Surface Properties of the Stainless Steel X10 CrNi 18/10 after Aplication of Plasma Polishing in Electrolyte

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Abstract: *The first part of the paper deals with surface properties of the stainless steel specimen after plasma polishing in electrolyte. The plasma polishing technology is considered as a more environmental friendly alternative to the common electrochemical polishing process (electropolish). The electrolyte solutions use no acids or toxicants, low concentrated water solutions of various salts are used instead. The surface roughness, the gloss level and the thickness of removed layer are in the focus, as well as their dependence upon the treatment time. The results are then compared to the surface properties of specimens treated by standard electrochemical polishing. The specimens of the same material, the same shape and the same surface state have been used for both polishing technologies. Finally, the differences between those polishing processes and the differences between the properties of the polished surfaces are discussed.*

Keywords: *electrolyte, plasma, polishing, stainless steel*

I. INTRODUCTION

Polishing of stainless steels is widely used, mainly in order to improve the corrosion resistance of its surface as well as to reduce the surface roughness. Polishing is the most common surface finishing operation of stainless steel products in the field of medical, pharmaceutical, food or chemical industry. The corrosion resistance improvement is not the only benefit of the polishing process, but the surface also becomes smoother and more resistant to dirt, bacteria, rot, etc. [2, 3].

Surface polishing in acids mixture under the influence of electric current, e.g. the electrochemical polishing, known as electro- polishing, is the most commonly used polishing method for surface finishing of complexly shaped industrial parts. A new polishing technology, the plasma polishing in electrolyte has been developed as an alternative to the standard electrochemical polishing. This paper deals with some properties of stainless steel surface treated by the plasma polishing process in electrolyte. Especially the surface roughness reduction, the rise of gloss level and the material removal rate are examined.

II. THE PRINCIPLE OF THE PLASMA POLISHING

The metal part, to be treated, is immersed into the electrolyte and is connected to the plus pole of the electric current supply. So the treated part is the anode. The second electrode is connected to the minus pole of the power supply. As it can be seen on the *Fig. 1*, the principal scheme of this process is similar to the electro-chemical polishing at first glance. The main differences are in the voltage value and in the chemical composition of the electrolyte. The high concentrated mixture of acids is displaced by low-concentrated water solution of chemically neutral salts. A water-steam film is forming on the entire treated surface due to high value of the voltage between electrodes. The film is electrically nonconductive and it separates the metal surface from the electrolyte. In this way the electric current is broken so the electric circuit gets disconnected. But if the voltage between the electrodes is high enough (a few hundreds of volts), the water-steam film becomes ionized due to high value of the electric field in the thin film. In this case, the electric current flows through the water-steam film in the form of glow discharge. In this way, the discharge act on the metal surface in this process. The discharge always runs toward the peaks of the surface profile in the form of thin columns since the distance between the electrodes (metal – wall of electrolyte) is here shortest. In this way the surface peak is quickly removed. When the material of the peak is removed, the column of discharge moves to another surface peak where the thickness of the water-steam layer is less. In this way the surface becomes smoother. If a proper chemical composition of the electrolyte and suitable parameters of the process are used, the treated surface becomes glossy [4].

One of the advantages of this technology is in the possibility to treat metal parts with complex and irregular shape. Furthermore, there is no shielding effect as it is in the case of electrochemical polishing. The next advantage of the plasma polishing process is in the harmless composition of the electrolyte. The electrolytes are based on environmental friendly solution of various salts. The solutions of concentrations 4 ÷ 6% are prepared by dissolving
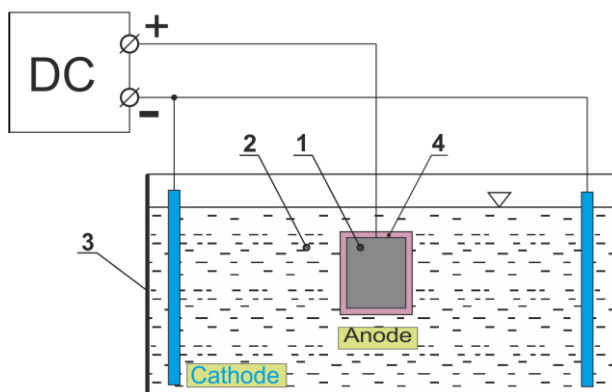

Fig. 1 The principal scheme of the process;
1 – treated specimen, 2 – electrolyte solution, 3 – work vessel, 4 – ionized water-steam film, 5 – power supply.

granulate in the water without extra demand on its quality. In the spent solution, iron is bound in the form of oxides. [2, 3].

