Improvement of the stainless steel electropolishing process by organic additives

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The influence of organic additives on the process of surface electropolishing of AISI 304 type steel was determined. Additives were selected in initial potentiodynamic tests pursuant to the plateau analysis on the current/potential curves. The assessment of the operational effectiveness of additives consisted in determining the relationship between surface gloss after electropolishing and the mass loss of the sample and in determining surface roughness. The applied electropolishing bath consisted of a mixture of concentrated acids: H_3PO_4 and H_2SO_4, and the following organic additives were used: triethylamine, ethanolamine, diethanolamine, triethanolamine, diethylene glycol monobutyl ether and glycerol. The best electropolishing result, i.e. low roughness and high gloss of stainless steel surface with a relatively low mass loss of the sample at the same time were obtained for baths containing triethanolamine.

Keywords: electropolishing, organic additives, surface roughness, stainless steel.

INTRODUCTION

Scientific and industrial research on the electropolishing (EP) of stainless steel, conducted in various units, focus on the improvement of bath compositions, development of more beneficial process parameters and intensification of the process[1–7]. The modification of bath composition for baths, whose main components are: phosphoric(V) and sulphuric(VI) acids, involves tests of surface active compounds. Their aim is to improve the smoothing of surface and to decrease bath contamination. Beneficial results may be obtained if these compounds are absorbed in indentations that exist on the surface of stainless steel[8–9]. The applied additives should also prolong the period of operation of the bath, i.e. improve the economic aspect of the process.

Currently, the most commonly used additive that improves the gloss and smoothening of the surface in the electropolishing process, is glycerol. It is applied in the electrochemical processing of various metals and alloys: titanium, cobalt, niobium, copper, palladium, aluminium, zirconium, chromium and stainless steels[10–12]. Its content in electropolishing baths varies within a wide range, depending on the applied technology and it depends on numerous factors, e.g.: bath composition, process conditions, type of the processed metal and the expected results. The concentration of glycerol in the bath may reach even up to 35% wt.

Examples of bath additives used in the stainless steel electropolishing process, described in scientific and patent literature are presented in Table 1.

Numerous authors emphasise the positive influence of organic additives on the smoothening of stainless steel surface in the electropolishing process. However, literature does not provide information that would allow to compare the efficiency of baths containing the most commonly used additives, i.e. glycerol and mono-, di- and triethanolamines with baths without such organic additives. Based on the review for presented research triethanolamine (TEA), triethylamine (TRE), ethanolamine (MEA), diethanolamine (DEA), glycerol (GLR) and diethylene glycol monobutyl ether, known as butyldiglycol (BDG) were selected for comparative tests. The latest two organic compounds (GLR and BDG) were listed as organic additives to electropolishing in the description of Best Available Techniques in the "Reference Document on Best Available Techniques for the Surface Treatment of Metals and Plastics"[20]. Our study focused on the improvement of surface properties of 304 stainless steel parts. The main objective of the study was to obtain better or faster effects of smoothing the surface samples after electrochemical treatment in baths that contained selected organic additives with a simultaneous decrease of the mass loss of the samples. Reducing the sample mass loss plays an important role in industrial practice and could significantly limit the contamination of electropolishing baths with ions of iron, chromium and nickel. On the one hand, lower bath contamination results in savings in power consumption during electropolishing, while on the other hand it brings benefits in form of cleaner water after washing and lower consumption of chemicals in the course of wastewater neutralization. These environmental aspects urge to examine organic additives.

EXPERIMENTAL PROCEDURES

Sample preparation

Specimens (90 x 25 x 1.5 mm with a 12 mm-diameter hole located at 5 mm distance from the shorter edge of the sample) were cut-off from the cold rolled stainless steel plate (AISI 304). The samples were designated as 2B-surface finish. The chemical material composition was (wt.%): 0.037 C, 0.42 Si, 0.057 N, 1.28 Mn, 0.029 P, 0.002 S, 18.13 Cr, 8.04 Ni and balance Fe. All the

Table 1. Examples of bath additives used in the stainless steel electropolishing process, described in scientific and patent literature

