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INFLUENCE OF THE SURFACE FINISHING ON ELECTROCHEMICAL CORROSION CHARACTERISTICS OF AISI 316L STAINLESS STEEL

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Resume

Stainless steels from 316 group are very often and successfully uses for medical applications where the good mechanical and chemical properties in combination with non-toxicity of the material assure its safe and long term usage. Corrosion properties of AISI 361L stainless steel are strongly influenced by surface roughness and treatment of the engineering parts (specimens) and testing temperature. Electrochemical characteristics of ground, mechanically polished and passivated AISI 316L stainless steel specimens were examined with the aim to identify the polarization resistance evolution due to the surface roughness decrease. Results obtained on mechanically prepared specimens where only natural oxide layer created due to the exposure of the material to the corrosion environment was protecting the materials were compared to the passivated specimens with artificial oxide layer. Also the influence of temperature and stabilization time before measurement were taken into account when discussing the obtained results.

Positive influence of decreasing surface roughness was obtained as well as increase of polarization resistance due to the chemical passivation of the surface. Increase of the testing temperature and short stabilization time of the specimen in the corrosion environment were observed negatively influencing corrosion resistance of AISI 316L stainless steel.

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1. Introduction

Due to their high strength, good corrosion resistance and oxidation resistance at temperatures up-to 550 °C are stainless steels often used engineering materials. Based on the microstructure four stainless steels groups can be identified: ferritic with application mainly for electrical industry, martensitic stainless steels in applications requiring high strength, austenitic stainless steels for applications requiring good corrosion resistance, weldability and good forging properties; and the last but not least group of "duplex" stainless steels for which are able to combine superior properties of previously described groups by combination of various microstructures [1].

Due to the good corrosion resistance and non toxicity a wide range of medical application are suitable for stainless steels as well. 316 type of stainless steels in the most widely used group for medical applications [2 - 9]. 316 group materials are used for production of medical devices, implants, drug transfer

materials, screws, and surgical equipment [4, 8, 9].

AISI 316 stainless steels microstructure consists of austenite, what means that chemical composition of these grades has to be optimized to maintain austenitic phase stability at room temperature as well as shifting of the martensitic transformation temperature well below room temperature, what is a result of the Ni addition to the material. Corrosion resistance of stainless steels is a result of the passivation effect of Cr addition. Mo and N addition improve the steels corrosion resistance against pitting and crevice corrosion. Ti is used for steel stabilization, thus minimizing carbide formation [2 - 7]. Microstructural changes due to the structural sensitization negatively influence corrosion resistance of AISI 316L in 0.9 % NaCl at various temperatures [8] and slightly decrease material fatigue properties [10].

AISI 316L has very good corrosion resistance in human liquid (salt solution) environments [8, 9]. The corrosion resistance can be even improved by addition of Mo or Cu. Mo improves its resistance against the acids and point corrosion. AISI 316L steel has good weldability without need of additional heat treatment, very good formability and it is possible to polish it to the mirror like surface [11].

Surface roughness is a factor influencing materials corrosion resistance. All the materials create a protective oxide layer when exposed to the corrosion environment [5, 6]. Quality of the oxide layer (thickness, how compact the layer is, its porosity etc.) influence following material protection against corrosion. Free material surface is more reactive when compared to the surface covered by compact oxide layer. AISI 316L stainless steel corrosion resistance is dependent on the surface roughness. In [12] the negative influence of surface roughness was proved, however after 50 days of exposure no difference between ground specimens (high surface roughness),

mechanically polished (small surface roughness) and chemically passivated specimens (artificial oxide layer) was observed performing immersion tests. The negative influence of temperature to the polarization potential was proved in [13] where decrease of polarization resistance of AISI 316L at 40 °C by 50 % at stabilization time of 30 min was observed when compared to polarization resistance values obtained at 20 °C. Polarization resistance measured at temperature of 50 °C was only 40 % of the value measured for 20 °C.

In the present work electrochemical characteristics of ground, mechanically polished and passivated AISI 316L stainless steel specimens were examined with the aim to identify the polarization resistance evolution due to the surface roughness decrease. Results obtained on mechanically prepared specimens where only natural oxide layer created due to the exposure of the material to the corrosion environment was protecting the materials were compared to the passivated specimens with artificial oxide layer. Also the influence of temperature and stabilization time before measurement were taken into account when discussing the obtained results.

2. Experimental material and procedures

AISI 316L stainless steel with different surface finishing treatment was used as the experimental material. Experimental material was delivered in a form of cold rolled sheets, however no visible deformation of the microstructure (grains deformation – elongation in the rolling direction) due to the rolling production process was observed.