III. THE SPECIMENS

A rolled sheet of austenitic stainless steel X10 CrNi 18/8 has been used for specimens. The specimens of dimensions 50 mm × 50 mm × 3 mm has been cut from the sheet using water jet cutter. Two holes of diameter 5 mm were drilled to fix the specimen to its holder during polishing. The surface of the specimens was not treated e.g. it was in crude (rolled) state. Due to untreated surface layer the initial (before polishing) gloss level of the specimen had the value of 7.5 GU only. Its average initial value of the surface roughness Ra was 1.7 μm.

IV. THE EXPERIMENTAL PROCEDURE

The experiment has been focused on the effect of plasma polishing upon the treated surface's properties. The properties have been examined are: the gloss level, the surface roughness and the layer thickness removal Δh. To know the exact material removal rate is important when precise machine parts with tight tolerances are polished. The total treatment time lasted 5 minutes and was divided into a few intervals to get the surface properties dependence on the treatment time. After each polishing step the examined properties were measured always on the same spot on the specimen. The conditions and the process parameters were kept on constant values during each plasma-polishing step. The electrolytic solution of concentration 6 % has been used at temperature 65°C and the immersion depth of the specimen was 100 mm.
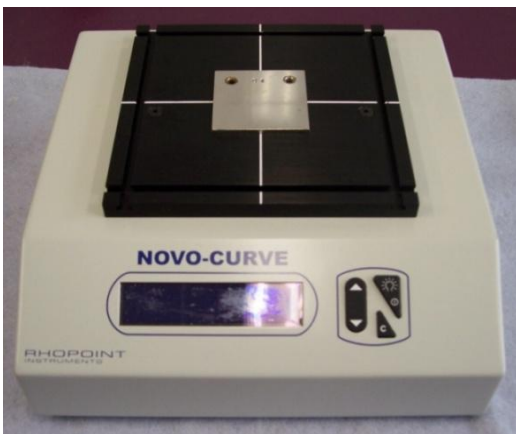

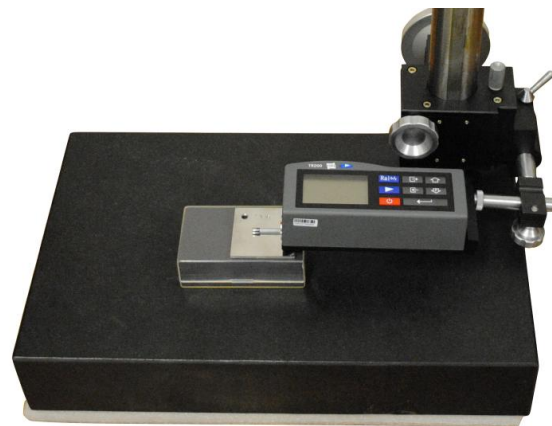Fig. 3 The gloss level measurement on a specimen

Fig. 2 The surface roughness measurement on a specimen using surface tester TR 200

For exact gloss level assessment of the treated specimen a gloss-meter of the type Novo Curve (Rhopoint Instruments, UK) has been used. The gloss meter uses 60° measurement angle and it conforms to ASTM D523 and to ISO 2813. The gloss level has been measured at the geometric centre of the specimen (*Fig. 2*) five times and the average values of these data are in third column of the *TABLE 2*. Surface tester of type TR 200 (*Fig.3*) has been used to evaluate the surface roughness. The resolution of used surface tester is 0.01 μm and it conforms to DIN4772. The tested area was the same as in the case of the gloss level measurements. The surface roughness of Ra has been measured five times at the same spot, the average values are in the *TABLE 1*, as well.