Organic additive	Bath type	Obtained results
≥1 wt.% 2-amino-2-methyl--1-propanol	≥1 wt.% nonoxidizing acid (e.g. H_3PO_4), citric acid, tartaric acid, oxalic acid, acetic acid, gluconic acid, glycolic acid, succinic acid, etc., H_2SO_4 and/or HF	Efficient electropolishing of stainless steel, esp. after welding and improvement of stainless steel corrosion resistant[14]
≥0.1 wt.% triethanolamine, diethanolamine and/or monoethanolamine	≥0.1 wt.% ≥ 1 of H_3PO_4, citric acid, tartaric acid, oxalic acid, acetic acid, gluconic acid, glycolic acid, succinic acid, H_2SO_4, and their salt with Na, K, and ammonium	The electrolytic solution also improves corrosion resistance of stainless steel[15]
3 wt.% triethanolamine	H_3PO_4 51 wt.% H_2SO_4 35 wt.%	After EP of 304 stainless steel pitting potential in 0.5 M NaCl solution increases up to 0.5 V_{SCE}. Both laboratory and industrial electrochemical treatment resulted in consistent test results[16]
2.27 vol.% ethanoloamine or diethanolamine or triethanolamine	H_3PO_4 56.82 vol.% H_2SO_4 40.91 vol.%	Hull cell studies indicated that best reflectance related to minimum electrical power consumption were obtained after EP with ethanoloamine[17]
25 or 35 vol.% glycerol	H_3PO_4 and H_2SO_4 (volume ratios: 3:2 and 2:1)	The bath temperature and EP time were highlighted as the main factors affecting Ra after treatment, but current density was examined only in the range 0.5–1.0 A/cm². The glycerol content and current density distinguished by the significantly lower impact in the levelling the surface compared to the main factors[18]
10 vol.% glycerol	H_3PO_4 and H_2SO_4 (volume rations: 5:5/6:4/7:3) Water content: 0/10/20/30 (vol.%)	Optimal conditions: 6:4 acids ratio, 85±10°C, 0.5 to 1.0 A/cm², 3–5 min EP time, addition of glycerol reduce surface roughness but anticorrosive performance in the $FeCl_3$ after 72 h is deteriorated[9]
10 vol.% glycerol	H_3PO_4:H_2SO_4: DI water ratio 60:20:10 (vol.%)	After EP treatment the passive films were thicker than naturally-formed passive films. In addition EP improves the surface biocompatibility and hemocompatibility. Nevertheless influence of addition glycerol weren't tested and evaluated[19]

samples were degreased with acetone, then washed in distilled water and named as-received. Later, the samples were pickled for 10 minutes in a solution composed of (wt.%): 4 HF, 20 HNO_3 and distilled water in the temperature 30 ±0.2°C.

Experimental circuit

The electropolishing process was carried out in a glass vessel of a volume of 400 cm³. The circuit consisted of: two cathodes and one anode (workpiece), a glass paddle stirrer, a thermometer, a thermostat Haake DC10 (Haake, Karlsruhe, Germany) with a spiral cooler, a power supply unit PS3010L (Velleman, Gavere, Belgium) and a charge counter KP-034 (KP-Elektronika, Dzierzoniow, Poland). The anode and cathodes were of the same size and made of the same stainless steel plate. A constant distance between the electrodes (20 mm) was assumed for test purposes. The samples of a surface area of 20 cm² were partly immersed in an electrolyte solution. The upper parts of samples were covered with Teflon tape, which allowed for a precise designation of the working area and eliminated the problem of uneven polishing on the border of the media: bath – air. The EP bath consisted of (wt.%): 51 phosphoric acid, 35 sulfuric acid, 3 organic additives and balance water. The electropolishing process was carried out at a stirring speed in the range of 49–50 rpm at bath temperature 55 ±1°C. Subsequently all the samples were washed with distilled water.

Organic compounds used as bath additives in the electropolishing process

Table 2 shows the structural formulas and molecular weights of some of the compounds used as additives to the bath.