Four different mechanical treatments of specimen surface and their combination with surface passivation were applied before the electrochemical characteristics measurement. Mechanical treatment consisted of mechanical grinding on SiC papers (no. 60 and 320) and polishing (diamond paste, 1 μm). After mechanical preparation followed passivation (P) of the surface in a solution of 20 % HNO_3 was

performed on mechanically treated specimens. For the passivation 500 ml of the 20 % HNO_3 solution heated to the temperature of 50 °C was used. Prepared specimens were inserted to the heated passivation solution for 30 minutes. Specimen surface finishing and marking is given in Table 1.

Electrochemical impedance spectroscopy (EIS) was used for the electrochemical characterization of AISI 316L stainless steel with different surface finishing. Experimental measurements were performed at temperatures of 22 °C and 40 °C in simulated body fluid solution. Chemical composition of the used solution is given in Table 2. For both the experimental temperatures the stabilizing time of the free potential between the specimen and electrolyte (simulated body fluid solution) was 1 and 30 minutes. EIS measurements were performed in frequency range from 100 kHz to 10 mHz (only to 5 mHz in some cases depending on the material reaction). Voltalab 4 software was used for analysis of obtained Nyquist plots. As a result of EIS measurements the values of polarization resistance were obtained.

3. Experimental results and discussion

The values of polarization resistance measured on mechanically treated and passivated specimens are given in Table 3.

From the results obtained for mechanically treated specimens a significant influence of the surface roughness can be considered. Decreasing the surface roughness by finer grinding and polishing by diamond paste increased the polarization resistance in the case of 22 °C as a testing temperature. In the case of 40 °C, as a testing temperature, small decrease of polarization resistance for specimens ground on paper 320 was observed when compared to specimens ground on paper no. 60, however the decrease is only minor. When the polishing by diamond paste for the mechanically treated specimens surface was applied the values of polarization resistance were higher by order of magnitude at both the experimental temperatures.

Longer stabilizing time (30 min) before EIS measurement always led to the polarization resistance values increase. In Fig. 1 the comparison between the polarization resistance for specimens tested at 22 °C with stabilization time of 1 min (Fig. 1a) and 30 min (Fig. 1b) are shown. Decreasing the surface roughness increased the polarization resistance of tested specimens. The increase of the polarization resistance values (the radius of semicircles − Nyquist plots) due to the passivation of the mechanically treated surface was observed in all the specimens surface finish state.

Table 1

Surface treatment of AISI 316L specimens used for corrosion testing.

marking	amount	surface treatment
60	13	grinding by a paper no. 60
60P	13	grinding by a paper no. 60 + passivation
320	13	grinding by a paper no. 320
320P	13	grinding by a paper no. 320 + passivation
LS	13	polishing
LSP	13	polishing + passivation
basic state	6	without treatment

Table 2

Chemical composition of solution used for the corrosion testing. The amounts are for 1 litre of the distilled water.

chemicals	weight (g)
NaCl	8.00
KCl	0.42
$CaCl_2*2H_2O$	0.32
$NaCO_3$	0.20

Polarization resistance a different temperatures and stabilizing time.