The thickness h_T and the exact weight of the specimens m_T have been measured after each plasma-polishing treatment step, e.g. after the treatment time T, to evaluate the value of the layer thickness removal Δh_T. The value of layer thickness removal can be calculated as the difference between thickness of specimen before and after polishing. Since this value is near the resolution and accuracy of common digital micrometres, such results are not precise enough. Therefore, the material removal rate is calculated from the weight difference of the specimen before and after polishing and the value of the layer thickness removal is computed from this value using own software, developed for this specific purpose. The balance of type PB 303-S

Table 1 *Properties of the specimen #1 after each plasma-polishing step;*

T [min]	Ra_T [μm]	G_T [GU]	m_T [g]	Δh_T [μm]	$\Delta \dot{h}_T$ [μm/min]
0	1.69	8.8	55.379	—	—
0.5	1.51	32.8	55.332	1.09	2.18
1	1.38	66.4	55.270	2.53	2.53
1.5	1.23	87.4	55.222	3.64	2.43
2	1.06	98.8	55.181	4.59	2.30
2.5	1.00	137.3	55.134	5.67	2.27
3	0.98	157.7	55.087	6.76	2.20
4	0.88	205.7	55.016	8.41	2.10
5	0.78	201.7	54.935	10.28	2.06

T – time of treatment, Ra – surface roughness, G – gloss level, m –weight of the specimen, Δh – layer thickness removal, $\Delta \dot{h}_T$ – speed of layer thickness removal

(Mettler-Toledo) with resolution 1 mg has been used. The measured values of the specimen's weight and the calculated data are given in *TABLE 1*. The layer thickness removal is the thickness of the removed material layer from one side of the treated surface, not the specimen's thickness difference. The speed of layer thickness removal $\Delta \dot{h}$ has been calculated from the value

of the layer thickness removal divided by the appropriate treatment time *T*. These data can be found in the last column of the *TABLE 1*.

V.   THE RESULTS

Graphical representation of the obtained data is shown on *Fig. 4*. The value of surface roughness *Ra* decreases by the treatment time nonlinearly, initially it declines quickly but then this decline slows down. So the roughness falls more quickly at the beginning of the polishing process. Using regression analysis of the measured data the following relation has been found:

$$Ra = 1.68 - 0.35T + 0.036\,T^2 \quad [\mu m, min] \tag{1}$$

The gloss level of the treated surface rises almost linearly by the treating time during the first four minutes (*Fig. 4*). After the maximal value is reached, there is no evident change of the gloss level. This relation can be described in the interval 0 to 5 minutes by the following equation:

$$G = 10.8 + 39.6\,T + 8.8\,T^2 - 1.8\,T^3 \quad [GU, min] \tag{2}$$

The layer thickness removal shown in *Fig. 4* seems to be constant by the treatment time at first glance, but as it can be seen in *TABLE 1* the values slightly decrease except the first value. The following equation has been used to describe this dependence in the range of $1 \div 5$ minutes:

$$\Delta h = -0.12 + 2.52\,T - 0.09T^2 \quad [\mu m, min] \tag{3}$$

This equation is not valid in the first minute of treatment, where the value of the layer thickness removal is less. It can be explained by the fact, that the initial specimen's surface had been covered by an oxide film. Metal oxides are electrically nonconductive so it takes longer to remove them for the plasma-polishing process. A graphical presentation of the dependence of the layer thickness removal Δh_T upon the treatment time *T* is shown on the *Fig. 5*.
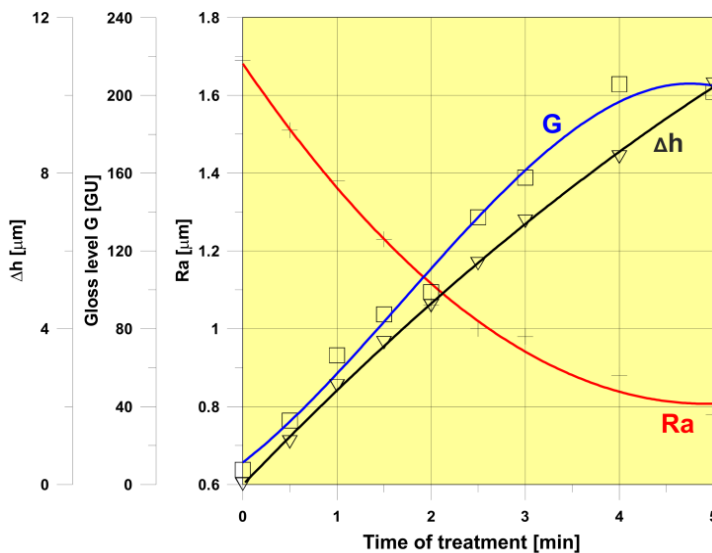