Potentiodynamic tests

Potentiodynamic tests were carried out in a tri-electrode system with use of SI 1286 potentiostat manufactured by Solartron. The tested electrode was 304 steel, reference electrode – saturated calomel electrode (SCE) and the counter electrode was a platinum electrode. The system was thermostated. The obtained data were analysed with use of CorrView software.
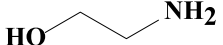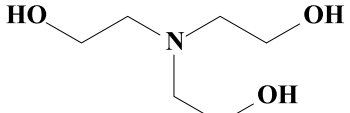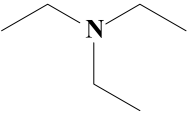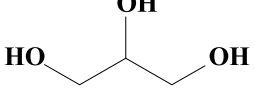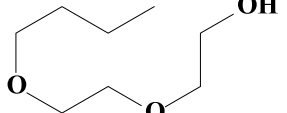
Gloss measurements

Surface gloss of 304 steel was measured with use of Elcometer 406L reflectometer. The device is equipped with a LED light source of a constant light intensity and it enables the gloss measurement of flat surfaces at 20° and 60° angles. Measurements were taken by recording the intensity of light reflected from the analysed surface. The gloss value is directly proportional to the amount of reflected light. Before the commencement of measurements, the apparatus was calibrated with use of a certified calibration plate. The applied measurement units were Gloss Units within the range 0–2000 GU for the 20° angle and 0–1000 GU for the 60° angle. For the purposes of the conducted tests it was decided to measure sample gloss for the measurement angle of 20°, which is suitable for glossy surfaces.

Surface roughness measurements

Roughness was measured with use of surface profiler Form Talysurf 120L manufactured by Taylor Hobson Limited. Measurement was taken with use of connection method with a needle with a conical diamond tip, of an opening angle of 90° and tip rounding radius of 2 μm. The length of the measured section λc was 0.8 mm. The surface profiler was controlled by Dell OptiPlex GX110 computer equipped with ULTRA software, rev. 6.0. Surface roughness assessment was based on the amplitude

Table 2. Organic additives used in tests on the electropolishing of AISI 304 steel

Organic additive	Abbreviation	Structure	Boiling temperature [°C]	Molecular weight [g/mol]
Ethanolamine	MEA		170	61.08
Diethanolamine	DEA		271	105.14
Triethanolamine	TEA		335	149.19
Triethylamine	TRE		90	101.19
Glycerol	GLR		290	99.09
Butyldiglycol	BDG		224–234	162.23

parameter R_a (arithmetic mean of the absolute departures of the roughness profile from the mean line) and R_{sk} skew (the asymmetry of the topography height distribution, showing if the holes or the hills are dominating features of the surface) and R_{ku} kurtosis (quantifies the width of the peak of the height histogram indicating how much the majority of the surface is close to the main plane). A summary list of the specified parameters allows for a more accurate description of surface topography[21–23].

RESULTS AND DISCUSSION

Potentiodynamic tests

In order to determine the influence of various organic additives on I–V curves, potentiodynamic tests were conducted for electropolishing solutions, which contained selected organic compounds. Initial tests for phosphoric and sulphuric acid bath without additives (Fig. 1A) and with the addition of triethanolamine (Fig. 1B), conducted at a scanning rate from 0.25 to 10 mV/s allowed us to determine that the potential change rate of 1 mV/s enables to precisely determine the current parameters of the occurrence of *plateau* in a relatively short measurement period.

