion of bare and anodized aluminium in a wide range of environmental conditions. Part I: Visual observation and gravimetric results, *Surface and Coatings Technology* **153**, 225-234 (2002).
- [10] B. Rachel Cheng, L. Hao, Comparative Study of the Effects of Sealing Processes on the Wear Resistance and Sealing Quality of Hard Anodic Coatings, *Metal Finishing* **98**, 48-55 (2000).
- [11] L. Hao, B.R. Cheng, Sealing Processes of Anodic Coatings – Past, Present and Future.
- [12] PN-EN ISO 2931:2010 “Anodizing of aluminium and its alloys – Assessment of quality of sealed anodic oxidation coatings by measurement of admittance.
- [13] PN-EN ISO 2143:2010 “Anodizing of aluminium and its alloys – Estimation of loss of absorptive power of anodic oxidation coatings after sealing – Dye-spot test with prior acid treatment”.
- [14] PN-EN ISO 3210:2010 “Anodizing of aluminium and its alloys – Assessment of quality of sealed anodic oxidation coating by measurement of the loss of mass after immersion in phosphoric acid/chromic acid solution”.
- [15] GMW 14665 “Anodic Oxidation Coating on Aluminum”.
- [16] Y. Shang, L. Wang, Z. Liu, D. Niu, Y. Wang, Ch. Liu, The effects of different sealing techniques for anodic film of Al-12.7Si-0.7Mg Alloys, *Int. J. Electrochem. Sci.* **11**, 5234-5344 (2016).
- [17] B.A. Manavbasi, K. Bodily, T. Clarke, K. Johnson, B. Estes, Alkaline Corrosion-Resistant Sealant for Anodized Aluminium Alloys, Technically speaking, www.metalfinishing.com, November/December 2013.