

## Corrosion Properties of Electropolished AISI 316L Austenitic Biomaterial in Relation to Electropolishing Conditions

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Electropolishing is recommended for various biomedical applications of austenitic biomaterials because it enables to achieve high brightness, very low surface roughness, excellent corrosion resistance and reduced bacterial attachment without residual surface tensions. The quality of an electropolished surface depends strongly on the composition of used electrolyte and on the applied electropolishing conditions. In this article the corrosion properties of eight AISI 316L surfaces electropolished in the same solution but at the different conditions were evaluated on the bases of the electrochemical impedance spectroscopy (EIS) test and exposure immersion test. The aim was to find whether in the frame of recommended electropolishing conditions (temperature, current density, time) the optimal combination exists which would provide the best corrosion properties of the surface passive film. The results showed that the electropolishing temperature 50 °C should be preferred due to the lower roughness parameters and minimizing the possibility of the bacterial attachment.

**Keywords:** austenitic biomaterial, electropolishing conditions, corrosion resistance, electrochemical impedance spectroscopy test, exposure immersion test.

### 1. INTRODUCTION

Polished stainless steel surfaces are required for various biomedical applications especially for surgical and dentistry instruments (sterilized instruments, endodontic files in root canal therapy, metal posts in root canal treated teeth), arch wires and brackets in orthodontics devices, coronary stents, orthopedic fixation plates and screws [1–4].

Bright smooth surfaces ensure better corrosion resistance [4–8], biocompatibility, cleanness, reduced bacterial attachment and biofilm formation [4, 9–11] compared to materials with higher surface roughness.

A smooth and bright material surface can be commonly obtained by various polishing processes. The main disadvantages of the mechanical polishing are connected with a deformed layer and residual stresses on the treated surface and with foreign particles deposited on the surface during processing [4, 8]. It consequently brings lower corrosion resistance and biocompatibility and finally it affects negatively the durability of produced implants and components [1, 2].

Contrary to this, electropolishing enables to achieve high surface brightness (mirror finish) with very low surface roughness ( $Ra < 0.2 \mu\text{m}$ ) [12], without residual surface tensions and with excellent corrosion resistance [4]. Electropolishing is based on the principle of anode metal dissolution in the electrolyte described by Faraday's law [13, 14]. A particular layer from the metal's surface is removed and a high quality passive oxide film, without impurities as oxygen, hydrogen, carbon, is created. The amount of the removed metal depends mostly on the

electrolyte composition, temperature and applied current density [14–16].

The high quality passive film on the stainless steels surface is related to the chromium enrichment. Iron and nickel atoms are more easily released from the structure than chromium atoms [7, 17], they are preferably removed and Cr/Fe ratio increases [7].

Electrolytic baths for electropolishing are mostly concentrated acid solutions, such as mixtures of sulphuric acid and phosphoric acid or mixtures of perchlorates with acetic anhydride and methanolic solutions of sulphuric acid [13, 14]. For the electropolishing of stainless steels biomaterials mixtures of orthophosphoric acid, sulphuric acid and water/glycerine (for the viscosity increasing) of various ratios of used acids [3, 4, 8, 9] are recommended. Recommended temperatures range is commonly from 40 to 90 °C [3, 4, 9, 18].

For the biomedical applications, electropolishing of austenitic steels is commonly applied after pickling (acid cleaning) used as the chemical surface pretreatment [3, 7].

AISI 316L stainless steel is used as the experimental biomaterial in this work. The corrosion properties of eight surfaces electropolished in the same solution (orthophosphoric acid + sulphuric acid + water) but at the different conditions (temperature 40 or 50 °C, current density 0.6 or 0.8 A/cm<sup>2</sup>, time 7 or 10 minutes) were evaluated on the bases of the electrochemical impedance spectroscopy (EIS) test and exposure immersion test. Both tests were carried out in the 0.9 % NaCl solution at the temperature of  $37 \pm 0.5$  °C for simulation of the internal environment of a human body. The aim was to find whether in the frame of recommended electropolishing conditions (given by the ranges of temperatures, current densities and electropolishing time) the optimal

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combination exists which would provide the best corrosion properties of the surface passive film.