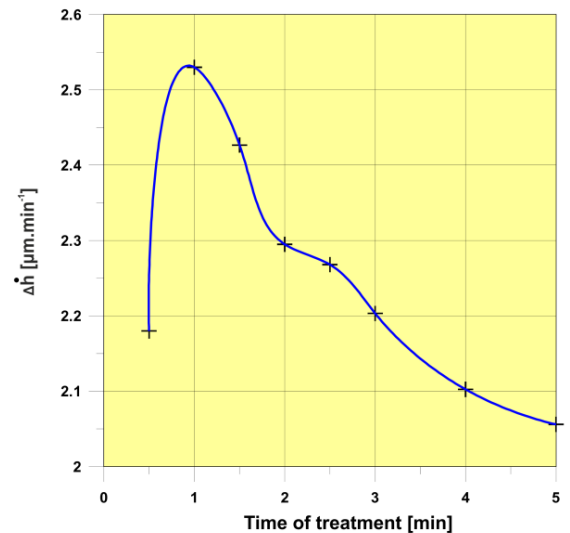Fig. 5 Surface roughness, gloss level and layer thickness removal dependence upon the treatment time

Fig. 4 Dependence of the layer thickness removal speed upon the treatment time

VI.   COMPARISON TO THE STANDARD ELECTROPOLISH

The plasma-polishing technology is an alternative to the commonly used electrochemical polishing process, known simply as "electropolish". There is the question, how the surface parameters will differ if the same samples are processed by both polishing methods. Therefore a few specimens have been polished electrochemically, as well. The specimens of the same material, the same shape and the same surface state as it was in the case of plasma-polishing have been used. The electrolyte solution for electrochemical polishing consisted of 64% H_3PO_4, 13% H_2SO_4 and 23% H_2O [5, 6]. The temperature of the electrolyte has been kept during polishing at 60°C. Four specimens (specimen #2 to #5) have been processed 3 minutes, each at different value of the electric current density: 40 A.dm^{-2}, 50 A.dm^{-2}, 60 A.dm^{-2} and 72 A.dm^{-2}.

Table 2 The measured and calculated properties of the specimens before and after 3 minutes of electrochemical polishing

Specimen No.	Ra_0 [μm]	Ra_3 [μm]	ΔRa_3 [%]	G_0 [GU]	G_3 [GU]	t_0 [mm]	m_0 [g]	m_3 [g]	Δh_3 [μm]	$\Delta \dot{h}_T$ [μm/min]
2	1.67	1.46	-12.6	8.8	116.0	2.8638	55.672	55.202	10.9	3.62
3	1.87	1.50	-19.8	8.8	141.6	2.8202	54.967	55.202	12.9	4.30
4	1.86	1.45	-22.0	8.8	148.5	2.8488	55.609	54.408	14.9	4.98
5	1.75	1.28	-26.9	8.8	184.4	2.8132	54.792	54.056	17.0	5.67