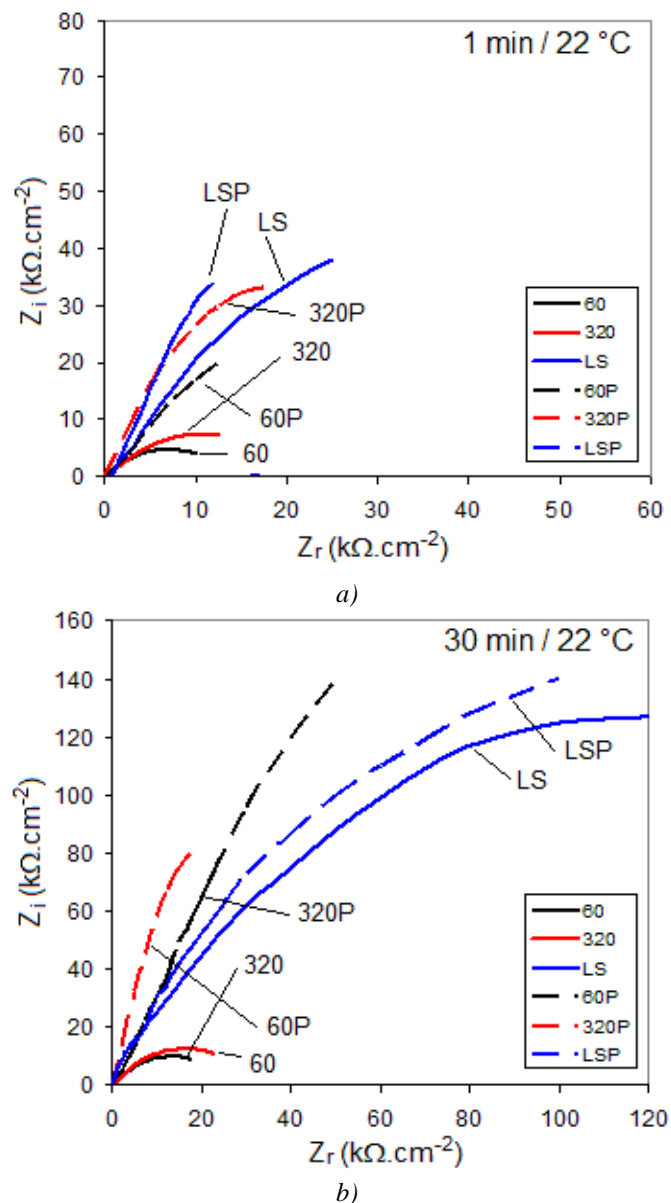| stabilizing time and temperature	1 min / 22 °C	30 min / 22 °C	1 min / 40 °C	30 min / 40 °C
mechanically treated specimens	polarization resistance (kΩ.cm^{-2})			
60	13.65	31.91	9.39	72.73
320	23.80	36.70	9.23	69.36
LS	320.97	449.00	151.06	425.25
passivated specimens	polarization resistance (kΩ.cm^{-2})			
60P	96.62	349.30	67.24	323.13
320P	118.95	445.10	135.73	289.77
LSP	466.80	447.77	432.87	316.93

a)

b)

*Fig. 1. Nyquist plots of AISI 316L stainless steel for 22 °C, a) Stabilization time 1 min at 22 °C,
b) stabilization time 30 min at 22 °C.*
(full colour version available online)

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This means that higher corrosion resistance was reached by passivation in solution of 20 % HNO_3 of mechanically treated specimens when compared to only polished specimens. Also the significant increase of polarization resistance values in the case of stabilization time of 30 min can be seen when compared to the 1 min stabilization in the simulated body fluids (Fig. 1b vs. Fig. 1a). This was caused by natural creation of protective oxide layer on specimen surfaces due to the exposure to the corrosion environment. In the case of longer stabilizing time more compact and thicker oxide layer was created on the specimen surface which protected the material against following corrosion.

Nyquist plots for specimens tested at 40 °C are given in Fig. 2. Also in this case a significant influence of passivation on the values of polarization resistance was observed.
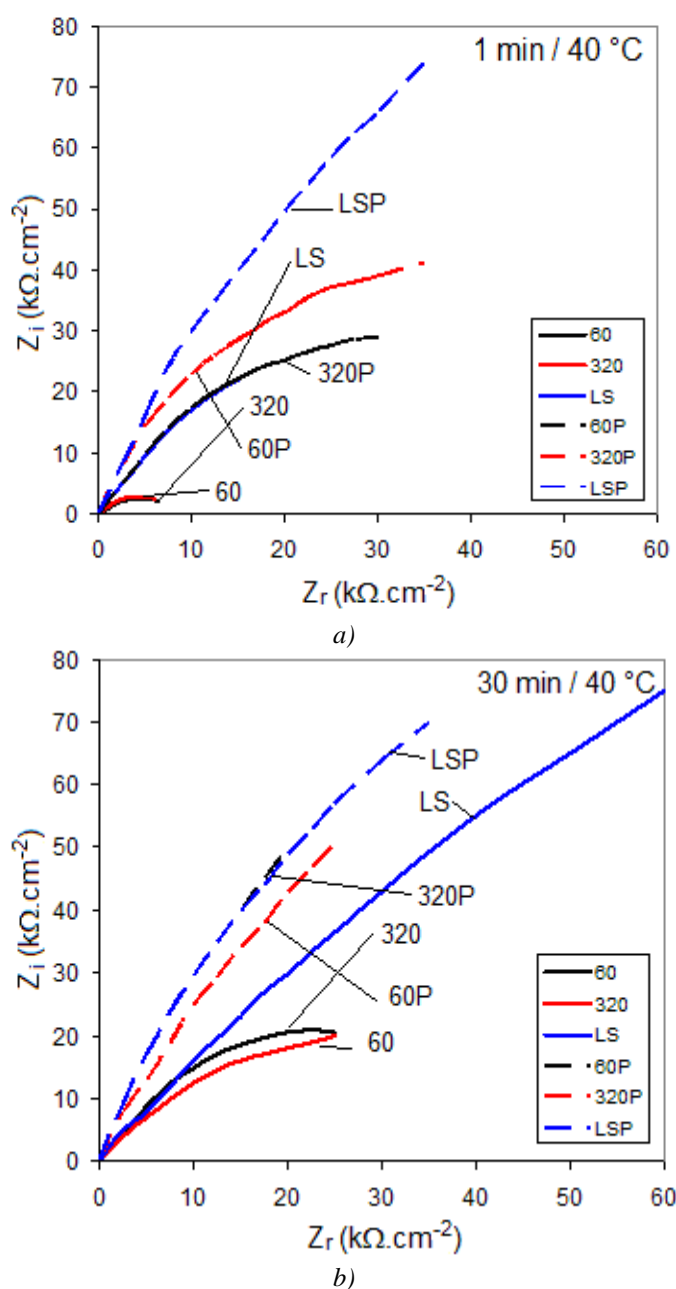