## 2. MATERIALS AND METHODS

The experimental material AISI 316L is Cr-Ni-Mo austenitic stainless steel (wt. %: Cr 16.79, Ni 10.14, Mo 2.03, Mn 0.82, N 0.05, C 0.02, Si 0.31, P 0.03, S 0.001, Fe balance). It was purchased in sheet (1000 × 2000 mm) of 1.5 mm thickness. Its production process was based on continuous casting in electric arc furnace, then it was annealed at 1050 °C. The IIB surface finish (smooth and matte metallic glossy surface) was obtained by pickling after slightly smoothing rolling.

Before the electropolishing the steel surface was mechanically (grinding 1200 mesh) and chemically (pickling) pretreated. The conditions of pickling (in Table 1) were adjusted according to the authors [3].

**Table 1.** Conditions of pickling (acid cleaning)

Component	Volume, ml	Temperature, °C	Time, s
HF	3	22 ± 3	3600
HNO <sub>3</sub>	9		
H <sub>2</sub> O	To 100 ml		

Due to the ecology and the safety of the manipulation, the electropolishing solution without perchloric acid operating at the lowest possible temperature was chosen. Electropolishing was performed in H<sub>3</sub>PO<sub>4</sub> + H<sub>2</sub>SO<sub>4</sub> + H<sub>2</sub>O electrolyte [3, 12, 18]. The polished specimen (15 × 40 × 1.5 mm) was immersed into electrolyte and connected to the positive pole of the power source as an anode. A cathode was of the same material as the anode. In the frame of the electropolishing conditions recommended by authors [3, 12, 18] and defined by the ranges of temperatures, current densities and electropolishing time, eight various combinations of the electropolishing parameters were chosen (Table 2).

**Table 2.** Chosen combinations of the electropolishing conditions

Specimen designation	Temperature, °C	Current density, A/cm <sup>2</sup>	Time, min
40-0.6-7	40	0.6	7
40-0.6-10	40	0.6	10
40-0.8-7	40	0.8	7
40-0.8-10	40	0.8	10
50-0.6-7	50	0.6	7
50-0.6-10	50	0.6	10
50-0.8-7	50	0.8	7
50-0.8-10	50	0.8	10

For comparison, corrosion resistance was tested also on the as received steel surface, i.e. original surface without additional mechanical and chemical treatment.

0.9 % sodium chloride solution (specific conductivity 15.51 mS/cm, pH 7.15) and the temperature of 37 ± 0.5 °C for simulation the internal environment of the human body was used as the corrosion environment for both exposure immersion test and EIS.

The specimen's shape for 50-days exposure immersion test was rectangular (15 mm × 40 mm × 1.5 mm). The specimens were degreased by ethanol and weighted out with accuracy ± 0.000 01 g before the test. The group of

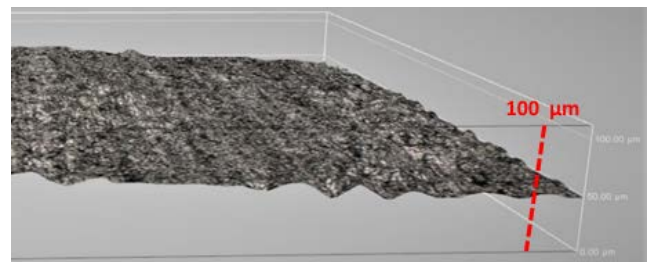
three parallel specimens was tested for each combination of the electropolishing conditions and for the as received surface as well. After exposure the specimens were carefully brushed, washed by de-mineralized water, freely dried up and weighted out again [19].