The measurement procedure of the specimens' parameters and the data processing was the same as it was described for the case of plasma-polishing. The measured data can be found in the *TABLE 2*. Subscript 0 is used for values measured before polishing (processing time $T = 0$ min), and subscript 3 is used for values measured after 3 minute of polishing ($T = 3$ min). The values of the layer thickness removal Δh for each specimen, calculated from the specimens' weight difference are also in in *TABLE 2*, as well as the relative values of the surface roughness reduction ΔRa. The results obtained for the specimen #1, which have been treated 3 minutes by plasma-polishing process, are graphically compared to the results obtained for the next four specimens (#2 to #5), which have been treated electrochemically also 3 minutes. *Fig. 6* shows the relative surface roughness reduction ΔRa of the treated specimens. It is obvious, that the plasma-polishing process is much more efficient in surface flattening than the traditional electrochemical polishing process, regardless the used anodic current density. The value of Ra decreases by 42 % when plasma-polishing has been used, the highest decrement of Ra in the case of electrochemical polishing is for the highest used value of anodic current density and its value is 27 % only.

The surface gloss levels of the specimens are compared in *Fig. 7*. As it can be seen on the figure, the reached gloss level of the polished surface is proportional to the electric current density used during electrochemical process. At its highest value (72 A.dm^{-2}) the gloss level is little higher than the gloss level of the plasma-polished specimen. The values of layer thickness removal from the specimens' surface after 3 minutes of treatment are compared at *Fig. 8*. This value is also proportional to the anodic current density in the case of electrochemical polishing as it was for the gloss level or for the surface roughness declension. As it is evident in the picture, all these values are much higher than as the value of layer thickness removal of plasma polished specimen. After 3 minutes of treatment, the plasma-polishing process removes 6.76 μm thick layer of the metal from the treated surface only.

VII. DISCUSSION

Analysing the above results of the accomplished experiments a few conclusions can be drawn. The plasma-polishing process removes the material from the treated surface starting to remove the top of the surface profile's peaks. Even a little amount of the material has been removed when the process starts, it corresponds to a relatively high value of layer thickness removal − marked as *h1* shown in *Fig. 8*. After the tops of the surface profile peaks are disappeared and the process removes the next
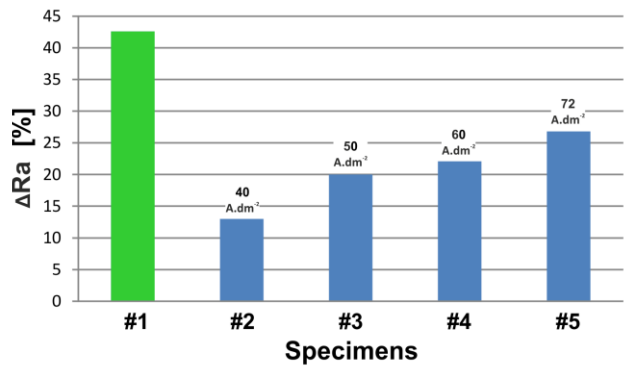

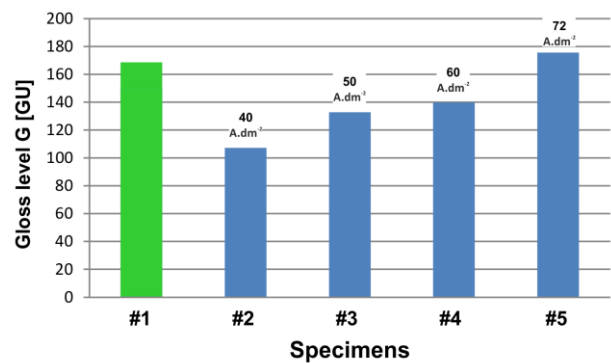Fig. 6 Surface roughness reduction of the specimens after 3 minutes of treatment

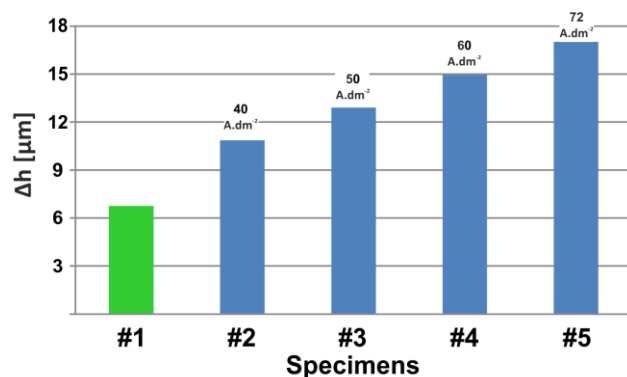Fig. 7 Surface gloss level of the specimens reached after3 minutes of treatment