Figure 2 shows the anodic curves obtained for the following bath composition (wt.%): 51 H_3PO_4, 35 H_2SO_4, 3 – addition of organic compound, 11 H_2O. Bath temperature was 55°C. For base composition of the bath (without added organic compounds) the *plateau* occurred in the potential range: 1.55–1.9 V_{SCE} and the corresponding current densities from $2.35 \cdot 10^{-2}$ to $2.50 \cdot 10^{-2}$ A/cm². The addition of triethylamine to the phosphoric and sulphuric bath changed this value only slightly (2.15–$2.28 \cdot 10^{-2}$ A/cm²) which demonstrates its low activity. In the presence of triethanolamine, the *pla-*
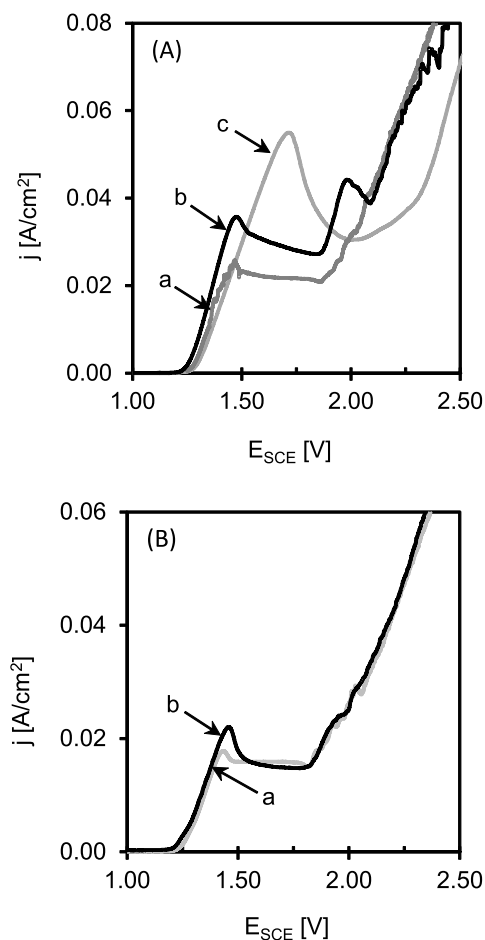

Figure 1. Anode polarisation curves obtained at different scanning rates: (a) 0.25 mV/s; (b) 1.0 mV/s; (c) 10 mV/s. Baths consisted of (wt.%): (A) 53% phosphoric and 36% sulphuric acids and balance water (T = 45°C); (B) 51% phosphoric and 35% sulphuric acids with the addition of 3% triethanolamine and balance water (T = 55°C)

teau level decreased to the value of $1.59 \cdot 10^{-2}$ A/cm^2 and for monoethanolamine to $1.60 \cdot 10^{-2}$ A/cm^2. One might conclude that all of the selected compounds, apart from triethylamine are active as additives to electropolishing baths (Fig. 2).
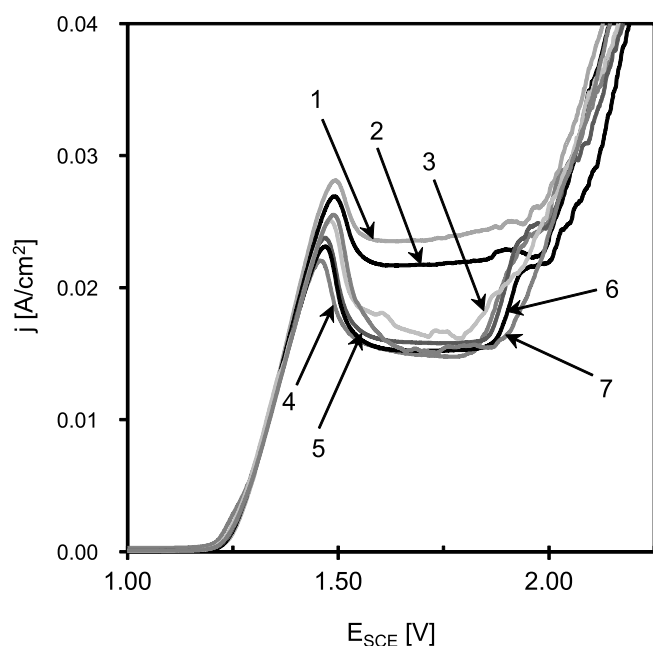

Figure 2. Anodic curves in a mixture of: (1) phosphoric acid, sulfuric acid and balance water (53:36:11 by wt.%) or phosphoric acid, sulfuric acid, balance water and selected organic additives (51:35:11:3 by wt.%) (2) triethylamine, (3) glycerol, (4) triethanolamine, (5) ethanolamine, (6) diethanolamine, (7) butyldiglycol; scan rate 1 mV/s, T = 55°C

Surface modification

In order to evaluate the role of the used bath additives, electropolishing was conducted, followed by the determination of the mass loss and change in gloss of the samples (Table 3). It was demonstrated that the used additives generally decrease mass loss. This will result in lower contamination of the phosphoric and sulphuric bath, but it may also point to decreased intensity of the electropolishing process. The mass loss in samples subject to electropolishing in bath containing glycerol was 27% lower, and in bath containing triethanolamine – 17% lower than in bath with base composition (without additives). Only triethylamine caused a 24% increase in mass loss.