Fig. 2. Nyquist plots of AISI 316L stainless steel for 40 °C, a) Stabilization time 1 min at 40 °C,
b) Stabilization time 30 min at 40 °C.
(full colour version available online)

Even without passivation the decrease in the surface roughness (mechanically ground and polished specimens) led to the increase of polarization resistance which means higher corrosion resistance. After passivation even higher values of polarization resistance were measured.

When compared mechanically treated states of specimens and passivated specimens a positive influence in the case of longer stabilizing time at temperature of 22 °C was observed. In the case of mechanically treated specimens a significant increase of polarization resistance due to the passivation was observed at 22 °C at both the stabilizing times (Fig. 3). By passivation of the surface the real surface was covered by compact and homogenous oxide layer, when compared to the naturally formed oxide layer of the specimen surface on the air and corrosion environment. Artificially created passivation layer protected the material against the corrosion much better than the natural one. By the longer time of stabilization both the surfaces reached similarly protective oxide layers (naturally created on the polished surface and due to the passivation).

In the case of the testing temperature of 40 °C and stabilization time of 1 min an increase of polarization resistance at 22 °C for all the mechanical treated specimens by surface passivation was observed, Fig. 4a. In the case of longer stabilization time the polarization resistance improvement only in the case of ground specimens was observed. In the case of polished specimen the surface passivation resulted in polarization resistance decrease, Fig. 4b, which could be explained by higher Cl ions activity at higher temperature. While the Cl ions were more active at 40 °C when compared to their activity at 22 °C the degradation of passive layer created during the surface passivation can be damaged earlier.

At stabilization time of 1 min higher temperature decreased the polarization resistance of mechanically treated specimens however an increase of polarization resistance for passivated specimens was observed, Fig. 3 vs. Fig. 4. At longer stabilizing time (30 min) the temperature negatively influenced the corrosion resistance of examined steel. The polarization resistance of individual specimens is lower at 40 °C when compared to the 22 °C, Fig. 4 vs. Fig. 3.
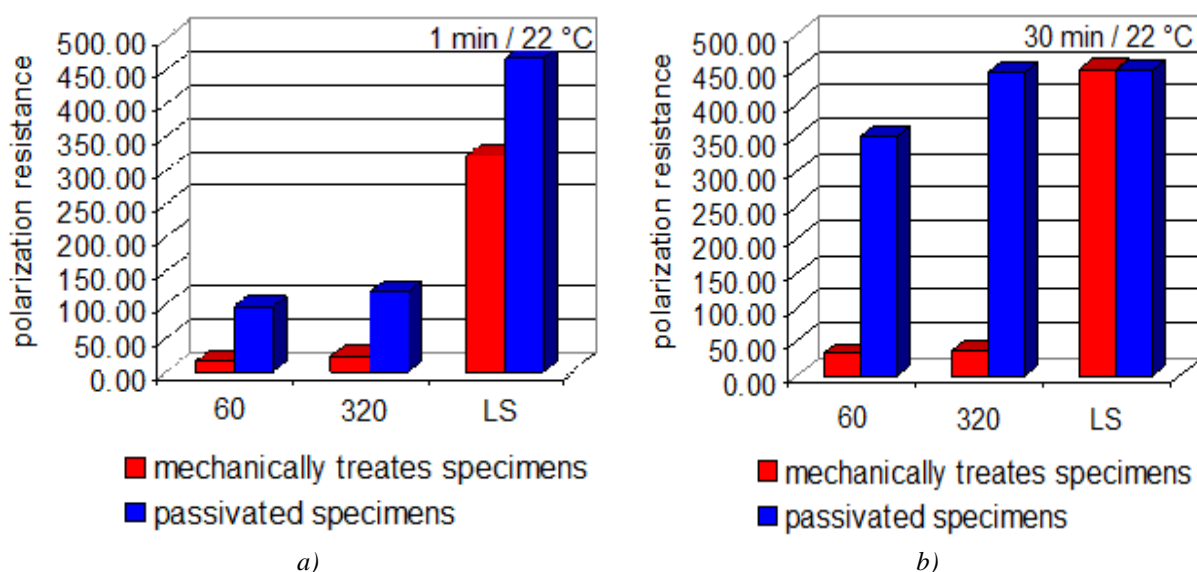