Fig. 8 The values of the layer thickness removal from the specimens' surface after 3 minutes of treatment

same amount of the material as in the previous case, e.g. *S2 = S1* on the *Fig. 8*, the thickness of this removed layer *h2* is much less as the thickness of the prior layer *h1*. Therefore the value of the layer thickness removal falls down quickly at the

beginning of the treatment and then it continually slows down probably to a constant value. The material removing progress is more complex if the treated surface is covered by a layer of oxides or any other dirt. Since this topmost layer of metal-oxides is electrically nonconductive it takes longer for plasma-polishing process to remove them. It explains the low material removal speed at the beginning of the treatment on the *Fig. 5*. As soon as the topmost layer is removed the surface roughness decreases according to above described theory.
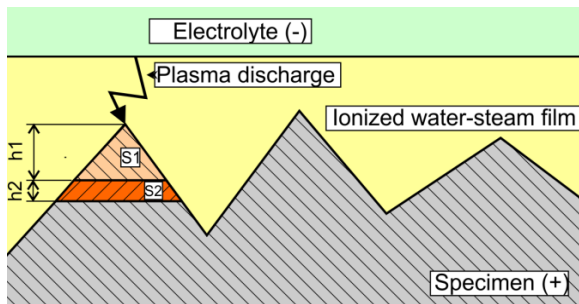

Fig. 9 Surface profile of the treated metal during plasma polishing
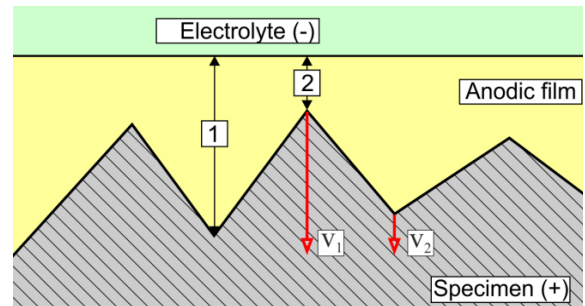

Fig. 10 Surface profile of the treated metal during electrochemical polishing

The material removal process during plasma polishing is different as it is in the case of standard electrolytic polishing, when the material from the surface is removed by anodic dissolution – *Fig. 10*. An anodic film forms on the treated surface during electrochemical polishing. The anodic film has lower electric conductivity then the surrounding electrolytic solution and the process of surface flattering is based on different velocity of anodic dissolving through this layer. Anodic dissolving is more intensive on the peaks of surface profile, where the anodic film is thinner so the electrical resistance is lower. Therefore the velocity of anodic dissolving v_1 is faster here than on the "valleys" of the surface profile – v_2. In this way, the metal is being removed not only on the peaks of the surface profile, but from whole surface although with different speed. This is the reason why it takes longer to reduce the surface roughness (*Fig. 6*) and why the thickness of layer removed from the surface is much higher (*Fig. 8*) then as it in the case of plasma-polishing, where the metal is removed mainly from the surface profile's peaks.

VIII. CONCLUSION

Surface properties of the specimens made from stainless steel X10 CrNi 18/10 have been plasma polished in electrolyte. The resulting properties of the polished surface depend on the treatment time. The gloss level rapidly increases during the first four minutes as it is described by the equation (2). The surface roughness reduction is most significant during the first four minutes, as well. The equation (1) will be useful to calculate the needful processing time when the desired value of the surface roughness is in focus. To calculate the dimension changes, for example when the precision machine parts are plasma-polished, the equation (3) can be used. All above equations can be also used for other stainless steels, although the resulting values may slightly differ.

The plasma polishing process is more efficient at surface roughness reduction than the common electrochemical polishing process. Furthermore, the value of the layer thickness removal is much less in the case of plasma polishing process. It can be supposed that this value will not depends on the spent state of the electrolytic solution such as it is in the state of electrochemical process. But this expectation must be confirmed experimentally in the next.

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