The gloss of samples subject to electropolishing in bath without additives was 890 (±20) GU. TEA slightly improved gloss: in the same electropolishing conditions

900 (±20) GU were measured. Glycerol has an inadequate effect – the gloss was decreased to 680 (±10) GU.

Electropolishing of samples for 6 minutes at current density 0.3 A/cm^2 in baths containing the addition of BDG or TEA resulted in similar mass losses, respectively: 43.4 mg/cm^2 and 44.6 mg/cm^2. However, the measured gloss values of both samples were quite different. TRE, on the other hand, enables to obtain high surface gloss (1045 GU) for electropolishing for 6 minutes at current density of 0.3 A/cm^2, but with a simultaneous high mass loss (67.0 mg/cm^2). Additionally, its relatively low boiling temperature of 90°C significantly limits the possibilities to apply this compound on an industrial scale.

Ethylendiamine, diethanolamine and triethanolamine have a similar influence on the gloss and mass loss of the sample during electropolishing in phosphoric and sulphuric bath. The best gloss effect with a simultaneous smallest mass loss of the sample – 20.2 GU · cm^2/mg was obtained for triethanolamine. From the practical point of view, a major advantage of this compound is the fact that its boiling temperature is the highest among the applied additives.

In the initial stage of electropolishing, the baths with base composition are changing their color. In baths without organic additives, after the flow of 1–2.5 Ah/dm^3 volume of the baths, metal ions originating from anodic dissolution of 304 steel changed the colour of transparent bath to brown. In the presence of MEA, DEA, TEA or GLR the colour of the bath turned green. The bath containing BDG was initially yellow, and then it darkened and became dark green. The addition of TRE initially dyed the bath brown, and, after several hours of operation – dark green. This is evidence of slow complexing of iron ions.

Electropolishing of 304 steel in in bath with base composition at a current density of 0.3 A/cm^2 and a specific charge input of 0.02 Ah/cm^2 (t = 4 min) reduce the sample roughness to R_a = 0.12 µm (Table 4). In the same electropolishing conditions, improved surface smoothness of AISI 304 steel was obtained after the application of bath containing TEA (R_a = 0.095 μm). GLR addition proved much less effective (R_a = 0.14 μm). Increasing the electric charge twice (to q = 0.04 Ah/cm^2) did not result in improved roughness of samples subject to electropolishing in bath without organic additives. However, as the time of electropolishing increased to 8 min, the smoothness of samples processed in baths containing TEA and GLR improved, respectively, to the values R_a = 0.079 μm and R_a = 0.090 μm for q = 0.04 Ah/cm^2.

Surface roughness assessment based only on the R_a parameter is quite often insufficient, as this parameter only

Table 3. Mass loss and changes in the surface gloss of 304 steel after electropolishing in phosphoric and sulphuric bath with the addition of 3 wt.% organic additives (q = 0.03 Ah/cm^2, j = 0.3 A/cm^2, t = 6 min, T = 55°C)

Organic additive	Abbreviation	After electropolishing		
		Gloss [GU]	Mass loss [mg/cm^2]	Gloss/ mass loss [GU · cm^2/mg]
None	–	890 (± 10)	54.1	16.5
Triethanolamine	TEA	900 (± 20)	44.6	20.2
Diethanolamine	DEA	880 (± 20)	46.8	18.8
Ethanolamine	MEA	840 (± 20)	48.0	17.5
Glycerol	GLR	680 (± 10)	39.7	17.1
Triethylamine	TRE	1045 (± 20)	67.0	15.6
Butyldiglycol	BDG	410 (± 10)	43.4	9.4

Table 4. Surface roughness after: pickling in a solution composed of HF, HNO$_3$ and H$_2$O (4:20:76 by wt.%), T = 30°C (A) t = 10 min; electropolishing in H$_3$PO$_4$, H$_2$SO$_4$, H$_2$O (53:36:11 by wt.%) and with addition of selected organic additive (51:35:11:3 by wt.%), T = 55°C (B) j = 0.3 A/cm^2, t = 4 min; (C) j = 0.3 A/cm^2, t = 6 min; (D) j = 0.4 A/cm^2, t = 6 min