The EIS test for the same types of surfaces was performed in the conventional three-electrode cell system with a calomel reference electrode (SCE) and a platinum auxiliary electrode (Pt) using Voltalab 10 corrosion measuring system with PGZ 100 measuring unit. The time for potential stabilization between the specimen and the electrolyte was set to 5 min. The exposed area of a specimen was 1 cm<sup>2</sup>. The measurement frequency ran in a range from 100 kHz to 5 mHz. Results of EIS measurements were displayed as the Nyquist diagrams plotted in coordinates of real and imaginary impedance components. The representative curve for each type of surface was selected from at least three measurements for the same type of surface. The polarization resistance ( $R_p$ ) values were obtained on the basis of the analysis of these representative Nyquist curves by EC-LAB software.

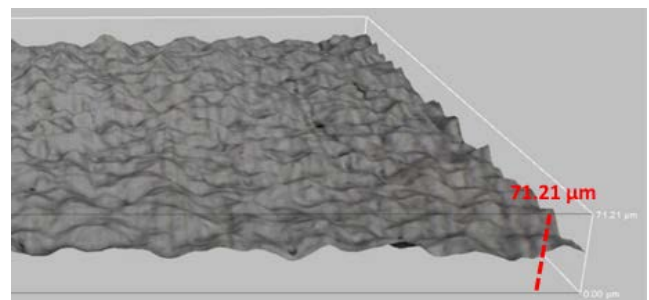
## 3. RESULTS AND DISCUSSION

### 3.1. Topography and roughness parameters

As can be considered from Fig. 1 and Fig. 2 there is a marked difference between the topography of the as received and the electropolished surface (Nikon AZ 100).



**Fig. 1.** Topography of the as received surface



**Fig. 2.** Topography of the electropolished surface on the example of 50-0.8-10 specimen

The roughness of all tested surfaces was evaluated by  $R_a$  (roughness average),  $R_z$  (average maximum peak to valley height) and  $R\Delta q$  (root mean square slope) parameters (measurements performed by Mitutoyo SJ 400). According to the authors [9]  $R\Delta q$  parameter is better for the description of the electropolished surfaces than commonly used roughness amplitude parameters and unlike to them it is insensitive to the scale. The above mentioned roughness parameters of all tested surfaces are listed in Table 3. According to the obtained results, the

electropolishing performed at the higher temperature (50 °C) ensured the lower values of all monitored roughness parameters.

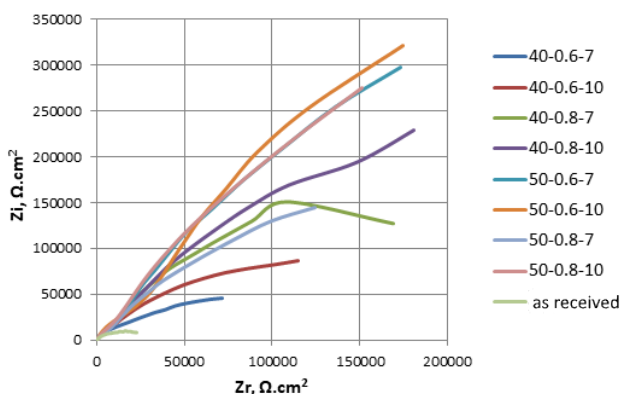
**Table 3.** Roughness parameters values of all tested surfaces

Specimen designation	$Ra$ , $\mu\text{m}$	$Rz$ , $\mu\text{m}$	$R\Delta q$
as received	0.22	2.3	–
40-0.6-7	0.14	1.1	0.04
40-0.6-10	0.06	0.3	0.02
40-0.8-7	0.12	1	0.04
40-0.8-10	0.11	0.7	0.04
50-0.6-7	0.06	0.3	0.02
50-0.6-10	0.05	0.3	0.02
50-0.8-7	0.09	0.4	0.02
50-0.8-10	0.05	0.3	0.02

### 3.2. EIS test

The impedance spectra measured on the basis of the EIS method were simple and the Nyquist curves were similar to those obtained for austenitic stainless steels by the authors [7, 21, 22]. Therefore, as in the aforementioned works, a single loop circuit consisting of  $R_Q$  (electrolyte resistance),  $R_p$  (polarization resistance) and CPE block connected to the circuit instead of the capacitance, was used for evaluation of the curves. The CPE block was used to simulate the inhomogeneities of the surface layer [7].