a)   *b)*

Fig. 3. Polarization resistance of AISI 316L stainless steel at 22 °C, a) Stabilization time 1 min at 22 °C,
b) Stabilization time 30 min at 22 °C.
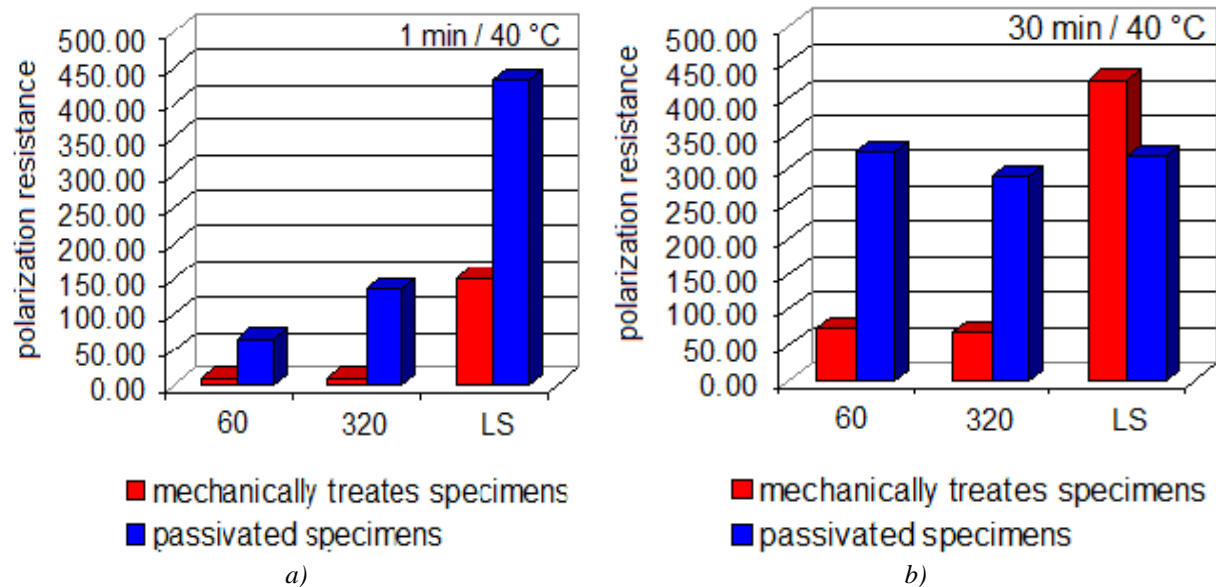*(full colour version available online)*

Fig. 4. Polarization resistance of AISI 316L stainless steel at 40 °C, a) Stabilization time 1 min at 40 °C,
b) Stabilization time 30 min at 40 °C.
(full colour version available online)

4. Conclusions

AISI 613L stainless steel commonly used for biomedical application exhibits good corrosion properties, however the surface finishing has an important influence on the reached corrosion characteristics of the material. From the experimental measurements presented in the paper following conclusions can be reached:

- by decreasing of the surface roughness the polarization resistance of AISI 316L increased,

- passivation in a solution of 20 % HNO_3 increased polarization resistance of AISI 316L stainless steel specimens,

- at longer stabilizing time in corrosion environment the naturally created protective layer oxides on specimens with mechanically polished surfaces reached similar values of polarization resistance as chemically passivated specimens,

- positive influence of chemical passivation on the AISI 316L polarization resistance was observed at 22 °C when compared to 40 °C.

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