Surface treatment	Organic additive					
	None		Glycerol		Triethanolamine	
	R$_a$ [μm]	Sd	R$_a$ [μm]	Sd	R$_a$ [μm]	Sd
(A) Pickling	0.38	0.005	0.40	0.017	0.38	0.012
(B) Electropolishing 0.02 Ah/cm^2	0.12	0.012	0.14	0.017	0.095	0.006
(A) Pickling	0.35	0.010	0.36	0.009	0.36	0.010
(C) Electropolishing 0.03 Ah/cm^2	0.10	0.005	0.10	0.005	0.090	0.006
(A) Pickling	0.35	0.010	0.36	0.009	0.36	0.005
(D) Electropolishing 0.04 Ah/cm^2	0.12	0.012	0.090	0.008	0.079	0.006

reflects the arithmetic mean of the height of all points in the topography within the analysed measurement section. The combination of the R$_a$, R$_{sk}$ and R$_{ku}$ parameters has a major significance in terms of the surface resistance to the dirt covering. The R$_{ku}$ parameter value close to 3 represents a Gaussian distribution, while higher and
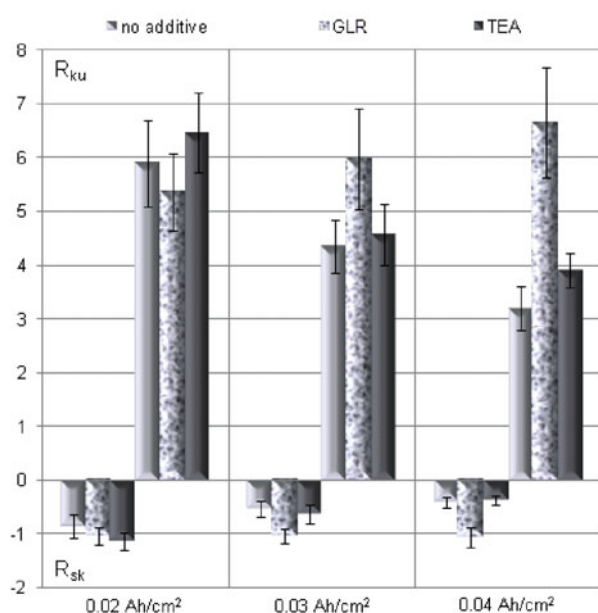

Figure 3. The average values and standard deviation bars of the R$_{ku}$ and R$_{sk}$ roughness parameters for the investigated samples electropolished in the baths with glycerol or triethanolamine and without organic additives

lower values gradually reflect, respectively, narrower and wider distribution. On the other hand, the approximation of the negative R$_{sk}$ value to 0 demonstrates that pores are no longer dominant surface features in relation to the hills. Electropolishing with an addition of TEA improved surface smoothness most effectively, which is illustrated by a progressive decrease of R$_a$ up to 0.079 μm and R$_{ku}$ to 3.9 and a simultaneous increase of R$_{sk}$ to –0.37 (Fig. 3).

CONCLUSIONS

The present investigation has demonstrated that the selection of additives to the electropolishing process may be initially verified by observing the changes in the current/potential characteristics in potentiodynamic tests. Lowering the *plateau* level (and increasing the range of potentials in which it occurs) constitutes proof of the activity of organic compounds and their adsorption on the electropolished surface. Considering the very high boiling temperature, relatively low mass loss during electropolishing, and the simultaneously obtained high gloss and low surface roughness, triethanolamine represents an optimal additive for electropolishing. The comparison of mass loss and gloss increase in samples from bath containing triethanolamine demonstrated that much better results per surface area unit of the electropolished surface were obtained (20.1 GU · cm^2/mg) in comparison to glycerol (17.5 GU · cm^2/mg) and to the control sample electropolished in bath with base composition – without additive (16.5 GU · cm^2/mg) (q = 0.03 Ah/cm^2, j = 0.3 A/cm^2, t = 6 min, T = 55°C). The application of triethanolamine also contributes to a smooth surface. The addition of glycerol requires a prolonged time for reaching similar electropolishing results.

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