The Nyquist curves for all types of surfaces are shown in Fig. 3, values of the electrochemical parameters calculated by the EC-LAB software are listed in Table 4.



**Fig. 3.** Nyquist curves for all tested AISI 316L surfaces types

The specimen electropolished at 50 °C, current density 0.6 A/cm<sup>2</sup>, 10 minutes (50-0.6-10) showed the highest polarization resistance  $R_p$ . This result is in accordance with the measured low roughness parameters  $Ra$ ,  $Rz$ ,  $R\Delta q$  and it points to the high quality of the passive film and high corrosion resistance. Also, authors [5, 6, 12] documented close relation between the low roughness and the high quality of the passive film.

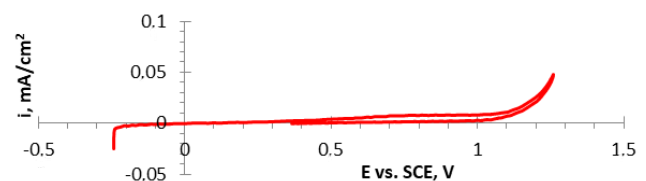
The lowest  $R_p$  value (among the electropolished specimens) was measured for the surface electropolished at 40 °C, current density 0.6 A/cm<sup>2</sup>, 7 minutes. The increase of the current density (0.8 A/cm<sup>2</sup>) at this temperature brought around double  $R_p$  increase. A simultaneous increase in current density and time at 40 °C caused a 3.5-fold increase in  $R_p$ . When assessing the surface passive film quality, the threshold  $R_p$  value could be considered

327 485  $\Omega$ , obtained for the specimen 40-0.8-7 (electropolished at 40 °C, 0.8 A/cm<sup>2</sup>, 7 minutes), therefore the same material, identically pretreated and electropolished in the same solution at the same conditions and tested by cyclic potentiodynamic polarization test showed according to the authors [23] high quality passive film without initiation of the pitting (potentiodynamic curve with low passive current density and without depassivation over the whole passivity region, Fig. 4).

Based on this consideration, the passive films of specimens with higher  $R_p$  values could be considered to be of the sufficient quality and corrosion resistance.

**Table 4.** Values of obtained EIS parameters

Specimen designation	Electrolyte resistance $R_Q$ , $\Omega$	Polarization resistance $R_p$ , $\Omega$
as received	47.82	22 170
40-0.6-7	36.87	77 372
40-0.6-10	67.25	154 072
40-0.8-7	25.37	327 485
40-0.8-10	47.71	559 267
50-0.6-7	29.15	931 730
50-0.6-10	82.26	1 299 000
50-0.8-7	20.05	371 489
50-0.8-10	28.34	891 427



**Fig. 4.** Cyclic potentiodynamic curve of electropolished AISI 316L working electrode identically pretreated and electropolished at 40 °C, 0.8 A/cm<sup>2</sup>, 7 min [23]

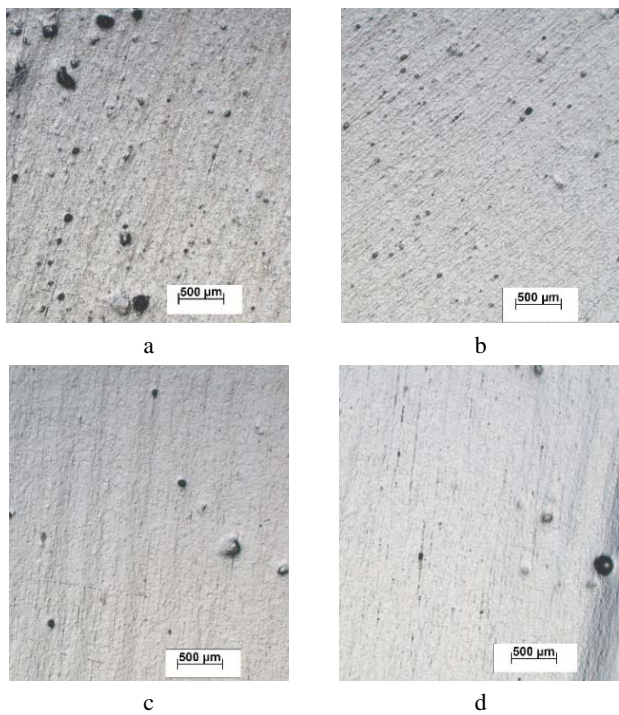
### 3.3. Exposure immersion test

Tested specimens were locally damaged by the pitting during 50-days immersion test. The corrosion damage was not visible to the naked eye, it was visualized at optical microscope (Zeiss Axio Imager). According to Fig. 5 and Fig. 6 corrosion pits on the electropolished surfaces are mostly round or oval in shape and they are normally located in the lines originated by mechanical grinding of the surface. The largest pits can be observed on the 40-0.6-7 surface which is the electropolished one with the lowest  $R_p$  value.

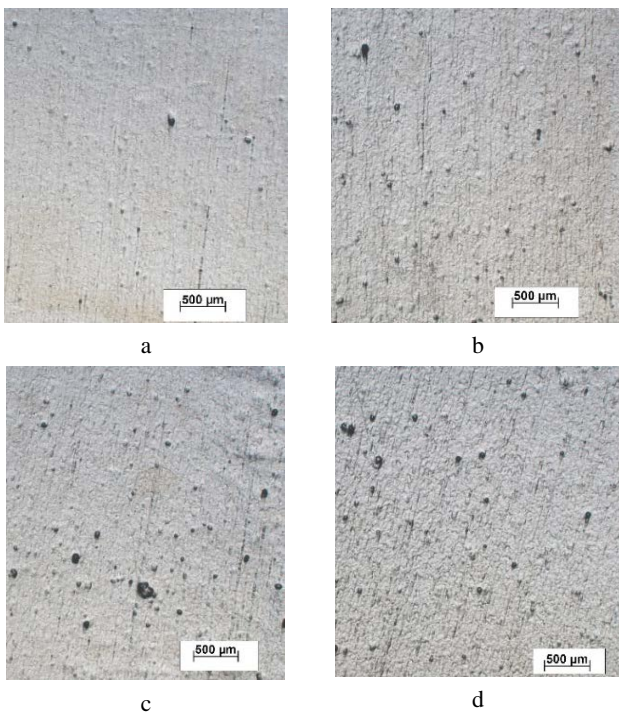
Corrosion pits on the as received surface (Fig. 7) are unlike the pits on the electropolished ones mostly irregular in shape and they are uniformly spread over the entire surface. It could be related to the markedly lower quality of the passive film which enabled the local penetration of the chloride anions in the numerous places.

The observed pitting corrosion of all tested specimens was reflected in their mass losses during 50-days immersion test and the average corrosion rates were calculated (Table 5).

It should be taken into account that the pitting corrosion attack is related to the locale surface imperfections and therefore the corrosion rate cannot be the substantial factor in the evaluation of the local corrosion resistance [19, 20, 24].



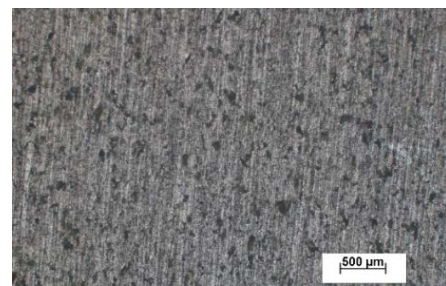
**Fig. 5.** Typical pitting corrosion damage of the specimens, electropolished at 40 °C: a–40-0.6-7; b–40-0.6-10; c–40-0.8-7; d–40-0.8-10



**Fig. 6.** Typical pitting corrosion damage of the specimens, electropolished at 50 °C: a–50-0.6-7; b–50-0.6-10; c–50-0.8-7; d–50-0.8-10

As can be considered from Table 5 there is marked difference between the average corrosion rate of the as received specimens and the electropolished ones.

The lowest average rate of the as received specimens, points to the local corrosion attack with high number of shallow pits (pitting corrosion with large anode area) which does not cause the marked loss of the material.



**Fig. 7.** Typical pitting corrosion damage of the as received specimen

**Table 5.** Average corrosion rates calculated from mass losses during the exposure test

Specimen designation	Average corrosion rate, $\text{g}\cdot\text{m}^{-2}\cdot\text{day}^{-1}$
as received	0.00108
40-0.6-7	0.02452
40-0.6-10	0.01831
40-0.8-7	0.01320
40-0.8-10	0.02348
50-0.6-7	0.02108
50-0.6-10	0.02015
50-0.8-7	0.01963
50-0.8-10	0.02381

The shallow pits on stainless steels after being subjected to a deformation without consequent surface treatment were also documented by the authors [25]. It should be remembered that the as received surface of austenitic biomaterial would not be appropriate in internal environment of human body because of the low quality of passive film ( $R_p$  only 22 170  $\Omega$ ) and high surface roughness ( $R_a = 0.22 \mu\text{m}$ ,  $R_z = 2.3 \mu\text{m}$ ) which would not provide required antibacterial properties.

Based on the photo documentation (Fig. 5 and Fig. 6) the corrosion of the electropolished specimens is taking place with a small anode surface (a smaller number of corrosion pits compared to the as received surface) and a large cathode surface (intact electropolished passivated surface). Although the number of corrosion pits is smaller, the higher material loss points to the deeper corrosion pits. This can cause serious damage of the biomaterial, which can have a negative impact when used in the human body environment (e. g. leakage of the toxic ions from the implant, [2]). Results of the exposure immersion test point to the fact that from the perspective of long-term application into the human body, the imperfect mechanical pretreatment of the electropolished austenitic biomaterial might have a worse impact on the corrosion damage than narrowly specified electropolishing conditions (electrolyte temperature, current density, time).

### 3. CONCLUSIONS

1. Electropolishing performed at the higher temperature (50 °C) ensured lower values of the monitored roughness parameters.
2. The surface electropolished at 50 °C, current density 0.6 A/cm<sup>2</sup>, 10 minutes, showed the highest polarization resistance  $R_p$ .

3. The lowest  $R_p$  value (of electropolished specimens) was measured for the surface electropolished at 40 °C, current density 0.6 A/cm<sup>2</sup>, 7 minutes.
4.  $R_p$  obtained for the surface electropolished at 40 °C, 0.8 A/cm<sup>2</sup> 7 minutes (327 485 Ω), which showed the high quality passive film also by cyclic potentiodynamic polarization test [23] could be considered the threshold  $R_p$  value. Based on this consideration, passive films of specimens with higher  $R_p$  values could be considered to be of the sufficient quality and corrosion resistance.
5. All tested specimens were locally damaged by pitting during 50-days immersion test.
6. Corrosion pits on the electropolished surfaces are mostly round or oval in shape and they are normally located in the lines originated by mechanical grinding.
7. Corrosion pits on the as received surface are irregular in shape and they are uniformly spread over the entire surface.
8. The obtain results of the exposure immersion test show that the factor of imperfect mechanical pretreatment of the surface probably had a more important influence on the corrosion damage than the narrow specified parameters of the electrochemical polishing and the associated quality of the passive film.

Based on the results of both independent corrosion tests. It can be summarized that in the frame of considered electropolishing conditions, the electropolishing at 50 °C should be preferred due to the lower roughness parameters and therefore minimizing the possibility of the bacterial attachment. It is also important to ensure the perfect mechanical surface pretreatment of the austenitic biomaterial.

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